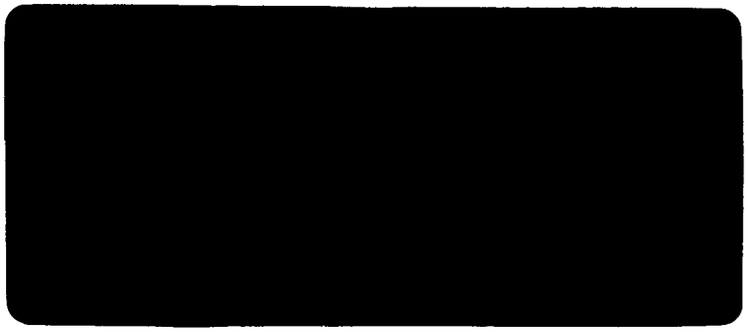

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September 10 to October 9, 1966

POLYMERS FOR SPACECRAFT HARDWARE

Prepared for:

Jet Propulsion Laboratory
California Institute of Technology
Pasadena, California 91103

Contract No. 950745

JPL Cognizant Engineer: E. Cleland

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SRI Project ASD-5046

Approved: R. F. Muraca, Director
Analyses and Instrumentation

Copy No. _____

CONTENTS

SCOPE

This report covers work performed during the period September 10 to October 9, 1966 on "Polymers for Spacecraft Hardware," SRI Project ASD-5046 under JPL Contract No. 950745.

The primary objectives of this program are to assist the Jet Propulsion Laboratory of the California Institute of Technology in the examination of polymeric materials to be used in connection with JPL spacecrafts, and to provide a study of the effects of simulated spacecraft environment on selected commercial polymeric products. The materials and products to be studied and the extent of work to be performed are specified by the JPL Cognizant Engineer.

WORK PERFORMED

Volatile Condensable Material

Micro-VCM

Maximum-VCM and total weight-loss results obtained with the micro-VCM apparatus are reported in Tables I to V for a series of protective coatings, wire enamels, seals and gaskets, and thermal coatings. A review of these data indicate that the most popular thermal coatings for spacecraft use cannot meet the suggested standards of less than 1% weight-loss, combined with less than 0.1% VCM (see M. R. No. 23), and that almost any Viton formulation is superior to other elastomers. As a whole, the wire enamels look like suitable candidates for spacecraft use; however, it must be remembered that the weight of polymer is but a fraction of the weight of bare wire plus polymer.

TABLE I

Micro-VCM Determinations: Protective Coatings

(24 hr at 125° C and 10^{-6} torr)
(VCM collector plates at 25° C)

Material	Mfr.*	Total Wt. Loss, %	VCM, wt-%	Noncondensable Wt. Loss %	Notes**
Epoxy					
PT-401/H-11	PTI	18.29	0.65	17.64	2,3
Diphenyl oxide					
Doryl B109-4	WEI	0.30	0.15	0.15	2,4
Doryl B109-5	WEI	0.18	0.14	0.04	2,4

*PTI, Product Techniques, Inc.
WEI, Westinghouse Electric Corporation, Insulating Materials Division

**2) Conditioned in 50% humidity for 24 hr prior to initial weighing and stored in desiccator for 30 minutes prior to final weighing.

3) Cured 20 minutes at 93° C.

4) Cured 2 hr at 250° C.

TABLE II

Micro-VCM Determinations: Wire Enamels

(24 hr at 125° C and 10^{-6} torr)

(VCM collector plates at 25° C)

Material	Mfr*	Total Wt. Loss, %	VCM wt-%	Noncondensable Wt. Loss, %	Notes **
Acetal (Formex)					
Magnet wire (AWG-35)	GEW	0.06	0.03	0.03	1,2
Polyurethane					
Magnet wire (AWG-22)	GEW	0.22	0.09	0.13	1,2

*GEW, General Electric Company, Wire and Cable Department

**1) As received.

2) Conditioned in 50% humidity for 24 hr prior to initial weighing and stored in desiccator for 30 minutes prior to final weighing.

TABLE III

Micro-VCM Determinations: Seals and Gaskets

(24 hr at 125° C and 10^{-6} torr)
(VCM collector plates at 25°C)

Material	Mfr*	Total Wt. Loss, %	VCM, Wt-%	Noncondensable Wt. Loss, %	Notes**
Vinylidene fluoride-hexafluoropropylene					
Viton A4411A-776	DUE	0.29	0.05	0.24	1,2
Viton A4411A-777	DUE	0.27	0.03	0.24	1,2
Viton A4411A-778	DUE	0.35	0.01	0.34	1,2
Butyl					
SR-634-70	SIS	1.55	0.21	1.34	1,2
Silicone					
SE-3604 (24/480)	GES	0.51	0.12	0.43	1,2

*DUE, E. I. du Pont de Nemours and Company, Elastomer Chemicals Department

SIS, Sargent Industries, Stillman Rubber Division

GES, General Electric Company, Silicone Products Department

** 1) As received.

2) Conditioned at 50% humidity for 24 hr prior to initial weighing and stored in desiccator for 30 minutes prior to final weighing.

TABLE IV

Micro-VCM Determinations: Tapes

(24 hr at 125° C and 10^{-6} torr)
(VCM collector plates at 25° C)

Material	Mfr. *	Total Wt. Loss, %	VCM, wt-%	Noncondensable Wt. Loss, %	Notes**
Polyester-aluminum					
Scotch Tape #852	MME	1.69	0.70	0.99	1,2
Scotch Tape #852	MME	0.57	0.40	0.17	2,3
Polyester-glass					
Scotch Tape #27	MME	6.08	2.27	3.81	1,2
Scotch Tape #27	MME	4.37	2.29	2.08	2,3
Fluorocarbon					
Ribbon Dope #520	PER	0.07	0.02	0.05	1,2,4
Ribbon Dope #520	PER	0.00	0.00	0.00	2,3

*MME, 3M Company, Adhesives, Coatings, and Sealers Divison
PER, Permacel

**1) As received.

2) Conditioned in 50% humidity for 24 hr prior to initial weighing and stored in desiccator for 30 minutes prior to final weighing.

3) Postcured 24 hr at 150° C.

4) This tape will be classified as a sealant in a forthcoming summary report.

TABLE V

Micro-VCM Determinations: Thermal Coatings
(24 hr at 125° C and 10^{-6} torr)
(VCM collector plates at 25° C)

Material	Mfr.*	Total Wt. Loss, %	VCM, wt-%	Noncondensable Wt. Loss, %	Notes**
Alkyd, modified					
Optical Black 101-C10	MMA	5.56	1.12	4.44	2,3
Optical Black 101-C10	MMA	0.57	0.25	0.32	2,4
Epoxy					
Cat-A-Lac Flat Black 463-1-8	FPC	13.00	1.52	11.48	2,3
Cat-A-Lac Flat Black 463-1-8	FPC	0.38	0.23	0.15	2,4
Cat-A-Lac Clear 473-1	FPC	25.48	3.30	22.18	2,3
Cat-A-Lac Clear 473-1	FPC	1.09	0.82	0.17	2,4
Cat-A-Lac White Gloss 443-1-500	FPC	15.79	0.95	14.74	2,3
Cat-A-Lac White Gloss 443-1-500	FPC	0.86	0.52	0.34	2,4

*MMA, 3M Company, Adhesives, Coatings, and Sealers Division
FPC, Finch Paint and Chemical Company

** 2) Conditioned at 50% humidity for 24 hr prior to initial weighing and stored in desiccator for 30 minutes prior to final weighing.

3) Cured 24 hr at 25° C.

4) Cured 24 hr at 150° C.

Macro-VCM

The current series of macro-VCM determinations is near completion; materials being examined are Lexan 141-112 (polycarbonate plastic), JPL-1002 (polyurethane conformal coatings), Epon 901/B-3 (epoxy adhesive), Eccobond 56C/9 (silver-epoxy solder), Hexcel HMH (polyester honeycomb core structure), and Thermofit RNF-100 (irradiated polyolefin shrinkable tubing). The data obtained thus far are in good agreement with the previously-determined micro-VCM results.

It has been demonstrated that the honeycomb core structure, Hexcel HMH, is not a suitable material for spacecraft use since it collapses (flattens) in the thermal-vacuum environment. (This same behavior was noted for the material when it was heated to 150° C in air.)

Comprehensive Polymer Test Program

The comprehensive polymer test program is designed to determine the changes in pertinent properties of polymeric materials subsequent to a decontamination treatment, a thermal-vacuum exposure, and a decontamination treatment followed by a thermal-vacuum exposure.

Operation Procedure

A. Each sample material is prepared in suitable sizes and shapes according to the tests to be performed. Where possible, a single specimen is used for more than one test (e. g., weight-loss and dimensional change). The test specimens are either suspended in the sample cell (see Figure 1) or laid flat on a wide-mesh screen. It is to be noted that only one polymeric product is contained in each cell.

B. The sample cells are sealed in place in the oven chamber which is brought to 50° C within one hour. The cells are then evacuated to about 10^{-3} torr and water vapor at 50° C is introduced through heated valving systems to provide a relative humidity of about 50%. The decontaminating agent (12:88 ethylene oxide-Freon-12, Matheson Company) is passed through a heat exchanger which bring it to 50° C and into the sample cells to provide an atmosphere of 455 mg/liter ETO.

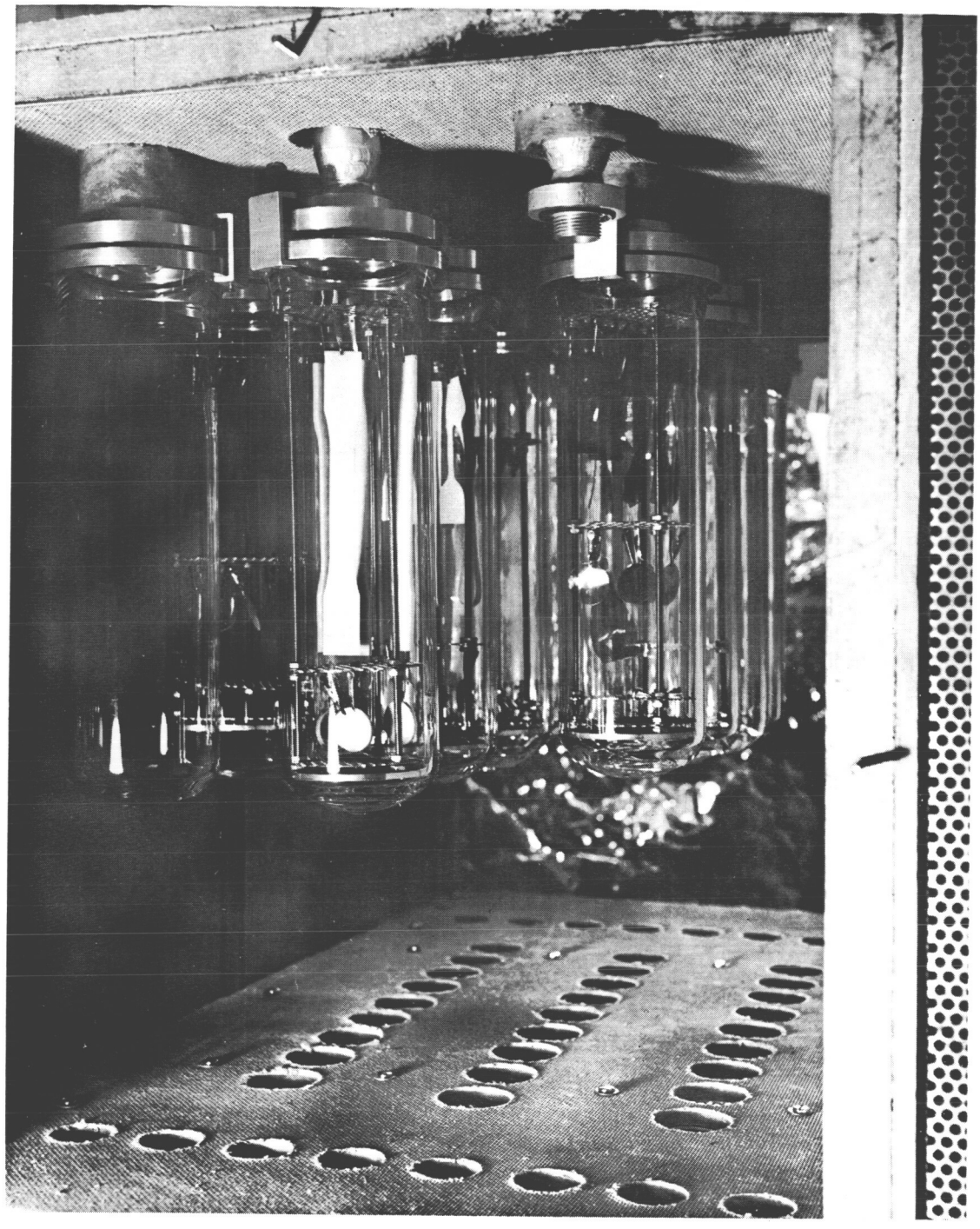

FIG. 1 VIEW OF MULTIPLE-CELL TEST UNIT SHOWING THE LOCATION OF SAMPLES IN THE TEST CELLS. Fifteen sample cells are contained in each of two ovens which comprise the assembly.

After a 28-hr period of 50° C in the humidified ETO atmosphere, the system is cooled to room temperature within one hour. During this period, the cells are evacuated for 10 minutes and vented to the atmosphere; evacuation and venting is repeated twice more to ensure complete flushing.

Six 30-hr cycles, as described above, complete the decontamination treatment.

C. Samples which are to be tested only for the effects of the decontamination treatment are withdrawn, and fresh samples are put in their place for tests of the effects of the thermal-vacuum exposure only. Othe samples are left in place for final tests of the effects of the decontamination treatment followed by a thermal-vacuum treatment.

D. The cells are then evacuated to 10^{-6} torr while the oven temperatures are raised to 135° C. The samples are maintained in this thermal-vacuum environment for 500 hours. At the end of this period, the ovens are cooled to room temperature and the cells are vented to the atmosphere. The samples are all removed for final testing.

Test Procedures

Duplicate samples of 1" x 6" are used for dimensional change, change in weight, and change in hardness.

(1) Dimensional change

The distance between bench marks on samples is measured to within ± 0.001 inch with vernier calipers with an accuracy considered to be ± 0.001 inch. The bench marks are located from 5.0-5.5 inches apart. The change in length is expressed as per cent of initial length.

(2) Change in weight

Samples are weighed either on an analytical balance or a micro-balance as required by the initial weight of the sample. The change in weight is expressed as percent of initial weight.

(3) Hardness

The hardness of structural materials is measured with a Wilson "Rockwell" Hardness Tester according to ASTM D785-62, using a 0.500-in ball with a 60-kg load.

The hardness of seal and gasket materials is measured with a Shore Durometer Type A-2 according to ASTM D2240-64T, using a 1-kg weight; readings are made within one second after application of the load.

An average of 5 determinations is made for each sample Results are reported as Rockwell or Shore numbers.

Results

Measurements of the changes in weight, hardness, and dimension have been completed for the polymeric materials which were exposed to the decontamination cycles. The effects of this treatment on films and sheets, hardware and structural materials, seals and gaskets, and tie cords/lacing tapes are shown in Tables VI to IX. The tests are near completion for changes in tensile strength, elongation, dielectric constant, and dissipation factor.

The thermal-vacuum cycle has been completed and testing is in progress.

TABLE VI

Effects of Decontamination Cycles on Films and Sheets

Material	Polymer Type	Mfr. *	Dimensional Change, %	Weight Change, %	Tensile/ Elongation	Dielectric Constant/ Dissipation Factor
Kapton 200XH667	Polyimide	DUM	n. c.	+1.89		
Kynar-200	Polyvinylidene-fluoride	PCC	n. c.	+0.18		
Mylar Type 150	Polyester	DUP	n. c.	+0.59		
Mylar Type 200	Polyester	DUP	n. c.	+0.40	Data	Data
P-2300	Polysulfone	UCP	n. c.	+1.13	being	being
PPO 681-111	Polypenylene oxide	GEC	n. c.	+1.76	completed	completed
Teflon FEP	Fluoroethylene-propylene	DUP	+0.21	+0.20		
Tedlar 100BG30TR	Polyvinyl fluoride	DUP	-1.73	+0.23		
Tedlar 200BG30WH	Polyvinyl fluoride	DUP	+2.82	+0.33		

*DUM, E. I. du Pont de Nemours and Company, Film Department
PCC, Pennsalt Chemicals Corporation, Plastics Department
DUP, E. E. du Pont de Nemours and Company, Plastics Department
UCP, Union Carbide Corporation, Plastics Division
GEC, General Electric Company, Chemical Materials Department

TABLE VII

Effects of Decontamination Cycles on Hardware and Structural Materials

Material [1]	Polymer Type	Mfr. [2]	Dimensional Change, %	Weight Change, %	Hardness [3]		Tensile/ Elongation	Dielectric Constant/ Dissipation Factor
					Control	Test		
Delrin 100NC10	Acetal	DUP	-0.71	+0.30	19.0	18.9		
Doryl H17511	Diphenyl oxide	WEM	n. c.	+0.35	23.1	23.8	Data	Data
Micarta H-2497	Epoxy-glass	WEM	n. c.	+0.10	24.9	25.2	being	being
Micarta H-5834	Phenolic-glass	WEM	n. c.	+0.30	23.2	23.6	completed	completed
Micarta 20201-2	Silicone-glass	WEM	n. c.	+0.01	17.8	17.4		

1 Postcured one hour at 1500 C.

2 DUP, E. I. du Pont de Nemours and Company, Plastics Department
WEM, Westinghouse Electric Corporation, Micarta Division

3 Rockwell

TABLE VIII

Effects of Decontamination Cycles on Seals and Gaskets

Material	Polymer Type	Mfr.[1]	Dimensional Change, %	Weight Change, %	Hardness[2]		Tensile/ Elongation
					Control	Test	
Hycar 520-67-108-1	Acrylic	BFG	n. c.	+0.64	86.3	85.4	
SE-3604	Silicone	GES	n. c.	+0.03	77.4	77.6	Data
SE-3613 (24/480)	Silicone	GES	n. c.	+0.14	70.6	70.5	being
SE-3713 (24/480)	Silicone	GES	n. c.	+0.17	77.9	81.4	completed
SE-3813 (24/480)	Silicone	GES	n. c.	+0.50	87.4	87.6	
Viton B	Vinylidene fluoride-hexafluoropropylene	AKR	+0.47	+1.47	85.4	84.6	

[1] BFG, B. F. Goodrich Chemical Company

GES, General Electric Company, Silicone Products Department

AKR, Akron

[2] Shore

TABLE IX

Effects of Decontamination Cycles on Tie Cords/Lacing Tapes

Material*	Polymer Type	Mfr. **	Dimensional Change, %	Weight Change, %	Tensile/ Elongation
Gude-Space 18D96	Polyester	GBE	n. c.	+1. 08	Data
Templace 256H	Fluorocarbon	GBE	n. c.	+0. 52	being completed

* Postcured 1 hour at 150° C.

** GBE, Gudebroad Brothers Silk Company, Inc., Electronics Division

Mechanical Properties

Long-Term Storage Tests

A 9-month exposure period has been terminated for samples stored at constant strain in an environment of 125° C and $<10^{-6}$ torr. Elastomers examined are General Electric SE-3604 (silicone), du Pont Viton A4411A-990 (vinylidene fluoride-hexafluoropropylene), and Goodrich Hycar 520-67-108-1 (polyacrylic). No samples ruptured during the cooling, venting, and disassembly procedures. Five samples ruptured during outgassing at 40° C (SE-3604 and Viton-990 at maximum strain), three samples ruptured during the first week at 125° C (Hycar-1 at maximum strain), and one sample ruptured after the 7th month, Hycar-1 at a strain of 50%.

The long-term constant-load tests of polyphenylene oxide films (General Electric) in the thermal-vacuum environment have been terminated after a duration of 7 months. One specimen at 1750 psi ruptured during the third month and one during the fifth month of storage; almost negligible creep has been observed for the remaining samples.

All of the stored materials, including control specimens, are being subjected to Instron testing. The thermal-vacuum equipment has been cleaned and is being maintained temporarily at stand-by vacuum.

Six-Week Storage Tests

The most recent thermal-vacuum storage program was completed on September 26. Stored specimens are being tested and data are being reduced.

The next program will start on October 10, when relaxometer and storage area temperatures will be brought to 125° C. Materials being evaluated in this program are:

(a) Continuous and intermittent stress relaxation
Viton A, batch 776; Viton B, batch 778

(b) Constant load (duplicate specimens at 500 and 650 psi)
Teflon FEP, Type A, 20 mil

FUTURE WORK

Volatile Condensable Material

Micro-VCM determinations for polymer screening and macro-VCM determinations for engineering information will be made on a continuing basis.

Comprehensive Polymer Test Program

Data will be completed for the first series of polymers to be tested under this program. Preparations will be made for the initiation of a decontamination/thermal-vacuum run on a series of 2-part polymers, such as sealants, adhesives, etc.

Mechanical Properties

Work will continue on the measurements of stress-relaxation changes for selected polymers during a 6-week exposure to the thermal-vacuum environment of 125° C and 10^{-6} torr.

Instron data will be obtained for the polymers which have undergone long-term storage tests.

Errata

Monthly Report No. 27, Table VIII:
Hycar-6 at intermittent f(t)/f(0)
should read 1.08 (not 1.80).