

December 15, 1965

POLYMERS FOR SPACECRAFT HARDWARE
MATERIALS SPECIFICATIONS AND ENGINEERING INFORMATION

Prepared for: Jet Propulsion Laboratory
California Institute of Technology
Pasadena, California 91103

JPL Contract No. 950745
under NAS7-100

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SRI Project ASD 5046

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FACILITY FORM 502

N66 24984

(ACCESSION NUMBER)

15
(PAGES)

CR-74666
(NASA CR OR TMX OR AD NUMBER)

(THRU)

(CODE)

(CATEGORY)

GPO PRICE \$ _____

CFSTI PRICE(S) \$ _____

Hard copy (HC) 1.00

Microfiche (MF) .50

Copy No. 5

SCOPE

This report covers the work performed during the period November 10 to December 9, 1965 on "Polymers for Spacecraft Hardware--Materials Specifications and Engineering Information," SRI Project ASD-5046 under JPL Contract No. 950745.

The primary objectives of this program are to assist the Jet Propulsion Laboratory in the development and preparation of polymeric material specifications to be used in connection with JPL spacecraft, and to provide a study of the effects of simulated space 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 Engineers.

The program is being conducted as two interrelated phases, running concurrently: The purpose of Phase I, Polymeric Materials Specifications, is to obtain quantitative values for parameters which may be used to assure the performance of a given batch of material in a spacecraft environment. The purpose of Phase II, Engineering Information, is to establish material limitations and to obtain detailed design information.

PHASE I. POLYMERIC MATERIALS SPECIFICATIONS

WORK PERFORMED

Two chemical test procedures have been established for RTV-type silicone potting-compound base materials: filler content, and hydrolyzable halide content. The results of these analyses for representative materials (General Electric Company RTV-11, -60, -560, -615) are summarized in Table I. Complete details of the procedures have been submitted to the JPL Cognizant Engineer as suggestions for incorporation into the appropriate Specifications.

The procedure for filler content is quite straightforward and typical of those which involve complete solution of a material in a suitable solvent in order to separate it from a material which is insoluble:

About 0.3-0.4 g of the silicone base material is mixed thoroughly with 50 ml of chloroform and 0.2-0.3 g of Celite filter-aid, and then the silicone-chloroform is extracted from the mixture by filtration. The material remaining on the filter is dried and weighed, and the weight is corrected for Celite filter-aid. The filler content is computed as wt-%.

Although a chlorinated silane is an intermediate product in the production of silicone polymers, virtually all chloride ion must be absent for qualification of the end-product as a space-grade material, since evolution of chloride ion as chlorine or hydrochloric acid would be detrimental to spacecraft operations. The following is a suggested procedure for determination of hydrolyzable halide in RTV-type silicones:

About 5-6 g of silicone base material is treated with 50 ml of ethanol. The resulting solution is titrated potentiometrically with 0.1N sodium hydroxide and results are reported as wt-% hydrolyzable halide (as chloride).

Table I
RESULTS OF ANALYSES OF REPRESENTATIVE RTV-TYPE*
SILICONE POTTING COMPOUNDS

Sample	Property	Value, wt-%
RTV-11	Filler content (TiO_2)	29.77 -29.90
	Hydrolyzable halide content	0.005- 0.003
RTV-60	Filler content (Fe_2O_3)	50.08 -50.33
	Hydrolyzable halide content	0.003- 0.002
RTV-560	Filler content (Fe_2O_3)	42.68 -42.71
	Hydrolyzable halide content	0.005- 0.004
RTV-615	Filler content	(none)
	Hydrolyzable halide content	0.002- 0.002

* General Electric Company

A simple extraction procedure with chlorobenzene or methylethyl ketone has been suitable for most epoxy adhesives, but questionable results have been obtained* for Epon 422J (Shell Chemical Company) glass-fiber tape impregnated with an epoxy-phenolic adhesive containing an aluminum filler. Continued work with this material has indicated that a Soxhlet extraction procedure is required, and that acetone is a suitable solvent:

About 10 g of the epoxy adhesive tape is weighed into a Soxhlet extraction cup, and extracted with 250 ml of acetone for a period of 4 hours. The cup and contents are dried overnight at 110°C , then cooled and weighed. The amount of acetone-soluble material is calculated and reported as wt-%.

The quantity of acetone-soluble material, 42.87-42.33 wt-%, consists of both the epoxy-phenolic resin and the curing agent. Details of the procedure have been submitted to the JPL Cognizant Engineer as a suggestion for incorporation into Specifications for epoxy-base adhesive tapes.

* Muraca, R. F., et al., Stanford Research Institute, Project 5046, Interim Report No. 1, August 9, 1965.

Vacuum-weight-loss determinations by a standardized procedure were made for epoxide coatings designated as Eccocoat EC/200 and Eccocoat VE, supplied by Emerson and Cuming, Inc. The base materials are suitable for application by brushing or dipping; they deposit an initial coating of about 5 mil thickness. The coating thickness is subsequently increased by successive applications after each coat is cured, according to manufacturer's instructions. Since given specifications are based on 10-mil coatings to simulate end-use, sample coatings of this thickness were prepared as follows:

In order to provide a sufficient amount of sample for weight loss determinations, the coatings were applied to copper wire coils of suitable size to fit in the sample chambers: three-foot lengths of 16-gage copper wire were wound on a half-inch mandrel. The coils were dipped in the coating material, drained, and cured; then a second coat was applied as required and treated in the same fashion.

Cure conditions: Eccocoat EC/200, 6 hours at room temperature and 2 hours at 120°C (two coats);

Eccocoat VE, 6 hours at room temperature and 2 hours at 95°C (one coat).

As indicated in Tables II and III, weight losses for both materials are far in excess of acceptable limits and no indication of leveling off is given after 192 hours of exposure in the thermal-vacuum environment. Evidence observed thus far indicates that these resins require more extended curing at elevated temperatures, that they undergo slow decomposition in the vacuum-thermal environment, or that the curing cycle is not long enough to remove any solvents that are present.

Weight loss data were also obtained for a modified-epoxy coating material, Clear Varnish B-276, supplied by Westinghouse Electric Corporation. Samples were applied to copper coils as described above and cured for 4 hours at room temperature followed by 1 hour at 165°C, according to manufacturer's instructions. As shown in Table IV, the weight loss apparently levels off in 96 hours, but nevertheless is well outside of acceptable limits. In this instance, an extended cure at the elevated temperature may help qualify this material for spacecraft use; this will be checked.

Table II

WEIGHT LOSS DATA FOR ECCOCOAT EC/200*
(125°C and 10⁻⁶ torr)

Time, Hrs.	S. Wt., g	Wt. Loss, g	Wt. Loss, %
48	0.7788	0.0235	3.02
	0.4901	0.0148	3.02
96	0.6727	0.0234	3.48
	0.8463	0.0288	3.40
192	0.5589	0.0311	5.56
	0.5123	0.0312	6.09

* Emerson and Cuming, Inc.

Table III

WEIGHT LOSS DATA FOR ECCOCOAT VE*
(125°C and 10⁻⁶ torr)

Time, Hrs.	S. Wt., g	Wt. Loss, g	Wt. Loss, %
48	1.5612	0.0435	2.79
	1.4761	0.0389	2.64
96	1.1800	0.0647	5.48
	1.4074	0.0978	6.95
192	0.8682	0.0892	10.27
	1.2895	0.0991	7.68

* Emerson and Cuming, Inc.

Table IV

WEIGHT LOSS DATA FOR CLEAR VARNISH B-276*
(125°C and 10⁻⁶ torr)

Time, Hrs.	S. Wt., g	Wt. Loss, g	Wt. Loss, %
48	0.0233	0.0222	2.40
	0.8092	0.0182	2.25
96	0.8341	0.0206	2.47
	0.7937	0.0223	2.81
192	0.7838	0.0201	2.56
	0.8442	0.0220	2.61

* Westinghouse Electric Corp., Micarta Division

Samples of a silver-filled epoxy paste solder, Eccobond Solder 56C/9, supplied by Emerson and Cuming, Inc., were applied to copper coils by brushing, and cured for 2 hours at 50°C, according to manufacturer's instructions. The nearly-acceptable weight loss of this material appears to level off at 48 hours (see Table V), but understandably would be much greater were not such a gross amount of silver present. Since this is a unique and important material, the effect of extended curing will be checked.

Table V

WEIGHT LOSS DATA FOR ECCOBOND SOLDER 56C/9*
(125°C and 10⁻⁶ torr)

Time, Hrs.	S. Wt., g	Wt. Loss, g	Wt. Loss, %
48	1.1956	0.0086	0.72
	1.7707	0.0095	0.54
96	1.6003	0.0121	0.76
	1.0117	0.0099	0.98
192	1.1778	0.0096	0.82
	2.0229	0.0121	0.60

* Emerson and Cuming, Inc.

FUTURE WORK

Vacuum weight loss determinations via standardized procedures will be made on a continuing basis in order to establish limitations for specifications requirements for various polymeric materials. Corollary studies will be conducted via thermogravimetry in order to verify and supplement the data obtained by standardized procedures.

Determinations of physical and chemical properties of polymers will be continued as required for incorporation into specifications requirements and test procedures.

PHASE II - ENGINEERING INFORMATION

WORK PERFORMED

Fabrication and assembly has been completed for the long-term mechanical-property test units. The four units, assembled on a single support-and-control stand, are being checked-out for vacuum operation, electrical continuity, and thermal uniformity. (A detailed description of the units and a photograph will be presented in the next report.)

The polymers in the long-term tests will be checked periodically for any observable effects of the thermal-vacuum environment (125°C and 10^{-6} torr). At the termination of an 8-month exposure period (representative of a flight to Mars), the tensile properties of surviving materials will be determined on an Instron machine.

Three of the units soon will be loaded for constant strain tests with materials which have been selected from prior work as those most qualified thus far for long-term spacecraft use: General Electric elastomer SE-3604, DuPont elastomer Viton A-4411A-990, and Goodrich elastomer Hycar-520-67-108-1. Loading of each unit will be confined to a single material in order to eliminate possible cross-contamination in such a long-term exposure. Also, in order to prevent any possible radiation effects (fluorescent lighting fixtures) during long-term storage, the glass "bell tubes" will be covered with aluminum shields. The fourth unit is temporarily reserved for selection of a significant material which may be subjected either to constant strain or constant load.

Necessary modifications to a basic vacuum console have been completed for the accommodation of the rapid, maximum-VCM test assembly. The max-VCM unit is near completion and will be placed in operation during the next working period. Initially, the max-VCM unit will provide maximum-volatile-condensable-material data on a weight basis for both engineering information and specifications requirements. Alternatively,

the unit may be adapted to provide semi-quantitative identification of the VCM which is released by polymeric materials subjected to a thermal-vacuum environment; work is underway on the latter modification. Materials to be tested in the max-VCM unit will be selected on the basis of interest to both phases of this program.

Work is continuing on the assembly of the "clam-shell" units which will permit establishment of VCM curves over long periods of time. It may be pointed out that these assemblies will accommodate polymeric samples of almost any configuration (within size limitations), i.e., films, elastomers, tapes, electronic component assemblies, etc.

Mass spectra have been obtained (CEC Model 21-103C mass spectrometer) of the substances evolved from several samples of General Electric Lexan (polycarbonates) during their exposure to the thermal-vacuum environment of 125°C and 10^{-6} torr. A sample is pumped overnight in a modified inlet system at ambient temperature to remove surface gases and moisture; then it is brought quickly to a temperature of 125°C and the evolved vapors are immediately scanned. Since the spectra for these materials have not yet been completely interpreted, it has not been determined whether the Lexan itself is decomposing under these conditions, but at least 3 or more extraneous materials are readily identifiable, e.g., water, plasticizer, etc.

In situ measurements of the changes in constant and intermittent stress relaxation are underway in the thermal-vacuum environment of 125°C and 10^{-6} torr for General Electric silicone elastomers SE-3613 (24/480) and SE-3713 (24/480). Samples of Lexan (General Electric, first annealed in air for 1 hour at 150°C, are being stored in the same environment for creep tests under 600 psi loading. Additionally, unloaded control specimens of all materials are being stored for post-test observations. Preliminary outgassing of materials at 50°C was completed after about 145 hours.

FUTURE WORK

Work will be completed on the installation and operation of the assembly for rapid, maximum-VCM determinations, and assembly of the VCM clam-shell units will be accelerated.

Long-term (8-month) mechanical properties tests will be initiated for several elastomers.

Work will continue on the spectral studies of polymers exposed to a thermal-vacuum environment and on the measurements of changes for selected mechanical properties of polymers during their exposure to a thermal-vacuum environment.

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