

Interim Report No. 3 - Part I March to December 1966

POLYMERS FOR SPACECRAFT HARDWARE: MATERIALS CHARACTERIZATION

Prepared for:

JET PROPULSION LABORATORY CALIFORNIA INSTITUTE OF TECHNOLOGY PASADENA, CALIFORNIA 91103

JPL CONTRACT NO. 950745 . UNDER NAS7-100





December 9, 1966

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JPL Cognizant Engineers: E. CLELAND, R. HARRINGTON, H. MAXWELL SRI Project Supervisor: R. F. MURACA

SRI Project No. ASD-5046

Approved: R. F. MURACA, DIRECTOR ANALYSES AND INSTRUMENTATION

> This work was performed for the Jet Propulsion Laboratory, California Institute of Technology, sponsored by the National Acronautics and Space Administration under Contract NAS7-100.

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FOREWORD

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This Interim Report summerizes the work performed by Stanford Research Institute during the period March to November 1966 under Contract No. 950745 for the Jet Propulsion Laboratory of the California Institute of Technology.

Cognizant Engineers of the Jet Propulsion Laboratory's Materials and Methods Group were Mr. E. Cleland, Mr. R. Harrington, and Mr. H. Maxwell.

The technical effort at Stanford Research Institute was under the supervision of Dr. R. F. Muraca, Director, Analyses and Instrumentation.

The work was performed largely within the Department of Analyses and Instrumentation under the leadership of J. S. Whittick, Chemist-Program Co-ordinator and A. A. Koch, Chemist. Project Leader for mechanicalproperty measurements <u>in situ</u> was N. Fishman, Manager, Propellants Evaluation, Division of Industrial Research.

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Acknowledgment is made of the work of a number of chemists, technicians, and machinists who have contributed to this program.

ABSTRACT

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Stanford Research Institute, Menlo Park, California POLYMERS FOR SPACECRAFT HARDWARE (in two parts) Interim Report No. 3, March to December 1966 R. F. Muraca, et al., December 9, 1966 (NASA Contract No. NAS7-100; JPL (Contract 950745; SRI Project ASD-5046)

The objectives of this program are to assist JPL in the selection of polymeric materials to be used in connection with spacecrafts, which includes the examination of the effects of simulated spacecraft environment on selected commercial products. The spacecraft environment is considered to be a protected thermal-vacuum environment of about $125^{\circ}C$ and 10^{-6} torr. Part I describes the technical work and the results thereof; Part II presents a display of drawings, photographs, and descriptions of the test equipment which has been designed for this program of work.

More than 200 polymeric products nave been screened by a special micro-method which gives values for total weight-loss and for the maximum amount of the volatile substances which condense on an adjacent cooler surface; i.e., volatile condensable materials (VCM). Information on VCM, that is, the deposition and removal of VCM as a function of time, is recorded for a number of polymeric products. Identification of the substances released by polymers in a thermal-vacuum environment was made possible by mass spectrometry.

The results of <u>in situ</u> measurements of selected mechanical properties of elastomers and plastics during 6-week storage periods in the thermal-vacuum environment, are discussed and compared with the performance of selected materials stored for as long as 9 months in the thermalvacuum environment. A comprehensive polymer test program is described which provides for physical/mechanical property testing of as many as 30 polymeric materials exposed simultaneously to a decontamination environment, or a thermalvacuum environment, or a decontamination environment followed by a thermalvacuum environment; results for 22 products are reported for properties such as hardness, dimensional change, tensile, dielectric constant, etc.

In accordance with the "New Technology" clause, three disclosures are made of advancement of technology under JPL/NASA sponsorship.

An Index is provided which lists every polymer examined by brand name and refers to the Tables in which tabulated data may be found.

Part II of the report provides details for the construction of (1) Micro-VCM Apparatus; (2) Macro-VCM Apparatus; (3) Multiple-Cell Test Unit; (4) Long-Term Storage Apparatus for Polymers (Under Constant Strain); and (5) Long-Term Storage Apparatus for Polymers (Under Constant Load).

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I. INTRODUCTION

This Interim Report summarizes the work performed for the Jet Propulsion Laboratory of the California Institute of Technology under Contract No. 350745, SRI Project ASD-5046, during the period March 1966 to November 1966. Earlier work under this contract is summarized in Interim Report No. 1 (August 1965) and Interim Report No. 2 (March 1966). The report is divided into two parts:

> Part I: Materials Characterization Part II: Test Equipment Design

The primary objectives of this program are to assist the Jet Propulsion Laboratory in the selection of polymeric materials to be used in the construction of spacecrafts. The program is predicated on the determination of the effects of simulated spacecraft environment on selected commercial products. The materials and products examined have been provided by the JPL Cognizant Engineers.

For the purpose of this program, a spacecraft environment is defined as the conditions existing within a scientific satellite or probe, or the unpressurized portions of a manned spacecraft. Since electronic assemblies, associated components, and various structures are protected from as many hazards as possible in space flights, the environment under consideration is obviously one shielded from the extremes of temperature, electromagnetic and particle radiation, and meteoroids. Therefore, the thermal-vacuum conditions employed during this program of work were established as consisting of pressures less than 10^{-5} torr (readily-attainable in batch-testing equipment) and temperatures less than 125° C (the maximum temperature anticipated in locations where polymeric materials might be used). The selection of materials for use in a spacecraft is based on a study of their behavior in a thermal-vacuum environment and a knowledge of the nature of the substances they release. Thus, results of determinations of

the loss of weight, of the amount of released material which may condense on cool surfaces adjacent to the warmed polymer, and the identification of the released substances in the thermal-vacuum environment conditions of interest must be combined with measurements of physical and mechanical property changes which may occur over a period of time in the environment in order to confirm the suitability of polymers for spacecraft construction.

Part I

The substances released by polymeric materials in a thermalvacuum environment are discussed in Section II. The section describes a macro-VCM procedure and a micro-VCM procedure which are used to obtain weight-losses and the amount of condensable material released by a polymer. The micro-VCM procedure is especially useful for screening polymers because it provides quantitative values for both maximum weight-loss and maximum volatile condensable material on 24 samples of about 0.1 gram within 48 hours. Micro-VCM information is given for more than 260 polymeric products; of these, about one-third can be recommended for further evaluation.

The information derived from micro-VCM screening is augmented in Section II by data obtained from macro-VCM determinations to provide information on the rate of deposition and removal of condensed material. The identification by mass spectroscopy of released substances indicates the nature of the substances and provides a basis for suggesting slight changes in the curing, processing, or formulation of the polymeric product.

A discussion is given in Section II of the correlation of regults of the three kinds of examinations for released substances, verification of the selection of one-third of the materials as being suitable candidates, and evidences of corroboration of micro-VCM screening by mechanical-property tests.

The work described in Section III has involved the measurement of selected mechanical property changes <u>in situ</u> during 1900-hr

exposures of elastomers and plastics in the thermal-vacuum environment as well as the examination of elastomers stored under constant-strain for 9 months and a plastic stored under constant load for 7 months, in similar thermal-vacuum environments. Comparative data are summarized in terms of polymer families: suggestions for use are made on the basis of poten ial applications.

In order to provide more data on pertinent physical and mechanical properties, a program was developed to test the effects of decontamination cycles and thermal-vacuum exposure on as many as 30 polymeric materials simultaneously; it is discussed in Section IV. The decontamination cycle consists of six 30-hr exposures to humidified ETO-Freon at 50°C; the thermal-vacuum exposure consists of 500 hours at 135°C. The comprehensive program includes testing after each exposure and after exposure to both environments. Data are given for 22 polymeric products for properties such as weight loss, dimensional change, hardness, tensile, elongation, dielectric constant, and dissipation factor.

Results are reported in Section V for standardized thermalvacuum weight-loss determinations on a number of polymeric materials. This method was established as a simple and inexpensive procedure for specifications purposes. Since the early months of this contract period, the equipment has been replaced by the micro-VCM apparatus which provides not only the same results more rapidly but also quantitative values for VCM.

A small continuing effort on the selection of test procedures for chemical and physical characterization of polymer base materials is discussed in Section VI. Chemical and physical data are given for an epoxy-silver solder and for two representative polyurethanes.

Section VII on "New Technology" provides pertinent information on technologies which have been advanced or developed under the JPL/NASA sponsorship of the program.

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A review is given in Section VIII of the program from its earliest inception; it outlines the transition from exploratory research of the behavior of polymeric materials in the thermal-vacuum environment to current recommendations for the most meaningful methods and procedures to obtain data for the evaluation and selection of polymeric materials.

The Appendices contain information on typical basic structures of polymeric materials, a code listing of manufacturers of polymeric materials which have been examined, detailed instructions for performing micro-VCM and macro-VCM determinations, and an alphabetical Index of polymer products with references to tabulated data in this report.

Part II

Part II of this report contains mechanical descriptions and design drawings for:

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Micro-VCM Apparatus for Screening Polymers Macro-VCM Apparatuss for Polymers Long-Term Storage Apparatuses for Polymers

1) Under constant strain

2) Under constant load

Multiple-Cell Test Unit for Polymers.

II. MATERIALS RELEASED BY POLYMERS IN A THERMAL-VACUUM ENVIRONMENT

The loss of matter by outgassing and by evaporation or sublimation is one of the most obvious effects of a thermal-vacuum environment on polymers. Because gross loss of material generally means that the physical properties of polymers are altered or that test chambers will be contaminated by the vaporized substances, the polymers which are considered suitable for use in spacecrafts are those which in laboratory tests exhibit a minimum loss of weight when exposed to the simulated conditions of the vacuum and thermal environment of space.

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The simplest quantitative value which can be obtained for the behavior of a polymer in a thermal-vacuum environment is the loss of weight. The value does not reveal the nature of the components released by the polymer but, nevertheless, is useful for selecting polymers suitable for spacecrafts since, in the majority of instances, it may be safely assumed that mechanical or other useful properties of the polymers are degraded when a substantial amount of loss of weight has been incurred. It is customary to assume that polymeric substances which lose less than 1% of their weight in a thermal-vacuum environment are suitable for spacecraft application.

Matter released by polymeric substances in a thermal-vacuum environment may be the ordinary gases (e.g., adsorbed air or carbon dioxide), ordinary liquids of relatively high varor pressure (e.g., water or solvents), and liquids or solids of relatively high molecular weight and low vapor pressure (e.g., lubricating oils, plasticizers, etc.).

A necessary adjunct to the evaluation of polymeric materials based on outgassing characteristics is the identification of the substances which are released by the materials under identical thermal-vacuum conditions. A schematic diagram of the general approach to acceptance for candidacy or disqualification of polymeric materials is given on the following page for "Brand X."

STRESS, IN-VACUO AT 125°C 24, 48, 96, and 336 HOURS <u>IN VACUO</u> AT 125°C 10-g SAMPLES, CONFIGURED % WT-LOSS WITH TIME % VCM WITH TIME MACRO-VCM EXAMINATION OF POLYMERIC SAMPLES . ۲ "BRAND X" 24 HOURS AT 125°C IN VACUO 01-0.2-9 SAMPLE, COMMINUTED % TOTAL WT-LOSS % MAXIMUM VCM 28,0 MICRO-VCM

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CONSTANT STRAIN, IN VACUO AT 125°C LONG-TERM STORAGE 1 MASS SPECTRUM 5 6 CYCLES OF MUMIDIFIED ETO-FREON AT 50°C 500 HOURS AT 135°C IN VACUO COMPREHENSIVE TEST PROGRAM TENSILE/ELONGATION DIELECTRIC/DISSIPATION HARDNESS CHANGE Dimensional change Compression set etc.

AT 125°C IN VACUO

2-mg SAMPLE

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Complete design drawings for VCM equipment are given in Part II of this report: "Test Equipment," and details of the procedures are given in Appendix C, Part I. A prior report includes a discussion of the principles underlying VCM determinations.^{**} ţ,

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Part A of this Section describes briefly the procedures used for screening polymers via the micro-VCM determination and the approach for establishing acceptance limits; screening data for more than 200 polymeric products are given and suggestions are made for candidacy of about one-third of the products.

In Part B, the general procedure for determining VCM with time is described, and data are summarized for 10 polymeric products suggested for use in several different spacecraft applications.

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The identification of substances released by polymeric materials under conditions of 125° C and $<10^{-6}$ torr is described in Part C of this Section for a number of commercial products.

The correlation of micro-VCM screening data, macro-VCM data, and the identification of released materials is discussed in Part D. Based on the correlations, suggestions are made for additional work or final elimination. References to corroborating mechanical-property testing are made as appropriate.

It has been shown that polymers which suffer sizable weight-losses usually release substances which condense on cooler surfaces and interfere with spacecraft functions (volatile condensable material). Volatile condensable material (VCM) may be defined as the weight of condensate obtainable at 25° C in a given interval of time from a given weight of material (<1/8 in thickness) maintained at 125° C in a vacuum (at least 5×10^{-6} torr). The temperature of 125° C was selected in 1962 as the upper limit of temperature which might be encountered in spacecraft operations. Space probes and satellites are generally designed to

^{*} Muraca, R. F. et al., Space Environment Effects on Polymeric Materials, Interim Technical Report No. 2, August 1964 to May 1965, JPL Contract No. 950324, May 30, 1965. maintain internal temperatures of the order of 25° C, but much higher temperatures may occur in the vicinity of power-dissipating components; for example, thermal data from SURVEYOR I (Lunar soft-lander)[#] reveal temperatures c⁺ the order of 60° C in various compartments of the spacecraft while comera temperatures were of the order of -80° C; thus, the migration of volatilized substances between spacecraft components which are at different temperatures is always possible and it becomes necessary to test every polymer which is used in a spacecraft to determine whether it releases materials which can condense and invalidate the craft's functions.

The development of the equipment and technique for the determination of VCM has been evolutionary during the program sponsored by the Jet Propulsion Laboratory: (1) A prototype unit was designed for determination of VCM as released by elastomers;[†](2) The value of this determination as a screening (and quality-control) procedure for polymeric materials invoked the design of equipment for making rapid determinations on multiple samples (micro-VCM);[§](3) The requirements for information on the rate of VCM release by any configuration of polymeric material led to the design of an improved macro-VCM apparatus and procedure as described in this Section and Part II of this report.

A. MICRO-VCM DETERMINATIONS

Data for the rapid screening of candidate polymeric materials for spacecraft applications are provided by the results of micro-VCM determinations made with an apparatus designed at SRI. Samples of 100-200 milligrams furnish values in duplicate for <u>maximum</u> VCM and total

^{*} Parks, R. J., Jet Propulsion Laboratory, Surveyor I Mission Report, Part I, T.R. No. 32-1023, August 31, 1966.

[†] Muraca, R. F., et al., Stanford Research Institute, Space Environment Effects on Polymeric Materials, Final Technical Report (Oct. 1962-Dec. 1963), JPL Contract 950324, Dec. 8, 1963.

[§] Muraca, R. F., et al., Stanford Research Institute, Polymers for Spacecraft Hardware, Interim Report No. 2, JPL Contract 950745, March 15, 1966. (See also Part II, this report)

weight loss. The determinations require a 24-hour exposure in the thermal-vacuum environment. The polymers are maintained at 125° C in a vacuum of about 10^{-6} torr; the condensing surface is at 25° C.

Figure 1 is a photograph of the vacuum console and the VCM assembly; visible are the front and back sections of two micro-VCM units, each of which accommodates 12 samples. Detailed drawings and mechanical descr..ptions of the micro-VCM assembly are given in Part II (Test Equipment) of this report. Details of the procedure for making micro-VCM determinations are given in App nd^4 : C (Part I of this report).

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The results of the micro-VCM determinations which are reported at this time are based on a minimum of 2 determinations per sample. Preliminary work and subsequent observations have shown the average reproducibility of determinations to be as follows:

Av. wt-loss of VCM	Av. reproducibility (abs.)
<1%	±0.05
1-5%	±0.20%
5-10%	±0.50
10-20%	±1. 0

From time to time at random, blank runs are made and the VCM collector plates are found to be free of condensed material; blanks have not been run routinely since it was established initially (March 1966) that no cross-contamination was occurring between sample compartments and that the change in weight of blank VCM collector plates did not vary more than ± 10 micrograms.

More than 200 ploymeric products, representing more than 20 classes of polymers, have been screened in the micro-VCM apparatus. A complete summary of all the results is given in Tables I-XVII at the end of this Section; the tables are arranged alphabetically, according to potential spacecraft applications.

The products were supplied by the JPL Cognizant Engineers and were prepared and/or cured according to manufacturer's instructions for hightemperature service. Additional runs were made after extended or modified curing cycles as recommended by the experience of spacecraft designers. Most weight-loss is known to be due to extraneous solvents, occluded gases, etc.; however, VCM may arise from incompletely-polymerized substances, high-molecular-weight additives, etc. Hence, many of the polymers were re-screened after postcures of 24 hours at 150°C; increased times and temperatures were selected in consideration of (1) possible effects on mechanical properties, (2) end-use of materials, and (3) feasibility of curing polymers in place.

As indicated in Tables I-XVII, a postcure of 24 hours at 150° C may reduce weight-loss and VCM, but it will not often be sufficient to upgrade the polymer so that it qualifies for spacecraft use. RTV-type silicones (Table X) are notorious for their VCM content; no further work with them should be contemplated. On the other hand, silicone <u>elastomers</u> which have been postcured for 24 hours at 250° C can qualify as potential candidates. (It has been found, however, that RTV-type silicones cannot withstand cure cycles at 250° C.)

Candidate Space-Grade Polymers

About one-third of the products screened can be considered as potential space-grade materials which merit further evaluation of physical and mechanical properties, more exacting VCM determinations, and identification of volatile substances. The selection of these materials, summarized in Table XVIII, is based on a combined VCM unper limit of 0.1% and weight-loss upper limit of 1%. It is commonly accepted that weightlosses of less than 1% do not affect polymer properties. On the other hand, results of calculations indicate that a pound of polymeric material which is known to release 0.1% of VCM at 125°C will deposit on a surface of one square foot at 25°C a film of about 0.00015 inch in thickness (0.15 mil); this amount is more than enough to foul lenses or electrical contact points. Ideally, the VCM content should be zero, or of a kind whick will subsequently b; evaporated from the cooler surface (e.g., see

Teflon FEP). It should be recognized that some condensed films eventually may evaporate while others may remain in place.

It appears at first sight that the candidates listed in Table XVIII may be improved by simple changes in formulation, processing, or curing since most of these materials qualify without harsh postcures. However, there is an urgent need for flexible sealants, honeycomb structures, lubricants, tapes, and temperature-control coatings since very few of the commercial products have been found suitable for spacecraft use.

B. MACRO-VCM DETERMINATIONS

The macro-VCM determinations provide information on the rate at which offending VCM is re-evaporated. At the same time, the results of these determinations provide insight into whether a modified cure or a modified blend of components will yield a polymeric product suitable for spacecraft use.

A photograph is given in Figure 2 of the assembly of 12 macro-VCM sample cells within the vacuum chamber, and Figure 3 illustrates the entire vacuum system used for obtaining macro-VCM data. Complete design drawings for the macro-VCM sample cells, fixtures, and assembly are given in Part II of this report: "Test Equipment." During a run, the sample cells are maintained at 125° C; operation records indicate that the chamber pressure ranges from about 10^{-6} torr in the 24- and 48-hr runs to about 10^{-7} torr in the 96- and 336-hr runs. Since preliminary work indicated that "blank" collector plates adjacent to plates on which material is deposited suffer no increase in weight, a full complement of 6 samples (in duplicate) has been used for making most of the macro-VCM determinations reported at this time.

A macro-VCM determination consists of measuring the amount of VCM collected (and weight-loss incurred) from polymeric products at intervals of 24, 48, 96, and 336 hours in an environment of 125° C and 10^{-6} torr. Details of the procedure for making macro-VCM determinations are given in Appendix C. It is to be emphasized that samples are not taken out and reinserted for additional periods of time; each run in the series

is made up of fresh samples. In fact, the 336-hr run is often performed before the 24-hr run for maximum utilization of hours over week-ends and holidays. (The use of a refrigerating system (Freon-502) eliminates the need for filling cold-traps by hand.)

Although samples must be comminuted for micro-VCM determinations (maximum VCM in wt- $\frac{4}{3}$), configurations such as sections of sleevings, honeycombs, electronics modules, etc. can easily be accommodated in the macro-VCM sample cells. The cells have a maximum utilizable volume of about 12 cubic inches. The results obtained with the macro-VCM apparatus are definitive and provide the data by which molymer candidacy for space-craft use is determined; micro-VCM results are only of use for eliminating grossly inadequate polymers. The macro-VCM apparatus is also useful in that it provides easily-visible evidence of the effect of a thermal-vacuum environment on polymers. For example, the micro-VCM results for the weight-loss and VCM content of Hexcel homeycomb core structure HMH qualify it as a good candidate for spacecraft use; however, a section $(2'' \times 2'' \times 1'')$ of the honeycomb collapsed during exposure to the thermal-vacuum environment, thus indicating its unsuitability for use at the temperature under consideration.

Eventually, data obtained from the macro-VCM determinations may be used for definitive studies of the diffusion rates of the volatile substances relative to thickness of materials or surface-to-volume ratios. At the present time, data are expressed in wt-% VCM/wt-sample since the available materials have been of the order of less than 0.1" in thickness and all surface areas have been exposed.

Macro-VCM data are summarized in Tables XIX to XXI for several seal and gasket materials, a sleeving and shrinkable tubing, and various structural materials; the behavior of the VCM with time, that is, accumulation of re-evaporation, is illustrated by the curves in Figures 4 to 6, plotted on a wt-\$ basis.

C. IDENTIFICATION OF VOLATILE MATERIAL

Confirmation of the effectiveness of the micro-VCM determination as a screening technique for the selection of candidate spacecraft materials is afforded by the macro-VCM determinations which provide information on the observed weight-loss and VCM deposition and or evaporation with time. Since all polymers release substances volatile at $125^{\circ}C$ in vacuo, mass spectrometric examination of the volatile substances has been undertaken with a two-fold purpose: (1) The identification of readily-volatile substances which cause the observed weight-loss in a thermal-vacuum environment, and (2) The identification of substances which may be responsible for the observed VCM (volatile condensable material).

The identification of the low-molecular-weight materials given of by polymers may be accomplished with simple mass spectrometer systems, such as quadrupole mass filters or residual gas analyzers; however, these materials are the volatile components which usually constitute the greater portion of the weight-loss (90%). The high-molecular weight materials, which contribute to VCM, such as low n-mer resins, plasticizers, etc. require examination by specially-designed instruments with mass range capabilities in excess of m.w. 500; since these kinds of materials have very low vapor pressures, samples must often be handled via a directintroduction system.

The mass spectrometer used at SRI for the identification of released polymeric substances is a CEC Model 21-103C, which has been modified to include an ion-pump exhaust system (which ensures that any detected hydrocarbons emanate only from the material under examination) and a versatile sample-inlet system (see Figure 7) which permits operation of the spectrometer with or without the restricting molecular leak as well as a direct line-of-sight path for vaporized molecules into the ionizing region. A representative mass spectrum obtained with the modified system is given in Figure 8 for RTV-60 (formulated by the General Electric Company). The presence of end-blocked $[Si(CH_3)_3]$ straight-chain dimethylsiloxanes is indicated (m/e 73, 147, 221, 295, etc.) as are cyclic dimethylsiloxane structures (m/e 207, 281, 355, etc.).

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The typical 74 a.m.u, sequence of intensified peaks corresponding to loss of $SiO(CH_3)_2$ is shown, as well as the decreasing intensity of significant peaks with increasing molecular weight.

The samples used for mass spectrometric examination have been finished polymers either provided by suppliers or cured (postcured) according to manufacturer's instructions. They are selected on the following bases:

(1) Qualification by micro-VCM determinations;

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- (2) Correlation with other phases of the program, e.g., mechanical properties;
- (3) Questions raised by suppliers, users, or JPL Cognizant Engineers.

Briefly, the procedure consists of placing the sample in the modified inlet system and pumping it overnight at room temperature to remove surface moisture and gases. Then the sample is heated quickly to 125° C, and the spectrum of volatilized substances is recorded within a few minutes. The volatile substances in samples of the order of 1-3 mg are nearly completely vaporized within 10-15 minutes (or in less than one hour) at this temperature.

The mass spectra may reveal a complexity of 2 to >10 volatilized substances; since reference spectra are not available for most of these materials, particularly low-molecular-weight resins and plasticizers, data cannot be reported as quantitative values but are reported rather as major and minor components, according to general structure. For example, of 15 phthalic acid esters used commercially as plasticizers, mass spectral information is readily available for only 4; on the other hand, identification as hydrocarbon oils, common solvents, and common gases is easily ascortained.

Mass spectrometric identification of volatile substances from a number of polymeric products is summarized in Table XXII. It is readily seen that substances which might contribute to VCM may be either a major or minor component of the volatilized substances. The utilization of this information, together with observations from micro- and macro-VCM

determinations, contributes to the ensuing discussion of results (Section II-D).

The importance of identification of volatile components is illustrated by a mass spectral study of Eccofoam SH (Emerson and Cuming, Inc.). Since it is commonly believed that the weight-loss of polyurethane foams is due almost entirely to the release of carbon dioxide, it was interesting to examine this typical commercial product (see Table V). Because carbon dioxide can be pumped away at ambient conditions, its loss was obviated in the following way: the sample was placed in a tube connected to the standard 3-liter reservoir of the mass spectrometer and evacuated after the sample was cooled to liquid nitrogen temperature. When a pressure of $<10^{-6}$ torr was indicated, the LN₂ Dewar was removed, and the substances released from the sample at 25°C were allowed to expand into the reservoir for 4 hours; the analysis of the mass spectrum of these substances is given in Table XXIII. Excluding water, which could easily be derived from the walls of the sample vessel, it is readily seen that nitrogen (not from air) is the major component of the evolved substances, and carbon dioxide is but a small contributor. Because it readily polymerizes, the styrene in the volatile materials is the most likely substance which can contribute to VCM.

Another sample of Eccofoam SH was pumped overnight at room temperature in the modified inlet system according to stand ' procedure, and then heated quickly to 125° C. The mass spectrum recorded at this time indicated the same components in relatively the same proportions (see Table XXIII) except for water content and nitrogen. Neither at 25° C conditions nor 125° C conditions does the carbon dioxide content of volatilized substances exceed 15%. Hence, experimental observations or conclusions made under the assumption that carbon dioxide is the principal gaseous component of this polyurethane foam should be re-examined.

D. DISCUSSION OF RESULTS

There are distinct correlations between the results of micro-VCM, macro-VCM, and mass-spectrometric determinations which provide the basis for qualification of the polymeric products which have been examined. The major correlations are summarized in Table XXIV and the following discussions of materials, arranged according to spacecraft applications, will note additional correlations and will suggest (for future work) means for up-grading existing polymers.

<u>Adhesives</u>. - The only suitable adhesives for spacecraft use are those based on pure epoxy structures. As shown in Table I, silicones are unacceptable and polyesters are marginal; the combination epoxy-polyamide adhesive films, FM-1000 series, have been shown to release low-molecular weight resins under thermal-vacuum conditions (Table XXII). The candidate adhesives listed in Table XVIII have qualified under mild curing conditions; thus, the first approach to up-grading these materials would be an extension of the curing time at the maximum recommended temperatures. The time devoted to curing prior to a space mission is wellspent if ultimate performance can be assured.

Circuit boards. - Three of the four circuit-board materials which have been screened (Table II) in the as-received condition qualify as candidate space-grade materials (Table XVIII) and consist of epoxy-glass fiber laminates. As shown in Table XXI and Figure 6, macro-VCM data not only confirms the eligibility of Micarta 65M25, but also indicates that the VCM is re-evaporating with time. However, mass spectrometer data indicates that the volatile material includes styrene (Table XXII) which vaporizes at relatively low temperatures but can also re-polymerize. Thus, it is suggested that the styrene be eliminated from the formulation if it cannot be removed by a reasonable postcure.

<u>Coated fabrics</u>. - As shown in Table III, silicone-coated fabrics are not acceptable for spacecraft use. The suitable fabrics (Table XVIII) are fluorocarbon-coated glass, polyester, or polyamide. Since these materials were used in the as-received condition, a simple postcure may be all that is needed for improved performance.

Films and sheets. - Most of the films and sheets listed in Table IV that could qualify for candidacy are fluorocarbon-type polymeric materials (Table XVIII). Since they have been screened only in the as-received condition, studies of postcures are in order as well as identification of possible degrading components. Nylons are not, in general, good spacecraft materials since they tend to de-polymerize at relatively low temperatures; the polyesters are marginal. Mass spectrometric identification (Table XXII) has shown that low-molecular-weight polyether glycols (as well as a plasticizer) are released by Mylar 500A. (See also "Mechanical Properties.") 1

Foams. - On the basis of VCM content, the silicones are entirely unacceptable as foams (Table V); the epoxy foams are suitable candidates (Table XVIII), and it is possible that some may be up-graded by more extensive curing. Acceptable VCM values are indicated for the pre-foamed polyurethanes, but the weight losses are excessive; identification of the released material from a sample of Eccofoam SH in the as-received condition (Table XXII) indicates the presence of a reactant, solvent, and additive as well as nitrogen and some carbon dioxide. As shown in Table V, a postcure of 24 nours at 150°C reduces drastically the weightloss value, but does not affect the VCM content. It seems probable that the VCM can be attributed to styrene, but another analysis would have to be made on a postcured material to determine whether only gases and solvents were driven off in postcuring or whether polymerization was enhanced. On the other hand, the mechanical performance of foams after postcuring or after exposure to a thermal-vacuum environment will be the deciding factor for selection; in this instance, weight-loss data may provide more screening information than VCM data.

Hardware and structural materials. - A great many of the hardware and structural materials screened appear to be suitable candidates for spacecraft use (Table XVIII). As shown in Table VI, the only class of structural materials disqualified at this time are the polyamides (nylons). The others might be up-graded by simple postcuring. Many of these products were screened in the as-received condition; postcures were carried out for the others on the recommendation of the Cognizant Engineer.

Further qualification of some of these materials indicates the generallygood outgassing characteristics; final recommendation requires mechanical property data.

Macro-VCM determination for PPO 681-111 (Table XXI) confirms the acceptance by micro-VCM criteria and also indicates the re-evaporation with time of VCM as shown in Figure 6; the most likely contributor to VCM has been identified as a terpene oil (Table XXII). The difference in reported weight-loss values is no doubt due to the fact that the film was annealed prior to micro-VCM determinations (which probably released excess solvents) but was used "as-received" for macro-VCM. 'See also "Mechanical Properties.")

Similar correlation of results for Lexan 141-112 is shown by the micro-VCM data in Table VI and the macro-VCM data in Table XXI; the VCM appears to be constant (Figure 6) but at an extremely low level, <0.01%; again, a terpene oil (Table XXII) may be the contributor to VCM. (Mechanical property data indicates the unsuitability of this material for structural use at temperatures of the order of 125°C.)

A phthalate plasticizer has been identified (Table XXII) as the probable contributor to the small VCM value for Delrin 100NC10 (Table VI).

The polyethylene plastic (Table VI) was examined because of its potential use in spacecraft optical experiments, since it is used as a window for far infrared spectrophotometers. However, low-molecular-weight polyethylenes are released at 125° C in vacuo (verified by mass spectroscopy) and deposit as VCM. Since this temperature is in excess of that feasible for photometric gear, this important material will be reexamined at a lower temperature.

Honeycomb structures. - As shown in Table VII, the silicone-based structures cannot be qualified. During the macro-VCM determination (Table XXI) the polyester composite was observed to collapse in the thermal-vacuum environment and is thus eliminated. The phenolic material might be upgraded by postcuring, but at the present time, no honeycomb structures can be suggested as suitable candidates for spacecraft use.

Lubricants. - Of the lubricants screened (Table VIII), none can approach candidacy for spacecraft use.

Protective coatings. - As indicated by the micro-VCM data in Table IX, few protective coatings can qualify for spacecraft candidacy as prepared according to manufacturer's instructions for general use. In some instances, curing temperatures in excess of 150° C are recommended for high-temperature applications; however, electronic components, such as printed-circuit boards or solid-state devices cannot be baked at such temperatures; perhaps an extension of curing time at moderate temperatures might be appropriate for a number of the coating materials. On the other hand, the nature of the substances contributing to weight-loss and VCM must be established before recommendations for curing or change in formulation can be made. As shown by the mass spectral data in Table XXJI for JPL-1002, a polyurethane coating, the weight-loss attributable to solvents could certainly be reduced by extended curing; whether the toluene diisocyanate released in the curing process (contributing to VCM) can be eliminated by extended curing must be determined.

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Sealants. - The only candidate sealants at this time are two epoxy products (Table XVIII) which were postcured 24 hours at $150^{\circ}C$; these materials possibly can be up-graded by extended curing at a more moderate temperature. The excellent fluorocarbon sealant is more properly a pipe-dope, whereas the real need is for potting compounds (or casting resins) and crack-fillers. As shown in Table X, RTV-type silicones, polyethylenes, and polyurethanes do not approach space-grade candidacy, even after harsh cures of 24 hours at $150^{\circ}C$.

Seals and Gaskets. - As with hardware and structural materials, seal and gasket materials in general (Table XI) are excellent candidates for spacecraft applications. Since these materials can be postcured at elevated temperatures prior to use, appropriate cures may up-grade the products. However, the small amounts of volatile material must still be identified, and the effect on their removal on mechanical properties must be determined. For example, the material released by Hycar-1 (not recommended) has been shown to be high-molecular-weight hydrocarbons, often used as

plasticizers; macro-VCM data summarized in Table XIX and Figure 4 indicate a rising increase in weight-loss and VCM with time. The effect of this loss in plasticizer is reflected in the relatively poor performance of this material in mechanical property tests (vide infra).

A note of cation must be made toward procurement of elastomeric products (as well as others) by code-names. For example, extreme variations in weight-loss and VCM values were observed for SE-3604, a silicone elastomer designed by the General Electric Company. It was found that the SE-3604 formulated by the Stillman Rubber Division (Sargent Industries) could not compare with that formulated by GE. After a suggested postcure of 3 hours at 205° C, the Stillman product still could not match the performance; subsequently, it was revealed that the GE material had been postcured for 24 hours at 250° C. Thus, the need for specifications and quality-control procedures is quite apparent. According to the macro-VCM data shown in Table XIX and Figure 4, the Stillman SE-3604 cannot be recommended for GE SE-3604 (vide infra) indicates a superior and recommended material.

The macro-VCM data in Table XIX for Viton-990 indicates the reevaporation of VCM with time. The most probable contributors to VCM have been identified as plasticizers such as dibenzyl ether and dioctylphthalate (see Table XXII); a postcure may be sufficient for removal of such substances.

The small amount of plasticizer identified with Teflon FEP 500A (Table XXII), and measurable after 24 hours in the micro-VCM apparatus (Table XI), apparently evaporates from the VCM collector plate during the first 48-hr run in the macro-VCM determination. Nevertheless, for critical applications, it is recommended that the FEP be postcured prior to use, preferably under vacuum. (See also "Mechanical Properties.") <u>Shrinkable tubing</u>. - The fluorocarbon shrinkable materials (Table XII) easily qualify as space-grade candidates in micro-VCM screening. Since two of the polyolefins had acceptable weight-losses, a further evaluation was made of Thermofit RNF-100 in order to determine whether the VCM

was of 1 kind that would re-evaporate with time. As shown by the data in Table XX and the increasing VCM value with time (Figure 5), the disqualification by micro-VCM screening was valid.

<u>Sleeving</u>. - The best sleeving materials tested appear to be formulated from acrylic-glass fiber combinations (Table XIII); however, macro-VCM determinations for Ben Har Acryl A indicate increasing VCM with time (Table XX and Figure 5). More work is needed before recommendations can be made for, say, Ben Har Lecton B and Ben Har Acryl A. It is surprising that no fluorocarbon materials have been submitted for evaluation as sleevings.

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Tapes. - Virtually none of the tapes, except Mystik 7452, a rubber-based resin/aluminum, can qualify as space-grade even under curing conditions of 24 hours at 150°C (Table XIV); a macro-VCM determination is in progress for Mystik 7452.

Temperature control coatings. - None of the temperature-control coatings screened thus far can qualify as space-g_ade candidates; however, several

er products have been submitted for testing. It is possible that the coatings listed in Table XV may be improved by additional curing. It is doubtful that apparatuses containing mirrors and lenses can withstand rigorous treatment, and thus extensive curing at moderate temperatures may have to be recommended.

Tie cord/lacing tapes. - The best materials for tip cord applications appear to be an impregnated fluorocarbon fiber and possibly a Dacronsynthetic rubber product after postcuring (Table XVI). These materials merit further consideration.

Wire enamels. - In general, the few wire enamels screened are satisfactory as shown by the data in Table XVII. However, it must be remembered that a large part of the sample weight of the magnet wires is comprised of bare wire. More work is needed really to qualify these enamels.

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MICRO-VCM DETERJINATIONS: ADHESIVES (24 hr at 125°C and 10⁻⁶ torr) (VCM collector plates at 25°C)

MATERIAL ¹	MFR. ²	TREATMENT	TOTAL WT. LOSS, 7	VCN, WT-%
Epoxy				
Armstrong A-2/A	APC	Cured 45 min at 74°C plus 45 min at 93°C	ə. 17	0.03
Armstrong A-2/E	APC	Cored 45 min at 93°C	0,26	0.03
Eccobond 45/15	EMC	Cured 1/2 hr at 70°C	0.55	0.05
Eccobond 55/9	EMC	Cured 24 hr at 25°C	0.40	0.06
Eccobond 55/9	EMC	Cured 16 hr at 65°C	0.17	0.07
Eccobond 55/9	EMC	Cured 24 hr at 150°C	0.15	0.10
Eccobond 55/11	EMC	Cured 1/2 hr at 150°C	3.76	3.40
Eccobond 55/11	EMC	Cured 24 hr at 150°C	0.43	0.43
Eccobond Solder 56C/9	EMC	Cured 16 hr at 50°C	0.20	0.03
Eccobond Solder 56C/9	EMC	Cured 16 hr at 65°C	0.42	0.03
Eccobond 104 A/B	EMC	Cured 3 hr at 150°C	0.30	0.24
Epon 828/Versamid-125	SCA	Cured 1 hr at 65°C plus 1 hr at 95°C plus 24 hr at 150°C	0.51	0.12
Epon 828/Versamid-125	SCA	Cured 1 hr at 65°C plus 1 hr at 95°C plus 1 hr at 150°C	1.65	0.77
Epon 901/B-1	SCA	Cured 1 hr at 93°C	0.34	0.14
Epon 901/B-3	SCA	Cured 1/2 hr at 115°C plus 1-1/2 hr at 175°C	0.19	0.01
Epon 903	SCA	Cured 1 hr at 175°C	0.36	0.21
Epon 914	SCA	Cured I hr at 175°C	0.24	0.15
Epon 917	SCA	Cured 15 min at 175°C	0.17	0.03
Е _{рол} 919 А/В	SCA	Cured 3 hr at 92°C	1.45	0.23
Epon 931 A/B	SCA	Cured 1 hr at 125°C	0.13	0.01
Epon 934 A/B	SCA	Cured 1 hr at 82°C	8.31	4.64
FM-1000	ACB	Cured 1 hr at 150°C	8,31	4.64
FM-1000	ACB	Cured 2 hr at 150°C	6.22	5.08
FM-1000	ACB	Cured 2 hr at 170°C	5.39	4.35
FM-1000	ACB	Cured 2 hr at 200°C	5.55	4.71
FM-1044	ЛСЬ	Cured 1 hr at 150°C	7.95	_3.76
rM-1044	ACB	Cured 24 hr at 150°C	5.84	2.99
Scotchweld EC-2216 A/B	MMA	Cured 8 hr at 25°C plus 1/2 hr ɛ. 125°C	0.60	0.24
Polyester				
Adhesive 46591	DUP	Cured 16 hr at 25°C	0,60	0.24
Cement 4684	DUP	Cured 16 hr at 25°C tlus 4 hr at 150°C	7.26	5,62

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MATERIAL	MFR. ²	TREATMENT	TOTAL WT. LOSS, 7	VCM, WT-%
Silicone				
Chenlok 607	HCC	Cured 24 hr at 25°C	12.62	1.02
Chemlok 607	HCC	Cured 24 hr at 150°C	2.95	0.56
Silicone primers				
SS-4004	GES	Cured 24 hr at 25°C	22.67	7.87
SS-4004	+ GES	Cured 24 hr at 150°C	0.40	0.20
SS-4044	GES	Cured 24 hr at 25°C	19,67	8.02
SS-4044	GES	Cured 24 hr at 150°C	1.05	0.35
SS-4101	GES	Cured 24 hr at 25°C	23.76	7.35
SS-4101	GES	Cured 24 hr at 150°C	6.72	4.16
SS-4120	GES	Cured 24 hr at 25°C	24.98	14.60
SS-4120	GES	Cured 24 hr at 150°C	2.00	0.00

Table I (Concluded)

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 1 See Appendix A for basic structure of polymers. 2 See Appendix B for Code List of Manufacturers.

Table II MICRO-VCM DETERMINATIONS: CIRCUIT BOARDS (24 hr at 125°C and 10⁻⁶ torr) (VCM collector plates at 25°C)

MATERIAL ¹	MFR. ²	TREATMENT	TOTAL WT. LOSS, %	VCM, WT-%
Epoxy-glass laminate				
Micarta II-8457	WEM	As received	0.80	0.12
Micarta H-17511	WEM	As received	0.44	0.04
Micarta H-2497	WEM	As received	0、18	0.00
Epoxy-glass laminate, copper-clad				
Micarta 65M25	WEM	As received	0.43	0.00

¹ See Appendix A. ² See Appendix B.
Table III

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

MATERIAL	MFB. ²	TREATMENT	TUTAL WT. LOSS, %	VCN, WT-%
Fluorocarbon-coated polyamide				
Armalon 98-101	DUF	As received	0.48	0.02
Fluorocarbon-coated polyester			1	
Fairprene 84-001	DTF	As received	1.33	0.21
Fairprene 80+060	DUF	As received	0.34	0.01
Fairprene 80-070	DUF	As received	0.30	0.01
Fairprene 80-080	DUF	As received	0.30	0.01
Fluorocarbon-coated glass				
TB5-PTFE	MMI	As received	0.05	0.05
Silicone-coated polyester				
SND-5903	MM1	As received	0,89	0.43
Silicone-coated glass				
SRG-1810	MME	As received	0.70	0.39
Silicone-coated aluminized glass				
SHGA-0214	MMI	As received	0.62	0.34

See Appendix A.
 See Appendix B.

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Table IV

MICRO-VCM DETERMINATIONS: FILMS AND SHEETS (24 hr at 125°C and 10⁻⁶ torr) (VCM collector plates at 25°C)

MATERIAL ¹	MFR. ²	TREATMENT	TOTAL WT. LOSS, %	VCM, WT-≫
Polyamide 6-Nylon	- Dum	As received	3.40	0.17
Folyimide Kapton 200X11667 Polyimide, fluorocarbon-coated	Dum	As received	0.14	0.09
Kapton 300XHF929A	DUM	As received	0.54	0.05
Polyterephthalate]
Mylar Type 500A	DUM	As received	0.24	0.06
Mylar Type 100S	DUM	As received	0.22	0.12
Mylar Type 100T	DUM	As received	0.20	0.12
Polyvinyl fluoride				
Tedlar Al30WH	DUM	As received	0.47	0.00
Tedlar 100BG30TR	DUM	As received	0.23	0.10
Tedlar 100BG30TL	DUM	As received	0.09	0.09
Polyvinylidene fluoride				
Kynar 200	PCC	As received	0.21	0.15

¹ See Appendix A.

² See Appendix B.

Table V

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MICRO-VCM DETERMINATIONS: FOAMS (24 hr at 125°C and 10⁻⁶ torr) (VCM collector plates at 25°C)

MATERIAL ¹	MFR. ²	TREATMENT	TOTAL WT. LOSS, %	VCM, WT-%
Ероху				
Stycast 1090/9	EMC	Cured 24 hr at 25°C	0.57	0.50
Stycast 1090/9	EMC	Cured 16 hr at 50°C	0.31	0.07
Stycast 1090/11	EMC	Cured 24 hr at 100°C	0.13	0.13
Stycast 1090/11	EMC	Cured 24 hr at 125°C	0.04	û.04
Stycast 1095/11	EMC	Cured 3 hr æt 95°C plus 3 hr at 156°C	0.92	0.11
Stycast 1095/11	EMC	Cured 24 hr at 125°C	0.50	0.11
Silicone				
Eccosil 5000 A/E	EMC	Cured 1 hr at 125°C	2.83	0.48
Eccosil 5000 A/B	EMC	Cured 24 hr at 150°C	2.01	0.50
Eccofoam Sil/25	EMC	Cured 1 hr at 95°C	14.95	11.55
Eccofoam Sil/25	EMC	Postcured 24 hr at 150°C	0.92	0.57 ⁻
Polyurethane				
Eccofoam S	EMC	As received	2.01	0.07
Eccofoam SH	EMC	As received	10.26	0.01
Eccofoam SH	EMC	Postcured 24 hr at 150°C	1,69	0.01
Eccofoam FPH/126H	EMC	Cured 24 hr at 25°C plus 3 hr at 150°C	2.25	0.15
Eccofoam FPH/126H	EMC	Postcured 24 hr at 150°C	1.39	0.20

¹ See Appendix A. ² See Appendix B.

Table VI

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MICRO-VCM DETERMINATIONS: HARDWARE AND STRUCTURAL MATERIALS (24 hr at 125°C and 16⁻⁶ torr)

(VCM collector plates at 25°C)

MATERIAU	MFR. ²	TREATMENT	TOTAL WT. LOSS, 7	VCM, WT-S
Acetal plastics				
Delrin 100NC10	DEP	As received	0.58	0.06
Detrin 150NC10	DOP	As received	0,56	0.06
Delrin 500NC10	DUP	As received	0.48	0.07
Detrin 507NC10	DIP	As received	3.12	0.11
Delrin 900NC10	DOP	As received	0,56	0.08
Diallylphthalate plastics				
Diall FS-4	ΛСМ	Postcured 24 hr at 150°C	0.58	0.02
Diall_FS-10	ACM	Postcured 24 hr at 150°C	0.70	0.03
Diall FS-40	ACM.	Postcured 24 hr at 150°C	1.00	0.02
Diphenyl oxide-glass laminate				
Doryl 11-17479	WEM	Postcured 24 hr at 150°C	0.34	0.13
Luoxy-glass fiber laminates				
Micaply G-284	TMC	As received	0.49	0.06
Micaply EG-725	TMC	As received	1.13	0.37
Micarta II-17690	WEM	As received	0.48	0.07
Epoxy-glass molding compound				
Epiall 1914	ЛСМ	Postcured 24 hr at 150°C	0.55	0.03
Phenolic-glass molding compounds				
Phenall 8010	۸СМ	Postcured 24 hr at 150°C	1.25	0.02
Phenall 8060	лСМ	Postcured 24 hr at 150°C	1.56	0.02
Phenall 8700	ACM	Postcured 24 hr at 150°C	1.25	0.01
Phenolic-glass laminates	-			
Micarta II-5834	WEM	As received	0.70	0.03
91-LD-1581	ARP	As received	2.51	0.08

MATER IAL ¹	MFR. ²	TREATMENT	TOTAL WT. LOSS, %	VCM, WT-%
Polyamide plastics				
Zytel 101NC10	DUP	As received	3.58	0.21
Zytel-31	DUP	As received	1.85	0.42
Zytel-42	DUP	As received	2.57	0.26
Polycarbonate plastics				
iexan 100-111	GEC	As received	0.06	0.02
Lexan 101-111	GEC	As received	0.08	0.01
Lexan 101-112	GEC	As received	0.09	0.04
Lexan 103-112	GEL.	As received	0.08	0.00
Lexan 130-111	GEC	As received	0.17	J.01
Lexan 131-111	GEC	As received	0.18	0.01
Lexan 131-112	GEC	As received	0.17	0.01
Lexan 133-112	GEC	As received	0.20	0.01
I.exan 140-111	GEC	As received	0.17	0.03
Lexan 141-111	GEC	As received	0.17	0.04
Lexan 141-112	GEC	As received	0.17	0.02
Lexan 243-112	GEC	As received	0.16	0.01
Polyethylene plastic				
Beckman #101577	DCC	As received	0.62	0.35
Polyimide plastic				
Vespel SP-1	DUP	As received	1.24	0.01
Polyphenylene oxide film				
PPO 681-111 (clear)	GEC	Annealed 1/2 hr at 180°C	0.07	0.05
PPO 531-081 (opaque)	GEC	As received	0.09	0,02
Polysulfone films				
P-2300	UCP	Annealed 1/2 hr at 125°C	0.03	0.01
P-7395-121-2	UCP	As received	0.33	0.01
Silicone-glass laminate				
Micarta 20201-2	WEM	As received	0.16	0.04

Table VI (Concluded)

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¹ See Appendix A. ² See Appendix B.

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Table VII

MICRO-VCM DETERMINATIONS: HONEYCOMB CORE STRUCTURES (24 hr at 125° C and 10^{-6} torr) (VCM collector plates at 25° C)

MATERIAL ¹	MFR. ²	TREATMENT	TOTAL WT. LOSS, %	VCM, WT-%
<u>Phenolic</u> Hexcel HRP Polyester	HFX	As received	1.30	0.20
Hexcel HMH	HEX	As received	0.18	0.17
<u>Silicone</u> Hexcel-HRS-asbestos Hexcel HRS-glass fiber	HEX HEX	As received As received	0.37 n,50	0.37 0.40

 $\stackrel{1}{\overset{}_{-}}$ See Appendix A for basic polymer structures.

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² See Appendix B for Code List of Manufacturers.

Table VIII

MICRO-VCM DETERMINATIONS: LUBRICANTS (24 hr at 125°C and 10⁻⁶ torr) (VCM collector plates at 25°C)

MATERIAL	MFR. ²	TREATMENT	TOTAL WT. LOSS, %	VCM, WT-%
<u>Silicate-MoS₂-G</u> Electrofilm 2396	EFI	Cured 2 hr at 80°C plus 2 hr at 205°C	3.19	0.87
Phenolic-MoS ₂ Electrofilm 4306	EFI	Cured 1-1/2 hr at 190°C	1.37	1.25
<u>Fluorocarbon</u> PR-240-AC	DUP	As received	20.02	7.42

¹ See Appendix A.

² See Appendix B.

Table IX

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MICRO-VCM DETERMINATIONS: PROTECTIVE COATINGS (24 hr at 125°C and 10⁻⁶ torr) (VCM collector plates at 25°C)

MATERIAL	MFR. ²	TREATMENT	TOTAL WT. LOSS, %	VCM, WT-%
Alkyd				
13224-2	WEI	Cured 3 hr at 95°C plus 1 hr at 150°C	8.99	3.86
Glyptal 1201	GEI	Cured 4 hr at 125°C	6,10	2.55
Glyptal 1201	GET	Cured 24 hr at 150°C	3.70	1.60
Glyptal 1202	GEI	Cured 4 hr at 125°C	5.55	2.46
Glyptal 1202	GEI	Cured 24 hr at 150°C	5.20	2.78
Diphenyl oxide	1			
Doryl B109-4	WEI	Cured 2 hr at 250°C	0.30	0.15
Dory1 B109-5	WEI	Cured 2 hr at 250°C	0.18	0.14
Epoxy	·			
<u>B-276</u>	WEI	Cured 3 hr at 95°C plus 1 hr at 150°C	3.52	1.15
Eccocoat C-26 A/B	EMC	Postcured 24 hr at 150°C	2.10	0.31
Eccocoat EC-200 A/B	EMC	Postcured 24 hr at 150°C	3.65	1.35
Eccocoat 210 A/B	EMC	Cured 24 hr at 25°C plus 1 hr at 75°C	2.86	0.36
Eccocoat 210 A/B	EMC	Cured 24 hr at 25°C plus 24 hr at 150°C	0.78	0.36
Eccocoat PCA/16	ENC	Cured 1 hr at 25°C plus 2 hr at 95°C plus 2 hr at 150°C	0.18	0.02
Eccocoat VE A/B	EMC	Postcured 24 hr at 150°C	1.51	0.52
Eccogel 1265 A/B	EMC	Postcured 24 hr at 150°C	16.59	8.66
PT-401/H-11	PTI	Cured 20 minutes at 93°C	18.29	0.65
Stycast 1217/9	EMC	Postcured 24 hr at 150°C	0.57	0.12
Polyester-phenolic				
Eccocoat PH7	EMC	Postcured 24 hr at 150°C	1.55	0.84
Phenolic-oil-modified				
Glyptal 9564	GEI	Cured 4 hr at 125°C	5.19	2.05
Glyptal 9564	GEI	Cured 24 hr at 150°C	3.62	1.35
Polyimide	}	· ·		
PYRE-M.L. RK-692	DUF	Cured 24 hr at 150°C	3.00	0.48
Polyurethane				
Eccocoat IC-2	EMC	Cured 1 hr at 25°C plus 2 hr at 95°C plus 24 hr at 150°C	6.99	1.48
JPL-1001	AAC	Cured 4 hr at 75°C	0.20	0.10
JPL-1002	AAC	Cured 4 hr at 75°C	0.19	0.03
Silicone	<u> </u>			
SR-17	GES	Postcured 24 hr at 150°C	1.22	0.80
SR-98	GES	Postcured 24 hr at 150°C	2.48	1.61
SR-220	GES	Postcured 24 hr at 150°C	3.52	2.86

¹ See Appendix A. ² See Appendix B.

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Table X MICBO-VCM DETERMINATIONS: SEALANTS (24 hr at 125°C and 10⁻⁶ torr) (VCM collector plates at 25°C)

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MATERIAL	MFR. ²	TREATMENT	TOTAL WT. LOSS, %	VCM, WT-%
Ероху				
Scotcheast 235	MMF	Cured 6 br at 95° C	6.20	0.31
Scotchcast-233	MME	Cured 6 hc at 95°C	2.97	0.04
Stycast 1210 A/B	EMC	Postcured 24 hr at 150°C	1.67	0.05
Stycast 1263 A/B	EMC	Postcured 24 hr at 150°C	0.12	0.01
Stycast 1264 A/B	EMC	Cured 8 hr at 25°C	2.80	0.14
Stycast 1264 A/B	EMC	Pestcured 24 hr at 150°C	2.73	0.32
Stycast 1269 A/B	EMC	Postcured 24 hr at 150°C	0,18	0.05
Stycast 2741/15	EMC	Cured 8 hr at 25°C	10.63	2.00
Stycast 2741/15	EMC	Postcured 24 hr at 150°C	1.65	0.10
Fluorocarbon				
Bibbo Frank #520	DED	An annal and	0.07	0.02
Ribber Dave #520	PEN DED	As received Deptembed 24 kp at 150°C	0.07	0.02
11000h Dope * 520	rin	rostcured 24 m at 150 C	0.00	0.00
Polyester				
Stycast 40/7	EMC	Postcured 24 hr at 150°C	2.26	0.35
Polyethylene				
TPM-2/10	EMC	Cured 12 hr at 50°C plus 4 hr at 80°C	6,41	0.42
TPM-2/10	LiC	Postcured 24 hr at 150°C	3.80	1.05
TPM-3/10	EMC	Cured 12 hr at 50 C plus 4 hr at 80°C	3.33	0.90
TPM-3/10	EMC	Postcured 24 hr at 150°C	3.06	0.27
TPM-6 A/B	EMC	Cured 16 hr at 105°C plus 4 hr at 145°C plus 4 hr at 175°C	2.29	0.47
T₽M-6 A/B	EMC	Postcured 24 hr at 150°C	1.28	0.27
Polyurethane				
CPC-21	EMC	Cured 6 hr at 100°C	17.33	4.18
CPC-21	EMC	Postcured 24 hr at 150°C	15.32	4.03
CPC-22	EMC	Cured 40 hr at 65°C	29.86	5.48
CPC-22	EMC	Postcured 24 hr at 150°C	28.25	5.12
CPC-41	EMC	Cured 48 hr at 65°C	3.58	0.65
CPC-41	EMC	Postcured 24 hr at 150°C	1.67	0.35

MATERIAL ¹	MFR. ²	TREATMENT	TOTAL WT. LOSS, %	VCM, WT-%
Polyarethane				
PR-1527 A/B	PRC	Cured 6 hr at 82°C	1.65	0.44
PR-1527 A/B	PRC	Postcured 24 hr at 150°C	2.07	0.56
PR-1538 A/B	PRC	Cured 6 hr at 82°C	2.07	0,56
PR-1538 A/B	PRC	Postcured 24 hr at 150°C	1.96	0,53
Silicone				
RTV-11	GES	Postcured 24 hr at 150°C	0.91	0.55
RTV-41/T-12	GES	Cured & hr at 25°C plus 4 hr at 50°C	2.06	0.45
RTV-60	GES	Postcured 24 hr at 150°C	0.69	0.54
RTV-77/T-12	GES	Postcured 24 hr at 150°C	1.69	1.02
RTV-90	GES	Postcured 24 hr at 150°C	0.62	0.49
RTV-560	GES	Postcured 24 hr at 150°C	1.03	0.68
RTV-580/T-12	GES	Postcured 24 hr at 150°C	1.81	0.81
RIV-615	GES	Postcured 24 hr at 150°C	1.01	0.77
RTV-630 A/B	GES	Postcured 24 hr at 150°C	1.30	0.81
BTV-632 A/B	GES	Postcured 24 hr at 150°C	1.25	0.74
RTV-655 A/B	GES	Postcured 24 hr at 150°C	2,72	1.27
RTV-757	GES	Postcured 24 hr et 150°C	12.94	1.39
Sylgard-184	DCC	Cured 4 hr at 65°C	1.77	0.89
Sylgard-184	DCC	Postcured 24 hr at 150°C	0.94	0.62

Table X (Concluded)

¹ See Appendix A. ² See Appendix B.

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Table XI

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MICRO-VCM DETERMINATIONS: SEALS AND GASKETS

(25 hr at 125°(, and 10⁻⁶ torr) (VCM collector plates at 25°C)

MATELIAL	MFB. ²	TREATMENT	TOTAL WT. LOSS, %	VCM, WT-%
Acrylic				
Hycar 520-67-108-1	BFG	As received	1.90	0.17
Hycar 520-67-108-2	BFG	As received	1.22	0.13
Hycar 520-67-108-3	BFG	As received	1.31	0.08
Hycar 520-67-108-4	BFG	As received	1.70	0.04
Hycar 520-67-108-5	БFG	As received	0.95	0.03
Hycar 520-67-108-6	BFG	As received	1.03	0.03
Butyl				
Enjay EC-1090	ENJ	Postcured 4 hr at 150°C	0.80	0.24
Enjay EX-1091	ENJ	Postcured 4 hr at 150°C	0.70	0.20
Enjay EX-1092	ENJ	Postcured 4 hr at 150°C	0.86	0.10
SR-634-70	SIS	As received	1.55	0.21
Fluoroethylene-				
propylene				
Teflor, FEP 500C	DUP	As received	0.02	0.01
Teflon FEP 100A	DUP	As received	0.06	0 06
Teflon FEP 500A	DUP	As received	0.05	0.05
Silicone				
SE-555 (white)	GES	As received	0.55	0.33
SE-555 (grav)	GES	As received	0.53	0.30
SE-556	GES	Postcured 24 hr at 250°C	0.10	0.01
SE-3694 (24/480)	GES	As received	0.51	0.12
SE-3604	SIS	As received	1.71	0.73
SE-3604	SIS	Postcured 3 hr at 200°C	1.40	0.80
SE-3704	SIS	As received	1.37	0.60
SE-3704	SIS	Postcured 3 hr at 200°C	1.12	0 67
SE-3804	SIS	As received	1.70	0.62
SE-3804	SIS	Pos cured 3 hr at 200°C	0.81	0.49
SE-3613	GES	As received	1.05	0.55
SF-3613 (24/480)	GES	As received	0.09	0.06
SE-3713	GES	As received	0,82	0.61
SE-3713 (24/480)	GES	As received	0.20	0.09
SE-3813	GES	As received	1 70	0.61
SE-3813 (24/480)	GES	As received	0.27	0.04
SE-4511 (24/480)	GES	As received	0.19	0.10
SE-5403 (24/480)	GES	As received	0,07	0.03

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MATERIAL	MLR. ²	FREATMENT	TOTAL WT. LOSS,%	VCM. WT-%
Vinylidene fluoride- hexafluoropropylene				
Viton A4411A-776	DUE	As received	0.29	0.05
Viton A4411A-776	DUE	Postcured 24 hr at 200°C	0.03	0.01
Viton A4411A-777	DUE	As received	0.27	0.03
Viton A4411A-777	DCL	Postcured 24 hr at 200°C	0.01	0.01
Viton A4411A-778	DUE	As received	0.35	0.01
Viton A4411A-778	DUE	Postcured 24 hr at 200°C	0.03	0.00
Viton A4411A-990	DUE	As received	0.54	0.03

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¹ See Appendix A. ² See Appendix B.

Table XII

MICRO-VCM DETERMINATIONS: SHRINKABLE MATERIALS

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

MATERIAL	MFR. ²	TREATMENT	TOTAL WT. LOSS, %	VCM, WT-%
Fluorocarbon				
Thermofit TFE (7-30-09)	RAY	Postcured 1 hr at 150°C	0.01	0.00
Thermofit TFE (7-32-16)	RAY	Postcured 1 hr at 150°C	0.00	0.00
Thermofit TFE-R	RAY	Postcured 1 hr at 150°C	0.01	0.00
Pennlube II-SMT	PFC	Postcured 1 hr at 150°C	0.00	0 00
Flucrocarbon, irradiated Thermofit Kynar	RAY	Postcured 1 hr at 150°C	0.27	0.09
Polyclefin, irradiated				
Thermofit RNF-100	RAY	Postcured 1 hr at 150°C	0.78	0.20
Thermofit CRN (clear)	RAY	Postc. ed 1 hr at 150°C	0.52	0.28
Thermofit CRN (white)	RAY	Postcured 1 hr at 150°C	2.01	0.60
Thermofit CRN (black)	RAY	Postcu. ed 1 hr at 150°C	2.09	0.39

¹ See Appendix A. ² See Appendix B.

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Table XIII

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MICRO-VCM DETERMINATIONS: SLEEVING (24 hr at 125°C and 10⁻⁶ torr) (VCM collector plates at 25°C)

MFR. ²	TREATMENT	TOTAL WT. LOSS, \$	VCM, WT-77
HM	Postcured 24 hr at 150°C	0.18	0.14
BEM	Postcured 24 hr at 150°C	0.13	0.11
BHM	Posscured 24 hr at 150°C	0.56	0.35
BIM	Fistcured 24 hr at 150°C	1.40	0.27
™∰.	Postcured 24 hr at i50°C	0.09	0.09
ын	Postcured 24 hr at 150°C	0.22	0.05
BHM	Postcured 24 hr at 150°C	0.54	0.42
ENIM	Postcured 24 hr at 150°C	0.66	0.43
BHM	Postcured 24 hr at 150°C	0.31	0.31
BIR(Postcured 24 hr at 150°C	0.29	0.13
. B.M	Postcured 24 hr at 150°C	0.42	0.24
	MFR. ² HIM HIM HIM HIM SIM BIM BIM BIM BIM	MFR. 2TREATMENTHIMPostcured 24 hr at 150°CHIMPostcured 24 hr at 150°C	WFR. ² TREATMENT TOTAL WT. LOSS, 5 HM Postcured 24 hr at 150°C 0.18 HM Postcured 24 hr at 150°C 0.13 HM Postcured 24 hr at 150°C 0.13 HM Postcured 24 hr at 150°C 0.56 HM Postcured 24 hr at 150°C 0.09 JM Fistoured 24 hr at 150°C 0.09 JM Postcured 24 hr at 150°C 0.22 HM Postcured 24 hr at 150°C 0.54 HM Postcured 24 hr at 150°C 0.54 HM Postcured 24 hr at 150°C 0.66 HM Postcured 24 hr at 150°C 0.31 HM Postcured 24 hr at 150°C 0.31 HM Postcured 24 hr at 150°C 0.42

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See Appandix A.
 See Appendix B.

Table XIV

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

MATERIAL ¹	MFis. ²	TREATMENT	TOTAL WT. LOSS, %	VCM, WT-%
Ероху				
Scotch Electric Tape #62	MME	As received	1.11	0.97
Scotch Electric Tape #62	MME	Cured 24 hr at 150°C	0.86	0.70
Fluorocarbon-silicone				
Mystik 7503	BCM	As received	1.17	0.73
Mystik 7503	ВСМ	Cured 24 hr at 150°C	1.02	0.81
Polyester				
Mystik 7352	BCM	As received	4.34	1.58
Mystik 7352	BCM	Cured 24 hr at 150°C	1.60	0.72
Polyester-aluminum				
Scotch Tape #852	MME	As received	1.69	0.70
Scotch Tape #852	MME	Cured 24 hr at 150°C	0.57	0.40
Polyester-glass				
Scotch Tape #27	MME	As received	6.08	2.27
Scotch Tape #27	MME	Cured 24 hr at 150°C	4.37	2.29
Polyester-silicone				
Mystik 7300	BCM	As received	1.90	1.28
Mystik 7300	ВСМ	Cured 24 hr at 150°C	1.44	1.03
Rubber resin-aluminum				
Mystik 7452	BCM	As received	0.37	0.(4
Rubber resin-glass-Al				
Mystik 7455	ВСМ	As received	3.82	2.62
Mystik 7455	BCM	Cured 24 hr at 150°C	1.71	1.34
Rubber resin-glass				
Mystik 7020	BCM	As received	2.63	0.62
Mystik 7020	BCM	Cured 24 hr at 150°C	1.74	0.56

¹ Sce A_rpendix A. ² See Appendix B.

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Table XV

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MICRO-VCM DETERMINATIONS. TEMPERATURE-CONTROL COATINGS (24 hr at 125°C and 10⁻⁶ torr) (VCM collector plates at 25°C)

MATERIAL ¹	MFR. ²	TREATMENT	TOTAL WT. LOSS, %	VCM, WT-%
Alkyd, modified Optical Black 101-C10 Optical Black 101-C10	MMA MMA	Cured 24 hr at 25°C Cured 24 hr at 150°C	5,56 0,57	1.12 0.25
Epoxy Cat-A-Lac Flat Black 463-1-8 Cat-A-Lac Flat Black 463-1-8 Cat-A-Lac Clear 473-1 Cat-A-Lac Clear 473-1 Cat-A-Lac White Gloss 443-1-500 Cat-A-Lac White Gloss 443-1-500	FPC FPC FPC FPC FPC FPC	Cured 24 hr at 25° C Cured 24 hr at 150° C Cured 24 hr at 25° C Cured 24 hr at 25° C Cured 24 hr at 150° C Cured 24 hr at 25° C Cured 24 hr at 150° C	13.00 0.38 25.48 1.09 15.79 0.86	1.52 0.23 3.30 ⁻ .82 0.95 0.52

¹ See Appendix A. ² See Appendix B.

Table XVI

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MICRO-VCM DETERMINATIONS: THE CORD/LACING TAPE

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

MATERIAL ¹	MFR. ²	TREATMENT	TOTAL WT. LOSS, %	VCM, WT-%
Nomex-silicone				
Gudebrod 72111	GBE	As received	3.07	0.25
Gudebrod 721H	GBE	Postcured 24 hr at 150°C	2.58	0.10
Gudebrod 722S	GBE	As received	4.92	1.80
Gudebrod 722S	GRE	Postcured 24 hr at 150°C	2.44	0.62
Dacron-synthetic rubber				
Gude-Space 181)96	GBE	As received	1.87	0.09
Gude-Space 18D96	GBE	Postcured 24 hr at 150°C	0.42	0.10
Stur-D-Luce 18DH	GBE	As received	1.26	0.20
Stur-D-Lace 18DH	GBE	Postcured 24 hr at 150°C	0.34	0.06
Impregnated fluorocarbon	[
fibre				
Temp-Lace 1125611	GBE	As received	0.69	0.05

¹ See Appendix A.

² See Appendix B.

Table XVII

MICRO-VCM DETERMINATIONS: WIRE ENAMELS

(24 hr at 125°C and 10⁻⁶ torr) (VCM collector plates at 25°C)

HATERIAL ¹	MFR. ²	TREATMENT	TOTAL WT. LOSS, %	VCM, WT-%
<u>Acetal (Formex)</u> Magnet wire (AWG-35)	GEW	As received	0.06	0.03
Polyimide PYRE-M.L. R.C5057	GEI	Cured 24 hr at 150°C	1.12	0.00
Polyurethane Magnet wire (AWG-22)	GEW	As received	0.22	0.09

¹ See Appendix A. ² See Appendix B.

Table XVIII

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POLYMER PRODUCTS RECOMMENDED FOR FURTHER QUALIFICATION

BASED ON MICRO-VCM VALUES

(wt-loss, 1.0% or less;

VCM, 0.1 wt-% or less)

APPLICATION	PRODUCT	TABLE	BASIC POLYMER
Adhesives	Armstrong A-2/A Armstrong A-2/E Eccobond 45/15 Eccobond 55/9 Eccobond Solder 50C/9 Epon 901 B-3 Epon 917	I I I I I I	ероху ероху ероху ероху ероху ероху ероху
Circuit boards	Epon 931 A/B	I	epoxy
	Micarta H-1754	II	epoxy-glass
	Micarta H-2497	II	epoxy-glass
	Micarta 65M25	II	epoxy-glass-copper
Coated fabrics	Armalon 98-101	111	fluorocarbon-polyamide
	Fairprene 80-series	111	fluorocarbon-polyester
	TB5-FTFE	111	fluorocarbon-glass
Films and sheets	Kapton 200XH667	IV	polyimide
	Kapton 300XHF929A	IV	polyimide-fluorocarbon
	Mylar Type 500A	IV	polyester
	Tedlar Al30WA	IV	polyvinyl fluoride
	Tedlar 100BG30TR	IV	polyvinyl fluoride
	Tedlar 100BG30TL	IV	polyvinyl fluoride
Foams	Stycast 1090/9	v	ероху
	Stycast 1090/11	v	ероху
Hardware and structural	Delrin (most) Diall (all) Epiall 1914 Lexan (all) Micaply G-284 Micarta H-17690 Micarta H-5834 Micarta 20201-2 PPO 581-111 PPO 531-081 P-2300 P-7395-121-2	VI VI VI VI VI VI VI VI VI VI VI	acetal diallylphthalate epoxy-glass polycarbonate epoxy-glass epoxy-glass phenolic-glass silicone-glass silicone-glass polyphenylene oxide polyphenylene oxide polysulfone polysulfone

APPLICATION	, PRODUCT	TABLE	BASIC POLYMER
Noneycomb core structures	No recommendations		
Lubricants	No recommendations		
	Eccocoat PCA/16	IX	ероху
Protective coatings	JPL-1001	IX	polyurethane
	JPL-1002	IX	polyurethane
	Ribbon Dope #520	X	fluorocarbon
Sealants	Stycast 1263 A7B	x	ероху
	Stycast 1269	x	ероху
	Enjay EX-1092	XI	butyl
	Hycar 520-67-108-5	XI	acrylic
	Hycar 520-67-108-5	XI	acrylic
Seals and gaskets	SE-556	XI	silicone
	SE-3613 (24/480)	XI	silicone
	Teflon FEP 100A	XI	fluorocarbon
	Teflon FEP 500A	XI	fluorocarbon
	Teflon FEP 500C	XI	fluorocarbon
	Viton A4411A-700 series	XI	vinylidene fluoride- hexafluoropropylene
	Penntube II-SMT	XII	fiuorocarbon
Shrinkable tubing	Thermofit Kynar	X11	fluorocarbon
-	Thermofit TFE	XII	fluorocarbon
Sleaving	Ben Har Acryl A	XIII	acrylic
STEEVINK	Ben Har Lecton B	XIII	acrylic-glass
Tapes	Mystik 7452	XIV	rubber resin-Al
Temperature contro) coatings	No recommendations		
······································	Cude-Space 18D96	XVI	Dacron-rubber
Tie cord/lacing tape	Stur-D-Lace 18DH	xvi	Dacron-rubber
	Temp-Lace H256A	XVI	fluorocarbon
Wire enamels	Magnet wire (Formex)	XVif	aceta
	Magnet wire (urethane)	XVII	polyurechane

Table XVIII (Concluded)

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MATERIAL BOLVMER TYDE	PROPERTY	HOI 12	HOURS OF EXPOSURE AT 125°C AND 10 ⁻⁶ torr			REMARKS
(Mfr Code)		24	48	96	336	
SE-3604 silicone (SIS) Hycar 520-67-108-1 acrylic (BFG)	wt-loss, % VCM, wt-% wt-los:, % VCM, wt-%	0.28 0.12 1.19 0.10	0.24 0.14 1.29 0.05	0.50 0.18 1.24 0.12	0.57 0.29 1.48 0.15	Postcured 4 hr/205°C; Dimensions: 2" x 1.5" x 0.084" Samples as received; Dimensions: 6" x 1" x 0.08"
Viton A4411A-990 vinylidene fluoride- hexafluoropropylene (SIS)	wt-ioss, % VCM, wt-%	0.44 0.02	0.46 0.02	0.53 0.01	0.61 0.01	Samples as received; Dimensions: 6" x l" x 0.08"
Teflon FEP 500A fluoroethylene- propylene (DLE)	wt-loss, % VCM, wt-%	< 0.01 < 0.01	< 0. J1 < 0. 01	< 0.01 < 0.01	<pre>< 0.01 < 0.01</pre>	Samples as received; Dimensions: 6" x 1" x 0.02"

Table XIX

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MACRO-VCM DETERMINATIONS: SEAL AND GASKET MATERIALS

Table	XX
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MACRO-VCM DETERMINATIONS: SLEEVING AND TUBING

MATERIAL POLYMER TYDE	HOURS OF EXPOSUR PROPERTY 125°C AND 10 ⁻⁶ t		PROPERT Y	HOURS OF EXPOSURE AT 125°C AND 10 ⁻⁶ torf			REMARKS
(Mfr Code)		24	48 .	96	336		
Ben Har Acryl A acrylic-glass (BHM)	wt-loss, % VCM, wt-%	0.09 0.03	0.10 0.05	0.33 0.05	0.26 0.06	Postcured 24 hr/150°C; Dimensions: 1-1/2" x 5/8" o.d. (2 pcs.)	
<u>Thermofit RNF-100</u> polyclefin (irrad.) (RAY)	wt-loss, % VCM, wt-%		0.59 0.20	0.62).24	0.62 0.27	Postcured 1 hr/150°C; Dimensions: 6" x 5/16" o.d. (5-5/16" after cure)	

Table XXI

MACRO-VCM DETERMINATIONS: VARIOUS STRUCTURAL MATERIALS

MATERIAL POLYMER, TYDE	PROPERTY	HO 1 2	JRS OF EX 25°C AND 1	(POSURE /	NT T	REMARKS
(Mfr Code)		24	48	96	336	
PPO 681-111 polyphenylene oxide (GEC)	wt-los≤, % VCM, wt-%	0.57 0.07	0.67 0.00	C.74 0.01	0.72 0.01	Samples as received; Dimensions: 30" x 2" x 0.001"
Micarta 65M25 epoxy-glass-copper (WEM)	wt-loss, % VCM, wt-%	0.36 0.01	0.36 0.02	0.44 0.01	0.35 <0.01	Somples as received; Dimensions: 2" x 2" x 0.075"
Lexan 141-112 polycarbonate (GEC)	wt-loss, % VCM, wt-%		0.15 < 0.01	0.15 <0.01	0.17 < 0.01	Samples as received; Dimensions: 2" x 2" x 0.10"
<u>Hexcel HMH</u> polyester (HEX)	wt-loss, % VQM, wt-%		0.15 0.13	0.15 0.02	C.20 0.02	Samples as received; Dimensions: 2" x 2" x 1" (collapses during exposure)

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Table XXII

MASS SPECTROMETRIC IDENTIFICATION IN SITU OF SUBSTANCES RELEASED AT 125 °C AND 10^{-6} TORE BY SELECTED POLYMERIC MATERIALS

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MATERIAL	MED 2	IDENTIFIED COMPONENTS		
POLYMER TYPE	Mr IS.	Major	Minor	
<u>Delrin NCL00</u> (acetal)	DUP	water	ester of phthalic acid; cresol	
<u>FM-1000</u> (epoxy)	ACB	low m.w. (to 600) epoxy resins		
Hycar 520-67-108-1 (acrylic)	BFG	water; hydrocarbon oils to m.w. `400		
<u>JP11002</u> (urethane)	AAC	water; aromatic solvent	toluene diisocyanate; cellosolve acetate	
Lexan 10X-11X (several) (carbonate)	GEC	water; mixed hexanes	carbon dioxide; phenols; n-propylamine; hydrochloric acid; C ₃₀ terpene oil	
<u>Micarta 65M25</u> (epoxy-glass fiber)	WEM	methyl cellosolve	slyrene	
Mylar Type 500A (terephthalate)	DUM	-glycol to m.w. >500	water; dioctylphthalate	
<u>PPO 681-111</u> (polyphenylene oxide)	GEC	aromatic solvents	water; C ₃₀ terpene oil	
<u>HTV-60</u> (silicone)	GES	low m.w. (>600) silicones, straight-chain and cyclic		
Teflon-FEP 500A (fluoroethylene- propylene)	DUP	dioctylphtk-late; C ₃₀ terpene oi!	water; carbon dioxide	
Viton A4411A-990 (vinylidene fluoride- hexafluoropropylene)	DUE	water; benzyl ether	carbon dioxide (CF ₂) _n and CILF; dioctylphthalate	

¹ See Appendix A.

² See Appendix B.

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Table XXIII

MASS SPECTROMETRIC ANALYSIS OF SUBSTANCES RELEASED IN VACUO BY ECCOFOAM SH (EMERSON AND CUMING, INC.)

	ESTIMATED MOL-%			
IDENTIFIED COMPONENT	at 25°C	at 125°C		
Styrene	C.3	1		
Toluene	1.5	6		
N-methyl morpholine	3.4	12		
Carbon dioxide	2.6	- 11		
Nitrogen	62.	54		
Water	70.	15		
Miscellaneous	0.2	1		

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Table XXIV

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CORRELATION OF MICRO-VCM, MACRO-VCM, AND MASS SPECTROMETRIC DETERMINATIONS LEADING TO FINAL SELECTION OF POLYMERIC PRODUCTS FOR SPACECRAFT USE*

	MICRO-VCM VALUES.	MACRO-VCM	VALUES	ADDING MUN A 1913300	
MATERIAI.	MAXIMUM VCM, WC-%	VCM at 336 hr, wt-%	VCM Status at 336 hr	(Mass Spec Identifcn.)	RECOMMENDAT IONS
Hycar 520-67-108-1	0.17	0.15	increasing	>C ₃₀ hydrocarbon oils	disqualified
Viton A4411A-990	0.03	0.01	leveling	hrnzyl ether, dioctyl- phthalate	elimination of plasticizers; postcure
Teflon-FEP 500A	0.05	< 0.01	increasing	dioctylphthalate, C ₃₀ terpene oil	elimination of plasticizers; postcure
PPO 683 111	0.05	< 0.01	decreasing	C ₃₀ terpene oil	elimination of plasticizers; postcure
Mi Jaa ta úSM25	0.00	< 0.01	decreasing	styrene	elimination of styrene; postcure
Lexan 141-112	C. 02	< 0.01	decreasing	unpolymerized reactants	postcure

* This Table is not to be construed as recommendation for a particular product; it is offered as an illustration of the correlations which are necessary before any product can be recommended for spacecraft use.

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FIG. 1 MICRO-VCM APPARATUS MOUNTED ON 6-INCH CONSOLE

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FIG. 5 VCM VALUES AT 125/25°C WITH TIME FOR SLEEVING AND TUBING







FIG. 7 VIEW OF MODIFIED MASS SPECTROMETER INLET SYSTEM SHOWING SAMPLE TUBE IN PLACE

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FIG. 8 MASS SPECTRUM OF SUBSTANCES VOLATILIZED FROM RTV-60 AT 125°C AND 10-6. TORR

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III. MECHANICAL PROPERTIES OF POLYMERIC MATERIALS IN THE THERMAL-VACUUM ENVIRONMENT

by Norman Fishman

The changes in selected mechanical properties which are incurred during the exposure of polymeric materials to a thermal-vacuum environment have been measured for a number of elastomeric and plastic products. Descriptions of these products are summarized in Tables XXV and XXVI, including those which were reported in Interim Report No. 2 (March 1966); test data for the latter are repeated in order to provide a basis for comparison among families of polymers. Exposure conditions, designated by letter references in Tables XXV and XXVI, are summarized in detail in Table XXVII.

The methods used to evaluate the behavior of the test materials when exposed to the vacuum-thermal environment can be categorized as active or passive. The active test is performed on the specimen in <u>situ</u>, or while it is in the vacuum-thermal environment; the passive test consists of storage in the vacuum-thermal environment, followed by tests of mechanical properties at atmospheric pressure. The tests performed during this program include:

Active tests

In situ continuous and intermittent stress relaxation tests of elastomeric materials in the vacuum-thermal environment;

In situ constant strain tests of elastomeric materials in the vacuum-thermal environment;

In situ constant load tests of plastic materials in the vacuum-thermal environment.

Passive tests

Storage of specimens of elastomeric and plastic materials in the vacuum-thermal environment, followed by constant strain rate tests of control and exposed specimens.

A. TEST RESULTS

In situ continuous and intermittent stress relaxation tests of several silicone, polyacrylic, and butyl elastomers were conducted in duplicate. Results of these tests (excluding those previously reported) are presented in Figures 9 to 16, inclusive; summary data are included in Tables XXVIII and XXIX.

Specimens of each of the above materials, as well as some Viton and additional silicone elastomers, were also stored, under no-load conditions within the test chamber at 125°C, for subsequent constant strain rate tests. The effects of the vacuum-thermal environment on the tensile properties of these materials are presented in Tables XXX, XXXI, and XXXII.

In addition to the 1000-hour tests reported above, long-term tests of three selected elastomers were undertaken using the apparatus described in Part II of this report. These materials were stored in the vacuumthermal environment for 6550 hours (condition H, Table XXVII) at several levels of constant 3 rain and under zero strain. Table XXXIII presents a summary of the conditions of ring storage, rupture time, and postexposure tensile properties. The effects of this long-term storage on tensile properties of unstrained specimens are compared with control and 1000-hour data in Tables XXXIV and XXXV.

Several plastic materials were subjected to constant load tests and to passive storage in the vacuum-thermal environment. In the constant load tests, the loads were applied shortly after the temperature of the specimens was raised to 125° C. Results of the constant load tests are summarized in Table XXXVI; the creep data for Tedlar are shown in Figure 17. The effects of the vacuum-thermal environment on the tensile properties of the plastic materials are presented in Table XXXVII. Included in Tables XXXVI and XXXVII are results of the long-term exposure of polyphenylene oxide (condition J, Table XXVII).

B. DISCUSSION OF RESULTS: ELASTOMERIC MATERIALS

The behavior of elastomeric materials in the vacuum-thermal environment was investigated with in situ continuous and intermittent stress relaxation tests and with constant strain rate tests of specimens which had been stored in the vacuum-thermal environment. Examination of the results of both tests provides a relatively complete picture of the suitability of the materials for use in space.

The results of continuous and intermittent stress relaxation tests can be interpreted to yield information concerning the relative rates of aggregative and disaggregative reactions occurring during exposure of rubbery materials to a stringent environment: Aggregation includes all those reactions which have the effect of cross-linking or chain lengthening; disaggregation includes chain-scission or cleavage. The decay of stress occurring in elastomeric materials maintained at constant extension is a direct measure of the scission reactions, since cross-linking generally does not affect the stress in p continuously stretched specimen. The net effect of the cross-linking and prostion reactions is measured by allowing the rubber to remain unstretured and testing its strength at intervals, as in the intermittent test.

In the intermittent test, if the cross-linking reactions proceed more rapidly than scission, intermittently measured values of stress increase; conversely, if cross-linking is slower than scission, stress decreases. Thus, the ratio of stress, or force, at time t to the stress at time o, f(t)/f(o), in a continuous stress relaxation test, is a measure of the fraction of original network chains remaining uncut after time t. The difference between the stress ratios, f(t)/f(o), measured by the continuous and intermittent test is a measure of the relative concentration of new network links formed by aggregation compared to the original concentration of network chains; the differences computed from the data of Table XXVIII are shown in Table XXIX.

The intermittent test yields stress values which can also be used as a measure of modulus change with time. Thus, the last data points obtained from intermittent tests should be comparable with the changed

values of stress at a strain of 0.25 obtained from constant strain rate tests of unstrained rings stored in the vacuum-thermal environment.

Another way of evaluating the data from continuous and intermittent tests is to consider how the elastomer is to be used in the spacecraft. Results of the continuous test are directly related to an application of the elastomer in which the material is maintained under a continuous tensile strain. If the material is normally relaxed or under slight compression (as in an O-ring application), the intermittent tests, or constant strain rate tests of the effects of storage on tensile properties, are more directly applicable.

With the above discussion as preamble, let us examine the relative stabilities of the various elastomers studied. Among the silicone rubbers, the results of Tables XXVIII and XXIX indicate that SE-3604 is the most stable, with SE-3613 showing almost equally good behavior. Both of these elastomers demonstrate low rates of stress decay, minimal scission, and the lowest degree of cross-linking as shown in Table XXIX. Examination of the effects of the vacuum-thermal environment on tensile properties presented in Table XXX yields the information that SE-3604 is generally more stable than any of the 10 silicone rubber formulations tested.

Among the Hycar rubbers, the same kind of analysis of test results yields the following relative ordering of formulation stability:

> The relative degree of chain-scission effected by exposure to the vacuum-thermal environment, as measured by the stress decay of the continuous test can be portrayed as

> > Hycar-1 < Hycar-2, -4, -5, -6 << Hycar-3

The relative rates of cross-linking, as measured by the difference between continuous and intermittent test results, can be shown as

Hycar-1, -6, < Hycar-3, -4, < Hycar-5 \ll Hycar-2

The results of constant strain rate tests (Table XXXI), particularly the effects of the vacuum-thermal environment on strain at rupture and on modulus, illustrate the superior stability of Hycers-5 and -6 and the inferior characteristics of Hycars-2, -3, and -4. If the application is utilization under no load conditions, Hycars-3 and -6 appear to be most suitable; if the materials are to be used under conditions of continuous strain, Hycars-1 and -6 are the most suitable. Hycar-6 appears to be the formulation least affected by the vacuum-thermal environment.

Similar considerations yield the information that EX-1090 is the most stable of the butyl rubbers. For no load application as well as for use under constant strain, EX-1090 is superior to both EX-1091 and EX-1092.

All the Viton rubbers stored during this period appear to be reasonably stable. On the basis of constant strain rate test results only, the following represents a possible relative order of stability:

Viton AHV > Viton B > Viton A

Comparisons among elastomeric families, based on very general considerations, yields the following possible ordering of resistance to degradation in the vacuum-thermal environment:

silicones > Vitons > butyls > Hycars

The above ordering of elastomeric stability is further illustrated by the results of the long-term tests reported in Tables XXXIV and XXXV. The effects of exposure to the vacuum-thermal environment for a period of 6550 hours (approximately 9 months) on modulus and strain at rupture were remarkably small for SE-3604, reasonable for Viton B (990), and excessive for Hycar-1.

Additional results of the long-term tests are shown in Table XXXIII. It is interesting to note that except for Hycar-1, no specimens ruptured during the test period. Even in the case of Hycar-1, except for one "accident," ruptures were in the nature of "infant mortality." Perhaps the most interesting results of these long-term tests were the tensile

properties of rings which had been stored for the entire period at various levels of strain. Although in almost all cases the rings remained fully deformed upon removal from the test apparatus, values of stress at rupture and extensibility were quite comparable to those of specimens stored unstrained. This illustrates that unless permanent deformation is disabling, the influence of the vacuum-thermal environment on the general mechanical strength of rubbers is essentially independent of the applied load or strain in use.

C. DISCUSSION OF RESULTS: PLASTIC MATERIALS

All the plastic materials studied during this program were found to be reasonably stable to the vacuum-thermal environment. Prior to each constant load test, dumbbell specimens were tested in air at 125°C to determine the maximum load the material was most likely to support for the entire 1000-hour test. In most cases, loads greater than those used would have resulted in failure during the test period. Thus, from Table XXXVI, one can see that Kapton has by far the greatest strength at 125°C, with polyphenylene oxide and Mylar films being next strongest. Although Lexan has good properties at lower temperatures, 125°C is apparently the upper limit; brief excursions above 125°C during the time of the test caused large deformations. Creep was almost negligible for all the materials tested except Tedlar, which exhibited considerable deformation during the 1000-hour test. Polyphenylene oxide appeared to be particularly resistant to the space envaronment, although there was some indication during the constant strain rate tests of exposed specimens, that what appeared to be a form of delamination was occurring at rupture.

The results of constant strain rate tests of control and exposed specimens (Table XXXVII) also provide an indication of serviceability in the space environment. Lexan appeared to be relatively unaffected, except that rupture of the exposed specimens occurred where yield had been observed 'n the controls. PPO was found to be stable, although specimens stored under load (D^* and J^*) exhibited higher moduli and smaller rupture strains. The modulus of Mylar increased about 2-fold, and although rupture properties remained relatively unaffected, yielding

of the exposed material occurred at a larger stress and smaller strain. The test results of the polysulfone material do not indicate significant effects of the space environment on properties at 125°C; however, the exposed material at room temperature was noticeably more brittle than the control.

Kapton was not noticeably affected by the vacuum-thermal environment and is probably the most stable of all the materials studied. The Tedlars and Kynar are relatively low modulus materials at 125°C and the 1-mil specimens tended to buckle and warp; results were somewhat erratic, but the materials did not appear to be seriously affected by the vacuumthermal environment. Teflon is a relatively soft material and is subject to flow; the changes noted upon exposure to the vacuum-thermal environment (i.e. increased modulus, higher yield stress) were probably due to loss of low molecular weight material.

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MATER AL	MATER AL POLYMER TYPE		EXLOSURE CONDITION	
			Active	Passive
SE-555 Red	sil·icone	GES	A	A
SE-556	silicone	GES		F
SE-3604	silicone	GES	A, H	А,Н
SE-3704	silicone	GES		E
SE-3804	silicone	ŒS		Е
SE-3613 (24/480)	silicone	GES	В	В
SE-3713 (24/480)	silicone	GES	В	В
SE-3813 (24/480)	silicone	GFS	D	D
SE-4511 (4/400)	silicone	GES		G
SE-5403 (21/480)	silicone	GES		G
Hycar-1 (520-67-108-1)	polyacrylic	BFG	C,H	С,Н
Hycar-2	polyacrylic	BFG	D	D
Hycar-3	polyacrylic	BFG	С	C
Hycar-4	polyacrylic	BFG	E	E
Hycar-5	polyacrylic	BFG	F	E
Hycar-6	polyacrylic	BFG	F	E
EX - 1 090 ¹	isobuty lene-isoprene	ENJ	E	D
EX-1091 ¹	isobutylene-isoprene	ENJ	G	D
EX-1092 ¹	isobutylene-isoprene	ENJ	G	D
Viton A4411A-776 (Viton A)	vinylidene fluoride- hexafluoro propylene	DUE		G
Viton A4411A-777 (Viton AHV)	vinylidene fluoride- hexafluoro propylene	DUE		G
Viton A4411A-778 (Viton B)	vinylidene fluoride- hexafluoro propylene	DUE		G

Table XXV ELASTOMERIC MATERIAL AND EXPOSURE CONDITION IDENTIFICATION

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Notes: (1) Postcured 4 hours at 150°C.

(2) Active tests of elastomers were in situ continuous and intermittent stress relaxation tests; passive tests consisted of no-load storage, followed by constant strain rate tests at 25°C and 125°C. Active tests in condition fi were constant strain tests at several elongations.

(3) See Table XXVII for detail of exposure conditions.

			EXPOSURE CONDITION		
MATERIAL	PULIMER (TPE	mr.	Active	Passive	
Lexan film (5-mil)	polycarbonate	GEC	В	13	
PPO film (5-mil)	polyphenylene oxide	GEC	D, J	D, J	
Mylar 500A (5-mil)	polyethylene terephthalatc	DIM	E	Ę	
P-2300 (5-mil)	polysuifone	1CP	-	E	
Kapton (2-mil)	polyimide	DEM	F	F	
Tedlar 200BG30TR	polyvinyl fluoride	DLM	G	G	
Tedlar 200SG40TR	polyvinyl fluoride	DUM		F	
Teflon-FEP 200A	fluoroeti:ylene-propylene	DUP	-	G	
Kynar (1-mil)	polyvinylidene fluoride	POC	-	G	

Table XXVI PLASTIC MATERIAL AND EXPOSURE CONDITION (DENTIFICATION)

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NOTES: (1) Active tests of plastic films were in <u>situ</u> constant load tests; passive tests consisted of no load storage, followed by constant strain rate tests at 125°C.

(2) See Table XXVII for detail of exposure conditions.

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Table XXVII DESCRIPTIONS OF EXPOSURE CONDITIONS

	FIRST STAGE		SECOND STAGE			
CONDITION	Hours at 50°C	Ave. Pressure, torr	Hours at 125°C	Ave. Pressure, torr		
A	280	4×10^{-6}	1040	3.5 x 10 ⁻⁶		
В	145	5×10^{-6}	910	2×10^{-6}		
С	215	4×10^{-6}	1055	4×10^{-6}		
D	140	5×10^{-6}	1000	3×10^{-6}		
Е	190	4×10^{-6}	1004	2.5×10^{-6}		
F	185	4×10^{-6}	1005	2×10^{-6}		
G	145	5×10^{-6}	1005	2×10^{-6}		
H	175 (40°C)	< 10 ⁻⁵	6550	0.5×10^{-6}		
J	100 (45°C)	< 10 ⁻⁵	5060	0.5×10^{-6}		
	80 (45-125°C)					
	INTERM	ITTENT		CONT I	NUOUS	
------------	-----------------	---------	------------	-----------------	---------	----------
MATERIAL	Approx. Time to	f(t)/f	(o) at	Approx. Time to	f(t)/f	'(o) at
	hours	20 hrs.	500 hrs.	hours	20 hrs.	500 hrs.
SE-555 Red	2	1.06	1.36	4	0.87	0.82
SE-3604	2	1.04	1.21	220	0.92	0.89
SE-3613	3	1.08	1.32	300	0.95	0.89
SE-3713	3	1.12	1.58	125	0.94	0.86
SE-3813	6	1.04	1.38	10	0.89	0.80
llycar-1	1	1.16	~1.65	0.6	0.84	0.80
llycar-2	10	1.03	~ 2.2	2	0.80	0.72
Hycar-3	3	1.06	1.38	0.3	0.59	0.48
Hycar-4	5	1.03	1.63	1	0.81	0.69
Hycar-5	2	1.20	1.82	0.15	0.79	P.66
Hycar-6	4	1.08	1.52	17	0.90	0.66
EX-1090	0.5	1.17	1.60	3	0.83	0.76
EX-1091	2	1.16	1.75	0.3	0.69	0.48
EX-1092	2	1.20	1.84	1.2	0.82	0.67

Table XXVIII

RESULTS OF IN SITU CONTINUOUS AND INTERMITTENT TESTS OF VARIOUS ELASTOMERS

NOTES: 1. All tests conducted at strains of approximately 0.25

2. Data obtained from best curves drawn through duplicate test results

3. Exposure conditions were as indicated in Tables XXV and XXVII

Table XXIX

DIFFERENCES BETWEEN CONTINUOUS AND INTERMITTENT TEST RESULTS

MATERIAL	[f(t)/f(o)] _I	- [[[([)/[(0)]]C
	20 hrs.	500 hrs.
SE-555 Red	0.19	0,54
SE-3604	0.12	0.32
SE-3613	0.13	0.43
SE-3713	0.18	0.72
SE-3813	0.15	0.58
Hycar-1	0.32	0,85
Hycar-2	0.23	1.48
Hycar-3	û.47	0.90
Hycar-4	0.22	0.94
Hycar-5	0.41	1.16
Hycar-6	0.18	0.86
EX-1090	0.34	0.84
EX-1091	0.47	1.27
EX-1092	0.38	1.17

Table XXX

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EFFECT OF VACUUM-THERMAL ENVIRONMENT ON TENSILE PROPERTIES OF SILICONE RUBBERS

MATERIAL	HISTORY	TEST TEMP.	STRESS AT STRAIN OF 0.25, psi	STRESS AT RUPTURE, psi	STRAIN AT RUPTURE, in/in.
SE-3604	Control Exposed Control Exposed	25 25 125 125	109 131 112 125	637 662 405 463	$ \begin{array}{r} 1.04 \\ 1.04 \\ 0.65 \\ 0.68 \end{array} $
SE- 3704	Control Exposed Control Exposed	25 25 125 125	130 259 199 277	595 600 475 515	$1.52 \\ 0.73 \\ 1.10 \\ 0.55$
SE-3804	Control	25	261	500	1.05
	Exposed	25	394	660	0.59
	Control	125	260	435	0.66
	Exposed	125	383	440	0.42
SE-3613	Control	25	87	422	0.88
	Exposed*	25	104	358	0.68
	Control	125	98	253	0.61
	Exposed*	125	116	190	0.40
SE-3713	Control	25	165	760	1.06
	Exposed*	25	195	643	0.79
	Control	125	168	546	0.84
	Exposed*	125	201	440	0.58
SE-3813	Control	25	328	935	0.98
	Exposed*	25	388	815	0.62
	Control	125	334	740	0.72
	Exposed*	125	400	840	0.67
SE-555 Red	Control Exposed Control Exposed	25 25 125 125	65 84 53 77	825 760 310 311	4.17 2.86 1.92 1.29
SE-556	Control	25	91	1120	4.21
	Exposed	25	120	1109	3.30
	Control	125	83	705	2.89
	Exposed	125	115	655	2.16
SE-4511	Control	25	57	605	2.84
	Exposed	25	67	550	2.23
	Control	125	65	312	1.43
	Exposed	125	76	315	1.22
SE-5403	Control	25	18.5	513	5.35
	Exposed	25	24	568	4.62
	Control	125	16.7	332	4.04
	Exposed	125	24	274	2.80

NOTES: (1) All data points are averages of measurements on duplicate specimens. *Indicates average of tests in triplicate.

(2) Tests were conducted at an extension rate of 0.1 in/min.

(3) Control specimens were stored at normal room conditions for the entire period from specimen preparation to final testing.

(4) Exposure conditions were as indicated in Tables XXV and XXVII.

Table XXXI

MATERIAL	HISTORY	TEST TEMP.	STRESS AT STRAIN OF 0.25, psi	STRESS AT RUPTURE, psi	STRAIN AT RUPTURE, in/in.
Hycar-1	Control	25	95	1180	1.87
	Exposed	25	1ó6	990	1.01
	Control	125	75	385	0.97
	Exposed	125	155	404	0.52
Hycar-2	Control	25	99	1225	2.24
	Exposed	25	227	810	1.17
	Control	125	77	375	0.66
	Exposed	125	226	330	0.35
hycar-3	Control	25	104	1240	1,31
	Exposed	25	184	980	0.68
	Control	125	108	435	0.72
	Exposed	125	192	505	0.40
Hycar-4	Control	25	121	92 0	1.25
	Exposed	25	243	630	0.49
	Control	125	128	365	0.68
	Exposed	125	260	380	0.35
Hycar-5	Centrol	25	41	815	2.12
	Exposed	25	60	930	2.14
	Contro!	125	30	270	1.65
	Exposed	125	45	285	1,15
Hycar-6	Control	25	243	1490	1.24
	Exposed	25	348	1590	1,07
	Control	125	162	800	0.95
	Exposed	125	232	735	0.70

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EFFECT OF VACUUM-THERMAL ENVIRONMENT ON TENSILE PROPERTIES OF HYCAR RUBBERS

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NOTES: (1) All data points are averages of measurements on duplicate specimens.

(2) Tests were conducted at an extension rate of 0.1 in/min.

- (3) Control specimens were stored at normal room conditions for the entire period from specimen preparation to final testing.
- (4) Exposure conditions were as indicated in Tables XXV and . XXVII.

Table XXXII

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MATERIAL	HISTORY	1FST TEMP. C	STRESS AT STRAIN OF 0.25, ps.	STRESS AT RUPTURE, PS 1	STRAIN AT RUPTURE, in/in.
EX-1090	Control	25	170	1860	2.14
	Exposed	25	246	1600	1.32
1	Control	125	152	925	1.40
	Exposed	125	232	849	0.95
EX-1091	Control	25	1 02	1515	2.78
i	Exposed	25	: 82	1415	1.59
	Control	125	77	750	2.43
1	Exposed	125	164	590	0.98
EX-1092	Control	25	148	1570	1.68
	Exposed	25	264	1125	0.83
	Control	125	150	790	1.22
	Exposed	125	240	575	0,58
Viten A	Control	25	113	2310	3.81
(776)	Exposed	25	136	2490	2.78
1	Control	125	91	433	1.18
	Exposed	125	120	484	0.98
Viton AilV	Control	25	180	1990	3.01
(777)	Exposed	25	192	2070	2.62
	Control	125	166	561	1.14
	Exposed	125	190	622	1.10
Viton B	Control	25	323	1910	0.89
(778)	Exposed	25	376	1980	1.07
	Control	125	353	630	0.39
	Exposed	125	461	566	0.30

EFFECT OF VACUUM-THEBMAL ENVIRONMENT ON TENSILE PROPERTIES OF BUTYL AND VITON RUBBERS

NOTES: (1) All data points are averages of measurements on duplicate specimens.

(2) Tests were conducted at an extension rate of 0.1 in/min.

- (3) Control specimens were stored at normal room conditions for the entire period from specimen preparation to final testing.
- (4) Exposire conditions were as indicated in Tables XXV and XXVII.

MATERIAL	APPROX. STRAIN, in,/in.	NO. OF RINGS	APPROX. RUPTURE TIME, hrs at 125°C	AVE. STRESS AT RUPTURE, psi	EXTENSION TO RUPTURE, in.
SE-3604	0	5		740	1,19
	0.25	4		600	1.20
	0.35	4		572	1,19
	0.50	4		584	1.21
	0.75	4		575	1.23
	1.00	4		562	1.21
	1.50	4	1 at -160: 1 at -7 1 at 0	500	1.28
Viton B	0	8		1885	3.14
A4411A-990	0.25	4		1940	3.64
	0.35	4		1610	3.40
	0.50	4		1720	3.90
	0.75	4		1600	3.46
	1.00	4		1440	3.70
	1.50	4		1360	3.23
	2.50	4		1625	3.83
	3.50	4	2 at -3	1030	3.92
Hycar 1	0	4		923	0.87
	0.25	2		1150	1.0!
	0.35	2		1170	1.12
	0.50	2	l at about 4900	1110	1,14
	0.75	4		840	1.13
	1,00	4		625	0.98
	1.50	4	2 between 100-160 1 between 170-185	738	1.05

Table XXXIII SUMMARY OF DATA FOR LONG-TERM EXPOSURE TESTS

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NOTES: (1) Exposure Condition H, Table XXVII

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(2) Average tensile properties of apecimens after exposure were obtained at a test temperature of 25°C, and an extension rate of 0.1 in/min.

Table XXXIV

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EFFECT OF VACUUM-THERMAL ENVIRONMENT ON TENSILE PROPERTIES OF VARIOUS ELASTOMERS: 1000 and 6500-hour Test Results

MATERIAL	EXPOSURE TIME, HRS.	TEST TEMP. °C	STRESS AT STRAIN OF 0.25, psi	STRESS AT HUPTURE, psi	STRAIN AT RUPTURE, in/in.
SE-3604	Control	25	109	637	1.04
	1040	25	131	662	1.04
	6550	25	170	740	0.97
	Control	125	112	405	0.65
	1040	125	125	463	0.68
	6550	125	189	505	0.56
Hycar-1	Control	25	95	1180	1.87
	1635	25	166	990	1.01
ĺ	6550	25	279	923	0.71
	Control	125	75	385	0.97
	1055	125	155	40 i	0.52
	6550	125	350	540	0.30
Viton [®] B	Conti 1	25	127	1495	4.65
A4411A-	820	25	130	1940	4.85
990	6550*	25	142	1885	2,60
	Control	125	79	338	2,10
	820	125	102	402	1.64
	6550*	125	155	577	1.18

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NOTES: (1) All data points are averages of measurements on duplicate specimens. * indicates averages of tests in quadruplicate.

(2) Tests were conducted at an extension rate of 0.1 in/min.

(3) Control specimens were stored at normal room conditions for the entire period from specimen preparation to final testing.

(4) Exposure conditions were as indicated in Tables XXV and XXVII.

Table XXXV

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EFFECT OF VACUUM-THERMAL ENVIRONMENT ON TENSILE PROPERTIES OF VARIOUS ELASTOMERS: 1000 and 6500-hour Test Results

	EXPOSURE	TECT	PERCENTAGE IN	CREASE OVER C	ONTROL VALUES
MATERIAL	TIME, HOURS	TEMP, °C	Stress at Strain of 0.25	Stress at Rupture	Strain at Rupture
SE-3604	1040	25	20	4	0
	6550	25	55	16	-7
	1040	125	12	14	5
	6550	125	69	25	-14
Hycar-1	1055	25	75	~16	-46
	6550	25	194	-22	-62
	1055	125	107	5	-46
	6550	125	380	40	-69
Viton B	820	25	2	30	4
A4411A-	6550	25	12	26	-44
990	820	125	30	19	-22
	6550	125	96	71	-44

NOTE: Percentage values represent comparisons of data from Table XXXIV.

Table XXXVI

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RESULTS OF CONSTANT LOAD TESTS OF VARIOUS PLASTIC MATERIALS

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MATERIAL	EXPOSURE CONDITIONS (Table XXVII)	LOAD CONDITION	RESULTS
Lexan, 5 mi1	В	4 specimens at 600 psi	erratic results; brief excursions to 128-130°C caused step defor- mations of up to 30%.
PPO, 5 mil	D	4 specimens at 2000 psi	one specimen ruptured at 500 hours, one at 850 hours; deformation less than 1% for whole test
	J	4 specimens at 2000 psi	one specimen ruptured at about 4,000 hours; deformation less than 1%
		2 specimens at 1750 psi 2 specimens at 1500 psi	One specimen (1750) ruptured at about 1540 hours; deformation less than 1%
Mylar, 5 mil	E	2 specimens at 2000 psi 2 specimens at 1500 psi	all defoimation occurred within first 100 hours; average € at 2000 psi wis 5%, average € at 1500 psi wis 3%
Kapton, 2 mil	F	2 specimens at 10,000 psi 2 specimens at 7500 psi	initial deformation of about 1% within first 4 hours for all specimens; no further deformation
Tedlar, 1 mil	G	2 specimens at 1750 psi 2 specimens at 1600 psi	considerable deformation during entire test, up to 50% (Fig. 17); no specimens ruptured

NOTE: Loads were applied shortly after environmental temperatures were raised to $125\,^{\rm o}{\rm C}.$

Table XXXVII

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EFFECT OF VACUUM-THERMAL ENVIRONMENT ON TENSILE PROPERTIES OF VARIOUS PLASTIC MATERIALS

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-	EXDOSIDE		YII	ELD	RUP	TURE
MATERIAL	CONDITIONS,	MODULUS.	Stress,	Strain,	Stress,	Strain,
	Table XXVII	psi	psi	in/in.	psi	in/in.
Lexan 5 mil	Control B	$50,000 \\ \sim 52,000$	3,560	0.08	2,860 3,800	0.58 0.14
Polyphenylene-oxide ("PO) 5 mil	Control D J D J	$105,000 \\ \sim 95,000 \\ 95,000 \\ \sim 140,000 \\ 135,000$	 	 	6,560 5,075 7,040 5,610 5,200	0.075 0.063 0.077 0.041 0.038
Mylar, Type 500A,	Control	33,000	2,900	0.09	12,300	1.74
5 mil	E	72,000	4,240	0.059	10,700	1.82
Polysulfone, P-2300	Control	82,000			4,940	0.085
5 mil	E	103,000			5,640	0.081
Kapton, 200XH667, 2 mil	Control F	92,800 112,000			12,800 11,800	0.20 0.17
Tedlar, 200SG40TR, 2 mil	Control F	2,900 8,700			4,080 3,720	3.2 2.7
Tedlar, ^a 100BG301R	Control	5,850	1,550	0.25	3,790	i.3
1 mil	G	6,900	2,150	0.45	4,500	2.2
Teflon FEP ^a	Control	1,800	390	0.22	830	3.0
Type A, 20 mil	G	2,860	540	0.19	840	2.8
Kynar, ^a 1 mil	Control	7,000	940	0.18	2,900	10.7
	G	6,800	1,040	0.22	2,400	5.7

NOTES: (1) All tests were conducted at a crosshead rate of 0.02 in/min. and at a temperature of 125°C. Superscript "a" indicates result: of tests conducted at 0.1 in/min.

- (2) Test specimens were dumbbell shapes; 0.125 inch width, 0.8 inch test length, thickness indicated. Strains were calculated on the basis of an 0.8 inch effective gage length.
- (3) Data represent, the averages of four tests with the following exceptions: Lexan-control, PPO; exposed U,J, and J*, 2 tests; rupture data for Mylar, 1 test Tediar 100BG30TR-control and exposed, 3 tests; Kynar-control and-exposed, 3 tests for yield values, single test for rupture values.
- (4) Lexan exhibited distinct yield point. All other yield values were obtained at the intersection of the modulus line with the tangent to the yielding curve.
- (5) Tests of PPO indicated as D^o and J^o were performed on specimens which had been under a 2000 psi losd for the entire period of exposure to vacuum at 125°C.
- (6) Results of tests on exposed specimens of Tedlar 1008G30TR and Kynar were erratic, presumably due to discortion of 1 mil specimens.











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FIG. 13 CONTINUOUS AND INTERMITTENT STRESS RELAXATION RESULTS FOR HYCAR-6 IN VACUUM AT 125°C







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FIG. 15 CONTINUOUS AND INTERMITTENT STRESS RELAXATION RESULTS FOR EX-1091 IN VACUUM AT 125°C







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FIG. 17 DEFORMATION OF TEDLAR 100BG30TR UNDER CONSTANT LOAD IN VACUUM AT 125°C

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IV. COMPREHENSIVE POLYMER TEST PROGRAM

The paucity of reliable data on the combined effect of spacecraft decontamination procedures and a thermal-vacuum environment on the physical and mechanical properties of polymeric spacecraft hardware materials prompted JPL Cognizant Engineers to initiate a comprehensive test program to provide parameters for selecting suitable materials for use in spacecrafts. Typical data required are: power factor, dielectric constant, dimensional change, hardness, elongation, etc.

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The results of tests from decontamination cycles alone are not sufficient to qualify a polymer for spacecraft use, for they must be followed by tests <u>in vacuo</u> (or after irradiation).* As a result, the comprehensive polymer test program includes the determination of 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.

A. APPARATUS

Design drawings and mechanical description of the multi-cell apparatus which contains individual compartments for each polymeric sample are given in Part II of this report: "Test Equipment." The design of the unit permits the pumping system to be valved-off for the decontamination cycles, and activated for the thermal-vacuum exposure,

B. OPERATION PROCEDURE

(1) 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-

^{*} Drummond, D., and Magistrale, V., Jet Propulsion Laboratory, JPL Spacecraft Sterilization Technology Program, T. R. No. 32-853, Dec. 31, 1965.

loss and dimensional change). The test specimens are either suspended in the sample cell (see Figure 18) or laid flat on a wide-mesh screen. It is to be noted that only one polymeric product is contained in each cell.

(2) 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 brings it to 50° C and into the sample cells to provide an atmosphere of 455 mg/liter ETO.

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.

(3) Samples 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. Other samples are left in place for final tests of the effects of the decontamination treatment followed by a thermal-vacuum treatment.

(4) 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.

C. TEST PROCEDURES

Duplicate samples of 1" \times 6" were used for dimensional change, change in weight, and change in hardness.

(1) Dimensional change

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

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(2) Change in weight

Samples were 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 was 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 was measured with a Shore Durometer Type A-2 according to ASTM D2240-64T, using a l-kg weight; readings are made within one second after application of the load.

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

(4) Tensile and Elongation

The tensile and elongation measurements were made at about 70° F using an Instron Model TTCLM-6.

Duplicate test specimens of film and sheet materials were cut with an ASTM die D: the speed of testing was 1.00 in/min.

Test specimens of structural materials were prepared and tested according to ASTM D638-64T. Duplicate type III test specimens were machined for each sample; the speed of testing was 0.05 in/min.

A modified method, employing a scaled-down die, was used for testing the seal and gasket materials in order to ensure sufficiency of specimens for the entire test program. The test specimens had a gage length of 1.000 inch and a width of 0.125 inch. Triplicate determinations were made on each sample at a testing speed of 1.00 in/min. Duplicate specimens of tie cord (lacing tape) materials were tested according to ASTM D638-64T at a testing speed of 1.00 in/min. NOTE: Tensile and elongation data obtained for a number of materials had to be invalidated since the operator failed to employ the same speed of testing for control and test specimens.

(5) Dielectric Constant and Dissipation Factor

Dielectric constant and dissipation factor measurements were made according to ASTM D150-64T. The test equipment consisted of a General Radio R-F bridge Type 916A, a Hewlett-Packard signal generator Model 606A, and a Hallicrafter SX71 radio-receiver in conjunction with a General Radio tuned amplifier and null detector Type 1232A.

Dielectric specimens 1.000 inch in diameter were machined from the structural materials. The measurements performed on these samples were made using a micrometer electrode system (General Radio "Dielectric Sample Holder," Type 1690A).

The dielectric specimens of film and sheet materials were die-cut at 1.000 inch in diameter. The electrodes were made from aluminum foil 0.7 mil thick by 0.950 inch in diameter. Silicone oil was used to increase the contact between the foil and the test specimen. The specimen and electrodes were then placed between a stiff wire connector for two-terminal measurement as shown in Figure 19.

It is to be noted that the majority of the samples tested were of a thickness of 2 mils or less; since the silicone oil was in series with the test specimen, it could cause the measured dielectric constant to be too low and the dissipation factor to be too high, which seems apparent. With this in mind, the data for film and sheet materials should be examined only from the point of view of comparison between control and test specimens and not as an absolute measurement of either dielectric constant or dissipation factor.

D. DISCUSSION OF RESULTS

The polymeric products which were examined during the first comprehensive test program are described in Table XXXVIII. Test data are reported in Tables XXXIX to L as per cent values for changes in weight, dimensions, and hardness; values for both control and test specimens are reported for tensile, elongation, and electrical properties. The Tables are arranged in a sequence to permit review of the effects of the various treatments for each class of materials according to potential spacecraft applications. Hardware and structural materials. - Data obtained after the three treatments of hardware and structural materials (Tables XXXIX, XL, and XLI) indicate negligible changes for dielectric constant, dissipation factor, and hardness. Tensile data were invalidated for the thermalvacuum and combined decontamination/thermal-vacuum treatments; after decontamination cycles only, tensile changed <10%. Dimensional changes were negligible after all treatments, except for Delrin 100NC10. Sample weights increased after ETO-Freon treatment, as expected, and decreased after thermal-vacuum creatment; the results of the combined exposure indicate that some substances adsorbed during the decontamination treatment were not able to be pumped away in a subsequent thermal-vacuum exposure. However, except for Delrin 100NC10, all weight losses after the thermal-vacuum exposure are acceptable and in agreement with polymer screening data.

<u>Seal and gasket materials</u>. - The effects of any of the three treatments on seal and gasket materials (Tables XLII, XLIII, XLIV) were virtually nonexistent for dimensions, and <2% for hardness. In general, tensile and elongation changes were within the range of $\pm 20\%$, with the greater changes incurred during the thermal-vacuum exposures. Sample weights increased after ETO-Freon cycles, and decreased after thermal-vacuum cycles in good agreement with micro-VCM screening data.

Films and Sheets. - Gross changes in dimensions, as shown in Tables XLVI and XLVII, are exhibited by several of the film and sheet materials after thermal-vacuum and decontamination/thermal-vacuum exposures. Weight changes are in agreement with polymer screening data except, of course, for the gein in weights incurred during decontamination cycles (Table XLV). Unfortunately, a great number of tensile and elongation tests have been invalidated; however, the remaining valid data indicate possible changes of 3-30% after the thermal-vacuum cycles. Data for dielectric constant and dissipation factor indicate essentially no change after any of the treatments.

<u>Tie cords/lacing tapes</u>. - As shown by the data for two samples of tie cords in Tables XLVIII, XLIX, and L, no changes except weight increase are effected by thermal-vacuum treatment. The data for the effects of thermal-vacuum, and decontamination/thermal-vacuum are quite cimilar, and indicate that the fluorocarbon tie cord is distinctly superior to the polyester tie cord.

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Table XXXVIII

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POLYMERIC PRODUCTS EXAMINED IN THE COMPREHENSIVE TEST PROGRAM

WATERIAL	LSE ¹	POLYMER-TYPE ²	MFH. 3	TREATMENT
		. 1	DI D	$D_{-1} = \frac{1}{1000} $
Delrin 100NC10	115	acetal	10.2	Postcured hr/150 C
Doryl H-1:511	IIS	diphenyl oxide	WEN	Postcured hr/150°C
Micarta II-2497	HS	epoxy-glass	WEM	Postcured hr/150°C
Nicarta H-5834	HS	phenolic-glass	WEM	Postcured 1 hr/150°C
Micarta 20201-2	HS	silicone-glass	WEM	Postcured hr/150°C
Kapton 200XH667	FS	polyimide	Dim	As received
Kynar 200	F	polyvinylidene fluoride	PCC	As received
Mylar Type 100T	FS	polyester	DUP	As received
Mylar Type 200A	FS	polyester	DUP	As received
P-2300	FS	polysul fone	ĽCΡ	As received
PPO 691-111	FS	polyphenylene oxide	CEC	As received
Teilon FEP 500A	FS	fluoroethylene- propylene	DUP	As received
Tedlar 100BG30TR	FS	polyvinyl fluoride	DUP	As received
Tedlar 2008G30WI	FS	polyvinyl fluoride (filled)	DUP	As received
Hycar 520-67-108-1	SG	acrylic	BFG	As received
SE- 3604	SG	silicone	GES	As received
SE-3613 (24/480)	SG	silicone	GES	As received
SE- 3713 (24/480)	SG	silicone	GES	is received
SE-3813 (24/480)	SG	silicone	GES	As received
Viton A4411A-990	SG	vinylidene fluoride- hexafluoropropylene	AKR	As received
Gude-Space 18D96	TC	polyester	GBE	Fostcured 1 hr/150°C
Temp-lace 256H	TC	fluorocarbon	GHE	Postcured 1 hr/150°C

¹ fS, hardware and structural; FS, films and sheets; SG, seals and giskets; TC, tie cordz/lacing tapes.

² See Appendix A.

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 ³ See Appendix B.

Table XXXIX

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EFFECTS OF DECONTAMINATION CYCLES ON HARDWARE AND STRUCTURAL MATERIALS (Six cycles of ETO---Freon for 30 hours at 50°C)

		PHYSIC	AL AND ME	SCHANIC/	AL PROPER'	TIES				ELECTRIC	AL PRO	PERTIES	
MA'TERIAI.	DIMENSIONAL	VEIGHT	Rock w Hardn	e l l e s s	Tensile	, eq	Elongati Bresk,	on at	FREQUENCY.	Dieles Const	tric Bnt	Dissi Fac	ation tor
	CHANGE, %	CHANGE, %	Control	Test	Control	Test	Control	Test	Milz	Control	Test	Control	Test
Delrin 100NC10	-0.71	+0.30	19.0	18.9	10,260	10,320	7.6	12.0	1	3.47	3.67	<0.0001	<0.0001
									15	3.64	3.56	0.0001	<0.0001
									25	3.37	3.58	0.0002	<0,0001
									35	3.33	3.36	<0.0001	<0.0001
			_						50	3.48	3.36	0.0007	<0.0001
Doryl H-17511	n.c.	+0.35	23.1	23.8	43,190	46,200	3.4	3.4	I	5.12	5.46	0.0015	9.0013
									15	4.94	5.38	0.0025	0.0020
									25	4.94	5.36	0.0026	0.0017
									35	4.83	5.38	0.0022	0.0008
									50	4.33	5.36	0.0031	0.0026
Micarta H-2497	n.c.	+0.10	24.9	25.2	46,000	51,000	4.3	4.6	н	4.75	4.70	0.0001	0.0001
									15	4.76	4.68	<0.0001	<0.0001
									25	4.57	4.63	0.0002	<0.0001
									35	4.32	4.43	0.0004	<0.001
						-			50	4.80	4.4]	<0.0601	<0.0001
Wicarta H-5834	n.c.	+0.30	23.2	23.ú	45,600	44,330	3.3	3.2	I	5.57	5.51	0.0018	0.0012
									15	5.46	5.46	0,0020	0.0023
									25	5.43	5.40	0.0010	0.0023
									35	5.35	5.40	0.0020	0.0012
									50	5.20	5.46	0.0023	<0.0001
Micarta 20201-2	n.c.	+0.01	17.8	17.4	28,600	29,880	2.3	2.5	1	4.94	4.89	0.0006	0.0004
									15	4.69	4.89	0.0007	0.0003
		<u> </u>							25	4.94	4.89	0.0010	0.0005
									35	4.96	4.89	0.0011	0.0004
									50	4.96	4.89	0.0011	0.0002

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Table XL

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EFFECTS OF THERMAL-VACUUM ENVIRONMENT ON HARDWARE AND STRUCTURAL MATERIALS (500 hours at 135°C and 10^{-6} torr)

		PHYSICA	VL AND MEC	CHANICA	L PROPERTIES				ELECTRICA	L PROP	ERTIES	
MATERIAL	DIMENSIONAL	WEIGHT	Rockwe Hardnes	11	Tensile, ps1	Elonga	tion at k, °	FREQUENCY,	Dielect Corste	tric Ant	Dissi Fac	ation
	CHANGE. %	CHANGE, %	Contro.	Test	Control Tes	t Contro	l Test	7 MW	Control	Test	Control	Test
Delrin 100NC10	-1.11	-0.95	19.0	18.2	invalid	invi	nlid	1	3.47	3.63	<0.0001	<0° 0001
								15	3.64	3.46	6.0001	0.0001
								25	3.37	3.44	0.0002	0.0001
								35	3.33	3.43	<0.0001	0.0002
								50	3.48	3.46	0.0007	0.0003
Doryl H-17511	-0.38	-0.06	23.1	24.1	invalid	invi	alid	-	5.12	5.12	0.0015	0.0012
								15	4.94	5.05	0.0025	0.0014
								25	4.94	5.03	0.0026	0.0916
-								35	4.83	5.03	0.0022	0.0012
								50	4.83	5.03	0.0031	0.0017
Micarta H 2497	-0.10	-0.20	24.9	25.5	invulid	inv	a lid	1	4.75	4.76	0.0001	0.0001
								15	4.76	5.52	<0.001	0.0001
								25	4.57	4.55	0.0002	0.0001
								35	4.32	4.67	0.0004	0.002
								20	4.80	4.67	<0.0001	0.0008
Micarta H-5834	п.с.	-0.38	23.2	24.0	invalid	101	alid		5.57	5.23	0.0018	0.0014
								15	5.46	5.23	0.0020	0.0017
								25	5.43	5.23	0.0010	0.0026
								35	5.35	5.20	0.0020	0.0020
								50	5.20	5.23	0.0023	0.0024
Micarta 20201-2	n.c.	-0.24	17.8	17.4	invalid	, ui	alid	~	4.94	4.96	0.0006	0.0002
								15	4.69	4.91	0.0007	0.0003
								25	4.94	4.91	0.0010	0.0004
				_				35	4.96	4.9Ì	0.0011	0.0005
								50	4.96	4,89	0.0011	0.0008

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Table XLI

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EFFECTS OF DECONTAMINATION CYCLES PLUS THERMAL-VACUUM EXPOSURE ON HARDWARE AND STRUCTURAL MATERIALS

		DUVSTCA		HANTCAL	I DRODERTIF				ELECTRICA	I. PROP	ERTIES	
		Unfeitul				, ,						
MATERIAL	DIMENSIONAL	WEIGHT	Rockw Hardn	e]] e s s	Tensile, j	p.s.i	Elongation at Break, %	FREQUENCY.	Dielect Consti	tric ant	Dissip Fac	ation tor
	CHANGE, %	CHANGE, %	Control	Test	Control T	Test	Control Test	MHz	Contro:	Test	Control	Test
Delrin 100NC10	-1.39	-2.34	19.0	17.6	invalid		invalid	1	3.47	3.41	<0,0001	<0.001
								15	3.64	3.44	0.0001	0.0001
								25	3.37	3.57	0.0002	0.0002
								35	3.33	3.38	-0.0001	0.0002
								50	3.48	3.42	0.0007	0.0003
Dorvl H-17511	-0.09	-0.20	23.1	24.0	 invalid		l invalid		5.12	5.13	0.0015	0.0012
								15	4.04	5.02	0.0025	0.0013
		_						25	4.94	5.04	0.0026	0.0015
								35	1.83	5.02	0.0022	0.0014
								50	4.83	5.02	0.0031	0.0013
Micarta H-2497	+0.05	-0.16	24.9	25.5	Invalid		 invalid	4	4.75	4.66	0.0001	0.0001
								15	4.76	4.49	<0.0001	n.0002
								25	4.57	4.39	0.0002	0.0001
								35	4.32	4.71	0.0004	0.0002
								50	4.80	4.40	<0.0001	1000.0
Micarta H-5834	-0.04	-0.2)	23.2	24.0	invalid		 invalid		5.57	5.20	0.0018	6.0013
								15	5.46	5.12	0.0020	0.0016
								25	5.43	5.12	0.0010	0.0016
								35	5.35	5.12	0.0020	0.0016
								50	5.20	5.15	0.0023	0.0015
Micarta 20201-2	-0.02	-0.19	17.8	17.5	invalid		invalid	1	4.94	4.90	0.0005	0.0002
								15	4.69	4.85	0.0007	0.0004
								25	4.94	4.85	0.0010	0.0004
								35	4.96	4.83	0.0011	0.0005
								50	4.96	4.30	0.0011	0.0008

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Table XLII

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MATERIAL	DIMENSIONAL	WEIGHT CHANCE 5	SHOR HARDNI	E ESS	TENSILE	, psi	ELONGAT I BREAL	ION AT (, %
		Change 1	Control	Test	Control	Test	Control	Test
Hycar 520-67-108-1	n.c.	+0.04	86.3	85.4	1,860	1,910	128	128
SE-3604	n.c.	+0.03	77.4	77.4	849	804	142	127
SE-3613(24/480)	n.c.	+0.14	70.6	70.5	1,020	891	212	150
SE-3713(24/480)	n.c.	+0,17	77.9	81.4	1,090	1,128	126	131
SE-3813(24/480)	n.c.	+0.50	87.4	87.6	1,074	1,123	79	92
Viton A4411A-990	+0.47	+1.47	85.4	84.6	2,030	2,080	358	417

EFFECTS OF DECONTAMINATION CYCLES ON SEAL AND GASKET MATERIALS (Six cycles of ETO-Freen for 30 hours at 50° C)

Table XLIII

EFFECTS OF THERMAL-VACUUM ENVIRONMENT ON SEAL AND GASKET MATERIALS (500 hr at 135°C and 10^{-6} torr)

MATERIAL	DIMENSIONAL	WE IGHT	SHORE HARDNE	ss	TENSILE	, ps:	ELONGATI BREAK,	ON AT %
	CHANGE, *	CHANGE, /	Control	Test	Control	Test	Control	Test
Hycar 520-67-108-1	n.c.	-1.06	86.3	89.1	1,860	2,140	128	88
SE-3604	n.^.	-0.18	77.4	78.4	849	917	142	105
SE-3613(24/480)	n.c.	-0.13	70.6	70.7	1,020	977	212	122
SE-3713(24/480)	л.с.	-0.25	77.9	77.9	1,090	1,176	126	117
SE-3813(24/480)	n.c.	-0.33	87.4	88.2	1,074	1,230	79	80
Viton A4411A-990	n.c.	-0.54	85.4	86.7	2,030	2,220	358	273

MATEBIAL	DIMENSIONAL	WEIGHT	SHOR HARDNI	IE ESS	TENSILE	, ps1	ELONGAT I BREAK	ON AT
	CHANGE, %	CHANGE. %	Control	Test	Control	Test	Control	Test
Hycar 520-67-108-1	п.с.	-1.44	86.3	90.8	1,860	2,120	128	85
SE-3604	n.c.	-0.22	77.4	77.6	849	1,070	142	125
SE-3613(24/480)	n.c.	-0.04	70.6	70.1	1,020	930	212	110
SE-3713(24/480)	n.c.	-0.20	77.9	79.4	1,090	1,103	126)12
SE-3813(24/480)	п.с.	-0.21	87.4	89.0	1,074	1,220	79	78
Viton A4411A-990	n.c.	+0.14	85.4	87.6	2,030	2,420	358	347

EFFECTS OF DECONTAMINATION CYCLES PLUS THERMAL-VACUUM EXPOSURE ON SEAL AND GASKET MATERIALS

Table XLIV

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Table XLV

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EFFECTS OF DECONTAMINATION CYCLES GN FILMS AND SHEETS (Six cycles of ETO-Freen for 30 hours at 50°C)

0,01.40.013 0.016 0.144 0.133 0.115 0.015 0.018 0.010 0.018 0.016 0.014 0.014 0.0120,018 0.005 0.007 0.023 0.066 0.109 0.14^C 0.020 <0.001 <0.001 <0.001 Test Dissipation Factor Control 0.018 0.015 0.013 0.013 0.016 0.013 0.012 0.013 0.005 0.010 0.018 0.112 0.126 0.125 0.122 0.016 0.0140.004 0.007 <0.001 0.044 0.031 0.101 <0.001 0.094 ELECTRICAL PROPERTIES 2.84 2.89 2.79 2.70 2.65 2.58 3.05 3.28 2.93 Test 2.90 2.75 2.75 2.30 3.47 2.68 2.44 2.44 1.92 2.79 2.30 2.58 2.58 2.35 3.30 3.36 2.76 Dielectric Constant 3.30 2.99 1.89 2.602.55 2.80 2.71 2.71 2.65 2.41 2.90 2.82 2.92 2.90 2.86 2.79 2.74 2.64 2.33 3.26 1.58 1.91 2.05 Control 2.61FREQUENCY, MHz 1 15 25 35 35 1 15 25 35 35 50 1 15 25 35 35 50 1 15 25 35 35 50 1 15 25 35 35 50 Test Elongation at Break, % 117 63 17 47 57 Control 117 93 2 22 51 6,400 9,540 23,300 37,730 29,030 28,350 Test MECHANICAL PROPERTIES Tensile, psi 38,680 Control 23,300 9,310 6,400 WEIGHT CHANGE, % +1.13 +l.89 +0.59 +0.40+0.18 DIMENSIONAL CHANGE, % n.c. n.c. n.c. п.с. n.c. Mylar Type 100T Mylar Type 200A Kapton 200X1667 MATERIAL Kynar-200 P-2300

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Table XLV (Concluded)

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		MECHANI	ICAL PROPE	ERTIES			ш 	LECTRICAL	PROPE	RTIES	
MATERIAI.	Dimensional	Weight	Tensile	rsd ,	Elongati Break,	on at	Frequency,	Dielecti Constai	rıc nt	Dissipa Facto	tıon r
	Change, %	Change, %	Control	Test	Centrol	Test	7 11	Control	Test	Control	Test
PPO 681-111	n.c.	+1.76	8,200	8,500	8.0	7.6	1	2.35	2.20	<0.001	<0.001
							15	2.35	2.19	<0.001	<0.001
							25	2.33	2.17	100 0>	<0.001
							35	2.24	2.11	<0.107	<0.100
							50	2.02	1.79	0.080	0.630
Teflon FEP-500A	n.c.	+0.20	2,560	2,475	315	316	-	2.02	2.09	<0.6301	1000.0>
	,		•				15	1.78	1.83	• 0.0001	<0.0001
							25	1.91	1.91	<0.0001	<0.0001
							35	1.55	1.60	<0.0001	<0.0001
							50	1.12	1.17	<0.0601	1000'0>
Tedlar 100BG30TR	п.с.	+0.23	13,400	12,500	137	127	1	4.02	4.02	0.081	0.081
							15	3.34	3.13	0.096	0.099
						_	25	3.15	3.36	0.080	0.086
							35	2.52	2.52	0.075	0.075
					-		50	9.55 45	2.52	0.086	0.086
Tedlar 200BG30WH	n.c.	+0.33	11,660	11,880	110	100		5.51	6.53	0,085	0.080
							15	4.66	5.37	0.086	0.086
	-	'					25	4.48	5.22	0.084	0.084
•							35	4.36	5.0R	0.082	0.083
	-						50	3.64	4.24	0.090	0.089

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Table XLVI

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EFFECTS OF THERMAL-VACUUM ENVIRONMENT ON FILMS AND SHEETS (500 hours at 135°C and 10⁻⁶ torr)

		MEC	HANICAL P	ROPERT	IES			ELECTRICA	L PROP	ERTIES	
MATERIAL	Dimensional Change, %	Weight Change, %	Tensile,	psi	Elongati Break,	on at	Frequency. MH,	Dielecti Constar	rıc nt	Dissip Fact	ation tor
			Control	Test	Control	Test	9	Control	Test	Control	Test
Kapton 200XH667	-0.09	+0.11	inval	id	inval	id	1	2.92	2.84	<0.001	<0.001
							15	2.90	2.82	<0.001	0.006
							25	2.86	2.80	0.044	0.005
							35				
							20				
Kynar-200	-1.10	-0.24	inval	bi	inval	Id		2.61	3.47	0,094	0.098
				_			15	3.26	2.86	0.126	0.133
							25	1.91	2.66	0.125	0.131
							35	1.89	2.54	0.122	0.102
							50	1.58	2.14	0.101	0.061
Mylar Type 150T	-9.94	-0.34	38,680	32,660	22	25		2.74	2.47	0.018	6.015
							15	2.64	2.41	0.015	0.013
							25	2.60	2.39	0.013	0.012
							35	2.55	2.22	0.013	0.012
							50	2.05	2.22	0.016	0.016
Mylar Type 200A	-1.03	-0.09	Ìnva	lid	inval	bi		2.30	2.53	0.016	0.014
							15	2.17	2.37	0.015	0.011
							25	2.71	2.38	0.013	0.011
							35	2.05	2.38	0.012	0.012
							50	2.41	2.18	0 013	0.008
P-2300	n.c.	-0.12	9,310	8,8,80	93	90		2.99	3.04	0.004	0.004
						<u>-</u>	15	3.30	2.89	0.007	0.003
							25	3.30	2.94	0.005	0.003
							35	2.90	2.84	0.010	<0.001
							50	2.82	2.77	0.018	0.003

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Table XLVI (Concluded)

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<0°.0001</p> <0.0001 <0.0001 0.001 0.002 0.003 <0.001 0.009 <0.0001 0.070 0.063 0.073 0.072 0.015 0.075 0.0800.082 0,076 0,096 Test Dissipation Factor Control <0.0010> <0.0001 <0.0001 <0.0001 <0.0001 0.107 0.080<0.001 <0.001 0.081 0.096 0.080 0.075 0.086 0.085 0.086 0.084 0.082 0.090 <0.001 ELECTRICAL PROPERTIES 2.13 2.33 2.12 2.08 1.84 1.90 1.99 1.56 1.23 Control Test 2.64 1.97 2.92 2.69 2.33 4.31 4.06 3.92 3.89 3.34 **Dielectric** Constant 2.35 2.33 2.24 2.02 2.35 1.55 3.15 2.021.78 1.12 3.34 2.52 2.52 4.66 4.48 4.36 1.91 4.02 5.51 3.64 Frequency, MHz 1 15 25 35 50 1 15 25 35 35 1 15 15 25 35 50 1 15 25 35 35 50 Elongation at Break, % Control Test 98 invalid invalid invalid 110 11,050 Test MECHANICAL PROPERTIES Tensile, psi invalid invalid invalid Gontrol 11,660 Weight Change, % -0.87 -6.01 -0.13 -0.19 Dimensional Change, % -12.88n.c. +0.46 +2.71 Tedlar 100BG30TR Tedlar 200BG30TR Teflon FEP-500A MATERIAL FPO 681-111

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Tabie XLVII

EFFECTS OF DECONTAMINATION CYCLES FLUS THERMAL-VACUUM EXPOSURE ON FILMS AND SHEETS

		MECHAN	ICAL PROPE	RTIES				ELECTRICA	L PROPI	ERTIES	
MATERIAL	Dimensional Change, %	Weight Change, %	Tensıle	tsd •	Elongati Break,	on at %	Frequency	Dielect Const	cric ant	Dissip	ation .or
	1		Control	Test	Control	Test	z HM	Control	Test	Control	Test
Kapton 200XH667	-0.12	+0.20	inval	id	inval	id	1	2.92	2.80	<0.001	200.0
		_					15	2.90	2.80	<0.001	0.007
							25	2.86	2.75	0.044	0.006
							35	2.79	2.71	0.112	0,006
							50	2.33	2.35	0.031	0.008
Kvnar-200	-1.34	-0.24	inval	Id	inval	1d	-1	2.61	3.21	0.094	0.088
							15	3.26	2.95	0.126	0.169
							25	1.91	2.88	0.125	0, 121
							35	1.89	2.74	0.122	0.119
							50	1.58	2.17	0.101	0.097
Wvlar Tvpe 150T	-9.73	-0.36	38,680	32.500	22	33	Π	2.74	2.29	0.018	0.013
							15	2.64	2.28	0.015	0.012
							25	2.60	2.29	0.013	0.011
							35	2.55	2.26	0.013	0.012
							50	2.05	2.02	0.016	0.014
Mylar Type 200A	-1.06	-0.13	inve	bile	inva	lid	l	2.80	2.31	0.016	0.014
							15	2.71	2.53	0.015	0.012
							25	2.71	2.57	0.013	0.011
							35	2.65	2.46	0.012	0,011
							50	2.41	2.31	0.013	0.006
P-2300	п.с.	-0.15	9,310	11,300	93	99	I	2.99	3.18	0.004	0.004
							15	3.30	3.04	0.007	0.003
							25	3.30	3.04	0.005	0.003
							35	2.90	2.98	0.010	0.901
							50	2.82	;	0.018	0.004

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Table XLVII (Concluded)

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		MECH	ANICAL PROP	ERTIES				ELECTRIC	AL PRO	PERTIES	
MATERIAL.	Dirensional	Weight	Tensile,	psi	Elongat Break	10n at %	Frequency,	Dielect Consta	tric ant	Dissip Fact	ation tor
	Change, %	Change, »	Control	Test	Control	Test	2 HW	Control	Test	Cuntrol	Test
PPO 681-111	•o•u	-0.70	ilavali	ų	l evnt	id	1	2.35	2.13	<6.001	0.001
							15	2.35	2.48	<0.001	0.002
							25	2.33	2.48	<0.001	0.003
							35	2.24	2.48	0.107	0.010
							50	2.02	2.12	0.080	0.006
Teflon FEP;500A	+0.25	-0.03	inval	p	inval	- pı	_	2.02	2.00	<0.0001	<0.0001
					-		15	1.98	1.96	<0.0001	<0.0001
							25	1.91	1.87	<0.001	<0.0001
							35	1.55	1.55	<0.0001	< 6.0001
							50	1.12	1.22	<0.0001	<0.001
Tellar 100BG30TR	-12.83	-0.08	invali	q	inval	id	I	4.02	3.74	0.081	0.064
							15	3.34	3.38	0,096	0.081
							25	3.15	3.31	0.680	0.078
							35	2.52	3.70	0.075	0.070
							50	2.52	2.68	0.086	0.079
	+2.82	-0.14	11.650	1021.11	011	100		רא עי	1 31	0 085	0 079
IMACOCINE TOTAL										2002	
							C 1	00.4	101.4	0.000	0.082
							25	4.48	4.00	0.084	0.080
							35	4.36	3.95	C.082	0.083
							50	3.04	3.34	0.090	250.0

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Table XLVIII

EFFECTS OF DECONTAMINATION CYCLES ON TIE CORDS'LACING TAPES (6 cycles of 30 hr at 50°C with ETO-Freen 12)

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MATERIAL.	DIMENSIONAL	WEIGHT	TENSILE	., psi	ELONGA AT BRE	TION AK, °₹
	CHANDE, «	UNADE, A	Control	Test	Control	Test
Gude-Space 18D96	Ŋ. C.	+}.08	42,000	42,900	29	32
Temp-Lace 256H	n.c.	+0.52	14,900	15,100	32	40

Table XLIX

EFFECTS OF THERMAL-VACUUM ENVIRONMENT ON THE CORDS/LACING TAPES (500 hr at 135° C and 10^{-6} torr)

NATERIAL	DIMENSIONAL	WEIGHT	TENSILE	, psi	ELONGA AT BREA	TION K, %
	CHANGE, A	CHANGE, A	Control	Test	Control	Test
Gude-Space 18D96	-0.23	-0.45	42,000	30,550	ź9	19
Temp-Lace 256H	-0.61	-0.49	14.900	14, v50	32	28

Table L

EFFECTS OF DECONTAMINATION CYCLE PLUS THERMAL-VACUUM ENVIRONMENT ON TIE CORDS/LACING "APES

MATERIAL		WEIGHT	TENSILI	f psi	ELONGA AT BRE	ATION AK, %
	CHANGE, *	CHANGE, 7	Control	Test	Control	Test
Gude-Space 19D96	-0.22	-0.43	42,000	29,400	29	20
Temp-Lace 256H	-1.35	~0.54	14,900	14,300	32	30



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FIG. 18 VIEW OF MULTIPLE-CELL TEST UNIT SHOWING THE LOCATION OF SAMPLES IN THE TEST CELLS



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FIG. 19 STIFF WIRE CONNECTOR FOR TWO-TERMINAL MEASUREMENTS OF DIELECTRIC CONSTANTS

V. STANDARDIZED '1 .RMAL-VACUUM WEIGHT-LOSS DETERMINATIONS

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Vacuum-weight-loss determinations have been carried out for a number of materials in accordance with the procedure (TM-5046-8) and utilizing the equipment described in Interim Report No. 2 under this Contract. The equipment and procedure were designed to provide a standardized method for the determination of the weight-loss of polymeric materials exposed to a thermal-vacuum environment of 125° C and 10^{-6} torr, which would serve as standards for materials specifications.

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Weight-loss determinations and limits were reported for many polymeric materials in Interim Report No. 2; the results reported here in Tables LI to LVI were obtained during the period March-May 1966; since that time, the equipment has been stored for future use or for transport to JPL because of other commitments for the vacuum system.

The schematic drawing of a standardized vacuum-weight-loss apparatus and a photograph of the vacuum assembly of the apparatuses are given in Figures 20 and 21.

Polyvinyl fluorides

The results of determinations for two polyvinyl fluoride films, Tedlar 200BG30WH and Tedlar 100BG30TR, candidates for structural and insulation applications in spacecrafts, are given in Table II. As produced by E. I. du Pont and de Nemours Company, Tedlar 100BG30CR is a "pure" transparent film and Tedlar 200BG30WH is a filled material; materials were used as received. Weight-loss results for the filled material are somewhat higher than for the unfilled, but both are within the lower limits for suitable polymeric materials. It is interesting to note, however, that both materials are subject to thermal-forming, i.e., flat films rolled for insertion in the sample chambers tended to maintain the roll curvature after removal from the thermal-vacuum environment.
Polyfluorocarbons

Although many creditable publications have referred to the superior thermal-vacuum weight-loss characteristics of polyfluorocarbons, it was nevertheless considered appropriate to determine the values for at least two representative materials in order to complement the systematic survey being undertaken for candidate spacecraft materials.

The data given in Table LII for Teflon TFE and FEP (du Pont) clearly confirm the superiority of these materials, as far as weight-loss criteria is concerned; materials were used as received. Correlative work has shown that the weight-loss (and VCM) of leflon FEP is real and is attributable to a small quantity of plasticizer and/or processing oil (see Section II).

Micartas

Two hardware and structural Micarta materials produced by the Westinghouse Electric Corporation were shown to be suitable candidates for spacecraft use by weight-loss criteria. These were: Micarta H-5834, a phenolic-glass fiber laminate and Micarta 20201-2, a silicone resinglass fiber laminate. The data reported in Table L are for the materials used as-received.

Polyphenylene oxide

Excellent weight-loss results were obtained for polyphenylene oxide films produced by the General Electric Company. As shown in Table LIV, little difference is indicated in the weight-loss incurred by PPO 681-111, a clear film annealed 1/2 hour at 180°C and PPO 531-801, an opaque film used as received, except that the PPO (clear) weight-loss has not leveled off as well as the PPO (opaque).

Vinylidene fluoride-hexafluoropropylene

Viton A4411A-990, a seal and gasket material produced by E. I. du Pont de Nemours and Company, has been subjected to every evaluation encompassed by this program. By this time, it would have been disappointing if the standardized weight-loss results, summarized in

Table LV, did not agree with all former good data. The sample material was used as received.

Polyacrylic

Weight-loss data are reported in Table LVI for another elastomer, Hycar 520-67-108-1 produced by the B. F. Goodrich Company. The weightloss value is in good agreement with that obtained from micro-VCM determinations.

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Since this equipment has been stored, the micro-VCM apparatus (Section II) has been in full routine operation and has demonstrated not only that equivalent weight-loss values can be obtained for samples of less than 1/10 the size in 1/5 the time, but also that additional information is provided, that is, maximum VCM content.

MATERIAL	TREATMENT	S. WT. (AV), g.	WT. <u>L</u> OSS (AV), %		
			48 hr	96 hr	192 hr
Tedlar 20013G30WH	As received	1.01	0.17		
		0.99		0.15	
		1.00			0.16
Tedlar 100BG36TR	As received	1.06	0.0		
		1.05		0.09	
		1.05		1	0.08

Table Ll

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THERMAL-VACUUM WEIGHT-LOSS DETERMINATIONS POLYVINYL FLUORIDE FILMS, Du Pont $(125^{\circ}C \text{ and } 10^{-6} \text{ torr})$

Table LII

THERMAL-VACUUM WEIGHT-LOSS DETERMINATIONS: POLYFLUOROCARBON FILMS, Du Pont (125°C and 10⁻³ torr)

WATERTAL	TREATMENT	S. ₩T. (AV), g.	WT. LOSS (AV), %		
			48 hr	96 hr	192 hr
Teflon FEP 500A	As received	0.72	0.01		
		0.60		0.03	
		0.72			0.04
Teflon TFE 100A	As received	1.14	0.01		
		1.30		< 0.01	
		1.25			< 0.01

Table LIII

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THERMAL-VACUUM WEIGHT-LOSS DETERMINATIONS: MICARTAS, WESTINGHOUSE $(125^{\circ}C \text{ and } 10^{-6} \text{ torr})$

MAቸድርጋ TAI	TOPATHONT	S. WT.	WT. LOSS (AV), %		
MAILNIAL	INCAIMENT	g.	48 hr	96 hr	142 hr
Phenolic-Glass Fiber	As received	1.35	0.38		
		1.36		0.39	
		1.27			0.40
Silicone-Glass Fiber					
Micarta 20201-2	As received	1.70	0.29		
		1.60		0.28	
		1.62			0.34

Table LIV

THERMAL-VACUUM WEIGHT-LOSS DETERMINATIONS: PPO FILMS, GENERAL ELECTRIC (125°C and 10⁻⁶ torr)

MATERIAL	TREATMENT	S. WT.	WT. LOSS (AV), %		
		8.	48 hr	96 hr	192 hr
PPO 681-111 (clear)	Annealed 1/2 hr at 180°C	1.10	0.06		
		1.05		0.11	
		1.02			0.22
PPO 531-801	As received	1.30	0.08		
(1.24		0.14	
		1.17			0.14

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Table LV

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THERMAL-VACUUM WEIGHT-LOSS DETERMINATIONS: VINYLIDENE FLUORIDE-HEXAFLUOROPROPYLENE, Du Pont

 $(125^{\circ}C \text{ and } 10^{-6} \text{ torr})$

матерта:	TREATMENT	S. WT. (AV), g.	WT. LOSS (AV), %		
MATERIAL			48 hr	96 hr	192 hr
Viton A4411A-990	As received	5.08	0.45		
		5.20		0.46	
		5.01			0.53

Table LVI

THERMAL-VACUUM WEIGHT-LOSS DETERMINATIONS: POLYACHYLIC, GOODRICH (125°C and 10⁻⁶ torr)

MATERYAL	TREATMENT	S. WT.	WT. LOSS (AV), %		
MAIENIAL		g.	48 hr	96 hr	192 hr
Hycar 520-67-108-1	As received	3.52	1.25		
		3.54		1.24	
		3.61			1.33

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FIG. 20 DESIGN DRAWING OF VACUUM-WEIGHT-LOSS APPARATUS



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VI. CHEMICAL AND PHYSICAL CHARACTERIZATION OF POLYMERIC MATERIALS

This phase of the program has been concerned with the selection and development of analytical procedures which will be most useful for the characterization of base polymeric materials and which may be applied as quality-control measures. Thus, determinations were reported for typical epoxy adhesives in Interim Report No. 1 (August 1965) and for typical RTV-silicone sealants in Interim Report No. 2 (March 1966).

During this last period, work has been performed on analytical procedures for polyurethane base materials: available methods for typical properties were found to be satisfactory without furthe: development.

Also characterized was an unique polymeric material, a silverfilled epoxy solder.

Polyurethanes

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Polyurethanes are especially interesting to spacecraft designers because of their resistance to high temperatures (coating materials) and because they can be produced as foams (structural materials, potting compounds). Two immediately available and representative polyurethanes were selected for characterization: (1) Eccocoat IC-2, a 2-part coating material and (2) Eccofoam FPH, a 2-part foamed-inplace material; both of the mate ials are produced by Emerson and Cuming, Inc.

The data for the chemical and physical properties of the two materials are summarized in Tables LVII and LVIII; immodiately below the tables are the infrared absorbance curves for the base materials (Figures 22 and 23). The isocyanate-group determination was made according to a procedure for its determination in terms of the

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combining weight with di-<u>n</u>-butylamine (so-called amine equivalent)¹ to form a corresponding urea:

The sample is dissolved in toluene and di-n-butylamine is added. The mixture is then diluted with isopropanol, and titrated with standard hydrochloric acid solution.

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Hydrolyzable halide was determined according to ASTM Method D 1638 for "Urethane Foam Raw Materials," except that the sample solution was not diluted with water since this caused immediate solidificatio of the reactants:

> The isocyanates are converted to carbamates by methanol; any liberated hydrochloric acid is retained by the methanol. The hydrochloric acid along with other chlorides are titrated potentiometrically, using standard silver nitrate solution.

Determinations for hydroxyl number were made as per ASTM Method D 1635 (A):

The hydroxyl growns setylated with a solution of acetic anhydride in pyrulne. The excess reagent is decomposed with water, the resulting acetic acid is titrated potenticmetrically with standard sodium hydroxide solution.

The density of the polyurethane pre-polymers was determined by ASTM wethod D 1875-61T. Filler content was determined by a straightforward procedure using toluene as a solvent for Eccocoat IC-2 and acctone for Ecc foam FPH. Nonvolatile content of the polyurethanes was determined by ASTM Method D 1259-61(b).

'nfinied absorbance curves of the materials were prepared by evaporating solvent solutions of the polyurethanes on optical salt flats and scanning the resulting film from 2-15 microns.

The amine nitrogen content of the secondary parts of the polymeric materials was determined by a nonaqueous procedure employing titration with perchloric acid in acetic acid (T.2-5046-2, Interim Report No. 2).

¹Dombrow, B.A., <u>Polyurethanes</u>, Reinhold Publishing Corporation, New York, 1965, Appendix A. The diluent recommended for use with Eccocoat IC-2 was readily identified by mass spectroscopy as ethyl acetate.

Epoxy-Silver Solder

The characterization of the epoxy-silver solder is of particular interest to this program since it is not only a unique and important material for spacecraft use, but also because it has been used successfully for more than 7 months as a conductor in the heated-glass system of the long-term constant-locd apparatus (see "Mechanical Properties"). Data for several properties of Eccobond Solder 56C (Emerson and Cuming, Inc.) are summarized in Table LIX and the infrared absorbance curve for the resinous material is given in Figure 24. detates the states and the states and show the states and show the states and state

Hydrolyzable halide was determined by a method previously modified at SRI (TM-5046-1, Interim Report No. 2), and based on ASTM D 1726-62T. Epoxy equivalent was determined by ASTM D 1652-62T, and filler content was determined by a modified Shell Company method (TM-5046-3, Interim Report No 2). The infrared absorbance curve was obtained by evaporating an acetone solution of the resinous material on an optical salt flat and scanning the resulting film from 2-15 microns.

Table LVII

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CHEMICAL AND PHYSICAL PROPERTIES: ECCOBOND 56C (EMERSON AND CUMING)

PROPERTY	VALUES	
Epoxy equivalent (g sample/g-equiv. of epoxy)	1,099 ~ 1,109	
Hydrolyzable halide	<0.01 %	
Filler content (silver)	79.61 - 79.92 wt-%	
Nonvolatile content	99.54 - 99.62 wt-%	



FIG. 22 INFRARED ABSORBANCE CURVE OF ECCOBOND SOLDER 56C

Table LVIII

CHEMICAL AND PHYSICAL PROPERTIES.

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ECCOCOAT IC-2 (EMERSON AND CUMING)

PROPERTY	VALUES			
Isocyanate content	6.85 - 5.88%			
Hydroxyl content	n.d.			
Hydrolyzable halide content	< 0.005%			
Filler content	none			
Density at 25°C, g/ml	1.064 - 1.066			
Nonvolatile Material content	98.4 - 98.4%			
PART B				
Amine nitrogen content	9.46 - 9.59 wt-%			





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Table LIX

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CHEMICAL AND PHYSICAL PROPERTIES: ECCOFOAM FPH (EMERSON AND CUMING)

PROPERTY	VALUES				
Isocyanate content	33.01 - 33.96%				
Hydroxyl content	n.d.				
Hydrolyzable halide content	< 0.005%				
Filler content	none				
Density at 25°C, g/ml	1.139 - 1.139				
Nonvolatile material content	48.7 - 50.7%				
PART B					
Amine nitrogen content	0.14 - 0.14%				





VII. NEW TECHNOLOGY

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In accordance with the New Technology Clause of Contract No. 950745 under NAS7-100, formal announcement is made of the various technologies which have been developed or advanced at Stanford Research Institute under JPL/NASA sponsorship during the period March to November 1966.

(1) MACRO-VOM APPARATUS FOR POLYMERS

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Innovator: R. F. Muraca First used: June 1966 Reports: Interim Report No. 2 (Mørch 1966) Interim Report No. 3 (December 1966)

The macro-VCM apparatus and accompanying vacuum system are designed to provide information on the deposition and subsequent removal of volatile condensable material released by all kinds of polymers exposed to a simulated spacecraft environment.

(2) MULTIPLE-CELL TEST UNIT FOR POLYMERS

Innovator:	R, F. Muraca
First used:	August 1966
Reports:	Interim Report No. 3 (December 1966)
	(Monthly Reports No. 22, April 1966, and following)

The nultiple-cell test unit is designed to accommodate 30 sample cells, each of which may contain several configurations of a single polymeric product according to the subsequent physic /mechanical property tests to be performed. The system is so designed that the materials may be exposed a* atmospheric pressure to decontamination cycles and subsequently exposed, in the same sample cells, to a thermal-vacuum environment.

 (3) LONG-TERM STORAGE APPARATUS FOR POLYMERS: CONSTANT LOAD Innovators: R. F. Muraca with N. Fishman First used: March 1966
 Reports: Interim Report No. 3 (December 1966) (Monthly Report No. 21, March 1966, and following)

The long-term storage apparatus, including a vacuum system, is designed specifically for subjecting plastic materials to constant load during long-term exposure to a simulated spacecraft environment.

Technical discussion of the work performed with these apparatuses is given in Part I of this report, "Materials Characterization." Complete design drawings and mechanical descriptions of the apparatuses are given in Part II of this report: "Test Equipment Design."

Also included in Part II of this report are complete design drawings for other apparatuses announced in Interim Report No. 2 as "New Technology" and currently in use.

- (1) Micro-VCM Apparatus for Polymers
- (2) Long-Term Storage Apparatus for Polymers Under Constant Strain).

It is a pleasure to announce the transfer of one of these technologies, MICRO-VOM APPARATUS FOR POLYMERS, to NASA centers which are concerned with the screening and quality-control of polymeric products. Duplicate units are being fabricated at the Goddard Space Flight Center and the Jet Propulsion Laboratory with the SRI design drawings. The procedure for performing micro-VOM determinations is described in detail in Part I (Appendix C) of this report.

VIII. PROGRAM REVIEW AND SUMMARY

The program at SRI began as Contract No. 950324 in 1962 (continuing as Contract No. 950745 in 1965) and has had as its objective the definition of the behavior of polymeric materials in a thermalvacuum environment so that test methods could be developed to obtain meaningful data for the selection of materials for use in spacecrafts and for providing quality-control parameters.

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The very first experiments with polymeric materials in the thermal-vacuum environment demonstrated that very few of them decompose at temperatures below about 150° C and that the volatile content of nearly all polymers (in thin sections) can be reduced to a very low value within a short time in the environment. (The polymers which decompose or evaporate at a moderate temperature have never been given serious consideration for use in spacecrafts, e.g., polystyrene, polyvinylchloride, polyethylene, etc.) Additional experiments showed that the loss of weight incurred by polymeric materials in a thermal-vacuum environment is due primarily to occluded gases and lo molecular-weight materials; loss of such material has little effect on mechanical properties. On the other hand, when mechanical properties are affected by the loss of weight, it was found that the low-molecular-weight materials had been deliberately added as plasticizers or they were substances generically related to the polymer matrix (low-molecular-weight polymers) which function as plasticizers. More importantly, however, it was shown that many of the components present in the volatile matter released by a polymer in a thermal-vacuum environment may condense on cool surfaces and become the cause of malfunctions of lenses, mirrors, electrical contacts, etc.

Based on the above findings, a sensitive and rapid micro-procedure was developed for the simultaneous screening of a multiplicity of polymeric products. The procedure (micro-VCM) furnishes for each

polymer sample an accurate value of the total weight-loss and the maximum weight of volatilized material which may condense on a cooler surface. The values obtained by the micro-VCM procedure have been used as the basis for acceptance or rejection of polymers as suitable candidates for further study. The reproducibility of the micro-VCM results recommends the procedure for use as a quality-control measure.

The accuracy of the total weight-loss values obtained in 24 hours by the micro-VCM procedure (samples of about 0.1 gram) has been verified by a number of ways:

- a) Carefully-controlled determinations of thermal-vacuum weightloss were made in a standardized apparatus in which samples up to 5 grams were treated for as long as 190 hours;
- b) Weight-loss values were also obtained by a macro-VCM procedure with samples up to 10 grams; the results from a series of runs as long as 350 hours are comparable to to those obtained by the micro-VCM procedure;
- c) Weight-loss determinations on tensile specimens which had been exposed for 1000 hours in the thermal-vacuum environment also furnished comparable checks;
- d) The accuracy and average reproducibility of the micro-VCM procedure were of the same order of magnitude obtainable from weight-loss determinations made with a thermogravimetric apparatus (Du Pont Model 990, operated at 10^{-6} torr).

A prototype <u>macro-VCM</u> unit was developed in response to the need for information on the rate of deposition of the condensable material released by elastomers in the thermal-vacuum environment and the rate of evaporation of condensed material from the cooler surface. There was also need for an apparatus which would accommodate materials such as sleevings, honeycombs, etc.; the apparatus described in this report can use samples of nearly any configuration as long as thickness does not exceed 1/16" (to minimize the effects of diffusion rates, etc.); however, the apparatus can accommodate samples up to 2" in diameter and 1" in height, and it is anticipated that determinations will be made of the influence of sample thickness (or surface-tovolume ratios) on the rates of evolution of volatile matter. The macro-VCM determination provides an important decision criterion in

one selection of spacecraft materials, for it can detect whether the substance which is responsible for an acceptably-low VCM value in the micro-VCM determination can evaporate from the cooler surface on which it has condensed.

Mass spectrometric identification of the volatile materials released by polymers has definitely confirmed that polymers are not decomposing in the thermal-vacuum environment of $125^{\circ}C$ and 10^{-6} torr, and has led to suggestions for up-grading the polymeric products.

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Equipment was designed to measure the changes in mechanical properties (uring the exposure of elastomers and plastics to the thermal-vacuum environment. Properties measured <u>in situ</u> included continuous and intermittent stress relaxation of elastomers and creep of plastics. Tensile properties were measured before and after exposure for both kinds of materials. In general, the materials examined have shown little enange of mechanical properties on exposure to the thermal-vacuum environment, and these data confirm the indications by micro-and macro-VCM techniques of the suitability of these materials for spacecraft use. Long-term storage tests (up to 9 months--a flight to Mars) on three elastomers and a plastic also confirmed the results obtained from other tests.

A comprehensive polymer test program was initiated to provide information on physical and mechanical properties of polymers after exposure to ETO-Freen decontamination cycles, a thermal-vacuum exposure, and a decontamination cycle followed by a thermal-vacuum exposure. A multiple-cell test unit was designed to accommodate all configurations of specimens as required by the tests to be performed, for example, dielectric constant, tensile, compression, hardness, dimensional change, etc. As many as 30 polymeric materials, from adhesives to tie-cords were examined. The results of the first run indicate that mechanical property data are comparable to those obtained by the time-consuming <u>in situ</u> measurements and that weightloss data are comparable to those obtained by other procedures. Except for an expected increase in weight during the decontamingtion

cycles, there is little difference in the results obtained for materials which have undergone both decontamination and thermalvacuum exposure or those which have been subjected to thermal-vacuum exposure only. At least one more run will be made; should sirilar results be obtained, it will be advantageous to substitute the multiple-cell test-unit procedure to obtain mechanical property data and electrical constants for the evaluation of polymeric materials. ł

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Data obtained thus far in the performance of this program of work suggests that candidate polymers be screened by the micro-VCM technique and that those which qualify for further consideration be examined by the multiple-cell test-unit procedure and the macro-VCM procedure so as to obtain the data which establishes their suitability for use in spacecrafts. Further work with mechanical-property testing <u>in situ</u> should not be considered. Work on preparation of quality assurance procedures should be continued.

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Name	Typical Structure	Spacecraft Applications
Acetal	$CH_{3} - C - O \begin{pmatrix} H \\ I \\ C \\ H \end{pmatrix} = O \begin{pmatrix} H \\ I \\ C \\ H \end{pmatrix} = O \begin{pmatrix} I \\ I \\ C \\ H \end{pmatrix} = O \begin{pmatrix} I \\ I \\ C \\ I \end{pmatrix} = O \begin{pmatrix} I \\ I \\ C \\ I \end{pmatrix} = O \begin{pmatrix} I \\ I \\ I \\ I \end{pmatrix} = O \begin{pmatrix} I \\ I \\ I \\ I \end{pmatrix} = O \begin{pmatrix} I \\ I \\ I \\ I \end{pmatrix} = O \begin{pmatrix} I \\ I \\ I \\ I \\ I \end{pmatrix} = O \begin{pmatrix} I \\ I \\ I \\ I \\ I \end{pmatrix} = O \begin{pmatrix} I \\ I \\ I \\ I \\ I \\ I \end{pmatrix} = O \begin{pmatrix} I \\ I \\ I \\ I \\ I \\ I \\ I \end{pmatrix} = O \begin{pmatrix} I \\ I \end{pmatrix} = O \begin{pmatrix} I \\ I$	Hardware and structural Wire enamels
Acrvlic	$ \begin{bmatrix} H & R \\ - & -C \\ - & - \\ H & -C \\ - & - \\ H & -C \\ - & - $	Seals and gaskets Sleeving
Alkyd	$\left \begin{array}{ccccc} 0 & 0 & H & H & H \\ 0 & 0 & H & H & H \\ c & c & c - 0 & c - c - c - 0 \\ 0 & H & 0 H & H \\ 0 & H & 0 H & H \\ \end{array} \right _{n}$	Protective coatings Thermal coatings
Amide	$- \left[\begin{array}{c} \begin{array}{c} H \\ H \\ N - \begin{pmatrix} H \\ I \\ C \\ H \\ B \\ 5 \end{array} \right]_{5} - \begin{array}{c} O \\ I \\ C \\ - O \\ D \\ n \end{array} \right]_{n}$	Coated fabrics Films and sheets Hardware and structural Tie cord/lacing tape
Butyl	$ \begin{bmatrix} CH_3 & H & H & C4_3 & H & H \\ I & 3 & I & I & 1 & 3 & I & I \\ C & -C & -C & -C & C & C & -C & - \\ I & I & I & I & I & I \\ CH_3 & H & H & H & H \end{bmatrix}_{n} $	Seals and gaskcts
Carbon _£	$\left[\begin{array}{c} 0 - \left(\begin{array}{c} 0 - \left(\end{array}{0 - \left(\begin{array}{c} 0 - \left(- \left($	Hardware and structural
Diallylphthalate	$ \begin{array}{c ccccccccccccccccccccccccccccccccccc$	Mardware and structural

Appendix A CANDIDATE POLYMERIC MATERIALS FOR SHACECRAFT APPLICATIONS

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· ·	Appendix A (Concluded)	Э.
Name	Typical Structure	Spacecraft Applications
Phenylene oxide	$ \begin{bmatrix} & & CH_3 \\ & & & -o \\ & & & CH_3 \end{bmatrix}_{n} $	Hardware a i structural
Silicone	$ \left[\begin{array}{ccc} R & R \\ \hline I \\ Si - O - Si - O \\ I \\ R & R \\ \end{bmatrix}_{R} $	Adhesives Coated fabrics Foams Hardware and structural Honeycomb structures Protective coatings Sealants Seals .nd gaskets Sleeving Tapes Tie cord/lacing tape
Sulfone	$\left\{ \left(\begin{array}{c} \overset{CH_{3}}{\overset{I}}{\overset{I}{\overset{I}{\overset{I}{\overset{I}{\overset{I}}{\overset{I}{\overset{I}{\overset{I}{\overset{I}{\overset{I}{\overset{I}{\overset{I}{\overset{I}{\overset{I}{\overset{I}{\overset{I}{\overset{I}{\overset{I}{\overset{I}{\overset{I}}{\overset{I}{\overset{I}}{\overset{I}{\overset{I}}{\overset{I}{\overset{I}{\overset{I}{\overset{I}{\overset{I}{\overset{I}{\overset{I}{\overset{I}{\overset{I}{\overset{I}{\overset{I}{\overset{I}{\overset{I}}{\overset{I}}{\overset{I}{\overset{I}}{\overset{I}}{\overset{I}{\overset{I}}{\overset{I}{\overset{I}}}}}}}}}$	Hardware and structural
ی Tetrafluoroethylene	$ \begin{bmatrix} F & F \\ I & I \\ C - C \\ I & I \\ F & F \end{bmatrix}_{n} $	Coated fabrics Hardware and structural Seals and gaskets Shrinkable materials
¢ Urethane	$ - \left(\begin{array}{c} H & O \\ i & \parallel \\ N - C - OR \end{array} \right)_{n} $	Foams Protective coatings Sealants Thermal coatings
Vinyl fluoride	$ \begin{bmatrix} H & H \\ I & I \\ C - C \\ I & I \\ H & F \end{bmatrix}_{n} $	Films and sheets
Vinylidene fluoride	$ \begin{bmatrix} \mathbf{F} & \mathbf{H} \\ \mathbf{i} & \mathbf{i} \\ \mathbf{C} - \mathbf{C} \\ \mathbf{i} & \mathbf{i} \\ \mathbf{F} & \mathbf{H} \end{bmatrix}_{\mathbf{R}} $	Films and sheets
Vinylidene fluoride- hexafluoropropylene co-polymer	$ \begin{array}{c} $	Scals and gaskets

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APPENDIX B

CODE LISTING OF MANUFACTURERS

AAC	Ablestik Adhesive Company		
ACB	American Cyanamid Company, Bloomingdale Department		
ACM	Allied Cherical Corporation, Mesa Products, Plastics Division		
APC	Armstrong Products Company		
ARP	American Reinforced Plastics Company		
BCM	The Borden Chemical Company, Mystik Tape, Inc.		
BFG	B. F. Goodrich Chemical Company		
BHM	Bentley-Har is Manufacturing Company		
DCC	Dow-Corning Company		
DUE	E. I. du Pont de Nemours and Company, Inc., Elastomer Chemicals Department		
DUF	E. I. du Pont de Nemours and Company, Inc., Fabrics and Finishes Department		
DUM	E. I. du Pont de Nemours and Cumpany, Inc., rilm Department		
DUP	E. I. du Pont de Nemours and Company, Plastics Department		
EFI	Electrofilm, Inc.		
EMC	Emerson and Cuming, Inc.		
ENJ	Enjay Chemical Company		
FPC	Finch Paint and Chemical Company		
GBE	Gudebrod Brothers Silk Company, Inc., Electronics Division		
GEC	General Electric Company, Chemical Materials Department		
GES	General Electric Company, Silicone Products Department		
GEW.	General Electric Company, Wire and Cable Department		
HCC	Hughson Chemical Company		
HEX	Hexcel Products, Inc.		
HYS	Hysol Corporation		
MCC	Magna Coatings and Chemical Corporation		
MMA	3M Company, Adhesives, Coatings, and Sealers Division		
MMC	3M Company, Chemical Division		

MME 3M Company, Electrical Products Division

MMI 3M Company, Irvington Division

MRC The Marblett Corporation

PER Permacel

Wist

PFC Pennsylvania Fluorocarbon Company

PRC Products Research and Chemical Corporation

PTI Product Techniques, Inc.

RAY Rayclad Tubes, Inc.

SCA Shell[°]Chemical Company, Adhesives Department

SIS Sargent Industries, Stillman Rubber Division

SPT Stone Paper Tube Company

TEC $^{\circ}$ The Epoxylite Corporation

TMC The Mica Corporation

UCP Union Carbide Corporation, Plastics Division

WEI Westinghouse Electric Corporation, Insulating Materials Division

Westinghouse Electric Corporation, Micarta Division

APPENDIX C

DETAILS OF MICRO-VCM DETERMINATIONS VCM AND WEIGHT-LOSS AT 125°C AND 10⁻⁶ TORR (VCM Collector Plates at 25°C)

Apparatus and Equipment

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Micro-VCM Apparatus. - See Part II: Test Equipment

Microbalance. - Capable of weighing to 20 g \pm 10 μ g

Aluminum Sample Boats. - A simple die is easily fabricated to form curved Al-foil boats (about $1/2" \times 1/4"$) which will fit the contour of the circular sample compartments. The formed boats must be degreased and oven-dried prior to use.

Polished Copper Collector Flates. - Fabricated as described in Part II. Plates are cleaned in 1:1 nitric acid solution or "Trisol," 1:1:1 methanol: benzene: chloroform, depending on condition. Frequently, they may be polished by machine buffing. They are stored in a desiccator which contains a slotted rack to support them.

Chamber for 50% Humidity. - May be made simply by filling the lower part of a standard laboratory glass desiccator with a saturated solution of $CA(NO_3)_2 \cdot 4 H_2O$; should contain numbered containers or supports for emplacement of samples.

Desiccator for Samples. - Charged with suitable desiccant; should contain numbered containers or supports for emplacement of exposed samples.

Samples and Recording

Identify samples as completely as possible. Note details of mixing (if any) and curing. Use as much sample as the boats will accommodate (100-200 mgm).

Finished Polymers. - Try to obtain representative portions of the sample, avoiding cuts from edges or surfaces.

Two-Part Polymers. - Weigh, on an analytical balance, sufficient quantities of base polymer and reactant portion 30 as to provide a representative batch.

General Techniques:

Adhesives, sealants, etc., may be poured directly into weighed Al boats before curing, or cut from large form after curing.

Finished polymers may be comminuted and cured before placing in weighed Al boats.

Paints, varnishes, coatings, etc. may be applied by brusning or dipping small pieces of clean and weighed fine-mesh stainless-steel screen; screen is then placed in weighed Al boat.

Tapes, etc. may be rolled tightly to fit into weighed Al boats.

Procedure

Prepare samples as described and place them in weighed Al boats. Place samples and boats in 50%-humidity cnamber for at least 24 hours prior to "initial" sample weighing.

Weigh the conditioned samples and place them in the sample compartments of the micro-VCM apparatus; note the location. Weigh the copper collector plates and affix them to the apparatus, noting the location in reference to specific samples.

When samples and collector plates are in position, cover the apparatus with the bell-jar and begin evacuation; at 10^{-3} corr, activate the liquid nitrogen trap and the cooling system for the collector plates. When the system is at least 10^{-5} torr, elevate the temperature of the copper blocks containing the samples to 125° C.

After the samples have been maintained at 125° C for 24 hours, allow the system to cool, <u>in vacuo</u>, to a temperature of at least 50° C. Then vent the system with dry nitrogen or helium.

Remove the samples and collector plates in a systematic order and store in appropriate desiccators.

Begin weighing of samples after 1/2 hour of storage in the desiccator, in the order of their removal from the thermal-vacuum system. Then weigh the collector plates.

Data to be reported are: sample weight in grams, sample weight-loss in wt-%, and VCM in wt-%.

APPENDIX C

DETAILS OF MACRO-VCM DETERMINATIONS VCM AND WEIGHT-LOSS WITH TIME AT 125°C AND 10⁻⁶ TORR (VCM Collector Plates at 25°C)

Apparatus and Equipment

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Macro-VCM Apparatus. - See Part II: Test Equipment

Analytical Balance. - Capable of weighing to ± 0.1 mg

Polished Aluminum Collector Plates. - Fabricated as described in Part II. Plates are cleaned by a <u>dip</u> in dilute NaOH solution (1%) or by rubbing with a wipe soaked in "Trisol,"]:1:1 benzene; methanol: chloroform, depending on condition. Frequently, they may be polished by machine buffing. They are stored in a desiccator, suspended from a rack by their weighing wires.

Sample Storage. - Any arrangement of simple, closed containers in which samples can be stored as a set for each run of the series (12 individually-wrapped packages in each of 4 containers) or stored according to polymer sample (8 individually-wrapped packages in each of 6 containers).

Desiccator for exposed samples. - Charged with suitable desiccant.

Samples and Recording

Fresh samples in <u>duplicate</u> are to be used for each run of the series.

Select sample sizes so as to provide for an optimum VCM weight of about 10 mgm (based on micro-VCM data); for example:

micro-VCM value	macro-VCM s. wt.
1.0%	. 1 g
0.1%	10 g
0,01%	(limited by compartment siz

Prepare sufficient amount of sample to prov'de for duplicate determinations in each of the 4 runs of the entire series and to provide for a re-run or a more extended run.

Record details of mixing and curing (if any), or other preparation.

All finished polymers or prepared samples should be of a thickness less than 1/8":

<u>Elastomers</u>. - generally about 1/16"; use as is, cut to appropriate size; may lay flat on sample-cell screen or be rolled loosely according to size. Record dimensions.

Films, Tapes, etc. - May be rolled loosely. Record dimensions.

<u>Plastics</u>. - If thickness exceeds 1/8", cut or saw into strips of <1/8". Record dimensions.

<u>Tubings</u>. - Use as-is, cut into short lengths as required by weight and compartment size. Record dimensions.

Adhesives, Sealants. - Spread to about 1/16" thick on clean and weighed Al foil strips of appropriate size before curing; may be rolled loosely to fit into sample-cells.

Where possible, the material may be released from one master plate and weighed into \mathbb{C}'' Al dishes.

<u>Coatings</u>. - May be brushed of lipped onto a weighed copperwire coil (about 3-ft length, is-gage).

Procedure

Weigh samples on analytical balance to ±0.1 mgm.

Weigh collector plates on analytical balance to ±0.1 mgm.

Set collector plates and samples in place in the macro-VCM apparatus.

Evacuate the system to 10^{-3} torr (1 micron) with forepump; in meantime, be sure refrigeration is on the baffles and the diffusion pump is operative.

Continue evacuation with diffusion pump to $<10^{-5}$ torr.

Bring temperature of sample compartments to 125°C; start timing.

Record every 24 hours: sample temperature, collectorplate temperature, pressure. Maintain sample at 125° C for prescribed period; then cut heat and let cool under high vacuum to less than 50° C.

Vent system with dry helium or nitrogen.

Remove samples and collector plates to desiccators; weigh within one hour after removal.

Store all exposed samples in labeled, paper envelopes.

Schedule

Duplicate samples are to be run for periods of 24, 43, 95, and 330 hours.

Twenty-four hours are allowed between each run for weighing, clean-up, and re-start. Samples and duplicate collector plates for a succeeding run are prepared and weighed while a run is in operation.

The following sequence is suggested [but any sequence may be used, providing no time is lost over week-ends (or holidays) and the series is completed within a 4-week period]:

Run #1 48-hr period - start Mon. aft; complete Wed. aft.
Run #2 96-hr period - start Thus. aft; complete Mon. aft.
Run #3 24-hr period - start Tues. aft; complete Wed. aft.
Run #4 330-hr period (2 wks) - start Thus. aft; complete Thus. morning.

Reporting

Report all recorded data.

Report weight-loss and VCM as wt-%.

Report any observations, such as change in color of samples, thermo-setting or other distortion, appearance of collector plates, etc.

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APPENDIX D INDEX OF TABULATED DATA

Polymeric Product	Table Reference	Polymeric Product	Table Reference
Armalon 98-101	111	Diall FS-4	VI
Armstrong A-2/A	I	Diall FS-10	VI
Armstrong A-2/E	I	Diall FS-40	VI
Adhesive 46591	I	Dory1 B109-4	IX
B-224-2	IX	Dery1 B109-5	IX
B-276	IX	Doryl H-17479	V L
Beckman #101577	VI	Doryl H-17511	TE XXXIX,XL,XLI
Ben-Har 263, G3	XIII	Eccobond 45/15	I
Ben-Har 1062, HAI	XIII	Eccobond 55/9	I
Ben-Har 1151,	IIIX	Eccobond 55/11	I .
Armasi1-2		Eccobond 56C/9	I,LVII
Ben-Har 1151, HAI	XIII	Eccobond 104 A/B	1
Ben-Har 1151, Superwall	XIII	Eccocoat 210 A/B	IX
Ben-Har 1151, UL	XIII	Eccocoat C-26 A/B	IX .
Ben-Har 1258-1, B	XIII	Eccocoat EC-200 A/B	IX
Ben-Har Acryl A	XIII.XX	Eccocoat IC-2	IX,LVIII
Ben-Har Ex-Flex 1500	XIII	Econcoat PCA/16	IX
Ben-Har Lecton B	XIII	Eccocoat PH7	IX
Ben-Har Pyro-Sleeve	XIII	Eccocoat VE A/B	IX
Cat-A-Lac Flat Back	XV	Eccofoam FPH/126H	V,LIX
(463-1-8)		Eccofoam S	v
Cat-A-Lac Clear	XV	Eccofoam SH	V,XXIII
(475 1) Cat-A-Lac White Glose	xv	Eccofoam Sil/25	v
(443-1-500)		Eccogel 1265 A/B	IX
Cement 4684	I	Eccosil 5000 A/B	v
Chemlok 507	I	Electrofilm 2396	VIII
CPC-21.	x	Electrofilm 4306	VIII
CPC-22	x	Epiall 1914	٧I
CPC-41	x	Epon 828/Versamid-125	I
Delrin 100MC10	VI,XXII,XXXIX XL,XLI	Epon 901,'B-1	I
		Epon 901/B-3	1
Delrin 150NC10	VI	Epon 903	I
Delrin 500NC10	vı	Epon 914	ĩ
Delrin 507NC10	VI	Epon 917	I ·
Delrin 900NC10	VI	Epon 919 A/B	I

Polymeric Product Epon 931 A/B Epon 934 A/3

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EX-1091 EX-1092 Fairprene 84-001 Fairprenc 80-060 Fairprene 80-070 Fairprenc 8C-080 FM-1000 FM-1044 Formex magnet wire Giyptal 1201 ~ Glyptal 1202 Glyptal 9564 Gudebrod 721H Gudebrod ?22S Gude-Space 18D96 Hexcel HRP

Hexcel HMH Hexcel-HRS Hycar-1

Hycar-2

Hycar-3

Hycer-4

Hycar-5 Hycar-6 JPL-1001 JPL-1002

Kapton 200XH667 Kapton, 300XHF929A Table Geference Polymeric Product Table Reference IV,XXXVII,XLV, Kynar 200 -91-1D-1581 XI,XXV111,XXIX, XXXII Lexan 100-111 XI,XXVIII,XXIX, Lexan 101-111 XXXII Lexan 101-112 XI,XXVIII,XXIX, Lexan 103-112 XXXII Lexan 130-111 Lexan 131-111 Lexan 131-112 Lexan 133-112 Lexan 140-111 I,XXII Lexan 141-111 Lexan 141-132 XVII Lexan 243-112 Micaply G-284 Micaply EG-725 Micarta 65M25 Micarta H-2497 Mi ... eta H-5834 XVI,XLVIII, XLVIX,L Micarta H-8457 Micarta H-17511 VII,XX1 Micarta H-17690 Micarta 20201-2 XI,XIX,XXII, XXIV, XXVIII, XXIX,XXXI, Mylar Type 100S XXXIII,XXXJV, Mylar Type 100T XXXV,XL11, XLIII,XLIV,LVI Mylar Type 200A XI,XXVIII,XXIX, Mylar Type 500A XXXI XI,XXVIII,XXIX, Mystik 7020 XXXI Mystik 7300 XI,XXVIII,XXIX, Mystik 7352 XXXI Mystik 7452 XI,XXVIII,XXIX, XXXI Mystik 7455 XI,XXVIII,XXIX, Mystik 7503 XXXI 6-Nylon **Optical Black 101-C10** IX,XXII P-2300 JV,XXXVI,XXXVII XLV, XLVI, XLVII P-7395-121-2

XLVI,XLVI1 VI VI,XXII VI,XXII VI,XXII VI,XXII VI ٧I VI VI VI VI VI,XXI,XXIV ٧I VI VI II,XXI,XXII,XXIV II,XXXIV,XL,XLI VI,XXXIX,XL,XLI LIII II 11 VI VI,XXXIX,XL,XLI, LIII IV IV,XLV,XLVI,XLVII XLV, XLVI, XLVII IV,XXII,XXXVI, XXXVII XIV XIV XIV XIV XIV XIV IV XV VI,XXXVII,XLV,XLVI XLVII VI XII

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Penntube II-SMT

Polymeric Product	Table Reference	Polymeric Product	Table Reference
Phenall 8010	VI	SE-3713	XI,XXVIIT,XXIX,
Phenall 8060	VI		XXX,XLII,XLIII,
Phenall 8700	VI	CF- 2913	ALIV
Polyurethane magnet wire	XVII	2013	XXX, XLII, XLIII,
PPO 681-111	VI,XXI,XXII, XXIV,XXXVI,XXXVII, XLV,XLVI,XLVI2	0P 4611	XLIV VI VVV
		5E-4511	х 1 , хлх
PPO 531-081	VI,LIV	SE-5403	XI, XXX
PR-240-AC	VIII	SR-17	
PR-1527 A/B	x	SR-98	
PR-1538 A/B	x	SR-220	1.7
PT-401 /h-11	IX	SR-634-70	XI
PYRE-M.L. R.C5057	XVII	SRD-5905	111
PYRE-M.L. RK-692	IX	SRG-1810	111
Ribbon Dope #520	x	SRGA-0214	J11
RTV-11	x	SS-4004	1
R:V-41/T-12	x	SS-4044	1
RTV-60	X,XXII	SS-4101	1
RTV-77/T-12	x	SS-4120	I
RTV-90	x	Stur-D-Lace 18DH	XVI
RTV-560	x	Stycast 40/7	Ä
RTV-580/T-12	x	Stycast 1090/9	v
RTV-615	x	Stycast 1099/11	v
RTV-630 A/B	x	Stycast 1095/11	v
RIV-632 A/~	х	Stycast 1210 A/B5	X
RTV-655 A/B	х	Stycast 1217/9	1X
RTV-757	x	Stycast 1263 A/B	x
Scotchcast-235	x	Stycast 1269 A/B	x
Scotchcast-241	x	Stycast 2741/15	X
Scotch Tape #62	XIV	Sylgard-184	X
Scotch Tape #27	XIV	TB5-PTFE	111
Scotch Tape #852	XIV	Tedlar 100BG30TL	1 V
Scotchweld EC-2216 A/B	I	Tedlar 100BG30TR	XLV, XLVI, XLVII, LI
SE-555	X1,XXVIII,XXX	Tedlar 200BG30WH	XLV,XLVI,XLVII,LI
SE-556	XI, XXX	Tedlar 200SG40TR	XXXVII
SE-3604	XI, XIX, XXVIII,	Tedlar A130WH	IV
	XXIX, XXX, XXXIII,	Teflon-FEP 100A	XI
	XLIII	Teflon-FEP 200A	XXXVII
SE-3704	XI, XXX	Teflor-FEP 500A	XI, XIX, XXII, XXIV,
SE-3804	XI, XXX	Toflon-FFD 6000	~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~
SE-3613	XI,XXVIII,XXIX, XXX,XLII,XLITI, XLIV	Teflon-TFE 100A	LII

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Polymeric Product	Table Reference
Temp-Lace H256H	XVI,XLVIII, XLVIX,L
Thermofit CRN (clear)	XII
Thermofit CPN (white)	XII
Thermofit CRN (black)	XII
Thermofit Kynar	XII
Thermofit RNF-100	XII, XX
Thermofit TFE (7-30-09)	XII
Thermofit TFE (7-32-19)	XII
Thermofit TFE-R	XII
TPM-2/10	Х
TPM-3/10	Х
TPM-6 A/B	x
Vespel SP-1	VI
Viton A4411A-776	XI,XXXII
Viton A4411A-777	XI,XXXII
Viton A4411A-778	XI,XXXII
Viton A4411A-990	XI,XIX,XXII, XXIV,XXXIII,XXXIV XXXV,XLII,XLIII, XLIV,
Zytel-31	VI ·
Zytel-42	VI
Zytel 101NC10	VI

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