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NASA CR-132725

### EFFECTS OF HIGH ENERGY SIMULATED SPACE RADIATION ON POLYMERIC SECOND - SURFACE MIRRORS

Lawrence B. Fogdall Sheridan S. Cannaday

Final Report to NASA-Langley Research Center for Contract NAS1-13530

October, 1975

# REPRODUCIBLE COPY (FACILITY CASEFILE COPY)

Boeing Aerospace Company Seattle, Washington 98124

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1.	REPORT NO. NASA CR-132725	2. GOVERNMENT A	CCESSION NO.	З.	RECIPIENT'S CA	TALOG NO.
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7.	Author(s) Lawrence B. Fogdall and She	ridan S. Canna	day	8. P	ERFORMING ORG D180-18014	ANIZATION REPORT #
9.	PERFORMING ORGANIZATION NAME AND A	DDRESS		10.	WORK UNIT NO.	
	Boeing Aerospace Company			<u> </u>	CONTRACT OF C	DANIT NO
	Seattle, Wash. 98124				NAS1-13530	RANT NU.
112	SPONSORING AGENCY NAME AND ADDRES	s		13.	TYPE OF REPORT	& PERIOD COVERED
	NASA Langley Research Cente	r			Contractor	Report
	Hampton, Virginia 23665				Aug. 15/4	- 000. 1975
				14.	SPONSORING AG	ENCY CODE
15.	SUPPLEMENTARY NOTES	• • • • • •		· .	•	
	Final Report. Technical Mo NASA Langley Research Cente	nıtor, Wayne S r, Hampton, Vi	Iemp, Materials D rginia	ivis	s10n,	
15.	ABSTRACT					
	A radiation effects experimental program has been performed, in which second- surface mirror type thermal control coatings were exposed to ultraviolet radiation, electrons, and protons simultaneously. Stability was assessed by making periodic spectral reflectance measurements in <u>situ</u> (and in air after testing for comparison). Solar absorptance coefficients were derived by com- puter. Many of the exposed materials showed large amounts of degradation in reflectance and absorptance, principally due to the electron exposure. A series of tests was conducted, leading to the identification of a modified second- surface mirror that shows considerable improvement and promise for stability during thermal control applications in a charged particle space radiation en- vironment.					cond- d by ter com- n in A series nd- lity n en-
17.	KEY WORDS	· · · · · · · · · · · · · · · · · · ·	18. DISTRIBUTION STAT	TEME	NT	
	Electron Ultraviolet Radiation High Energy Reflectance Radiation Solar Absorptance Proton Thermal Control Coating		U.S.A.			
19.	SECURITY CLASSIF, (of this report)	20. SECURITY CLAS	SIF, (of this page)	21.	NO. OF PAGES	22. PRICE
	Unclassified	Unclassi	fied	i c	164	

For sale by National Technical Information Service, Springfield, Virginia 22151

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#### INTRODUCTION

This document is a final report to NASA-Langley Research Center for experimental radiation effects work performed by the Boeing Aerospace Company between August 1974 and June 1975 under contract NASI-13530. In this contract Boeing has utilized a multi-radiation source facility to investigate the stability of certain spacecraft materials <u>in situ</u> (in vacuum). The materials, furnished by NASA, are dielectric surfaces and composites suitable for temperature-control of spacecraft.

The materials studied are the latest candidates for application on NASA spacecraft. Included are derivatives of commercial films such as FEP Teflon and Kapton, and also some of the product of NASA laboratory efforts in the polymer field. The commercial films, being optically transparent over much or all of the visible wavelength region, have been metallized through commercial processes to convert them to highly reflective second surface mirrors, then adhered to aluminum substrates. The latter have been applied to reflective aluminum substrates directly. No paints or other "Lambertian" (diffusely reflecting) candidates have been investigated. (An approximation of a diffusely reflecting surface—"embossed" metallized FEP Teflon—has been included.) More complete descriptions are given in the Section "Test Materials".

The materials selection is indicative of trends in this field: there is no longer an emphasis or reliance on "white paints". Along with this is the emphasis on utilizing a state-of-the-art facility for the evaluation testing and measurements. The 3 primary space radiation sources—ultraviolet radiation, protons, and electrons—are all simulated, simultaneously. The vacuum environment inside the chamber is also an excellent simulation of space conditions, having been obtained by techniques that preclude use of pumping fluids or oils. Reflectance measurements of these coatings have been made with highly accurate techniques as will be shown in the Section "Test Results". The most significant features of this facility will be presented in detail in the Section "Facility Description".

#### PROGRAM REPORT

This Section describes the facility used for the irradiation tests, reviews the test materials and samples, presents the detailed results from the 3 tests conducted, and discusses the findings and various comparisons of results from 2 or more tests.

#### Facility Description

The work was done in a Boeing vacuum chamber known as the CRETC (combined radiation effects test chamber). This facility was originally designed, and over the years has continually been upgraded, to optimize the capability for as complete and accurate simulation of the space radiation environment as knowledge and the state of the art will allow. For example, conventional techniques for vacuum pumping—still in widespread use elsewhere—have been discarded in the CRETC to eliminate contamination. Energy capabilities have been expanded as the need has risen for investigating the effects of charged particle spectra on spacecraft materials.

The capabilities of the CRETC facility can be summarized as follows:

- Continuum ultraviolet radiation (xenon arc discharge) at selectable intensities ranging from less than one solar constant to 5 solar constants (1 A.U.), simultaneously with:
- Electrons with energies between approximately 10 and 125 keV (higher energies with conditioning) and/or protons with energies from 0.5 to 80 keV (kilo electron volts).
- 3. Controlled temperatures for test and reference (standard) samples; temperatures range from -195  $^{\circ}$ C (-320  $^{\circ}$ F) to +180  $^{\circ}$ C (+360  $^{\circ}$ F). Temperature control is not interrupted for measurements in the chamber's integrating sphere.
- 4. Vacuum pumping (both rough and hard) without resorting to organic and other contaminating fluids. The sequence used is (a) dry nitrogen gas aspiration, (b) cryo-sorption, (c) large-surface LN<sub>2</sub> cryogenic, and (d) ion pumping, to obtain a 10<sup>-8</sup> torr vacuum before testing begins.

- 5. Extensive automation, interlocks, and sequential shutdown procedures during unmanned night-time operations, to allow as high a reliability as possible during long-term, continuous testing.
- High-precision spectral reflectance data system, consisting of a double-beam spectrophotometer coupled to a data-logging module whose output is ready for computer processing.

#### Test Parameters and Operations

From the available range within which the experimental apparatus operates, the following test parameters were chosen for the first test in accordance with the contract work statement:

- (1) UV at a one-sun rate in the 0.2 to 0.4 micrometer wavelength region.
- (2) Electrons with an energy of 125 keV (before scattering——115 keV after scattering) and with a continuous arrival rate (flux) at the sample plane of 2 x  $10^9$  e/cm<sup>2</sup>-second.
- (3) Protons with an energy of 50 keV and with a continuous flux of  $2 \times 10^9$  p/cm<sup>2</sup>-sec at the sample plane.
- (4) Sample substrate temperature of 20 <sup>O</sup>C, maintained throughout the test.

The experimental apparatus is pictured in Figures 1, 2, 3, and 4. Figure 1 is an overall view of the CRETC facility. Figure 2 is a closer view of the electron high-voltage feedthrough portion of the chamber, a recent addition allowing testing at the relatively high energies desired for this program. Figure 3 shows the sample reflectance measurement system. Figure 4 shows the first array of NASA-Langley-supplied samples, mounted in the chamber. These samples were quite specular before irradiation; hence in Figure 4 they appear in various shadings of gray depending upon what portion of the room environment is reflected by each of them into the camera. In reality the samples are "gold" or "silver" as to general color tone. Figure 4 is intended to show the sample array configuration on the temperature-controlled sample block in the CRETC chamber, and the size of each 1-cm-square sample relative to adjacent apparatus.



Figure 1. Boeing CRETC Facility

The contract also called for measurements of each sample's reflectance properties periodically throughout the test, and frequently enough to measure well the rate of reflectance degradation and onset of damage saturation. Both in-air and in-vacuum measurements were included. Once the test exposure began, all measurements were made in vacuum (<u>in situ</u>). The exact test-hour points at which measurements were made, are listed in a table later, in the Section "Test Sequence".



Figure 2. View Above CRETC Chamber Showing Electron Gun Power Supply and Feedthroughs

As for sample measurement technique, the CRETC utilizes an integrating sphere reflectometer with detectors <u>in situ</u>. Only the measurement light sources, monochromator, and electronic and light chopping apparatus are external to the chamber. Sample reflectance measurements are made relative to the reflectance of the integrating sphere's magnesium oxide wall. Normalization to absolute reflectance, assisted by NBS and other known reference



Figure 3. Sample Reflectance Measurement System

measurements, is handled by computer since all original sample data is computerprocessed routinely. The thermophysical property of interest for this program (solar absorptance) is derived from solar reflectance, which is defined as



Figure 4. Second-Surface Mirror Coating Samples for NASA-Langley Test 1

Solar reflectance, 
$$R_s = \frac{\int I_s(\lambda)R(\lambda)d\lambda}{I_s(\lambda) d\lambda}$$

where  $I_{s}(\lambda)$  is the solar irradiance as a function of wavelength  $\lambda$ , and  $R(\lambda)$  is sample reflectance, generally a function of  $\lambda$ . Of course, solar absorptance is, by definition, unity minus  $R_{s}$  in opaque samples. The integral  $\int I_{s}(\lambda)d\lambda$  in the denominator is an expression of the solar "constant". When data processing is computerized it is appropriate to replace the integral evaluations with numerical summations, so that

$$\alpha_{s} = 1 - R_{s} = 1 - \frac{\sum_{i=1}^{100} R_{\lambda}}{100}$$

This simplified  $\alpha_s$  equation is valid when the wavelengths used in the computer summation are selected in accordance with the solar spectral shape or weighting, which is to say, in accordance with the shape of the  $I_s(\lambda)$  function.

Figure 5 is a set of typical computer-processed data plots of reflectance vs. wavelength. The reflectance data are from an aluminum control sample measured 5 times over a period of approximately 600 hours. The precision and reproducibility of the CRETC measurement system are evident from these data.



Figure 5. Effects of Vacuum on Control Sample (Polished Aluminum Substrate)

Other salient points regarding test operations can be summarized as follows. Checking and calibration of UV intensity is done before beginning a test and periodically during the test. The sun rate is determined from radiometer output levels taken with and without a UV-absorbing filter over the radiometer detector. Since the UV-absorbing filter also excludes ten percent of the incident radiation at wavelengths longer than the ultraviolet (five percent reflection at each surface of the filter), a correction is made for radiometer readings taken with the filter over the radiometer sensor. For a total radiation reading T and a UV-filtered reading F,

Ultraviolet Sun Rate =  $\frac{T - \frac{10}{9}F}{(8.0)(0.091)} = 1.37 (T - 1.11 F),$ 

where 8.0 is the radiometer sensitivity in millivolts per incident sun  $(\approx 0.14 \text{ watts/cm}^2)$  and 0.091 represents the ultraviolet content of the sun (at air mass zero). The uniformity of ultraviolet radiation intensity across the sample array is determined by "mapping" with the radiometer held in a precise jig. Spatial uniformity of ultraviolet radiation can be maintained within plus or minus 10 percent across the sample array. The F and T values indicate that the ultraviolet content of the long-arc xenon sources is approximately 10 percent of their total output.

Similarly, electron and proton intensities are measured just after the chamber is evacuated, before the pre-exposure sample measurements commit to a go-ahead. These intensities are also checked frequently during testing, via continuous monitors (Faraday cups) and an array of metallic tabs that measures beam uniformity.

The proton beam originates in an RF-excited plasma more than 5 meters from the sample plane. The proton beam steadily expands after leaving the plasma orifice and attains a size larger than the sample array as the protons reach the sample plane. Spatial uniformity is typically  $\pm 15$  percent; temporal uniformity typically is  $\pm 10$  percent. The proton beam is not rastered, but it is passed through a bending magnet to separate H<sup>+</sup> ions from H<sup>+</sup><sub>2</sub> and other species.

Electrons are emitted thermionically from a filament that is part of an electron gun inside the CRETC chamber. Accelerating and focusing electrodes

between the filament and the sample plane form the electrons into a beam. A thin foil scatters the electrons as the beam continues toward the sample array. No electrons are stopped in the foil, but electron energy degrades (approximately 10 keV) as the beam traverses the foil, emerging with a Gaussian distribution. No rastering occurs. The array of samples constitutes a small portion of the straight-ahead solid angle, so that spatial uniformity of electrons across the samples is  $\pm 10$  percent, and temporal uniformity a like value.

#### Test Sequence

The contract originally called for one test lasting 1400 hours. The first half of this was normal, including the collection of several "rounds" or sets of reflectance data on each sample. At 690 hours the proton source became erratic, necessitating an up-to-air repair. A second test was instituted to obtain comparative data for exposure rates of 1.5 times the rates used in the first test. The principal finding of this second test, as will be discussed more fully following presentation of test results, was that several materials reacted in an appreciably different manner when exposed to electrons and protons at the 50 percent higher rates. This second test was ended at 610 hours. A third test was added to the program which included more early-hour test data and the use of a silver-filled-adhesive with the 5-mil, silvered Teflon material.

The parameters that were varied during these three tests are summarized in Table I. The exposure hours and fluences at which reflectance measurements were made in each test are indicated in Table II. In Table II ultraviolet sun hours (ESH) are equal to the number of exposure hours stated for Tests 1 and 3; for Test 2, ultraviolet exposure equals the number of equivalent hours stated.

#### Test Materials

In the series of tests that was performed for this contract the emphasis was on studying Teflon, Kapton, and specialized polymers that could be applied to metal substrates. Variations in film thickness, in second-surface metal, and in lamination of Teflon and Kapton were included. In the third test a

	1		
PARAMETER	TEST 1	TEST 2	TEST 3
Test Length	690 hours	610 hours	850 hours
Sun Rate	1.0	1.5	1.0
Max. Equivalent Solar Hours	690 ESH	915 ESH	850 ESH
Proton Flux	2x10 <sup>9</sup> p/cm <sup>2</sup> -sec	3x10 <sup>9</sup> p/cm <sup>2</sup> -sec	2x10 <sup>9</sup> p/cm <sup>2</sup> -sec
Max. Proton Fluence	5x10 <sup>15</sup> p/cm <sup>2</sup>	7x10 <sup>15</sup> p/cm <sup>2</sup>	6x10 <sup>15</sup> p/cm <sup>3</sup>
Electron Flux	2x10 <sup>9</sup> e/cm <sup>2</sup> -sec	3x10 <sup>9</sup> e/cm <sup>2</sup> -sec	2x10 <sup>9</sup> e/cm <sup>2</sup> -sec
Max. Electron Fluence	5x10 <sup>15</sup> e/cm <sup>2</sup>	7x10 <sup>15</sup> e/cm <sup>2</sup>	6x10 <sup>15</sup> e/cm <sup>2</sup>
Last <u>in situ</u> Reflectance Data	588 hours	610 hours	636 hours
Additional Data	Pre-test % in air	Post-test, in- vacuum %R re- covery	Post-test, %R recovery in air

Table I. Test Parameters and Variants

newly-developed modification to silver-backed Teflon was added. The silverbacked Teflon was bonded to the aluminum substrate with an adhesive which was filled with finely divided silver, providing a partially conducting path from the vapor-deposited silver to the sample's aluminum substrate. All other commercial second-surface mirror coatings in these three tests were bonded to the 6061 aluminum substrates with a flight-qualified, double-back, pressuresensitive silicone adhesive which uses a 0.025 mm Kapton carrier (Mystic 7366).

The Kapton/Teflon laminates were processed using Type C, FEP Teflon, which was bonded to the Kapton by use of heat and pressure. The BTDA/ODA and PPQ are experimental polymers made at Langley and applied directly to the Boeing-supplied substrates. The high solar absorptance values for these materials are indicative of their slight coloration and the "polish" of these substrates. Table II. Exposure Hours and Fluences for Sample Reflectance Measurements

TEST 1	TEST 2	TEST 3
0; pre-exposure,	1.5 Sun Rate	
samples in air		
O; pre-exposure, samples in vacuum	O; pre-exposure, samples in vacuum	O; pre-exposure, samples in vacuum
100 hours	64 hours (x1.5=96 equiv. hours)	44 hours 3.2 x $10^{14}_{14} \text{ p/cm}^2_2$ 3.2 x $10^{14} \text{ e/cm}^2_2$
7.2 x $10^{14}_{14} \text{ p/cm}^2_2$ 7.2 x $10^{14} \text{ e/cm}^2$	$6.9 \times 10^{14} \text{ p/cm}^2$ $6.9 \times 10^{14} \text{ e/cm}^2$	197 hours
303 hours	193 hours (x1.5=290 equiv. hours)	$1.4 \times 10^{13} \text{ p/cm}^2$ 1.4 x 10 <sup>15</sup> e/cm <sup>2</sup>
2.2 x $10^{15}$ p/cm <sup>2</sup> 2.2 x $10^{15}$ e/cm <sup>2</sup>	2.1 x 10 <sup>15</sup> p/cm <sup>2</sup> 2.1 x 10 <sup>15</sup> e/cm <sup>2</sup>	391 hours 2.8 x $10^{15}$ p/cm <sup>2</sup>
588 hours	391 hours (x1.5=586 equiv. hours)	$2.8 \times 10^{15} \text{ e/cm}^2$
4.2 x $10^{15}_{15} \text{ p/cm}^2_{4.2 \text{ x } 10^{15}_{15} \text{ e/cm}^2_{15}}$	4.2 x $10^{15} \text{ p/cm}^2$ 4.2 x $10^{15} \text{ e/cm}^2$	636 hours 4.6 $\times$ 10 <sup>15</sup> p/cm <sup>2</sup> 4.6 $\times$ 10 <sup>15</sup> e/cm <sup>2</sup>
	610 hours (x1.5=915 equiv. hours) 6.6 x 10 <sup>15</sup> p/cm <sup>2</sup> 6.6 x 10 e/cm	in air after test exposure ended at 850 hours.
	<u>in situ</u> stability check 310 hours after previous reflectance measurements	

è

The materials tested are listed and described in Table III. A multiple number of like specimens was exposed in each test, and this number is given in Table III. Testing multiple numbers of specimens provided a statistical base for averaging changes in the values of properties that depend on microscopic phenomena (see Discussion).

#### Test Results

<u>Principal Results, Test 1</u>.- All the samples exposed in this test showed measurable (but not extreme) degradation of reflectance, and therefore increased solar absorptance, as the test progressed. All  $\alpha_s$  values derived from the reflectance measurements made during the first test are gathered in Table IV. A comparison of results obtained on the 5-mil aluminized FEP Teflon and on the 5-mil silvered FEP Teflon shows somewhat greater damage in the aluminized material (Figure 6). This is consistent with the wider band-gap or wavelength



Figure 6. Change in Solar Absorptance for Teflon Samples During First Test

	Number of	Samples Exp	osed in
SAMPLE DESCRIPTION	Test l	Test 2	Test 3
5-mil <sup>①</sup> Type A FEP Teflon/ 2000 A Aluminum/Adhesive	3®	4Q3	30
5-mil Type A FEP Teflon/1800 Å Silver/ 400 Å Inconel/Adhesive	3®	30	30
5-mil Type A FEP Teflon/1800 A Silver/ 400 A Inconel/Silver-Filled Adhesive			3
2-mil Type A FEP Teflon/1800 Å Silver/ 400 Å Inconel/Adhesive	2	2	2
Diffuse <sup>④</sup> 5-mil FEP Teflon/1800 Å Silver/ 400 Å Inconel/Adhesive	2	2	2
l-mil Kapton/2000 A Aluminum	2	2	2
0.5-mil Kapton/2000 Å Aluminum	3	2	2
Laminate No. 1 (0.3-mil Kapton/5-mil Type C Teflon/Silver/Inconel/ Adhesive)	3	3	3
Laminate No. 2 (O.15-mil H-film/ 5-mil Type C Teflon/Aluminum/ Adhesive)	2	2	
BTDA/ODA polyimide film/ Polished Aluminum	2	2	2
PPQ (polyphenylquinoxaline)/ Polished Aluminum	2	2	2

#### Table III. Materials Tested and Number of Specimens Exposed

NOTES: ① Thickness in mils stated so as to conform with manufacturer's specification. In metric units, each mil equals 25.4 micrometers, and 5 mils equals 0.127 millimeter. ② An additional sample of this material was present in the vacuum environment (but shielded from radiation exposure) as a control or "standard". ③ One of these 4 samples was perforated at 3 places within its 1 cm<sup>2</sup> area just prior to mounting in the test chamber.
④ An approximation to diffuse reflection was produced by an embossing technique prior to metallization.

Sample Description	Number	Before In Air	Exposure In Vacuum	100 hrs.	<u>n Situ</u> Afte 303 hrs.	er 588 hrs.	Change in $\alpha_s$ After 588 hrs.
Aluminum (control	) (27)	(0.118)	(0.108)	(0.105)	(0.106)	(0.106)	(-0.002)
5-mil FEP o	(11)	(0.158)	(0.152)	(0.146)	(0.151)	(0.152)	(0.000)
Teflon/2000 A		0.155	0.147	0.163	0.190	0.226	0.079
Aluminum	2	0.155	0.148	0.164	0.193	0.230	0.032
	3	0.155	0.149	0.164	0.195	0.228	0.079
5-mil <sub>o</sub> FEP Teflon/	(18) 24	(0.103)	(0.096)	(0.088)	(0.088)	(0.089)	(-0.007)
1800 <sub>0</sub> A Silver/ 400 A Inconel	24 25 26	0.099 0.102	0.092 0.093 0.096	0.099 0.098 0.099	0.119 0.125	0.151 0.142 0.166	0.059 0.049 0.070
Embossed Silvered	15	0.096	0.089	0.096	0.124	0.143	0.059
5-mil FEP Teflon	16	0.099	0.099	0.097	0.126	0.150	0.051
2-mil FEP Teflon/	13	0.080	0.071	0.078	0.096	0.107	0.036
Silver/Inconel	14	0.078	0.069	0.076	0.095	0.106	0.037
l-mil <sub>o</sub> Kapton/	5	0.360	0.352	0.376	0.403	0.417	0.065
2000 A Aluminum	9	0.361	0.352	0.381	0.416	0.432	0.080
1/2-mil Kapton/ 2000 A Aluminum	6 7 3	0.323 0.324 0.323	0.313 0.312 0.314	0.336 0.339 0.342	0.360 0.364 0.371	0.373 0.379 0.386	0.060 0.067 0.072
PPQ/Polished	12	0.374	0.365	0.392	0.420	0.437	0.072
Aluminum	17	0.394	0.386	0.424	0.461	0.478	0.092
BTDA-ODA/	19	0.452	0.440	0.474	0.495	0.521	0.081
Polished Aluminum	23	0.437	0.428	0.465	0.486	0.510	0.082
Laminate No. 1	20	0.246	0.236	0.259	0.285	0.308	0.072
(Kapton/Teflon/	21	0.244	0.238	0.261	0.239	0.337	0.099
Silver/Inconel)	22	0.247	0.242	0.264	0.295	0.318	0.076
Laminate No. 2 (H-film/Teflon/ Aluminum)	4 10	0.362 0.356	0.350 0.346	0.367 0.373	0.415 0.409	0.455 0.447	0.105 0.091

## Table IV. Test 1 Solar Absorptance Values (1-Sun Rate)

NOTES: ① Sample numbers and data in parentheses represent unexposed control samples. For the other, exposed samples, the test hours listed in column headings are numerically equal to UV sun hours (ESH). Proton and electron fluences that correspond to these test hours are listed in Table II. ② Change in solar absorptance is the final <u>in situ</u> value after 588 hours, minus the value in vacuum before exposure.

region over which aluminum is highly reflective and therefore more sensitive to apparent degradation. Figure 6 also shows the  $\alpha_s$  changes in diffuse (embossed) 5-mil FEP Teflon and in the 2-mil FEP Teflon, for further comparison.

Data from the 3 types of Kapton surfaces exposed in Test 1 are shown in Figure 7, which indicates the highly consistent results obtained from the measurements on these similar materials. The precision of the CRETC reflectance measurements, as evidenced by stability of control (standard) samples, was discussed earlier in the Section, "Test Parameters and Operations" (see Figure 5).



Figure 7. Change in Solar Absorptance for Kapton Samples During First Test

A graph of each sample's reflectance as a function of wavelength for all measurement times in Test 1 (equivalent to one full row of data in Table IV) is included in Appendix A.

Principal Results, Test 2.-Following the end of the first test, a 610hour exposure was conducted on like samples using a 1.5-sun rate, for an equivalent exposure of 915 hours (Table I). It was determined from the data in this test that the dielectric properties of certain sample formulations led to a high density of electrical discharge breakdowns in the polymer layer(s) and underlying metals, in response to the simultaneous charged particle flux of 3 x 10<sup>9</sup> electrons cm<sup>-2</sup> sec<sup>-1</sup> and 3 x 10<sup>9</sup> protons cm<sup>-2</sup> sec<sup>-1</sup>. Other sample formulations withstood this charge arrival and buildup rate. The electrical discharge phenomena were visually manifested as an alteration of the polymer films from a transparent condition to a translucent nature. Therefore those showing breakdown became diffusely transmitting and reflecting (gray) instead of allowing a specular reflection from the second-surface metal. Large  $\alpha_r$ changes were noted in samples with a high density of electrical breakdown sites. Sites were concentrated in different portions of the 1 cm<sup>2</sup> area of each sample; since the spectrophotometer measurement beam illuminates a 4 mm square in the center of any given sample, some variation in reflectance changes was noted from sample to sample depending on local breakdown-site density. The tabular  $\alpha_s$  data for this test (Table V) indicates the extent of these sample-to-sample differences, which have been averaged in the 2 figures that follow. The  $\alpha_s$  changes in several Teflon coatings are shown in Figure 8. Figure 9 is for samples having a Kapton outer (exposed) layer.

Appendix B contains plots of reflectance versus wavelength for every sample in Test 2. The large decreases in hemispherical reflectance during the first exposure increment (from 0 to 64 hours) are evident for those samples which underwent severe discharge breakdown.

<u>Principal Results, Test 3</u>.-The third test involved a return to exposure at a 1.0-sun ("real-time") rate (see Table I). As previously indicated, a new sample formulation was added, having finely divided silver in the adhesive (Table III). Another emphasis in this test (previously stated) was the acquisition of more early-test-hour reflectance data. Thus the first exposure

Table V. Test 2 Solar Absorptance Values (1.5-Sun Rate)

Sample Description Number(	D Pre-irrad. In Vacuum	64 hrs.	Situ After 193 hrs.	Exposure 391 hrs.	for 610 hrs.	$\Delta \alpha_{S}$ After 610 hrs.	<u>In Situ</u> 310 hours after the 610-hr Exposure
Aluminum (control) (27)	(0.114)	(0.113)	(0.115)	(0.114)	(0.114)	(0.00)	
5-mil FEP (11) Teflon/2000 A 2 Aluminum 3 3 perforations → 8	(0.134) 0.118 0.115 0.116 0.122	(0.137) 0.128 0.128 0.128 0.141 0.130	$\begin{pmatrix} 0.135 \\ 0.130 \\ 0.130 \\ 0.130 \\ 0.132 \\ 0.132 \end{pmatrix}$	(0.134) 0.132 0.133 0.133 0.133 0.138	(0.136) 0.137 0.137 0.137 0.140	(0.002) 0.019 0.022 0.024 0.020	(0.134)
5-miloFEP Teflon/ (18) 1800 <sub>0</sub> A Silver/ 24 400 A Inconel 26	(0.091) 0.090 0.089 0.092	(0.089) 0.325 0.298 0.299	(0.091) 0.343 0.309 0.309	(0.091) 0.375 0.341 0.335	(0.091) 0.392 0.359 0.351	(0.000) 0.302 0.270 0.259	0.357
Embossed Silvered 15 5-mil FEP Teflon 16	0.091	0.246 0.274	0.260 0.279	0.303	0.325 0.328	0.234 0.236	0.305
2-mil FEP Teflon/ 13 Silver/Inconel 14	0.073 0.078	0.097 0.097	0.094 0.099	0.099	0.115 0.120	0.042 0.042	0.115
l-mil <sub>o</sub> Kapton/ 5 2000 A Aluminum 9	0.354 0.357	0.431 0.434	0.424 0.436	0.434 0.450	0.439 0.468	0.085 0.111	0.453
1/2-mil Kapton/ 6 2000 Å Aluminum 7	0.318 0.317	0.377 0.373	0.369 0.368	0.380 0.382	0.397	0.073 0.080	0.384
PPQ/Polished 12 Aluminum 17	0.491 0.531	0.536 0.584	0.540 0.597	0.551	0.559	0.068 0.099	0.553
BTDA-ODA/ Polished Aluminum 23	0.645 0.747	0.671 0.773	0.666 0.768	0.669 0.773	0.677 0.778	0.032 0.031	
Laminate No. 1 20 (Kapton/Teflon/ 21 Silver/Inconel) 22	0.238 0.239 0.244	$\begin{array}{c} 0.409\\ 0.409\\ 0.405\\ 0.405 \end{array}$	0.410 0.412 0.407	0.444 0.448 0.441	0.457 0.462 0.461	0.219 0.223 0.217	0.452
Laminate No. 2 4 (H-film/Teflon/ 10 Aluminum)	0.357 0.363	0.478 0.463	0.482 0.476	0.523 0.505	0.536 0.525	0.179 0.162	0.515
NOTES: () Sample numbe for the other, exposed si fluences corresponding to value measured after 610	rs and data in pa amples are equal o these test hour test hours, minu	rentheses to to l.5 time s are indic s the value	represent ( es the test cated in Ta	unexposed t hours li able II. ( in vacuum	control sam sted in the Change i before exp	ples. UV equi column headin n solar absorp osure began.	valent sun hours (ESH) gs. Proton and electron tance ( $\Delta\alpha_{\text{S}}$ ) is the in situ



Figure 8. Change in Solar Absorptance for Teflon Samples During Second Test

increment was from 0 just to 44 hours. This small increment successfully caught the threshold for reflectance changes in the test samples. Figure 10 compares the various Teflon-surface coatings tested. Spectral reflectance and solar absorptance changes in the samples with silver-filled adhesive were confined to the low values previously obtainable only in 2-mil silvered Teflon (compare Figure 10 with Figures 6 and 8). In contrast to the first test, the aluminized 5-mil FEP Teflon samples exhibited less degradation than the 5-mil silvered FEP Teflon samples. No explanation can be given for this difference between test 1 and test 3 data, although the difference is only approximately 0.02 in  $\Delta \alpha_s$  values.



Figure 9. Change in Solar Absorptance for Kapton Samples During Second Test

Figure 11 presents data on samples with a Kapton outer layer. Data presented in Figures 10 and 11 are drawn from Table VI, which indicates  $\alpha_s$  values for all samples in Test 3. Appendix C consists of spectral reflectance plots, one for each sample tested in Test 3.

The test samples used in this program were prepared over a period of approximately 8 months, and it appears from the data values that batch-to-batch differences occurred in some cases. For this reason no attempt has been made to gather  $\alpha_s$  values from Tables IV to VI for the purpose of summarizing results on the PPQ and BTDA/ODA polymers in figures similar to those above. Moreover, we observed that the reflectance of individual samples of a given type of coating sometimes degraded at appreciably different rates, though tested at

Sample		Pro-irrad		Citu After		f	Ac. 264000	5
Description Nu	umber (	In Vacuum	44 hrs.	197 hrs.	391 hrs.	636 hrs.	636 hours (2)	after test
Aluminum (control)	(27)	(0.116)	(0.114)	(0.113)	(0.115)	(0.115)	(-0.001)	(0.129)
5-mil FEP o Teflon/2000 A Aluminum	(11) 1 2 3	(0.159) 0.159 0.158 0.157	(0.160) 0.172 0.172 0.173	(0.159) 0.185 0.186 0.186 0.184	(0.161) 0.209 0.208 0.207	(0.163) 0.228 0.229 0.226	(0.004) 0.069 0.071 0.069	(0.173) 0.239 0.258 0.251
5-mil FEP Teflon/ Silver/Inconel/ Conductive Adhesive	4 3 10	0.081 0.080 0.082	0.086 0.084 0.084	0.092 0.094 0.093	0.104 0.107 0.105	0.121 0.123 0.123 0.123	0.040 0.043 0.043	0.143 0.144 0.150
5-mil <sub>o</sub> FEP Teflon/ 1800 <sub>0</sub> Å Silver/ 400 Å Inconel	(18) 24 25 26	(0.089) 0.088 0.087 0.088	(0.091) 0.098 0.098 0.098	(0.092) 0.156 0.111 0.134	(0.092) 0.178 0.133 0.169	(0.092) 0.197 0.152 0.152 0.184	(0.003) 0.109 0.065 0.096	(0.106) 0.209 0.163 0.206
Embossed Silvered	15	0.099	0.109	0.123	0.141	0.156	0.057	0.174
5-mil FEP Teflon	16	0.099	0.108	0.126	0.147	0.164	0.065	0.187
2-mil FEP Teflon/	13	0.076	0.081	0.088	0.099	0.112	0.036	0.132
Silver/Inconel	14	0.076	0.032	0.091		0.115	0.039	0.136
l-mil <sub>o</sub> Kapton/	9	0.355	0.383	0.403	0.437	0.462	0.107	0.426
2000 A Aluminum		0.358	0.383	0.409	0.439	0.466	0.108	0.430
1/2-mil Kapton/	6	0.319	0.339	0.362	0.399	0.433	0.114	0.414
2000 Å Aluminum	7	0.321	0.341	0.369	0.406	0.442	0.121	0.421
PPQ/Polished	12	0.473	0.486	0.507	0.533	0.564	0.095	0.554
Aluminum	17	0.443	0.461	0.479	0.507	0.533		0.530
BTDA-ODA/	19	0.497	0.542	0.567	0.603	0.644	0.147	0.581
Polished Aluminum	23	0.501	0.540	0.567	0.606	0.649	0.148	0.601
Laminate No. 1	20	0.243	0.273	0.295	0.337	0.386	0.143	0.383
(Kapton/Teflon/	21	0.241	0.268	0.295	0.338	0.388	0.147	0.389
Silver/Inconel)	22	0.242	0.272	0.296	0.336	0.389	0.147	0.384
NOTES: (1) Sample nu the test hours liste correspond to these 636 hours, minus the	umbers ar ed in col test hou value i	nd data in par umn headings urs are listed n vacuum befo	entheses r are numeri in Table re exposur	epresent u cally equa II. 2 Ch	unexposed o il to UV su ange in so	control sample in hours (ESH Jar absorpta	es. For the other, ). Proton and elect nce is the final in	exposed samples, cron fluence that situ value after

Test 3 Solar Absorptance Values (1-Sun Rate) Table VI.



Figure 10. Change in Solar Absorptance for Teflon Samples During Third Test

adjacent positions in the chamber. In some cases this is a matter of how early in the test dielectric breakdown began in that portion of the sample illuminated by the DK-2A reflectance measurement beam (which was always at the center of the sample). Figure 12 illustrates this; it shows changes in  $\alpha_s$  in 3 samples of 5-mil silvered FEP Teflon during Test 3 (samples 24, 25, and 26 in Table VI). A few other similar cases can be **discern**ed from Tables IV, V, and VI.

Secondary Results, Test 1.-Though the materials investigated in this program are intended for orbital use, it was also desired to obtain data indicating the coatings' reflectance and absorptance characteristics in air, which would have pre-launch applicability. In nearly all cases, pre-test reflectance values in air have been found to be equivalent to values measured when the sample is in vacuum and before exposure takes place. Exact  $\alpha_s$  values have





Figure 11. Change in Solar Absorptance for Kapton Samples During Third Test



Figure 12. Change in Solar Absorptance for 3 5-mil Silvered FEP Teflon Samples During Third Test

been included in Table IV, and reflectance curve shapes are included in the figures of Appendix A.

<u>Secondary Results, Test 2</u>.-At the end of the radiation exposure period for Test 2 the samples were purposely left in vacuum for a period of 310 hours following the 610-hour measurements; then 10 of them were measured again. This kind of study has come to be known as an <u>in situ</u> or in-vacuum recovery check, or a test of coating reflectance stability in the damaged state. In the past (Reference 1), some thermal control coatings, especially diffusely reflecting white paints, have shown substantial recovery toward original
(pre-test) reflectance values, even when they remain in ultra-high-vacuum (between, say,  $10^{-9}$  torr and  $10^{-8}$  torr). In the case of this program the vacuum measured during the 310 hours was between 2 and 3 x  $10^{-9}$  torr. Recovery in this vacuum environment was less than 0.01 (as to solar absorptance changes). For the 10 samples measured, the exact  $\alpha_s$  differences following 310 hours in the dark in vacuum have been included in Table V. Ten graphs showing the reflectance curves measured, from which the  $\alpha_s$  values were derived, are included herein as Appendix D.

Secondary Results, Test 3.-Post-test, in-air reflectance characteristics of the materials investigated for this program were measured following Test 3. The final in situ reflectance data was obtained as scheduled (Tables II and VI) after 636 hours of exposure to UV, electrons, and protons. Further exposure was begun to obtain longer-term data, but facility vacuum was lost at 850 hours, and final in-air reflectance data was obtained immediately thereafter. Thus instead of a "short-time" comparison of sample reflectance for 636 hours, in situ, and 636 hours, in air, one has, for the last 2 sets of data from Test 3, 636 hours, in situ and 850 hours, in air. Rates of degradation in situ such as those shown in Figures 10 and 11, if extrapolated to 850 hours would show well the base from which recovery was made in air by each sample, ending with reflectance characteristics shown in the graphs of Appendix E, and the  $\alpha_{\text{s}}$  values of Table VI. For several materials the amount of reflectance recovery in air approximately offset the degradation in situ between 636 and 850 hours, so that the final data in air is numerically comparable to the 636-hour data taken in situ.

# Discussion

The phenomenon of dielectric breakdown in polymeric thermal control coatings has never been revealed as graphically as in this program. We have conducted a series of tests under contamination-free vacuum/radiation simulation conditions, the results of which include a discerning of which polymer thicknesses and formulations can tolerate what charged particle fluxes or orbital environment. While much remains to be done in the area of damage mechanisms, a "charge model" can be described, and further developed once a larger data base is established in the future.

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The combined thickness of 5-mil Teflon plus an adhesive layer is required to stop all incident ll5-keV electrons; but 50-keV protons are stopped even in the thinnest polymers exposed during these tests. A model with positive- and negative-charge zones emerges, from which one can derive strengths of electric fields as a function of depth and/or time, the latter being a function of orbital conditions (environmental charged-particle fluxes). Protons tend to accept electrons, once stopped, and diffuse as hydrogen gas at rates dependent upon material properties. In many cases, it can be shown, E fields from electron irradiation build up to values beyond a material's dielectric strength, resulting in breakdown along a locally preferred and momentarily advantageous path. Figure 13 illustrates the geometry and charging conditions for 2-mil and 5-mil Teflon as prepared for these tests.



(PROTONS STOPPED IN ALL DIELECTRIC THICKNESSES STUDIED)



Figure 13. Expanded View of Adhesive-Bonded Thermal Control Coating Samples

The effects of irradiation of 5-mil Teflon and its adhesive layer by the combination of 115-keV electrons and 50-keV protons have been studied by the Monte Carlo code ETRAP. This program is a modification of an existing electron transport code to include the effects of charge trapping in dielectric materials. The electrons are followed by the Monte Carlo process, with collective effects such as induced fields, potentials, and current flow, as well as radiation-induced transient conductivity, included in the modeling.

Figure 13 (left half) shows the problem geometry. The incident electrons are stopped in the Teflon and adhesive backing, with a charge distribution shown in Figure 14. Due to electron multiple scattering the distribution of stopped electrons extends from near the irradiated surface to the adhesive layer, with the peak charge density occurring at a depth of 6 mils (in the upper silicone adhesive layer). Positive charges, consisting of incident protons at the front surface and induced charge in the aluminum substrate, will result in both surfaces of the dielectric sandwich being near zero potential. There is, therefore, little decelerating effect exerted by the test samples upon energetic particulate radiation arriving at the sample plane. But the resulting electric fields and internal potentials create dielectric stress of great intensity on a microscopic scale. This is a mechanism of damage in polymers. The resulting fields and potentials developed in FEP Teflon and its adhesive after  $10^3$  seconds irradiation are shown in Figure 14. The peak field strength after  $10^3$  seconds already exceeds the dielectric failure level of some dielectrics (500-1000 volts/mil or 2-4 x  $10^5$  volts/cm). and additional exposure produces dielectric stress exceeding the most optimistic breakdown data for Teflon.

Figure 15 shows the increase of front and rear surface field strength with time. The slight non-linearity shown is due to the radiation-induced conductivity in the material, and the resulting current flow with increasing electric field. The initial conductivity used for these calculations is  $10^{-16}$  mho/meter. The radiation-induced conductivity is increased to  $10^{-14}$  mho/meter when an electron flux (dose rate) of 2 x  $10^9$  electrons/cm<sup>2</sup>-sec is used for sample exposure.

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Figure 14. Electric Field Strength, Potential, and Density of Trapped Charge in 10 Mjls of FEP Teflon and Adhesive, After 2 x 10<sup>12</sup> e/cm<sup>2</sup> (115 keV)

These exploratory calculations have modeled the general physical processes at work in an irradiated dielectric. More precise calculations using recently developed field-strength-dependent conductivity data, and taking into account the temperature-dependent conductivity of illuminated Teflon (or Kapton), are recommended as future work for more complete predictions.



Figure 15. Front and Rear Surface Electric Fields in a 10-mil Teflon/Adhesive Slab with Time

# CONCLUSIONS

Correlating all of the data from this series of 3 tests with electrons, protons, and UV, we conclude:

It is possible to select a polymer thermal control coating from among those investigated that will not degrade catastrophically under simultaneous exposure. Some trade of  $\alpha_s$ ,  $\varepsilon$ , or  $\alpha_s/\varepsilon$  ratio may be necessary in making this selection.

On the other hand, many choices of film thickness or other parameters involved in coating formulation will surely result in substantial dielectric breakdown in the materials under orbital conditions similar to those simulated for this program.

A substantial reduction in degradation, in this high energy radiation test environment, was obtained by the use of a partially conducting adhesive.

### RECOMMENDATIONS

Therefore Boeing can unreservedly recommend that experimental and developmental work of the type conducted herein be continued, to examine the widest possible orbital environment conditions and to optimize the selection of spacecraft thermal control coatings for all conditions that NASA will have to meet.

In particular, experiments should be continued with UV and protons combined with lower energy electrons (on the order of 30 keV), to further survey the high-flux, low-energy end of the spectrum. This energy region has recently been shown to have great significance in spacecraft discharge phenomena (References 2 and 3).

In parallel with these experiments, Boeing recommends undertaking efforts to improve the polymers used in thermal control coatings. Recent amalgamation of efforts at Boeing has led to development of several promising approaches that should be tried.

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#### REFERENCES

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#### ACKNOWLEDGMENTS

We are indebted to Loren Milliman, Maurice Wilkinson, and E. R. Crutcher III of Boeing for their contributions to this program and report. Also appreciated are the beneficial discussions held with Wayne Slemp at NASA-Langley Research Center.

# APPENDIX A

# Test 1

Appendix A contains one computer plot of reflectance data for each sample tested <u>in situ</u> in the 588-hour Test 1, including 3 control samples. The data plots are presented here in the same order as samples listed in Table IV, the table that lists solar absorptance values and changes measured during Test 1. The solar absorptance values listed in Table IV and in the legends on the plots of Appendix A are derived from the computed reflectance curves and values shown in Appendix A.









































A-19



A-20



A-21







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APPENDIX B

Test 2

Appendix B contains one computer plot of reflectance data for each sample tested <u>in situ</u> during Test 2 (610 hours, 915 equivalent sun hours). The data plots are presented here in the same order as samples are listed in Table V.



B-2


B-3























B-12



B-13



B-14



B-15





















. 1

B-24







B-27



## APPENDIX C

## Test 3

Appendix C consists of one computer plot of reflectance data from each sample tested during Test 3 (636 hours in situ). The data plots in this Appendix are presented in the same order as samples are seen to be listed in Table VI.







C-4











C-8



C-9




C-11









C - 14



C-15















C-22



C-23





C-25









C-28

## APPENDIX D

Test 2

Appendix D consists of 10 computer-generated reflectance data plots. Each data plot contains 2 curves that also appear in Appendix B — curves 1 and 5. The third curve — curve 7 indicates the degree of reflectance stability <u>in situ</u> for the 10 samples measured 310 hours after a previous measurement (curve 5) that took place just following exposure for 610 test hours. The solar absorptance values indicated along with curve 7 correspond with those given in the right-hand column of Table V.







D-4













D-10



## APPENDIX E

## Test 3

Appendix E contains one plot of spectral reflectance as a function of wavelength for each sample tested during the third test for this program. Curves 1 and 5 on these plots repeat data presented earlier with the same numbers in Appendix C. Curve 6 appears only in this appendix, and indicates solar absorptance for a sample in air following the end of Test 3 at 850 hours. The  $\alpha_s$  data herein also appears in the right-hand column of Table VI.


















E-10







E-13



E-14





E-16





' E-18













g HEMISPHERICAL SPECTRAL ABSORPTANCE 0 ğ 8 8 g B 8 R 8 80. 100 8 0.383 · D 2.8 RND ELECTRONS ON THE REFLECTRNCE / TEFLON / SILVER / INCONEL ) H ъ S 2.0 2 6, {{ 2,4 2.2 0, כי 1.2 1.4 1.6 1.8 MAYELENGTH (MICROMETERS) EFFECTS OF UV. PROTONS. LAMINATE NO. 1 (KAPTON 1.0 0 ഹ ပ် á Ę FIGURE E-24 5 Ņ ğ 8 8 -90 R B 9 B 8 R 2 HEMISPHERICAL SPECTRAL REFLECTANCE



