D2-84118-7

UNCLASSIFIED TITLE: In Situ Electron Rate Effects Studies on Thermal Control Coatings

RESEARCH CONTRACT NO.: NAS 5-9650

7th Quarterly Progress Report to NASA Goddard Space Flight Center, Greenbelt, Maryland, for 'Space Environment Effects on Thermal Control Coatings'

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ABSTRACT

This document constitutes the seventh quarterly progress report for NASA Goddard Contract NAS 5-9650. Included is a description of the test progress on the research program, "Space Environment Effects on Thermal Control Coatings," which was accomplished during the period 15 March through 15 June 1967. During this quarter, low energy (50 kev) electron irradiation rate testing of thermal control coatings was performed in situ. Changes in spectral reflectance of coating specimens were observed in vacuum throughout the 0.25 to 2.5 micron wavelength region, after exposures to electron fluences of 1×10^{13}, 1.6×10^{13}, 5×10^{13}, 1×10^{14}, 2×10^{14}, 5.5×10^{14}, 8.5×10^{14}, and 8×10^{15} electrons cm^{-2}. Exposures were conducted using electron fluxes ranging from those typical of maximum space rates (4×10^{8} electrons cm^{-2} sec^{-1}) to accelerated fluxes as much as 4×10^{3} more intense. Recovery of spectral reflectance was also observed following re-exposure of test samples to dry air.

Significant conclusions reached during this quarter are:

1. In the types of coatings studied for this contract, degradation in spectral reflectance due to 50-kev electron exposure is independent of irradiation rate. Thus, irradiation of thermal control coatings with 50-kev electrons at accelerated laboratory rates appears to be valid, at least to an acceleration factor of 4×10^{3}.

2. Coatings employing methyl silicone binders sustain the greatest degree of reflectance degradation in the infrared. Coatings using potassium silicate binders suffer the largest reflectance losses in the visible region.

3. Threshold for reflectance degradation, and rate of buildup of degradation, are highly variable, even among coating formulations of a single class (e.g. TiO_2-methyl silicone). In ZnO-methyl silicone coatings, degradation proceeds to very high reflectance change values before saturation occurs.

4. Non-linear damage is present in one or more wavelength regions of several coating types examined. Degradation proceeds most nearly linearly with electron fluence in type F, zinc oxide/aluminum oxide-potassium silicate.

5. Based upon substantially different degradation phenomena (various relative amounts of reflectance change) in the several wavelength regions studied, the operation of more than one damage mechanism is indicated.

6. Substantial restoration of spectral reflectance toward pre-irradiation values occurs in most diffuse coatings upon re-exposure to air.

7. The results of these tests on thermal control coatings tie together (1) the results of early in-air tests (samples exposed at high fluences) which indicated strong effects only in the visible region, and (2) the more recent results of <u>in situ</u> testing that indicate that contamination-free electron tests show reflectance changes primarily in the infrared region.

The following recommendations are made:

1. That a study of the dependence of reflectance degradation in selected thermal control coating types, upon the incident energy of electrons between 20 and 80 kev, be performed as an add-on to this contract.

2. That in the interest of being able to characterize test results more fully, and to arrive at more meaningful conclusions, more information on the formulation and preparation of coating specimens be passed along to Boeing whenever it is shared with NASA-Goddard by suppliers.

KEY WORDS

Electron gun	Non-linear damage
Electron irradiation	Rate effects
Fluence	Spectral reflectance
Flux	Thermal control coatings
In situ	Vacuum

1.0 INTRODUCTION

1.1 Program Description

The overall objective of the program is the determination of the effects of electrons and ultraviolet radiation on thermal control coatings in the presence of environmental conditions similar to those encountered in space. The environment criterion selected is that for a near-earth orbiting spacecraft. Those features selected for simulation are ultrahigh vacuum, temperature, ultraviolet radiation, and magnetically trapped electrons. The specific objectives of the program are to:

1. Study the concomitant effects of radiation, vacuum, and temperature over a range of -70°C to +90°C.

2. Study the effects of simultaneous electron and ultraviolet irradiation on the thermophysical properties of thermal control coatings.

3. Establish the validity of accelerated-rate laboratory irradiation through a study of radiation damage as a function of exposure rate.

4. Investigate physical property changes as well as optical characteristic changes due to irradiation in a simulated space environment.

Approximately 1,000 test specimens have been supplied by the Thermal Systems Branch of NASA Goddard. These samples are being exposed to electrons and ultraviolet radiation, both separately and simultaneously. Spectral reflectance measurements of these samples are being made *in situ* over the 240 to 2500 millimicron wavelength region before, periodically during, and after irradiation, using the Combined Radiation Effects Test Chamber (CRETC). Selected test specimens are also measured after samples are re-exposed to dry air.

Specifically, the following tests have been completed:

1. A screening test involving exposure of 17 sample types at 22°C, to 50 kev electrons at fluences from 1.6×10^{13} to 8.5×10^{14} electrons cm^{-2}. (See D2-84118-6.)

2. An electron rate test, again using 50 kev electrons at fluxes from 4×10^{8} to 1.7×10^{12} electrons cm^{-2} sec^{-1}, to establish the equivalence between effects produced by accelerated laboratory exposures and the lower flux levels of space exposure, on 12 sample types at 22°C. The fluence range covered was 1×10^{13} to 8×10^{15} electrons cm^{-2}.

Future tests include the following:

1. An ultraviolet radiation effects study to provide information needed to determine if the effects of low energy electrons and ultraviolet radiation are additive for simultaneous or sequential irradiations. Sixteen types of samples will be exposed to more than 1000 equivalent UV-sun hours, while their substrates are maintained at 20°C, and measured at selected increments of exposure.

2. Electron-ultraviolet intensity ratio studies to determine the influence of this ratio on damage and damage rates, and to study other synergistic effects. Periodic reflectance measurements will be made in vacuum at 10°C during combined ultraviolet and 50-kev electron exposure tests.

1.2 Progress Summary

Selected test and control specimens of 12 sample types were placed in the Combined Radiation Effects Test Chamber (CRETC) and exposed to electrons following the obtaining of base-line, preirradiation spectral reflectance measurements. Exposures, designed to yield data on the presence or absence of effects due to various electron irradiation rates, were made to the following fluences: 1×10^{13}, 1.6×10^{13}, 5×10^{13}, 1×10^{14}, 2×10^{14}, 5.5×10^{14}, 8.5×10^{14}, and 8×10^{15} electrons cm^{-2}. Rates at which electrons were incident upon test samples ranged from 4×10^{8} electrons cm^{-2} sec^{-1}, which is typical of peak electron fluxes in space, to as high as 1.7×10^{12}, representing a laboratory test-acceleration factor of approximately 4×10^{3}. Spectral reflectance of test and control samples was obtained in situ following each exposure. No test sample was exposed at more than one rate.

Following each irradiation test, the CRETC was backfilled with dry air, and in-air reflectance curves were obtained on selected samples. Partial recovery was seen in all diffuse samples. A second pumpdown of the chamber afforded the opportunity to observe whether any types of coatings tested had a memory for previous in situ reflectance changes due to electron exposure. No evidence of memory for previous damage was seen.

2.0 DETAILED PROGRESS REPORT

2.1 Test Specimens

Table 1 lists and describes the types of coatings which were exposed to electrons during the seventh quarter. Earlier electron testing of more coating types was described in the sixth quarterly report, "In Situ Electron Damage to Thermal Control Coatings." Tested samples were stored in a clean environment prior to irradiation. They were handled on edge with clean gloves during mounting in the test array cover plates. Copper shims were placed behind the aluminum substrate of each sample to assure maximum thermal contact with the temperature-controlled copper sample wheel. Sample temperature was 22°C throughout the tests.

2.2 Electron Exposure

Electrons for the screening test were obtained from an electron gun mounted in the CRETC. The electron beam was scattered through a thin aluminum foil to obtain uniform exposure (axial symmetry) of samples mounted in the array being irradiated. The energy of the electrons, after passing through the scattering foil, was adjusted by the accelerating electrodes to be 50 kev. Direct beam current was measured in a fixed Faraday cup located behind the sample wheel. A hole in the center of the sample array serves as an aperture for that insulated Faraday cup. Thus the cup can also be used to monitor the forward scattered beam during exposure in order to provide a means of obtaining total exposure fluence. Two remotely controlled Faraday cups (90° with respect to each other) were rotated about the scattering foil in order to map the Gaussian electron scattering.

Table 2 shows the irradiation test points employed in these electron exposures, and the respective irradiation rates for each exposure which were

Table 1

Sample Types Tested

Type	Description
B	Zinc oxide-methyl silicone (approximately 9 mils of type S-13 on top of a thin coat of General Electric S54044 primer).
F_3	Zinc oxide/aluminum oxide-potassium silicate (approximately 5 mils of paint pigment SP500 from New Jersey Zinc).
H	Silicon dioxide on polished aluminum (vacuum-deposited SiO_x coating).
I	Leafing aluminum-silicone (approximately 3 mils of coating, fine leafing aluminum in a phenolate silicone, mixture of Dow Corning 805 and 806A).
J	Vapor-deposited aluminum on a lacquered substrate (transparent lacquer from Bee Chemical, thermosetting type).
K	Polished aluminum (buffed and chemically cleaned by vapor degreasing).
L_1	Titanium dioxide-methyl silicone (approximately 5 mils of paint on top of approximately 2 mils of Cat-a-Lac white primer, Dow Corning methyl Q9-0090, mixed three parts paint to one part catalyst).
L_2	A methyl silicone type L with a black water emulsified strippable coating.
M	Zinc oxide-methyl silicone (same as type B except S-13G of a thicker coating of approximately 10 to 12 mils).
N	Kapton H-film (2 mils thick).
O	Titanium dioxide-methyl silicone (vehicle is General Electric RTV 602, 2 parts pigment to 1 part vehicle).
P	Titanium dioxide-methyl silicone (vehicle is Dow Corning XR 6-3488, 1.5 parts pigment to 1 part vehicle).
Q	Mixed zinc oxide and titanium dioxide pigments in a mixed silicone-silicate vehicle.
Y	Titanium dioxide-methyl phenyl silicone coating. (Pyromark)

Table 2. Electron Exposure Rates

Average Rates of and Exposures to 50 kev Electrons	
Fluence (electrons cm^{-2})	Flux (electrons cm^{-2} sec^{-1})
1×10^{13}	4×10^{8}, 1×10^{10}
1.6×10^{13}	2×10^{10}
5×10^{13}	2×10^{9}, 2×10^{10}
1×10^{14}	2×10^{9}, 2×10^{10}
2×10^{14}	2×10^{10}, 1×10^{11}
5.5×10^{14}	2×10^{10}
8.5×10^{14}	5×10^{11}
8×10^{15}	1.7×10^{12}

used to establish the findings concerning electron rate effects. The findings are summarized by the statement that, for 50-kev electron fluxes ranging from those typical of peak space fluxes (4×10^{8} electrons cm^{-2} sec^{-1}) to accelerated testing rates up to 1.7×10^{12} electrons cm^{-2} sec^{-1}, no significant rate effects exist in any of the 12 types of coatings tested.

2.3 Electron Rate Test Results

The conclusion regarding absence of rate effects was obtained after detailed analysis of sample spectral reflectance data at four significant wavelengths, 590, 900, 950, and 2100 nm. The wavelengths were chosen from continuous wavelength scan charts on the basis of significant reflectance changes and apparent operation of damage mechanisms in surrounding wavelength regions. Probable error in the reflectance percentages expressed is plus or minus two percent. In the figures following, R_i is the pre-irradiation reflectance value, and R_f is the "final" in situ value at any given fluence. Each value has been corrected very nearly to absolute reflectance, by normalizing the integrating sphere MgO wall reference curve to 100 percent, and the sample reflectance data point proportionally.

Figure 1 shows the building up of reflectance changes at 2100 nm with increasing electron fluence in two zinc oxide-methyl silicone types of samples, S-13 (B) and S-13G (M). Data for each irradiation rate of flux bears a different symbol, allowing the determination to be made that exposure at each rate used causes equivalent damage via reflectance change.

Figure 2 shows similar data at 2100 nm for two types of titanium dioxide-methyl silicone coatings (L and P). Reflectance changes in L, an anatase TiO_2, are larger than those in P, a rutile TiO_2, at all but the highest fluence. Again, reflectance changes are seen to be independent of irradiation rate.

Figure 3 shows data on two zinc oxide-pigmented sample types, M and F, at 950 nm. The differences in damage buildup may be seen for zinc oxide-potassium silicate (F) as contrasted with zinc oxide-methyl silicone (M, S-13G). The data point spread indicates absence of rate effects.

Figure 4 contains near-infrared wavelength data for two titanium dioxide-methyl silicone coatings, L and P. The data for anatase TiO_2 (L), at 900 nm, have the greatest spread seen in all groupings of reflectance values analyzed for rate effects phenomena. Because the spreads are within experimental error, however, there is again no evidence of irradiation rate effects.

Figure 5 shows reflectance data at 590 nm for S-13G (M), zinc oxide-methyl silicone, and for type F, zinc oxide-potassium silicate. A comparison of the relative spreads of data from F and M is believed to point out greater sample-to-sample differences in M than in F. Figure 6 contains 590 nm reflectance data for sample type L, TiO_2-methyl silicone.

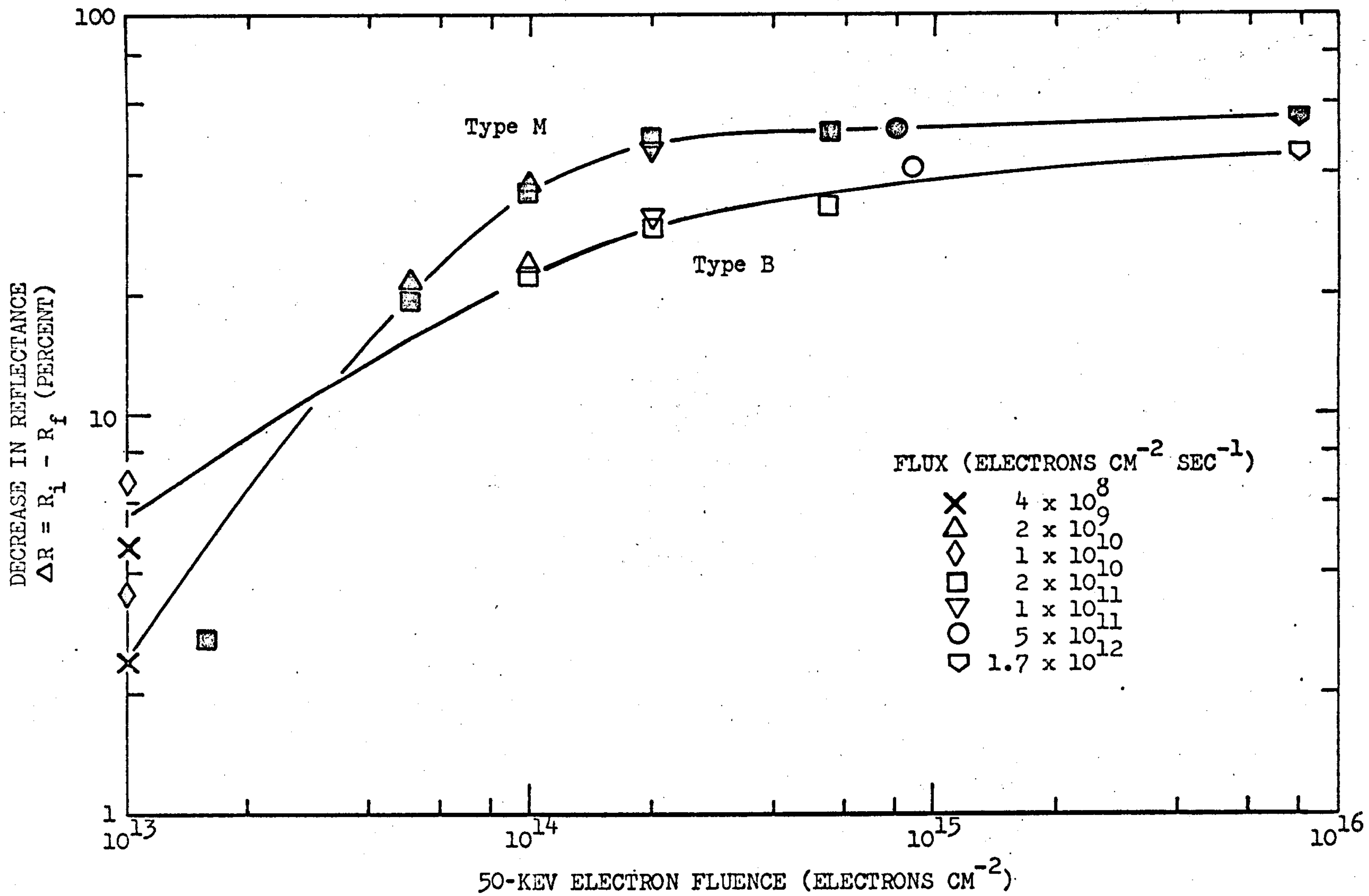


Figure 1. Electron Rate Study in ZnO-Methyl Silicone Coatings at 2100 nm.

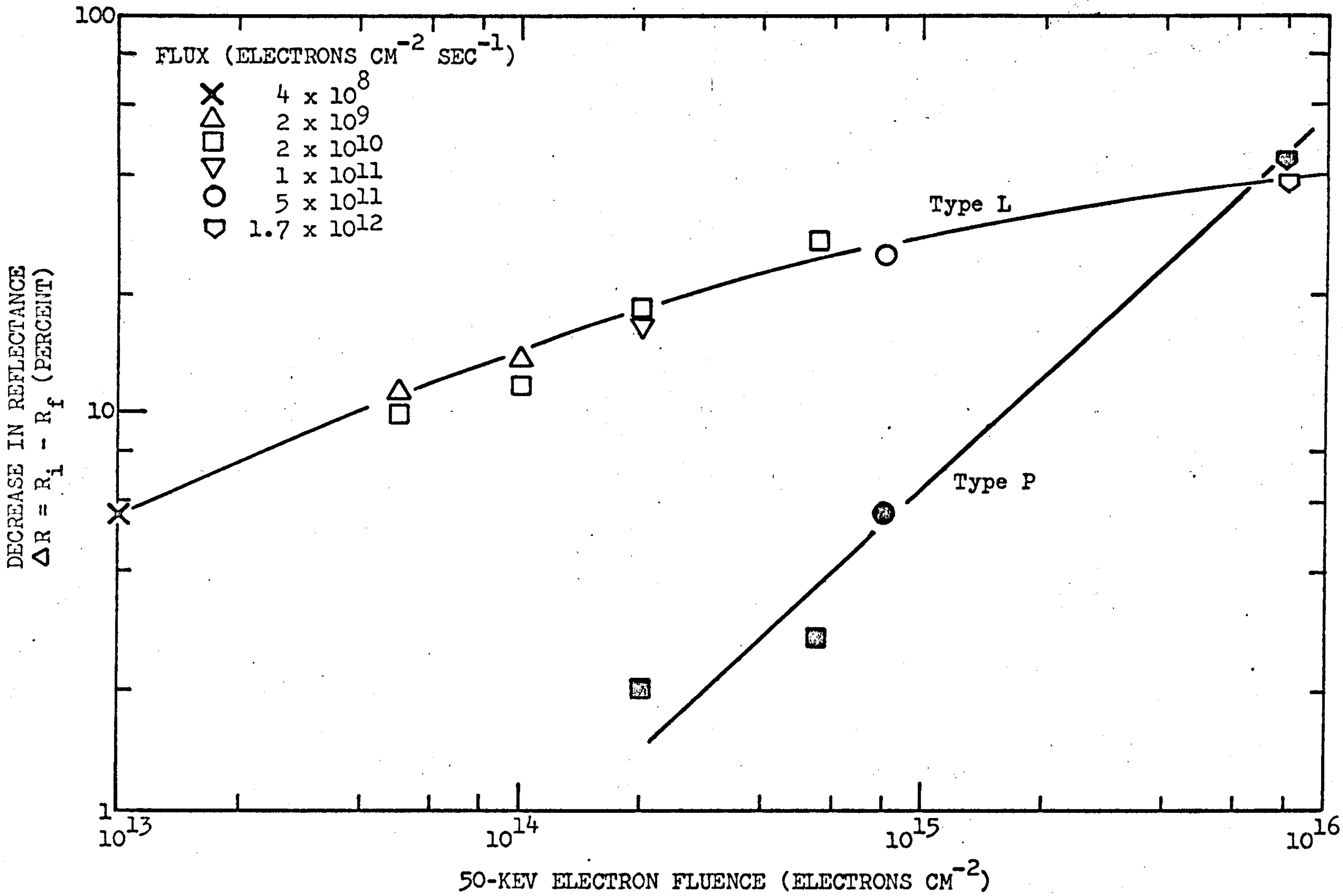


Figure 2. Electron Rate Study in TiO_2-Methyl Silicone Coatings at 2100 nm.

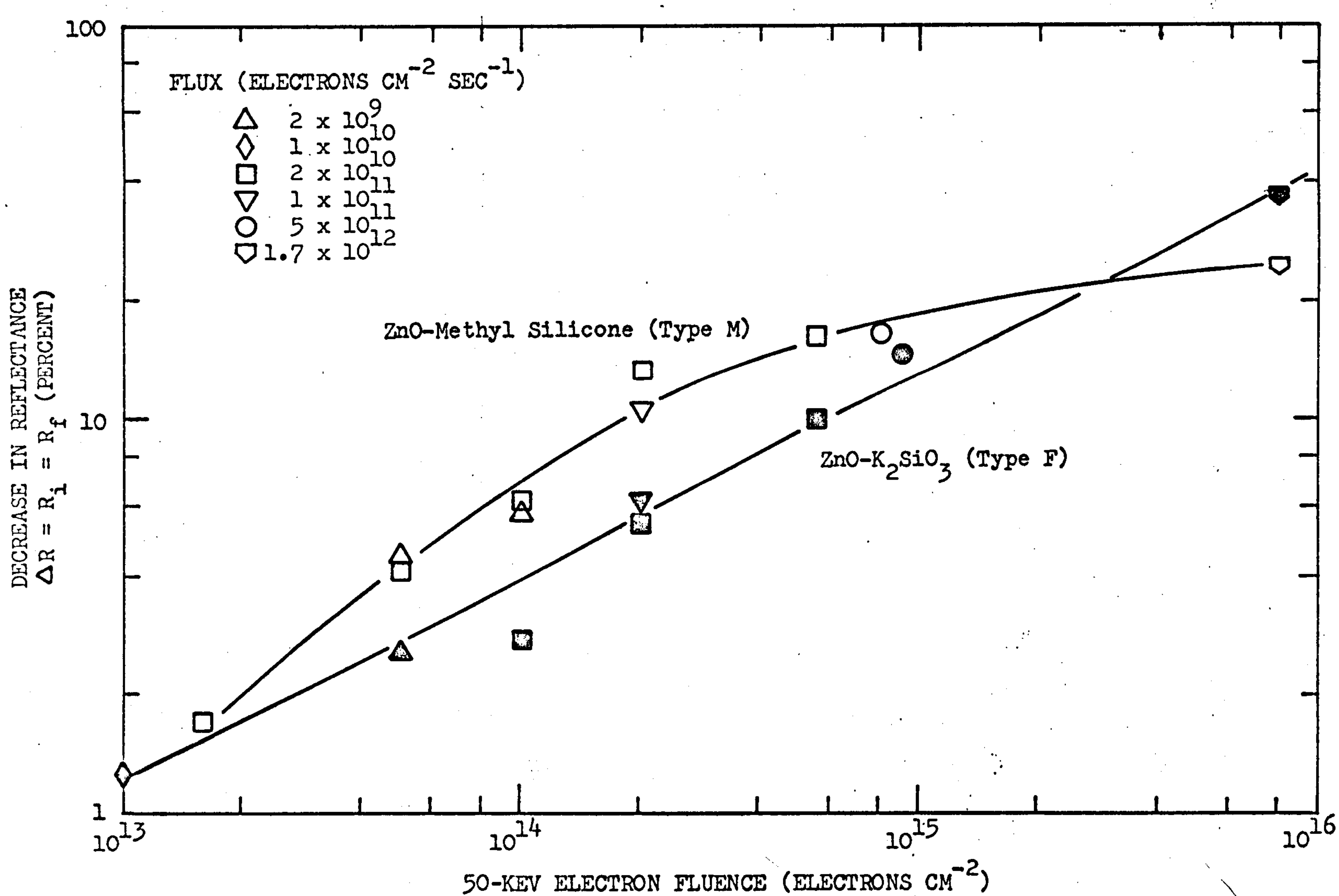


Figure 3. Electron Rate Study in ZnO-Pigmented Coatings at 950 nm.

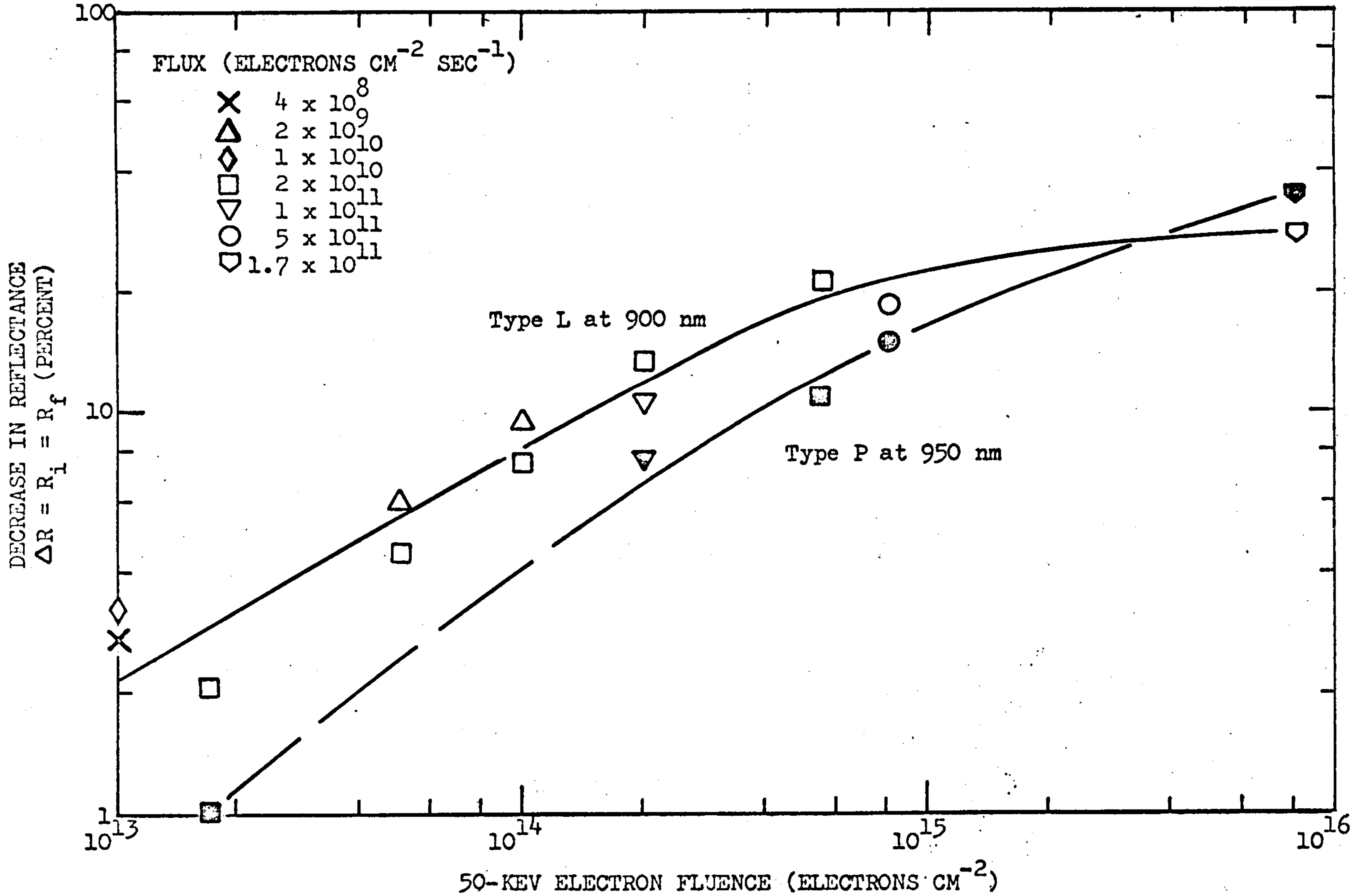


Figure 4. Electron Rate Study in TiO_2-Methyl Silicone Coatings

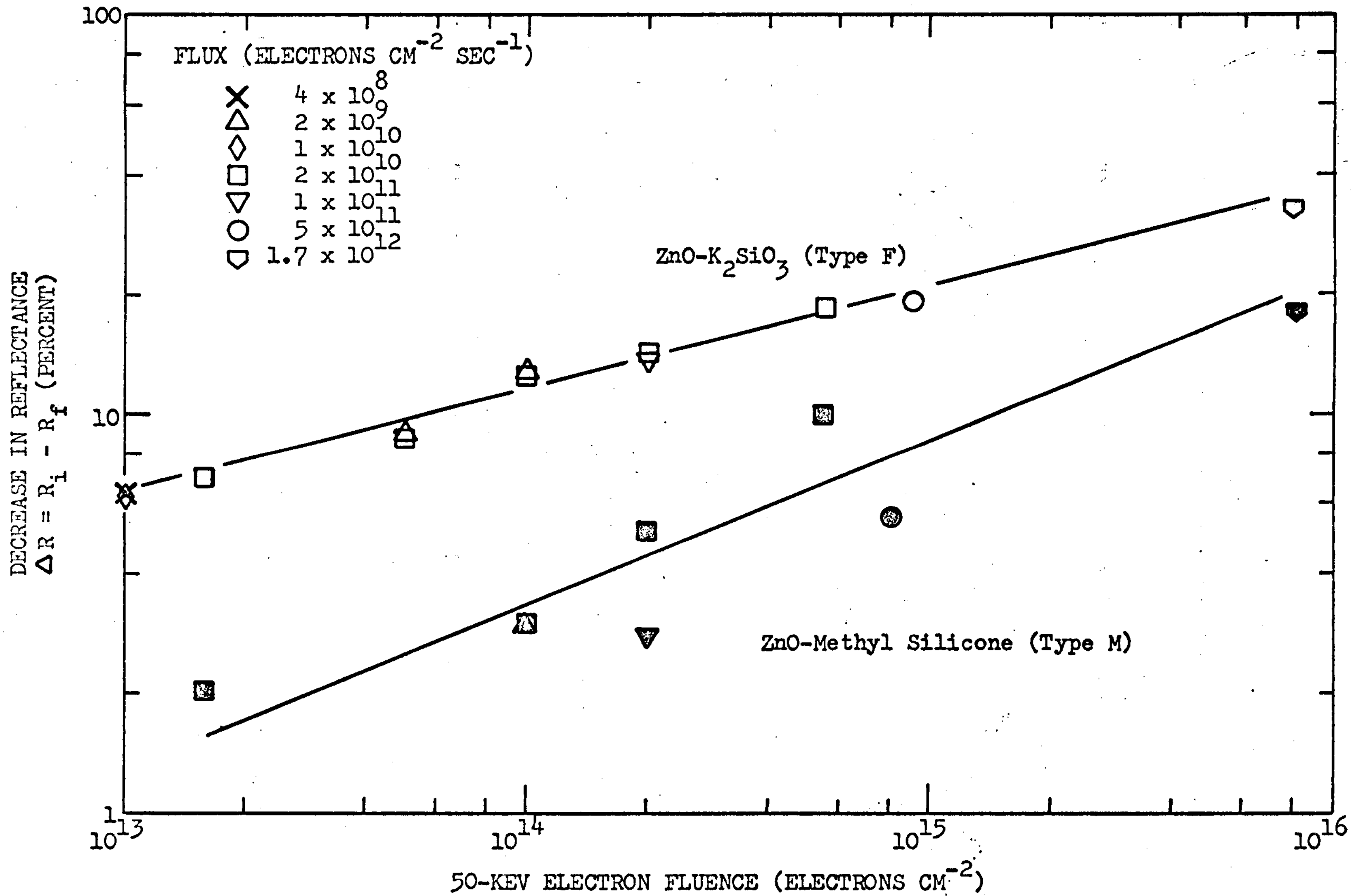


Figure 5. Electron Rate Study in ZnO-Pigmented Coatings at 590 nm.

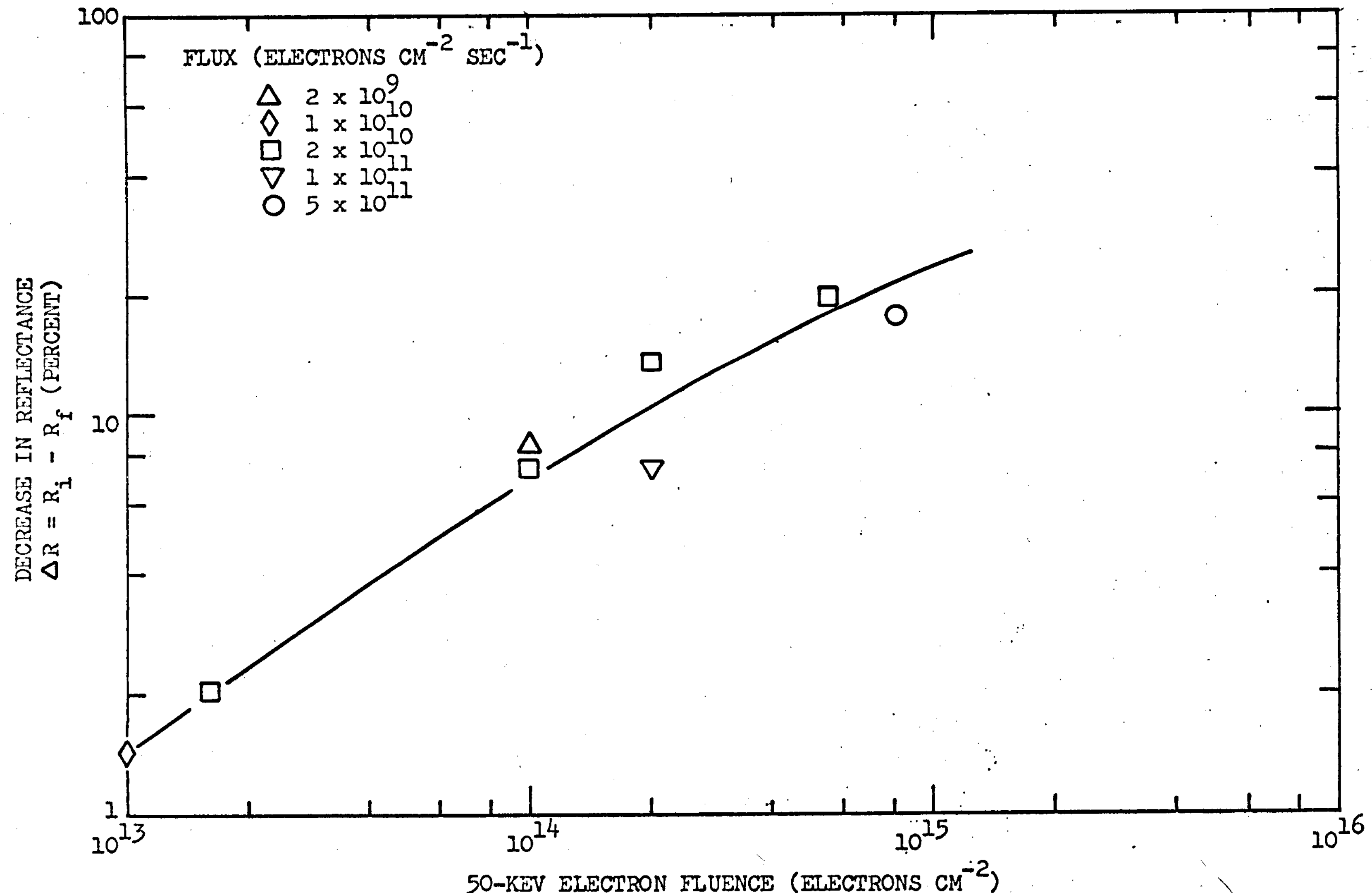


Figure 6. Electron Rate Study in Type L TiO_2-Methyl Silicone at 590 nm.

In Figure 7, total hemispherical reflectance data at 2100 nm shows absence of irradiation rate effects in 2 specular sample types, H (SiO_2 vapor-deposited onto aluminum) and K (buffed aluminum).

Figures 1 through 6 also provide additional means of characterizing coating degradation. Table 3 summarizes two aspects of degradation shown in the six figures: extent of damage buildup before saturation, and presence of non-linear damage, (i.e., reflectance changes which do not depend linearly on electron fluence). Saturation of degradation is seen to take place after various amounts of reflectance change have occurred, and occasionally (such as type M at 2100 nm) only as reflectance approaches zero. Reflectance changes in sample type F are seen to be more linearly dependent on fluence than other coating types studied.

Tables 4, 5, and 6 indicate at the selected wavelengths of 2100, 950, and 590 nm (chosen from 230 to 2500 nm continuous-scan charts), the relative sensitivity to 50-kev electrons of the types of thermal control coatings studied. Buildup of degradation in these coatings can be summarized as follows:

(1) At 2,100 nm (Table 4) zinc oxide-methyl silicone coatings (Figure 1) showed the greatest sensitivity. Of the titanium dioxide-methyl silicones, sample type L (Figure 2) was the most sensitive while types O and P exhibited much higher thresholds for damage. At 2,100 nm type F was the least sensitive of the paints. Kapton H-film showed no evidence for degradation at that wavelength.

(2) The resistance to reflectance change was, in general, slightly greater at 950 nm for most coatings (Table 5, Figures 3 and 4). The most notable exception was type F, which appeared to be significantly more sensitive at 950 nm.

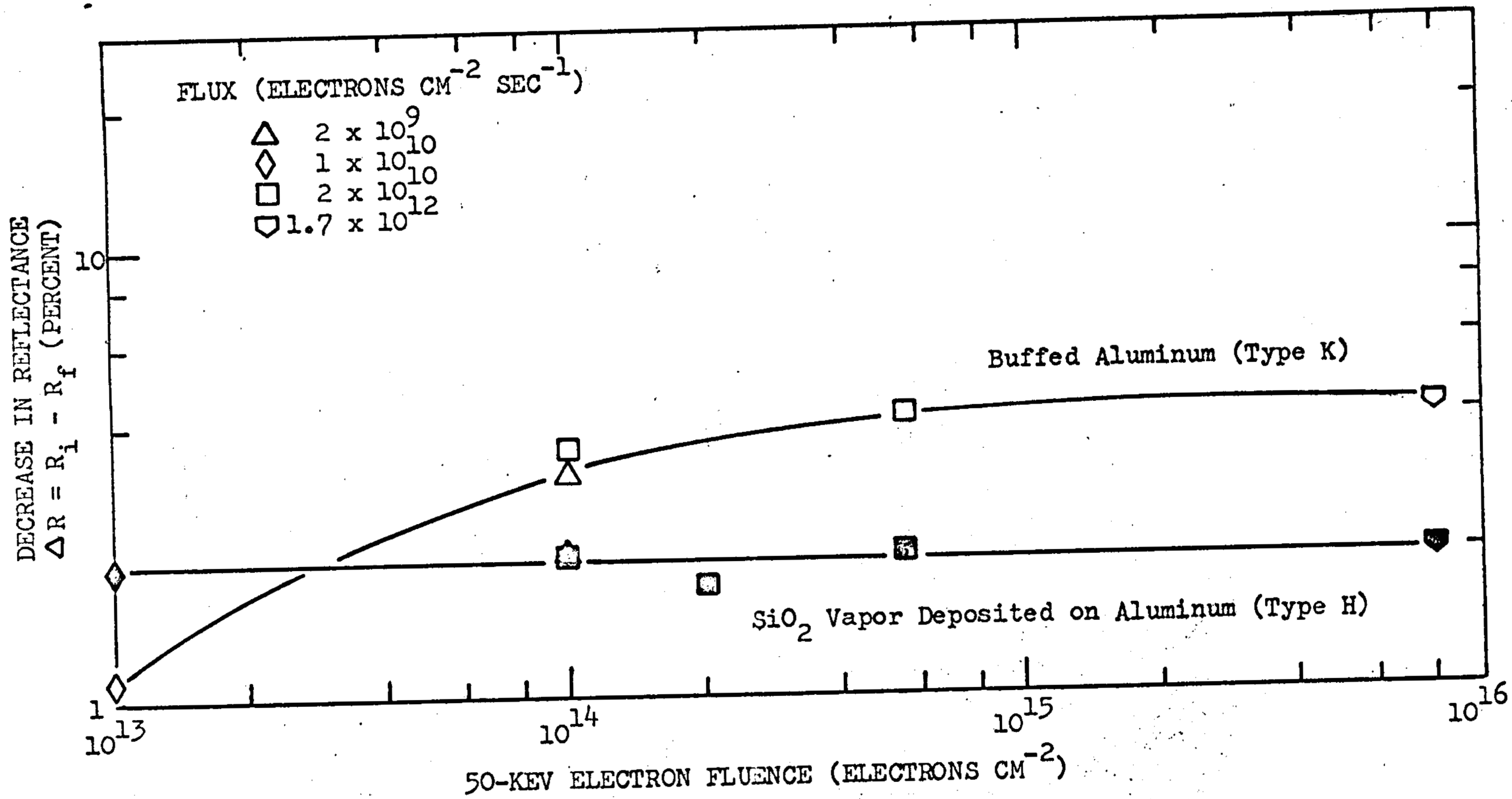


Figure 7. Electron Rate Study in Specular Coatings at 2100 nm.

Table 3. Characterization of Degradation in Selected Coatings

Wavelength Region	Coating Type Encircled number refers to Figure with same number				
	L Anatase TiO_2-Methyl Silicone	P Rutile TiO_2-Methyl Silicone	B ZnO-Methyl Silicone	M ZnO-Methyl Silicone	F_3 ZnO-Potassium Silicate
Middle Infrared	(2) Begins to saturate at $\Delta R \approx$ 40%.	Linear damage (within limits of data.) (2)	Non-linear damage. (1) Begins to saturate at $\Delta R \approx$ 40%.	Non-linear damage. (1) Saturates at limit of reflectance.	(Mostly small reflectance changes.)
Near Infrared	Non-linear damage. (4) Begins to saturate above ΔR $\approx$20%	Non-linear damage. (4) Begins to saturate above ΔR $\approx$20%.	(Small reflectance changes.)	Non-linear damage. (3) Begins to saturate at $\Delta R \approx$ 20%.	Linear damage to highest fluence tested. (3)
Visible	Non-linear damage. (6) Begins to saturate above ΔR $\approx$10%.	(Mostly small reflectance changes.)	(Very little reflectance change.)	Within data limits, linear damage to highest fluence tested. (5)	Linear damage to highest fluence tested. (5)

Table 4. Decrease in Reflectance at 2100 nm.

Sample Type	$\Delta R = R_i - R_f$, (percent) for 50 kev electron exposures (electrons cm^{-2}) of:					
	1×10^{13}	5×10^{13}	1×10^{14}	2×10^{14}	5.5×10^{14}	8×10^{15}
B	6	--	23	30	33	45
F_3	--	--	0	0	7	29
H	2	--	2	2	2	2
I	--	--	--	--	3	8
J	--	--	--	--	3	7
K	1	--	3	3	4	4
L	6	11	13	17	28	39
M	3	20	37	48	51	55
N	--	--	--	0	0	--
O	--	--	--	1	2	37
P	0	--	--	2	3	43
Q	--	--	--	22	31	--
Y	--	--	--	2	12	--

Table 5. Decrease in Reflectance at 950 nm.

Sample Type*	$\Delta R = R_i - R_f$, (percent) for 50 kev electron exposures (electrons cm^{-2}) of:					
	1×10^{13}	5×10^{13}	1×10^{14}	2×10^{14}	5.5×10^{14}	8×10^{15}
B	--	--	0	0	2	9
F_3	1	2	2	6	10	37
L	3	5	8	13	21	28
M	--	4	6	13	16	24
N	0	0	0	0	6	17
O	--	--	--	4	9	46
P	--	--	--	7	11	35
Q	--	--	--	22	33	--
Y	--	--	--	6	18	--

*5.5×10^{14}: I - 3%; H, J, K No change

(3) In the visible region (590 nm) type F was the most sensitive and types B and O the least sensitive paints. Kapton H-film showed very large degradation, but only at very high fluence. See Table 6.

A multiplicity of 3 samples per sample type was used at most data points. Very close agreement was found between samples of the same type. No systematic differences were found between samples of type L which had or had not had a strippable coating.

A detailed illustration of the buildup of degradation in sample type M with electron exposure is shown in Figure 8. There occurred in the infrared wavelength region an initially rapid decrease in reflectance, which eventually saturated at high ΔR values. In the visible region, however, the buildup of damage was slow at first and more rapid at high exposure. Upon re-exposure to (dry) air, this sample exhibited contrasting behavior.

2.4 Post-Exposure Recovery of Reflectance in Air

Dry air was admitted to the CRETC to an absolute level of one atmosphere, after completion of all *in situ* reflectance measurements. Reflectance measurements were made on selected samples after in-air stabilization. Results are given in Table 7. In addition, a set of reflectance measurements was made on type M, zinc oxide-methyl silicone, while and after the backfilling of the chamber was done. This set of data is reduced to reflectance-change form in Figure 9. Re-exposure to air causes a rapid rate of reflectance recovery in the infrared region; this rate decreases with time, as Figure 10 shows. In the visible region, however, reflectance is restored much more slowly, with

Table 6. Decrease in Reflectance at 590 nm.

Sample Type*	$\Delta R = R_i - R_f$, (percent) for 50 kev electron exposures (electrons cm^{-2}) of:					
	1×10^{13}	5×10^{13}	1×10^{14}	2×10^{14}	5.5×10^{14}	8×10^{15}
B	--	--	--	0	0	--
F	6	9	13	14	19	32
L	1	--	8	14	20	--
M	0	--	3	5	10	18
N	0	--	--	4	13	60
O	--	--	--	2	3	--
P	--	--	--	3	14	20

*H, I, J, K: Very small changes even after exposures of 8×10^{15} electrons cm^{-2}.

Table 7. "Permanent" Reflectance Losses

Sample Types*	Approximate Loss of Reflectance (percent) for Exposure of:					
	5.5×10^{14} Electrons cm^{-2}			8×10^{15} Electrons cm^{-2}		
	U.V. Edge	Visible	Infrared	U.V. Edge	Visible	Infrared
B	0	0	0	4	0	0
F	0	1	0	3	1	1
M	0	0	0	4	0	0
O	6	0	0	6	4	0
P	4	3	0	6	4	0
L	2	6	13	7	9	20

*Types H, I, J, and K exhibited no permanent reflectance losses.

IN-SITU REFLECTANCE LOSS

FIGURE 8

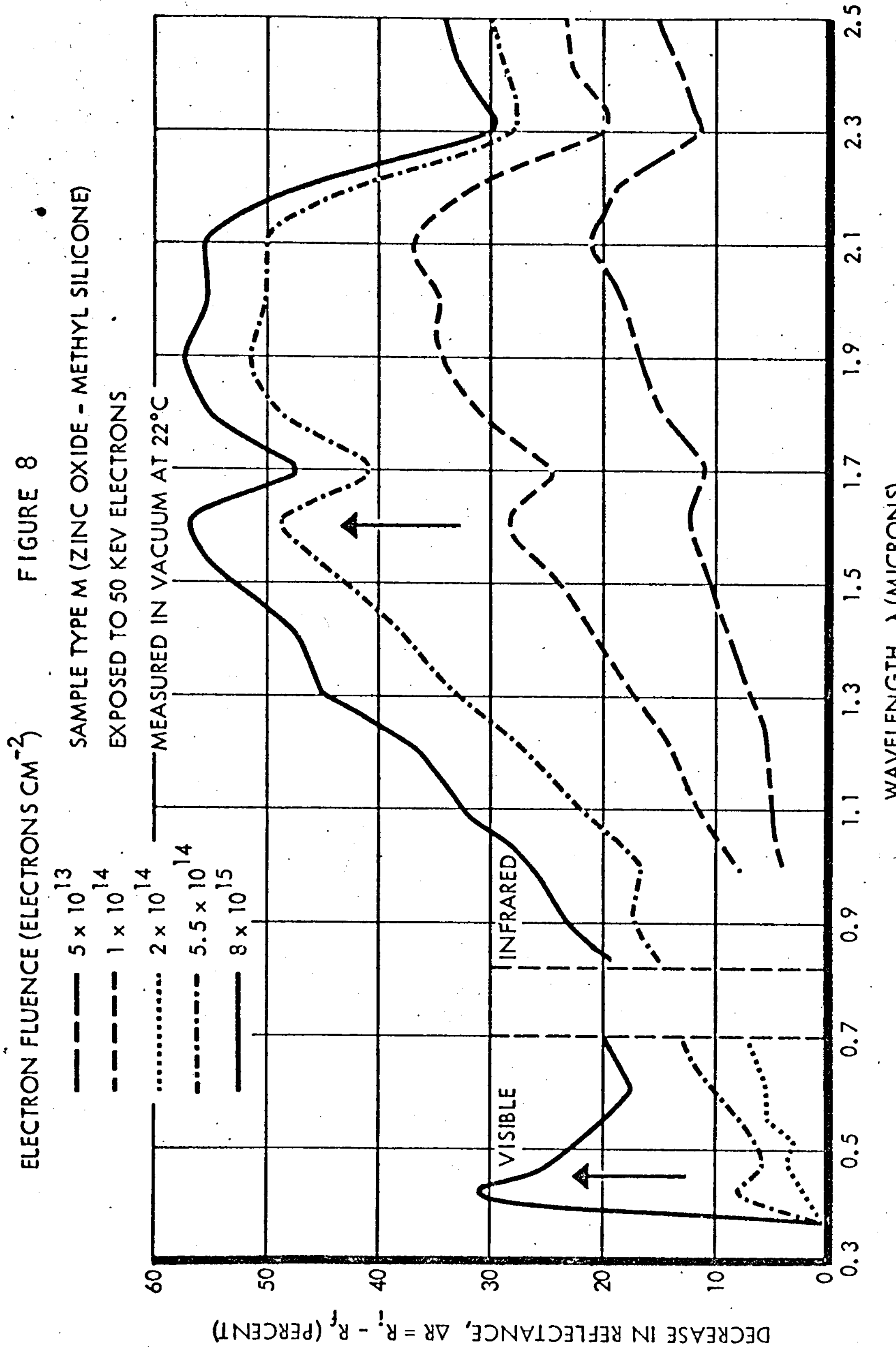


FIGURE 9

POST IRRADIATION RECOVERY

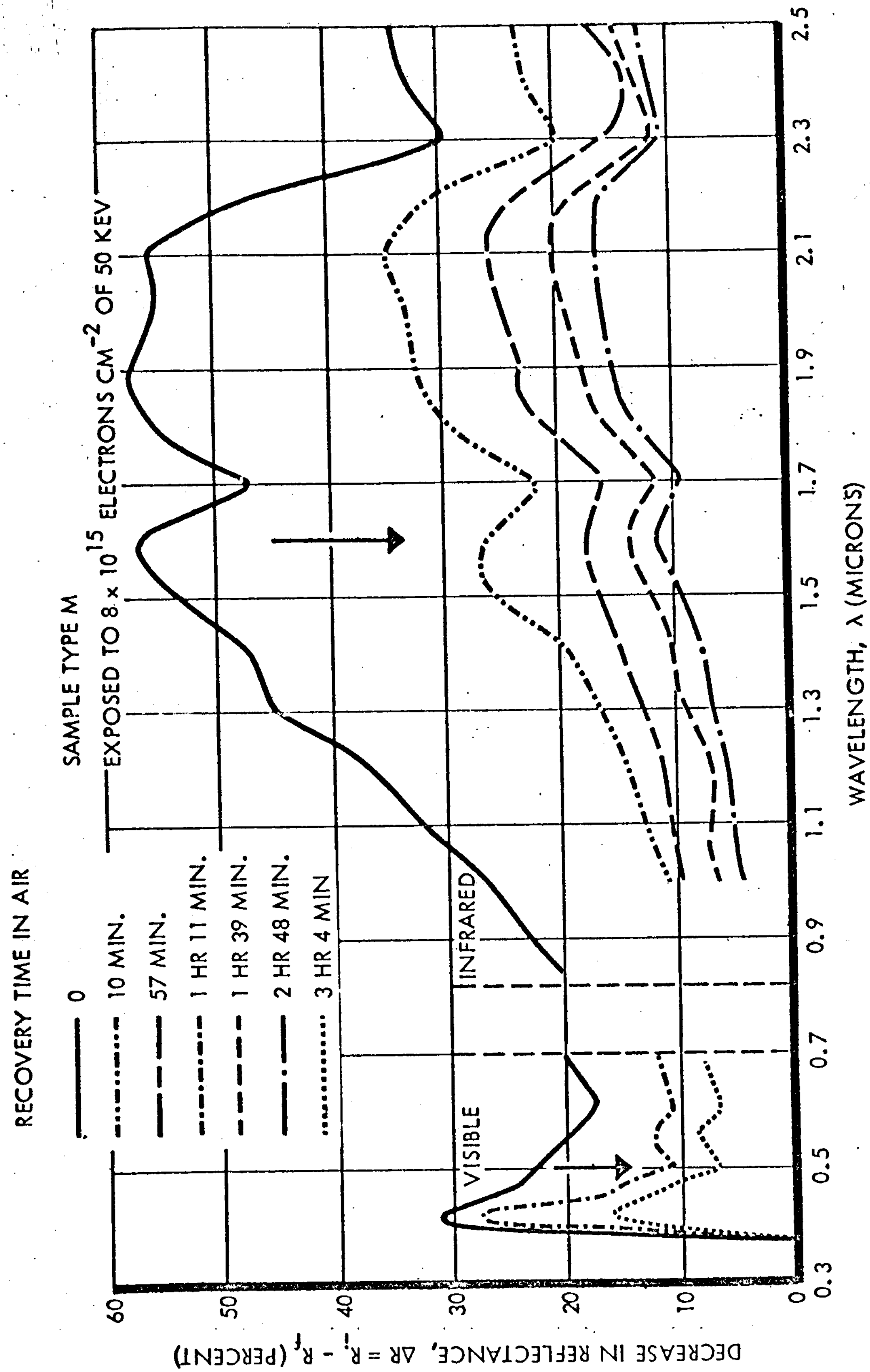
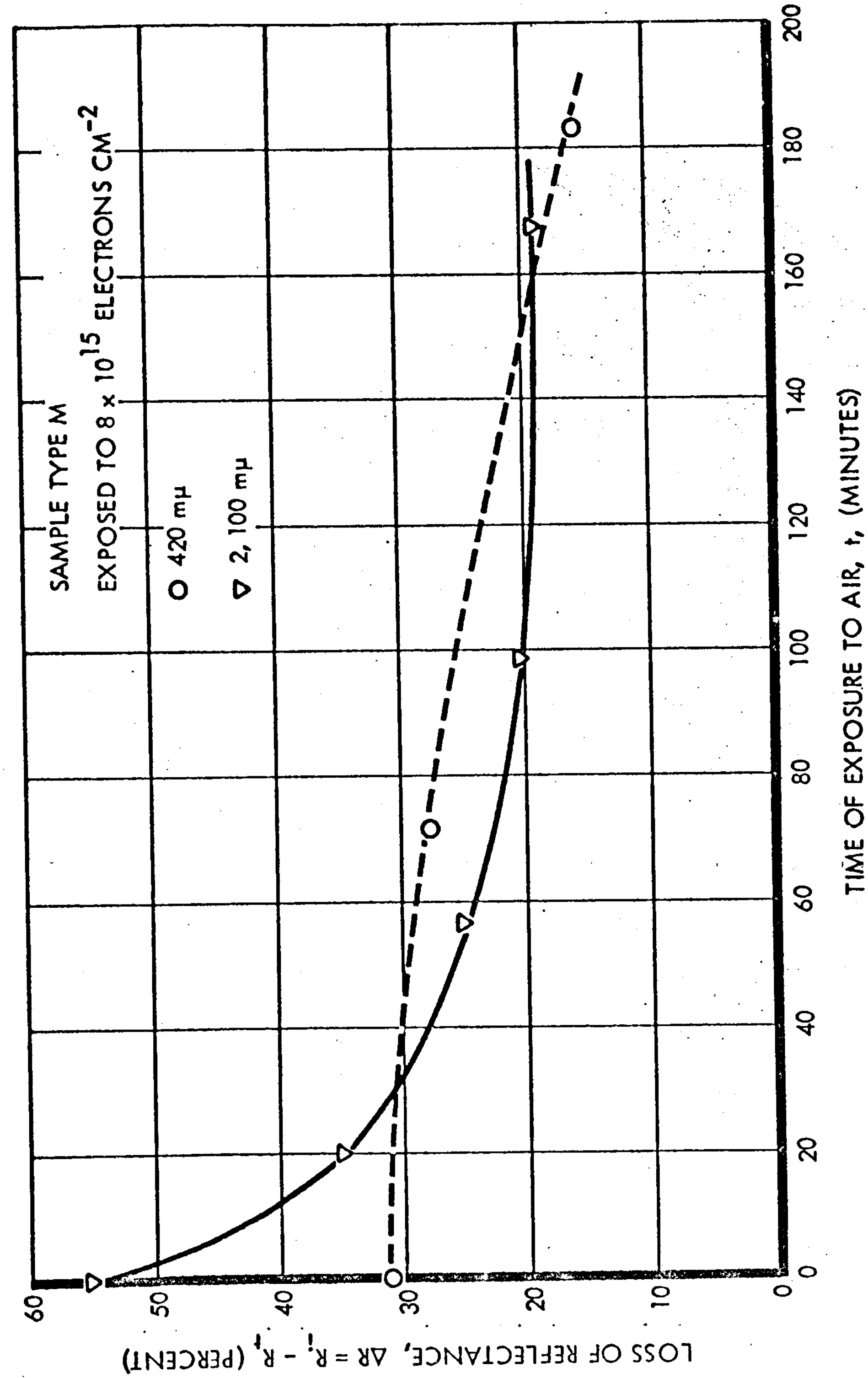


FIGURE 10

RATE OF RECOVERY IN AIR



considerable "permanent" damage remaining in the diffuse coatings even days after backfilling the chamber with air.

This data is seen to be consistent with results from early in-air tests, in which coatings exposed to high fluences showed signs of permanent effects mainly in the visible wavelength region.

After completion of in-air reflectance measurements, the CRETC was re-evacuated in an attempt to confirm results reported elsewhere, to the effect that certain specimens have a memory for damage sustained during a previous pumpdown.[1] In our tests results were negative on all coatings measured, as no memory for previous damage was exhibited for the two cases of (1) pumpdown only, and (2) pumpdown followed by a small electron exposure. (The exposure was made to see if it would "trigger" a return to the degraded state.)

More recent discussions with G. A. Zerlaut of IIT Research Institute have indicated that, when placed in vacuum a second time, unbound ZnO pigment has a memory for previous damage.

[1] J. E. Gilligan, oral remark at the 2nd AIAA Thermophysics Specialist Conference, New Orleans, Louisiana, April 19, 1967.

3.0 NEW TECHNOLOGY

Research work performed on this contract has been reviewed. To the best of our knowledge, there is no new technology to report to date.

4.0 PROGRAM FOR THE NEXT REPORTING INTERVAL

The next reporting interval will be for the period of 15 June through 15 September. During that quarter the exposure of samples to ultraviolet-rich electromagnetic radiation will be conducted.

5.0 SUMMARY OF RESULTS, CONCLUSIONS, AND RECOMMENDATIONS

5.1 Summary of Results

1. In the types of coatings studied for this contract, degradation in spectral reflectance due to 50-kev electron exposure appears to be independent of irradiation rate, over a range of rates from those typical of peak space rates (4×10^8 electrons cm^{-2} sec^{-1}) to laboratory rates as much as 4×10^3 higher. The fluence range over which this result was obtained spans the various damage thresholds of the coatings tested -- 1×10^{13} to 8×10^{15} electrons cm^{-2}.

2. Thresholds for reflectance degradation, and rate of buildup of degradation vary considerably, even among coating formulations of a single class (e.g. TiO_2-methyl silicone). In ZnO-methyl silicone coatings, degradation proceeds to very high reflectance change values before saturation occurs.

3. Coatings employing methyl silicone binders sustain the greatest degree of reflectance degradation in the infrared wavelength region. Coatings using potassium silicate binders suffer the largest electron-induced reflectance losses in the visible region.

4. Substantial restoration of spectral reflectance toward pre-irradiation values occurs in most diffuse coatings upon re-exposure to air.

5.2 Conclusions

1. The results of these tests on thermal control coatings tie together (1) the results of early in-air tests (samples exposed at high fluences), in which specimens evidenced lasting effects only in the visible region, and (2) the more recent results of <u>in situ</u> testing that

indicate that contamination-free exposures to electrons show reflectance changes in the infrared region as well.

2. Irradiation of thermal control coatings with 50-kev electrons at accelerated laboratory rates appears to be valid, at least to an acceleration factor of 4×10^3.

3. Non-linear damage is present in one or more wavelength regions of several coating types examined. Degradation proceeds most nearly linearly with electron fluence in type F, zinc oxide/aluminum oxide-potassium silicate.

4. Based upon substantially different degradation phenomena (various relative amounts of reflectance change) in the several wavelength regions studied, the operation of more than one damage mechanism is indicated.

5.3 Recommendations

The following recommendations are made:

1. That a study of the dependence of reflectance degradation in selected thermal control coating types, upon the incident energy of electrons between 20 and 80 kev, be performed as an add-on to this contract.

2. That in the interest of being able to characterize test results more fully, and to arrive at more meaningful conclusions, more information on the formulation and preparation of coating specimens be passed along to Boeing whenever it is shared with NASA-Goddard by suppliers.