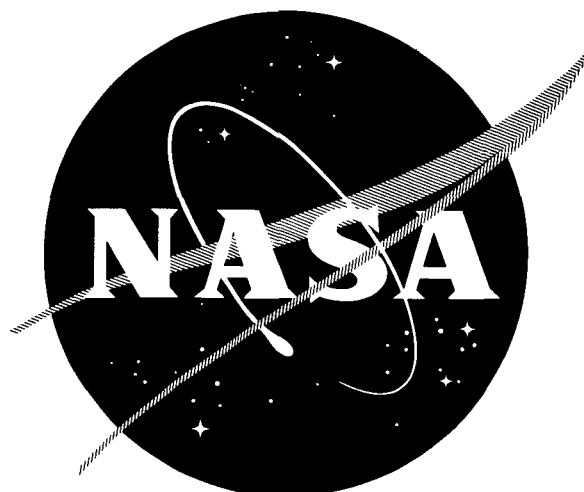


NASA-CR-72161



**EMISSIVITY COATINGS
FOR LOW-TEMPERATURE
SPACE RADIATORS**

**Quarterly Progress Report No. 6
For Quarter Ending 31 December 1966
CONTRACT NAS-3-7630**

**Prepared for
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
LEWIS RESEARCH CENTER
CLEVELAND, OHIO**

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FOREWORD

The results of the research activities carried out during the sixth quarterly period (10 October through 31 December 1966) of Contract NAS 3-7630, entitled Emissivity Coatings for Low-Temperature Space Radiators, are reported in this document. This work is being done by Lockheed Missiles & Space Company for the Lewis Research Center of the National Aeronautics and Space Administration.

PREVIOUS QUARTERLY REPORTS

1. F. J. Smith et al., Emissivity Coatings for Low-Temperature Space Radiators, QPR-1, NASA-CR-54807, Lockheed Missiles & Space Company, Sunnyvale, Calif., Sep 1965. Contents: Literature survey, coating selection, description of experimental apparatus
2. F. J. Smith et al., Emissivity Coatings for Low-Temperature Space Radiators, QPR-2, NASA-CR-54807, Lockheed Missiles & Space Company, Sunnyvale, Calif., Dec 1965. Contents: Apparatus description, uv source description, initial properties of coatings
3. F. J. Smith et al., Emissivity Coatings for Low-Temperature Space Radiators, QPR-3, NASA-CR-54807, Lockheed Missiles & Space Company, Sunnyvale, Calif., Mar 1966. Contents: Experimental procedure, spectral band absorptance measurements, initial properties of coatings
4. F. J. Smith et al., Emissivity Coatings for Low-Temperature Space Radiators, QPR-4, NASA-CR-72089, Lockheed Missiles & Space Company, Sunnyvale, Calif., Jun 1966. Contents: Initial properties of coatings, thermal cycling results, experimental procedures, exposure test results on titanium oxide silicone (thermatrol)

5. J. R. Grammer, Emissivity Coatings for Low-Temperature Space Radiators, QPR-5, NASA-CR-72130, Lockheed Missiles & Space Company, Sunnyvale, Calif., Sep 1966. Contents: Exposure test results on zinc oxide/silicone (S-13), zinc oxide/silicone (S-13G), zinc oxide/potassium silicate (Z-93) and zirconium silicate/potassium silicate

CONTENTS

Section		Page
	FOREWORD	ii
	ILLUSTRATIONS	v
	TABLES	vi
1	INTRODUCTION AND SUMMARY	1
2	EXPERIMENTAL RESULTS	3
	2.1 Apparatus	3
	2.2 Sample Preparation and Description	3
	2.3 Test Procedure	3
	2.4 Test Results	4
3	DISCUSSION OF SCREENING TESTS	21
4	REFERENCES	25

ILLUSTRATIONS

Figure		Page
2-1	Solar Absorptance of Aluminum Silicate/Potassium Silicate Coating as a Function of Exposure Time	7
2-2	Pre- and Post-Test Room Temperature Spectral Reflectance of Aluminum Silicate/Potassium Silicate Coating, Sample No. 19	8
2-3	Pre- and Post-Test Room Temperature Spectral Reflectance of Aluminum Silicate/Potassium Silicate Coating, Sample No. 21	9
2-4	Total Hemispherical Emittance of Aluminum Silicate/Potassium Silicate Samples as a Function of Exposure Time at a Temperature of 535° K (500° F)	11
2-5	Solar Absorptance of Z-93 (Zinc Oxide/Potassium Silicate) Coating as a Function of Exposure Time at 535° F (500° F)	13
2-6	Pre- and Post-Test Room Temperature Spectral Reflectance of Z-93 (Zinc Oxide/Potassium Silicate), Sample No. 36	14
2-7	Total Hemispherical Emittance of Z-93 (Zinc Oxide/Potassium Silicate) as a Function of Exposure Time at a Temperature of 535° K (500° F), Sample No. 36	15
2-8	Solar Absorptance of Z-93 (Zinc Oxide/Potassium Silicate) as a Function of Exposure Time at a Temperature of 452° K (500° F), Sample No. 39	18
2-9	Room Temperature Spectral Reflectance of Z-93 (Zinc Oxide/Potassium Silicate), Sample No. 39	19
2-10	Total Hemispherical Emittance of Z-93 (Zinc Oxide/Potassium Silicate) as a Function of Exposure Time at a Temperature of 452° K (300° F), Sample No. 39	20
3-1	Effect of Temperature on Degradation Rate of Solar Absorptance of Zinc Oxide/Potassium Silicate	22

TABLES

Table		Page
2-1	Calorimetric Total Hemispherical Emittance and In Situ Absorptance Data for Aluminum Silicate/Potassium Silicate Coating, Sample No. 19	5
2-2	Calorimetric Total Hemispherical Emittance and In Situ Absorptance Data for Aluminum Silicate/Potassium Silicate Coating, Sample No. 21	6
2-3	Calorimetric Total Hemispherical Emittance and In Situ Absorptance Data for Zinc Oxide/Potassium Silicate, Z-93, Sample No. 36	12
2-4	Calorimetric Total Hemispherical Emittance and In Situ Absorptance Data for Zinc Oxide/Potassium Silicate, Z-93, Sample No. 39	17
3-1	Summary of Data Obtained on Aluminum Silicate/Potassium Silicate and Zinc Oxide/Potassium Silicate	21

Section 1
INTRODUCTION AND SUMMARY

The major objective of this program is the evaluation of candidate coatings for low-temperature spacecraft radiators. The use of certain classes of power sources on spacecraft having long operational lifetimes presents several complex problems with regard to the choice of radiator coatings. The surfaces must show a high total hemispherical emittance to operating temperature with a low absorptance to energy in the solar spectrum. The latter is of importance because of the low operating temperature level of some radiators, 530°K or less. The stability of the radiative properties of the coating in the space environment is a critical factor as changes in either absorptance or emittance during the mission would result in severe problems with the operating power system. Changes in the radiative properties, particularly in solar absorptance, may occur because of exposure to temperature, vacuum, temperature cycling, and the complex radiation fields attributed to the space environment.

This initial program has as its objective the evaluation of the properties and stability of candidate coatings under conditions of exposure to ultraviolet radiation, vacuum of less than 10^{-7} Torr, elevated temperatures, and temperature cycling. During the first phase of the program, candidate materials have been exposed simultaneously to ultraviolet radiation at a 1-sun level, maximum operating temperature, and vacuum for 500-hr periods. The effects of temperature cycling have also been investigated.

The second phase of this program will be the exposure of one material, selected on the basis of the results of this initial phase, for 5,000 hr.

The major tasks completed during this report period included the following:

- Exposure tests of aluminum silicate/potassium silicate coating at 534° K (500° F)
- Exposure test of zinc oxide/potassium silicate coating (Z-93) at 534° K (500° F)
- Exposure test of zinc oxide/potassium silicate coating (Z-93) at 422° K (300° F)

Detailed discussion of the activities for the quarterly period are presented in the following sections.

Section 2
EXPERIMENTAL RESULTS

In order to evaluate the stability of the solar absorptance and total hemispherical emittance of candidate space power radiation coatings in the space environment, samples have been exposed for a period of 500-hr duration to ultraviolet radiation at a 1-sun level, defined by the 0.2- to 0.4- μ spectral band, from a xenon source; vacuum of 10^{-7} Torr or less; and maximum intended operating temperature level. The sample was temperature cycled from its operating temperature to room temperature approximately 110 times during the 500-hr exposure test. The results of these screening tests have been used to form the basis for the selection of the coating to be subjected to long-term exposure testing.

2.1 APPARATUS

The ultraviolet exposure apparatus used for these tests is described in detail in the first and second quarterly progress reports of this contract (Refs. 1 and 2).

2.2 SAMPLE PREPARATION AND DESCRIPTION

Preparation and description of all samples discussed in this report are described in detail in the second and fourth quarterly progress reports of this contract (Refs. 2 and 3).

2.3 TEST PROCEDURE

The test procedure and data reduction are described in detail in the third quarterly progress report of this contract (Ref. 4).

2.4 TEST RESULTS

2.4.1 Aluminum Silicate/Potassium Silicate Coating at 535°K (500°F)

Two samples of the aluminum silicate/potassium silicate (Hughes) coating were exposed to uv irradiation at a 1-sun level from a xenon lamp. The samples were maintained at a temperature of 535°K (500°F) and temperature cycled to room temperature every 4.7 hr. The chamber pressure was maintained at less than 1×10^{-7} Torr. The test on Sample No. 19 was terminated at 122 hr because of a broken thermocouple support wire.

The results of the calorimetric in situ measurements made during the exposure tests on the duplicate samples, Nos. 19 and 21, are tabulated in Tables 2-1 and 2-2. The solar absorptance of the two samples as a function of exposure time is shown graphically by Fig. 2-1. It is seen that the solar absorptance of both samples degraded very rapidly in the first 100 hr of exposure; with the absorptance increasing from 0.15 to over 0.40. After exposure both samples showed a definite ring-like discoloration around the outer periphery of the sample. It is believed that this may have been due to contact with the strippable protective coating on the edge of the sample during application and curing of coating. The strippable coatings were also baked on and were extremely hard to remove. They were finally removed by scrapping with a knife blade. The sample color after exposure was a medium grey with a slightly darker border around the outer edge of sample. An analysis of the coating will be made in an attempt to determine if any contamination is present which could have caused the ring-like discolorations.

Pre- and post-test room temperature reflectance data for the two samples are shown in Figs. 2-2 and 2-3. The spectral band absorptances were determined from these reflectance curves and are presented in the data tables for comparison. Very little infrared recovery is shown for these samples upon exposure to air.

Table 2-1

CALORIMETRIC TOTAL HEMISPHERICAL EMITTANCE AND IN SITU ABSORPTANCE DATA
FOR ALUMINUM SILICATE/POTASSIUM SILICATE COATING, SAMPLE NO. 19

Hours	Cycles	Temperature		ϵ_{TH}	α_H (a)				α_S (b)	
		(°K)	(°F)		Total	0.2 - 0.41	0.41 - 0.60	0.60 - 0.85		0.85 →
0	0	290	62	0.90	0.14	-	-	-	-	-
0	0	342	156	.89	.15	-	-	-	-	-
0	0	395	250	.92	.18	-	-	-	-	-
0	0	535	500	.72	-	-	-	-	-	-
0	1	535	500	.72	.18	0.35	0.22	0.13	0.13	0.20
24	7	535	500	.72	.30	.41	.36	.35	.18	.31
75	19	535	500	.72	.44	.65	.50	.48	.32	.44
122(c)	31	535	500	.72	.46	.60	.55	.50	.38	.46
Before Exposure(d)					.14	.32	.11	.10	.12	.14
After Exposure(d)					.42	.60	.54	.40	.33	.41

- (a) Absorptance for xenon source.
- (b) Absorptance for solar source.
- (c) Test terminated due to thermocouple failure.
- (d) From Cary reflectance measurements.

Table 2-2

CALORIMETRIC TOTAL HEMISPHERICAL EMITTANCE AND IN SITU ABSORPTANCE DATA
FOR ALUMINUM SILICATE/POTASSIUM SILICATE COATING, SAMPLE NO. 21

Hours	Cycles	Temperature		ϵ_{TH}	α_H (a)				α_S (b)
		(°K)	(°F)		Total	0.2 - 0.41	0.41 - 0.6	0.6 - 0.85	
0	0	290	62	0.94	0.15	-	-	-	0.15
0	0	342	156	.96	.15	-	-	-	.15
0	0	395	250	.93	.15	-	-	-	.15
0	0	535	500	.82	.16	0.47	0.11	0.10	.16
24	3	535	500	.80	.25	.49	.35	.18	.25
72	13	535	500	.80	.37	.61	.38	.38	.37
168	39	535	500	.79	.43	.58	.38	.38	.43
218	52	535	500	.79	.44	.60	.44	.38	.44
290	68	535	500	.80	.48	.64	.48	.41	.48
338	72	535	500	.80	.46	.61	.44	.43	.46
386	82	535	500	.78	.47	.59	.47	.43	.47
458	97	535	500	.80	.51	.60	.51	.45	.50
530	112	535	500	.81	.50	.63	.54	.47	.50
Before Exposure(c)					.13	.40	.11	.09	.14
After Exposure(c)					.46	.65	.55	.46	.45

(a) Absorptance for xenon source.

(b) Absorptance for solar source.

(c) From room temperature reflectance measurements.

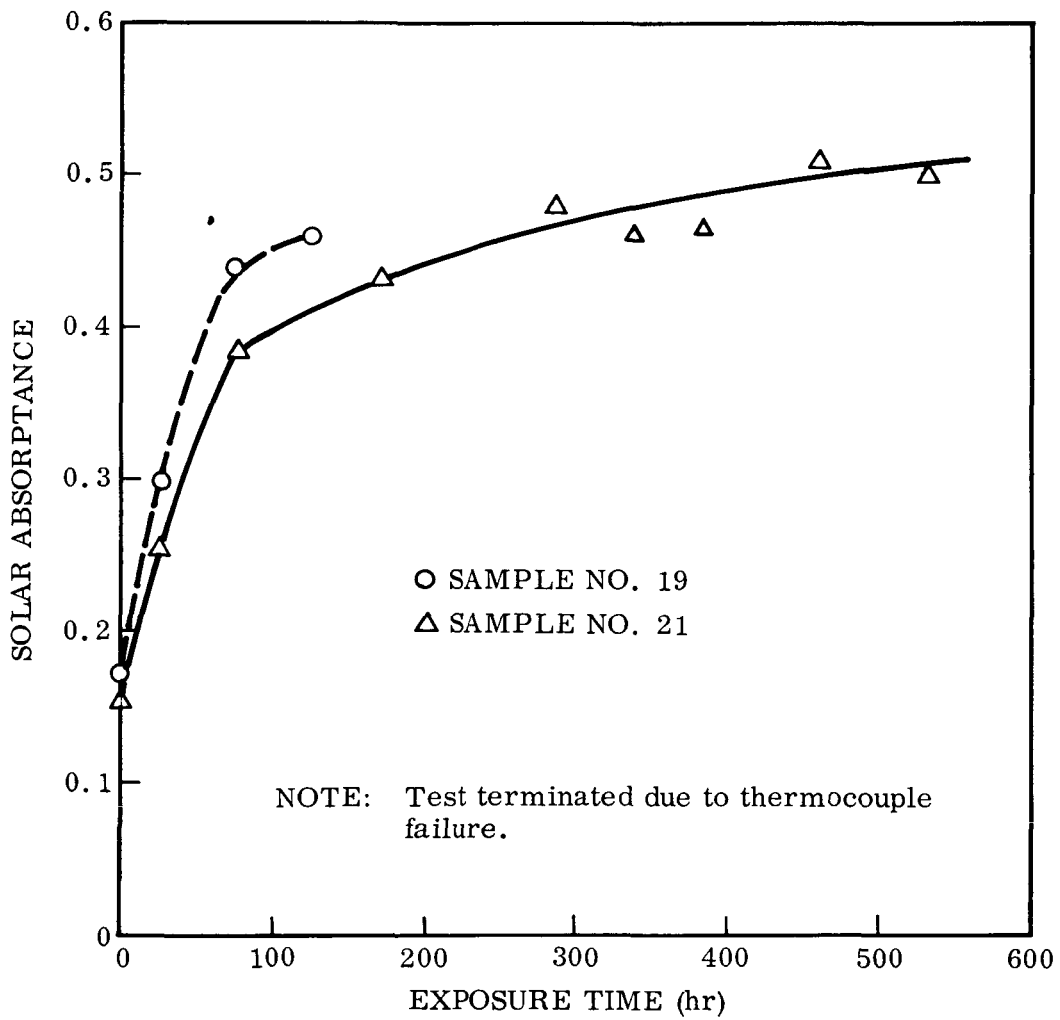


Fig. 2-1 Solar Absorptance of Aluminum Silicate/Potassium Silicate Coating as a Function of Exposure Time

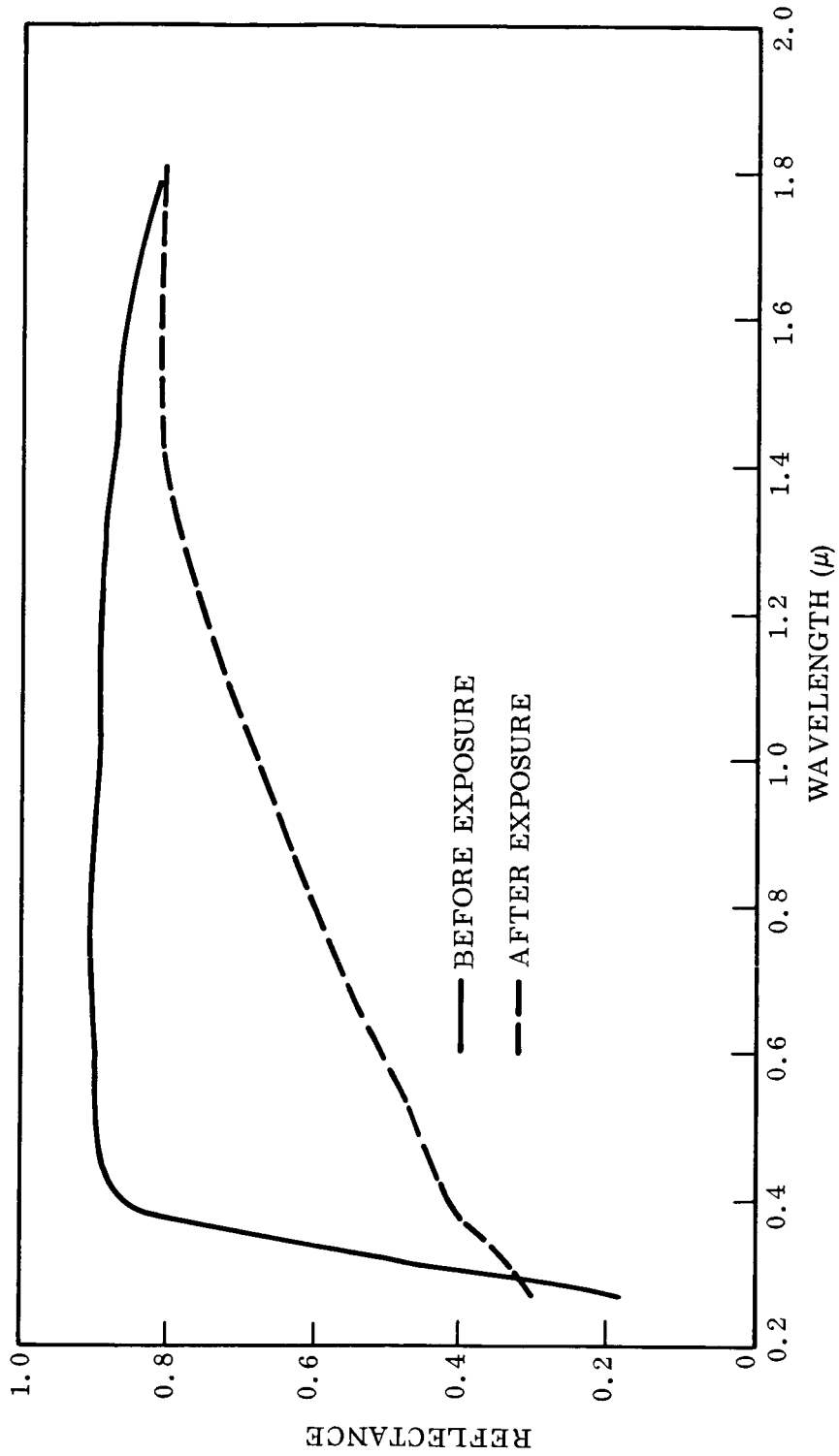


Fig. 2-2 Pre- and Post-Test Room Temperature Spectral Reflectance of Aluminum Silicate/Potassium Silicate Coating, Sample No. 19

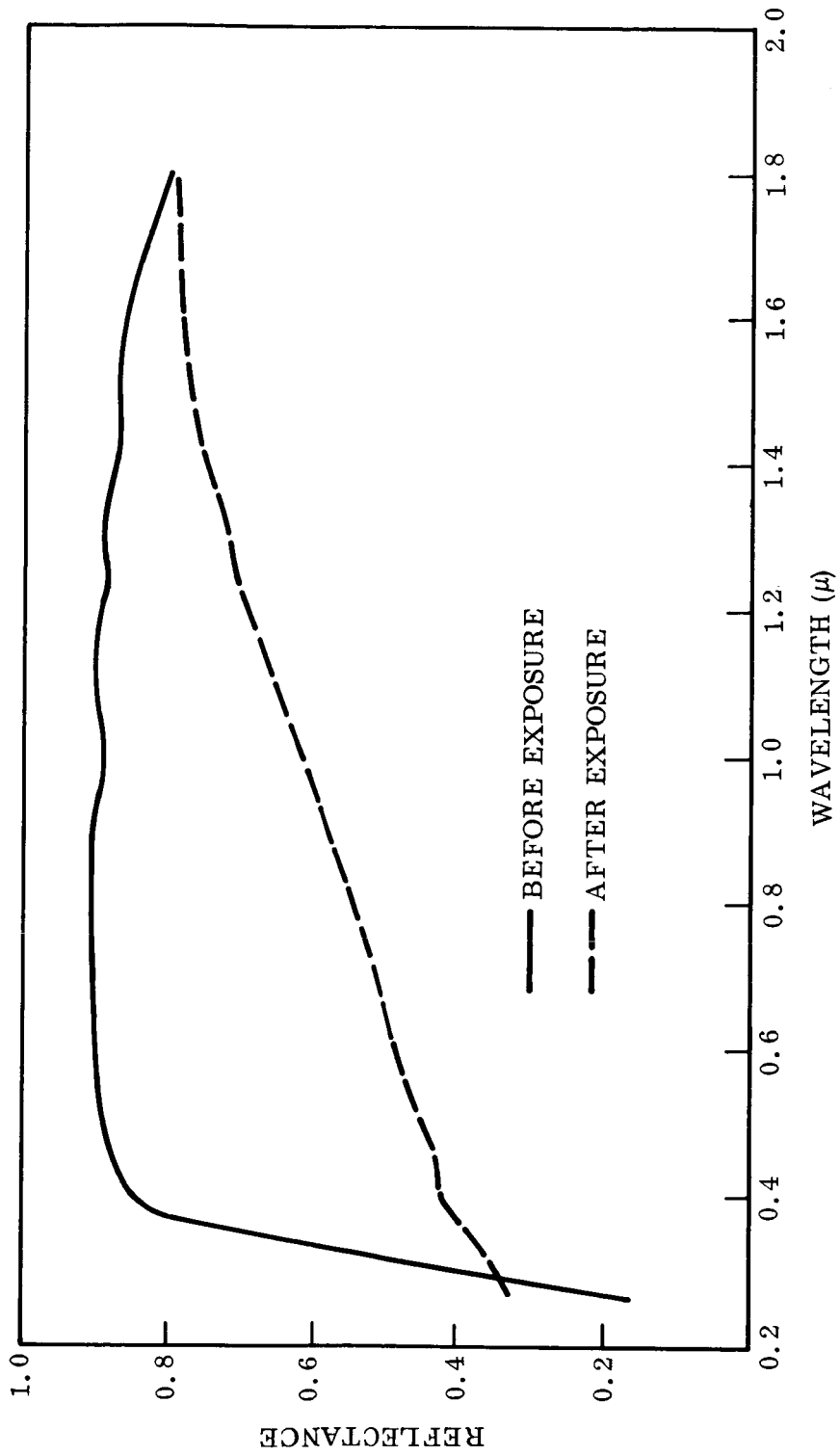


Fig. 2-3 Pre- and Post-Test Room Temperature Spectral Reflectance of Aluminum Silicate/Potassium Silicate Coating, Sample No. 21

The total hemispherical emittance measured calorimetrically during the exposure test is shown graphically in Fig. 2-4. Sample No. 19 decreased from 0.90 at room temperature to 0.72 at 535°K (500°F). Sample No. 21 decreased from 0.94 at room temperature to 0.80 at 535°K (500°F). There is no evident indication of why this large difference occurs between the two samples.

2.4.2 Zinc Oxide/Potassium Silicate Coating (Z-93) at 535°K (500°F)

The second sample of zinc oxide/potassium silicate (Z-93), No. 36, was exposed to uv irradiation at a 1-sun level from a xenon lamp. The sample was maintained at a temperature of 535°K (500°F) and temperature cycled to room temperature every 4.7 hr. The chamber pressure was maintained at less than 1×10^{-7} Torr. This test was terminated at 331 hr due to a vacuum failure.

The results of the calorimetric in situ measurements made during the exposure test on Sample No. 36 are tabulated in Table 2-3. The solar absorptance as a function of exposure time is shown graphically in Fig. 2-5. The data from Sample No. 35 are shown for comparison. The data on this sample were presented and discussed in the previous report (Ref. 5). The data show close similarity of the solar absorptance degradation of the two samples. After 331 hr, a loss of vacuum occurred with an increase in absorptance of the sample. The test was continued for a short time but the absorptance remained about 0.05 above the original level before the vacuum failure so the test was terminated.

Pre- and post-test room temperature reflectance data for the sample are shown in Fig. 2-6. These data are converted to broadband spectral absorptance for comparison with in situ broadband data and presented in the table.

The total hemispherical emittance measured calorimetrically during the exposure test is shown by Fig. 2-7. The emittance decreased from a value of 0.93 at room temperature to 0.80 at 535°K (500°F).

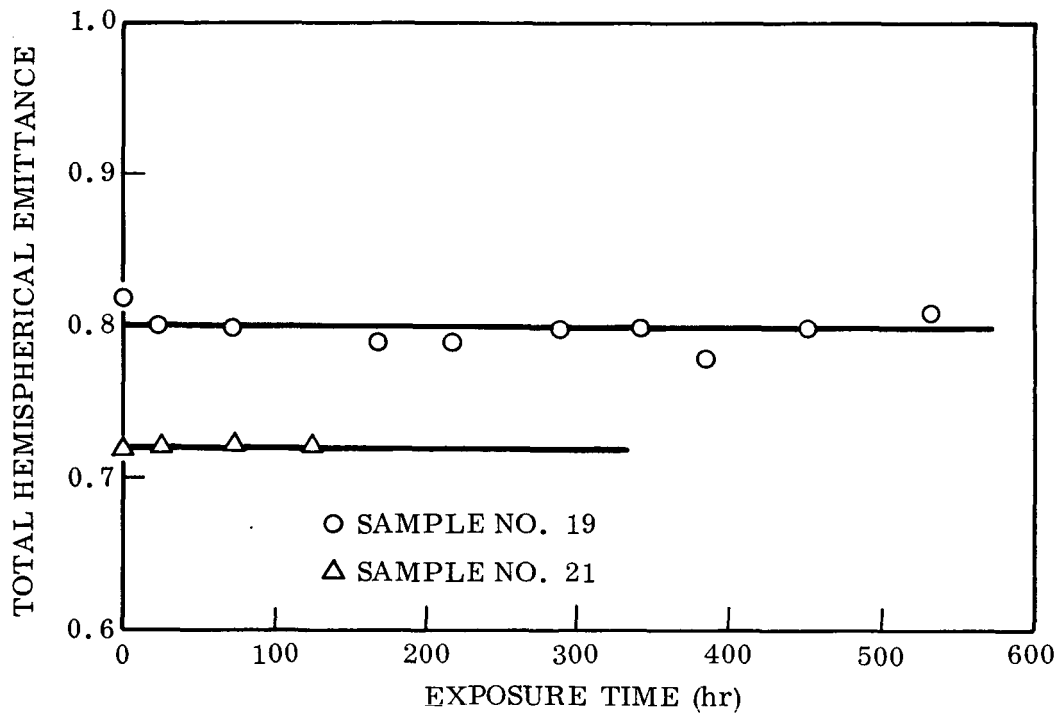


Fig. 2-4 Total Hemispherical Emittance of Aluminum Silicate/Potassium Silicate Samples as a Function of Exposure Time at a Temperature of 535° K (500° F)

Table 2-3

CALORIMETRIC TOTAL HEMISPHERICAL EMITTANCE AND IN SITU ABSORPTANCE DATA
FOR ZINC OXIDE/POTASSIUM SILICATE, Z-93, SAMPLE NO. 36

Hours	Cycles	Temperature		ϵ_{TH}	α'_H (a)				α'_S (b)
		(°K)	(°F)		0.2-0.41	0.41-0.6	0.6-0.84	0.85-→	
0	0	290	62	0.93	0.13	-	-	-	0.14
0	0	342	156	.91	.13	-	-	-	.14
0	0	535	500	.82	.11	-	-	-	.12
0	0	535	500	.81	.10	0.50	0.05	0.05	.11
15	12	535	500	.79	.15	.50	.08	.10	.15
87	30	535	500	.81	.23	.55	.20	.19	.24
187	54	535	500	.79	.25	.65	.22	.19	.25
235	66	535	500	.79	.24	.65	.22	.20	.24
283	78	535	500	.79	.24	.65	.22	.20	.24
331(c)	90	535	500	.81	.31	.75	.32	.25	.32
Before Exposure	(d)				.13	.60	.05	.06	.14
After Exposure	(d)				.42				.42

(a) Absorptance for xenon source.

(b) Absorptance for solar source.

(c) Vacuum failure pressure > 100 μ .

(d) From room temperature reflectance measurements.

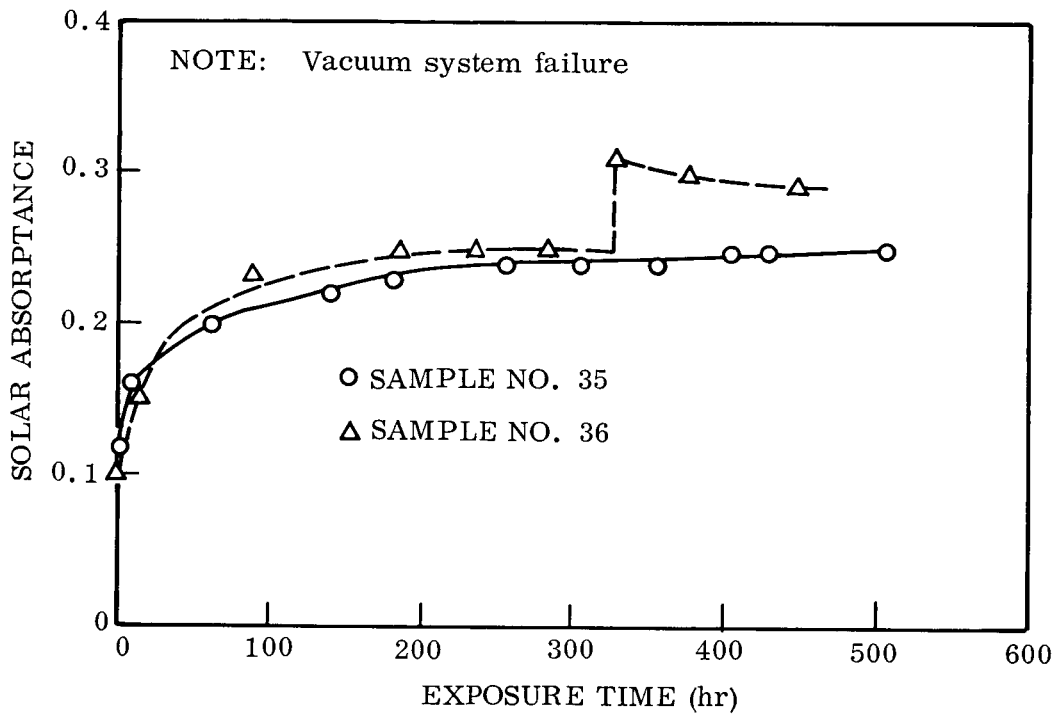


Fig. 2-5 Solar Absorptance of Z-93 (Zinc Oxide/Potassium Silicate) Coating as a Function of Exposure Time at 535°K (500° F)

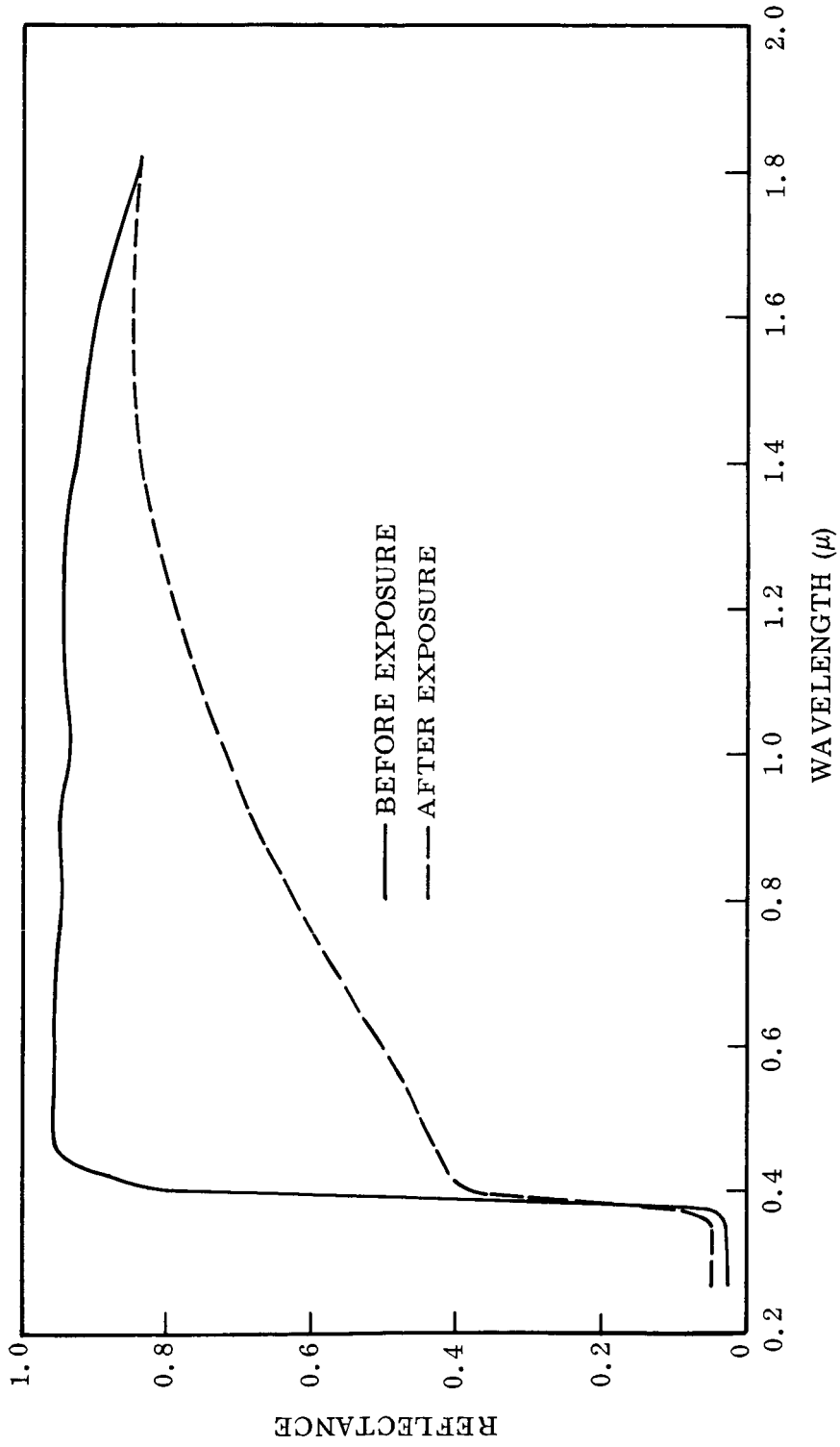


Fig. 2-6 Pre- and Post-Test Room Temperature Spectral Reflectance of Z-93 (Zinc Oxide/Potassium Silicate), Sample No. 36

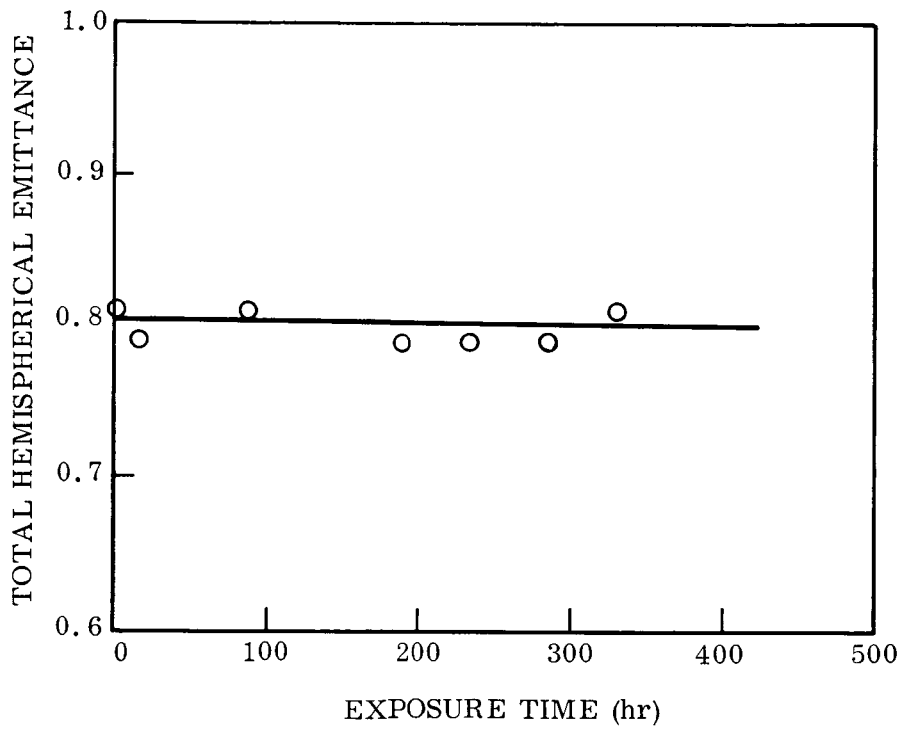


Fig. 2-7 Total Hemispherical Emittance of Z-93 (Zinc Oxide/Potassium Silicate) as a Function of Exposure Time at a Temperature of 535°K (500° F), Sample No. 36

2.4.3 Zinc Oxide/Potassium Silicate Coating (Z-93) at 452°K (300°F)

A sample of Z-93, Sample No. 39, was exposed to uv irradiation at a 1-sun level from a xenon lamp. The sample was maintained at a temperature of 452°K (300°F) and temperature cycled to room temperature every 4.7 hr. The chamber pressure was maintained at less than 1×10^{-7} Torr for the test duration.

The results of the calorimetric in situ measurements made during the exposure test on Sample No. 39 are tabulated in Table 2-4. The solar absorptance of the sample as a function of exposure time is shown graphically by Fig. 2-8. At this temperature the solar absorptance increased very slowly from 0.12 to 0.15 during the exposure test.

Pre- and post-test room temperature spectral reflectance data for the sample are shown in Fig. 2-9. Conversion of these data to broadband absorptance is shown in the tables for comparison.

The total hemispherical emittance measured calorimetrically during the exposure test is shown in Fig. 2-10. The total hemispherical emittance of this sample remained essentially constant at 0.90 for the test duration.

Table 2-4

CALORIMETRIC TOTAL HEMISPHERICAL EMITTANCE AND IN SITU ABSORPTANCE DATA
FOR ZINC OXIDE/POTASSIUM SILICATE, Z-93, SAMPLE NO. 39

Hours	Cycles	Temperature		ϵ_{TH}	α_H (a)				α_S (b)
		(°K)	(°F)		Total	0.2-0.41	0.41-0.6	0.6-0.85	
0	0	290	62	0.90	0.12	-	-	-	0.13
0	0	342	156	.91	.11	-	-	-	.12
0	0	452	300	.91	.11	-	-	-	.12
0	1	452	300	.91	.11	0.65	0.06	0.06	.12
0	4	452	300	.91	.10	.65	.06	.06	.11
24	8	452	300	.90	.10	.65	.07	.08	.11
48	13	452	300	.89	.10	.70	.07	.08	.11
98	25	452	300	.90	.10	.75	.07	.09	.11
148	37	452	300	.89	.10	.75	.07	.08	.11
198	50	452	300	.89	.11	.75	.08	.10	.12
248	62	452	300	.89	.10	.75	.08	.09	.11
298	24	452	300	.89	.12	.80	.08	.10	.13
322	80	452	300	.89	.13	.85	.08	.09	.14
400	95	452	300	.89	.13	.90	.07	.09	.14
450	106	452	300	.89	.14	.90	.07	.08	.15
475	113	452	300	.89	.13	.90	.07	.09	.14
Before Exposure (c)					.13	.64	.05	.07	.14

(a) Absorptance for xenon source.

(b) Absorptance for solar source.

(c) From room temperature reflectance measurements.

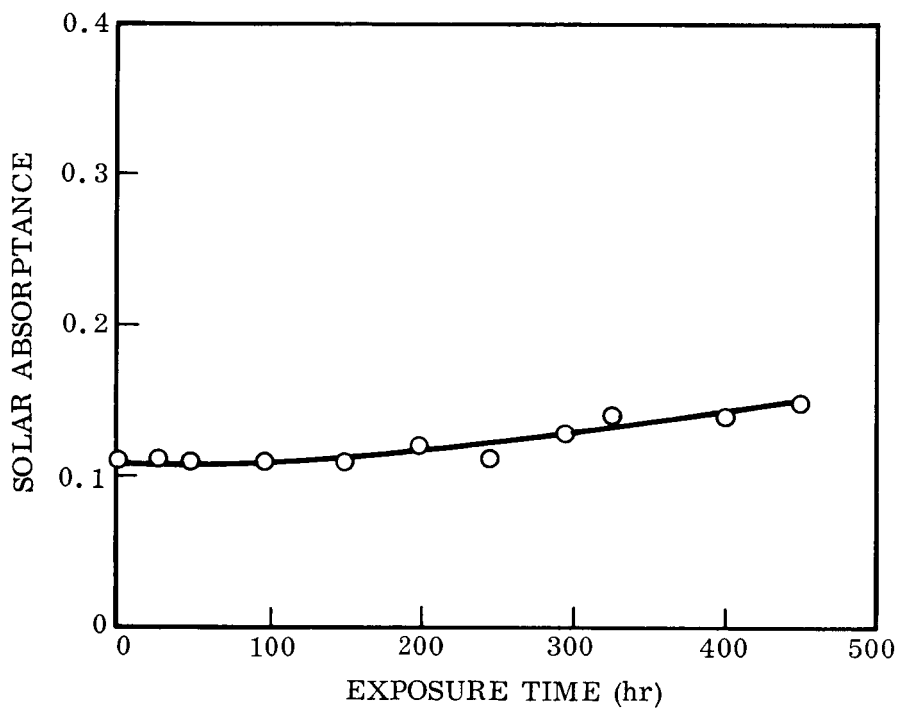


Fig. 2-8 Solar Absorptance of Z-93 (Zinc Oxide/Potassium Silicate) as a Function of Exposure Time at a Temperature of 452°K (500° F), Sample No. 39

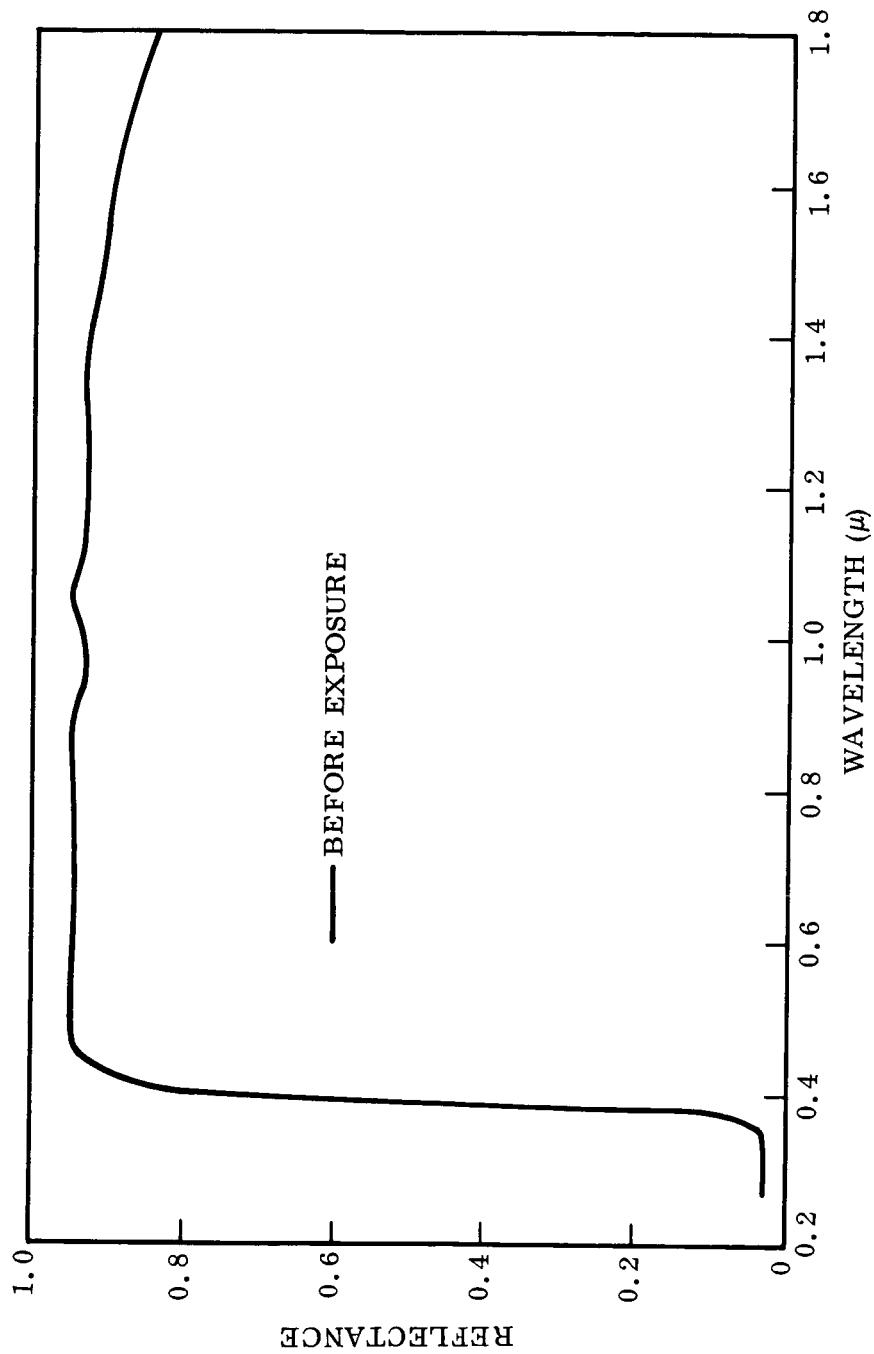


Fig. 2-9 Room Temperature Spectral Reflectance of Z-93 (Zinc Oxide/Potassium Silicate),
Sample No. 39

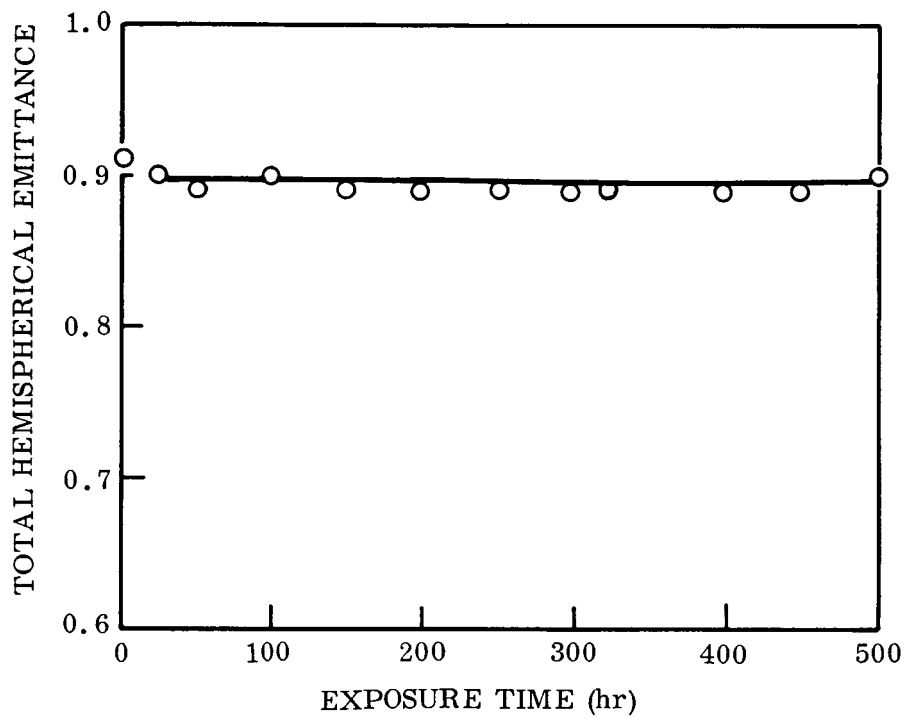


Fig. 2-10 Total Hemispherical Emittance of Z-93 (Zinc Oxide/Potassium Silicate) as a Function of Exposure Time at a Temperature of 452°K (300° F), Sample No. 39

Section 3
DISCUSSION OF SCREENING TESTS

The 500-hr endurance testing at 535°K (500°F) has been completed on both the aluminum silicate/potassium silicate and the zinc oxide/potassium silicate (Z-93) coatings. A 500-hr endurance test of Z-93 was also made at 452°K (300°F). The data from these tests are summarized in Table 3-1. No data have been found for uv irradiation of the aluminum silicate/potassium silicate system at these temperatures for comparison with the data presented.

The data obtained for Z-93 for both the 535°K (500°F) and 452°K (300°F) runs are shown in Fig. 3-1. Also shown are data presented by Streed (Ref. 6) for a zinc oxide/potassium silicate coating at approximately the same temperatures. The curves show the same general characteristics although the initial absorptance of Streed's samples were much higher, the irradiation was at approximately 10-sun level, and all measurements were made by reflectance techniques out of the exposure chambers.

Table 3-1

SUMMARY OF DATA OBTAINED ON ALUMINUM SILICATE/POTASSIUM SILICATE
AND ZINC OXIDE/POTASSIUM SILICATE

Sample	α_s				Emittance		
	Temperature		Before	After	Temperature		ϵ_{TH}
	(°K)	(°F)			(°K)	(°F)	
Aluminum Silicate/Potassium Silicate	535	500	0.14	0.45	290	62	0.94
					535	500	.80
Zinc Oxide/Potassium Silicate	535	500	.11	.24	290	62	.93
					535	500	.82
Zinc Oxide/Potassium Silicate	452	300	.12	.15	290	62	.90
					452	300	.90

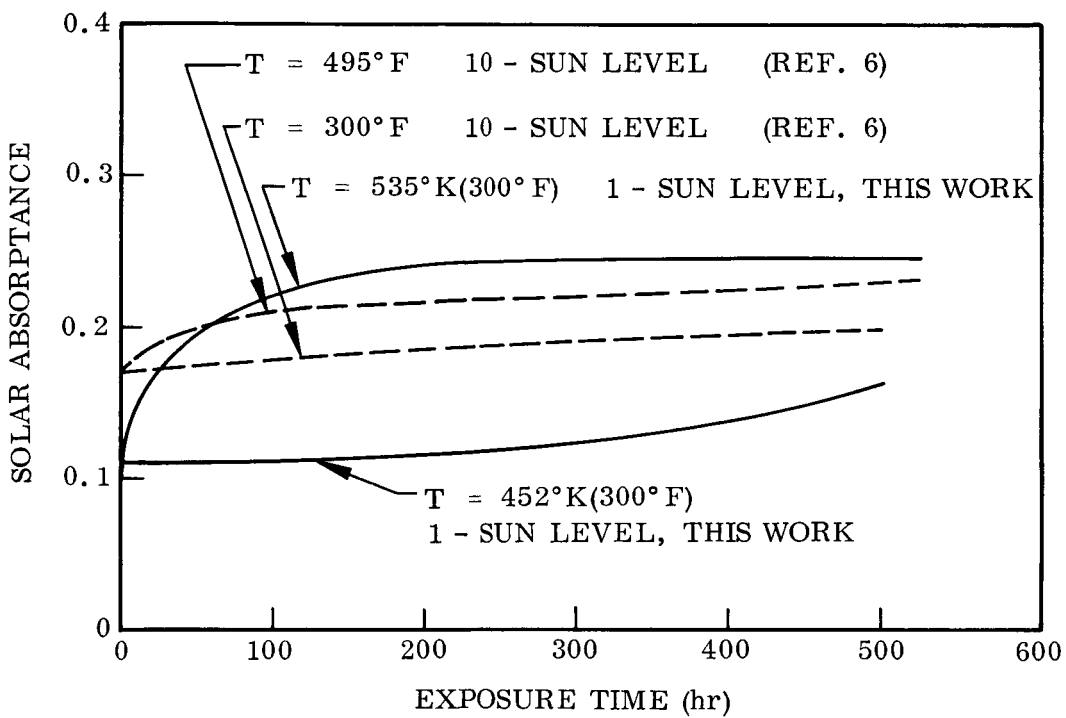


Fig. 3-1 Effect of Temperature on Degradation Rate of Solar Absorptance of Zinc Oxide/Potassium Silicate

The change in solar absorptance, $\Delta\alpha_s$, of the titanium dioxide/silicone coating measured by this method is in good agreement with that measured by S. Greenberg's (Ref. 7), using an in situ bidirectional reflectance technique. He reported a value of $\Delta\alpha_s$ of 0.11 at 500 equivalent sun hours (ESH) with the sample at approximately 320°K. This compares with a $\Delta\alpha_s$ of 0.16 at 395°K as determined by the calorimetric method. The larger value at the higher temperature is believed to be due to a rate effect which is temperature dependent. After 1330 ESH, Greenberg's data showed a $\Delta\alpha_s$ of 0.15 which appears to have reached a saturation level. Data reported by Arvesen et al. (Ref. 8) for a titanium dioxide/silicone coating show a large spread with temperature, source, and intensity. The $\Delta\alpha_s$'s reported are considerably lower than those obtained from the in situ methods. This is attributed to the fact that solar absorptances were computed on the basis of post-exposure reflectance measurements made in air, and the recovery of the TiO_2 in the near infrared upon re-exposure to air resulted in a lower value of $\Delta\alpha_s$.

For both zinc oxide/silicone coatings, S-13 and S-14G, the change in solar absorptance was 0.08. This agrees with the data of McMillan et al. (Ref. 9) and Greenberg (Ref. 7) using the bi-directional in situ method which resulted in a $\Delta\alpha_s$ for S-13 of 0.07 to 0.08 for 500 ESH. Data reported by Pearson (Ref. 10) on flight test results for a zinc oxide/silicone system show a $\Delta\alpha_s$ of approximately 0.05 after 500 hr of exposure to 1 sun. However, the temperature of this flight sample was not reported. No data were found in the literature for comparison with the S-13G coating. Both the post-test spectral reflectance data and the broadband spectral data showed the improvement of the S-13G in the near infrared region as reported by IITRI (Ref. 11). However a greater degradation was observed in the 0.2- to 0.85- μ region with the result that the $\Delta\alpha_s$ of both systems were equal.

Of the two coatings tested at 534°K, the zinc oxide/potassium silicate system was the more stable. The $\Delta\alpha_s$ was 0.13 for this system, whereas it was 0.30 for the zirconium silicate/potassium silicate coating. For both coatings, the degradation appeared to remain essentially constant after 300 hr of exposure. If this degradation did in fact reach a saturation level before the 500 hr of exposure, the data are in reasonably

good agreement with those reported by Streed et al. (Ref. 6). They exposed zinc oxide/potassium silicate and zirconium silicate/potassium silicate coating to 7000 ESH at 527° to 530°K with $\Delta\alpha_s$'s of 0.16 and 0.29, respectively.

Section 4
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