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EMISSIVITY COATINGS FOR LOW-TEMPERATURE SPACE RADIATORS

Quarterly Progress Report No. 4

For Quarter Ending 30 June 1966

CONTRACT NAS-3-7630

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

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Aerospace Sciences Laboratory
Lockheed Palo Alto Research Laboratory
OCKHEED MISSILES & SPACE COMPANY
Sunnyvale, California

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ERRATA

EMISSIVITY COATINGS FOR LOW-TEMPERATURE SPACE COATINGS

Aerospace Sciences Laboratory
Lockheed Palo Alto Research Laboratory
Lockheed Missiles and Space Company
Sunnyvale, California

Quarterly Progress Report 1 (for quarter ending 30 September 1965), Report 2 (for quarter ending 31 December 1965), and Report 3 (for quarter ending 31 March 1966) were all incorrectly numbered NASA CR-54807. Report 1 should continue as NASA CR-54807. However, please change this number on the covers and title pages of reports 2 and 3 to NASA CR-72059 and NASA CR-72060, respectively.

NASA-CR-72089

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FOREWORD

The results of the research activities carried out during the fourth quarterly period (1 April through 30 June 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.

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Section 1

INTRODUCTION AND SUMMARY

The major objective of this program is the evaluation of 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 a radiator coating. The surface must show a high total hemispherical emittance at 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 temperature, and temperature cycling. During the first phase of the program, candidate materials are being exposed simultaneously to ultraviolet radiation at a 1-sun level, maximum operating temperature, and vacuum for 500-hr periods. The effects of temperature cycling are also being investigated.

The second phase of the program will be the exposure of two materials, selected on the basis of the results of the initial phase, for 5,000 hr to ultraviolet radiation at a 1-sun level in vacuum at maximum operating temperature.

The major tasks completed during this report period are as follows:

- Continuation of literature survey
- Procurement and initial room temperature measurements of the sixth coating selected for the program, ZnO/Silicone (S-13G)
- Thermal cycling tests of five coating systems
- 500-hr uv exposure test of the TiO_2/silicone coating.

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

Section 2
LITERATURE SURVEY

No major disclosures of direct bearing to this program have been found in the open literature. However, the results of some work done at Lockheed Missiles & Space Company are of significant importance to this program.

In situ spectral reflectance measurements have been performed (Ref. 1) on ZnO/silicone (S-13) and TiO_2/silicone (Thermatrol 2A-100) as a function of time and ultraviolet radiation intensity. Irradiation of both materials was performed for 256 hr at 1 equivalent sun, 2000 – 4000 Å, and then accelerated to the 10-sun level for a total exposure of 1,600 equivalent sun hours (ESH). The changes in solar absorptances were in precise agreement with the flight experiment data from OSO-I (Ref. 2) and OSO-II (Ref. 3) for these coatings. The limiting values of the change in solar absorptance for Thermatrol and S-13 were 0.13 and 0.09 respectively. Accelerated testing employing 10 and 20 equivalent suns for 1,500 equivalent sun hours demonstrated substantially different damage kinetics, with approximately 50 percent of the degradation occurring in the first 50 equivalent sun hours. Despite the widely differing damage rates, the 1, 10, and 20 sun data appear to converge after approximately 1,000 ESH.

In all cases, pre-and postexposure measurements of solar absorptances showed that upon exposure to air, there was substantial recovery of the uv damage, the rate of recovery being dependent upon the partial pressure of oxygen in contact with the samples. These results again demonstrate the necessity of performing in situ measurements for evaluation of the change in solar absorptances of coating systems of this type.

Section 3
CANDIDATE COATINGS

During the report period specimens of the sixth candidate coating system were received from IIT Research Institute. This coating is a modified zinc oxide-methyl silicone (S-13G) selected on the recommendation of the Lewis Research Center. Initial optical properties were measured and are presented in Section 3.2. The coating description presented below is based upon the information provided by the supplier.

3.1 COATING DESCRIPTION

Source

IIT Research Institute (cost not given by supplier)

Formulation

Material	Part By Weight
New Jersey Zinc Co.,SP 500 zinc oxide, PS7-treated	240
General Electric Co.,RTV-602 silicone	100
Toluene, USP	175
	515

Silicate Treated -- ZnO

The PS7 treated ZnO is used to prepare the S-13G paint. Approximately 600 grams of "as received" SP500 zinc oxide are thoroughly mixed with 1200 grams of PS7 potassium silicate (Sylvania) in a 2-quart capacity ball-mill jar. Approximately

20 cylindrical grinding stones, 1 × 1/2-in. diameter, are added to the mill, and the slurry is ground at approximately 75% of critical speed for 45 min. The ball-jar is removed from the mill, and the ground mixture is allowed to stand approximately 16 hr. The slurry is then reground for 10 min. The ground slurry is transferred to a 3-liter beaker and diluted with 800 ml of distilled water. The mixture is thoroughly stirred and transferred to a large Buchner funnel and filtered at reduced pressure through a No. 597 filter paper.* The filter cake is washed with 3 liters of distilled water and pumped dry. The filter cake is removed from the funnel and spread on an aluminum foil tray. The contents are placed in a forced-air oven and dried for 16 hr at 100°C. The dried treated-pigment is then placed in a completely dry mill jar and dry ground with approximately 20 grinding stones for 15 min. The resultant treated-pigment is then reheated for 1 hr at 100°C.

The zinc oxide, the RTV-602, and 100 parts by weight of the toluene are premixed and charged to a porcelain ball mill in a quantity sufficient to just fill the void space when the mill is one-half full of grinding stones 0.5-in. in diameter. The paint is ground for 4 hr at approximately 70% critical speed. The critical speed (rpm) is given by:

$$W_{C_S} = \frac{54.2}{\sqrt{R}}$$

where R is the radius of the mill in feet. The basic charge is then removed, and the remaining toluene is added to the mill. The mill residue and the solvent are ground until the contents are uniformly thin, but not for more than 5 min. The contents are then added to the main charge, and the whole charge is mixed thoroughly. Note: The SRC-05 catalyst is not added until the paint is applied.

---

*Schleicher and Schuell Analytical Filter Paper.

Particle Size and Shape

Not stated by supplier.

Substrate

1-in. diameter disc, 0.050-in. thick, of 6061-T6 aluminum machined to a 30 - rms finish.

Method of Application

Preparation of Paint for Application:

The paint is furnished without the SRC-05 catalyst. The catalyst is added as 1 part SRC-05 in 10 parts of toluene: The catalyst solution is added to the paint with thorough stirring. A low catalyst concentration is recommended in order to ensure optimum stability to ultraviolet irradiation in vacuum. A concentration of 0.4% based upon RTV-602 provides optimum stability without greatly sacrificing thermal-cure properties, although a coating prepared at this concentration represents the lower limit without sacrificing cure and physical properties. Somewhat better physical properties are obtained with a catalyst concentration of 0.5% based on RTV-602 and still better properties are obtained at 0.75% SRC-05. The SRC-05 catalyst-to-paint ratio is 1 part SRC-05 in 10 parts of toluene to 1030 parts of S-13G (by weight). The catalyst solution is added only as the paint is used and to only the amount that can be applied in a 30-min period. Allow the catalyzed paint to set for 10 to 15 min before application to the primed surfaces. The paint should be thoroughly stirred before transfer to other containers or before addition of catalyst.

Preparation of Surfaces for Painting

Standard surface cleaning procedures should be used to prepare the surface for application of the S-13G paint. The S-13G paint can, in general, be applied to any surface to which the required primer can be applied. The primer, General Electric's proprietary SS-4044, can be applied to either anodized or zinc chromate-primed surfaces. It is preferable that it be applied to clean bare metal or to anodized surfaces, however. Greasy surfaces should be cleaned with standard detergent and water prior to priming; they should be thoroughly dry.

Application of Paint

The primer can be spray-applied (Binks model 18, Paasche Autch, or comparable gun) at about 30 psi. Only about 0.5 mil of primer is required (just enough to provide a base for the S-13G paint). The primer should be allowed to air-dry for 1 to 2 hr before application of the S-13G paint.

The S-13G paint can be spray-applied with a Binks model 18 spray gun (or comparable gun) at a gas pressure of about 60 psi. Unless clean, dry air is available, prepurified nitrogen or prepurified air must be used. The S-13G paint should be allowed to air-cure 16 hr before handling. It is imperative that dust and debris be kept off the surface during the curing process.

The wet film thickness of the paint can be measured by either the Pfund or the Interchemical wet-film thickness gage, or a suitable bridge-type gage. Dry film thickness can be measured with a Fischer Permascope nondestructive thickness tester, type ECTH.

Reapplication

Soiled or damaged areas can be recoated. Soiled areas must be cleaned thoroughly with detergent and water and dried before application of additional S-13G paint. Damaged or gouged areas can be recoated by making a paste of S-13G in which the bulk of the solvent is omitted. Such a material can be trowelled or brushed over the damaged areas and cures tack-free within a few hours.

Storage of Paint

The paint is supplied in various quantities. Since the paint cannot be mixed with catalyst solutions in larger lots than can be applied at one time, it may be desirable to store the paint in smaller containers. If smaller containers are utilized, only glass, nickel, or unlined, unleaded, unsoldered steel cans can be used. The caps, tops, or closures of these containers should not possess gummed seals or any material soluble in toluene.

Coating Thickness

5.0 to 8.0 mils recommended, actual thickness not determined. Coating weight 0.006 lb/ft^2, as measured from test specimens.

Weight Loss During Vacuum Testing

Not stated by supplier.

Final Composition

Not stated by supplier.

3.2 INITIAL OPTICAL PROPERTIES

The solar absorptance α_s and total emittance ϵ_T of the S-13G samples were measured at room temperature in air. These data are reported in Table 3-1. Total emittance measurements were carried out using the Lion Optical Surface Comparator (Ref. 4). Solar absorptances were calculated using spectral reflectance data obtained from measurements made using a Cary Model 14 spectrophotometer with integrating sphere and a Gier-Dunkel integrating sphere with a Perkin-Elmer Model 98 monochrometer (Ref. 4). A comparison of typical spectral reflectance curves are presented in Fig. 3-1.

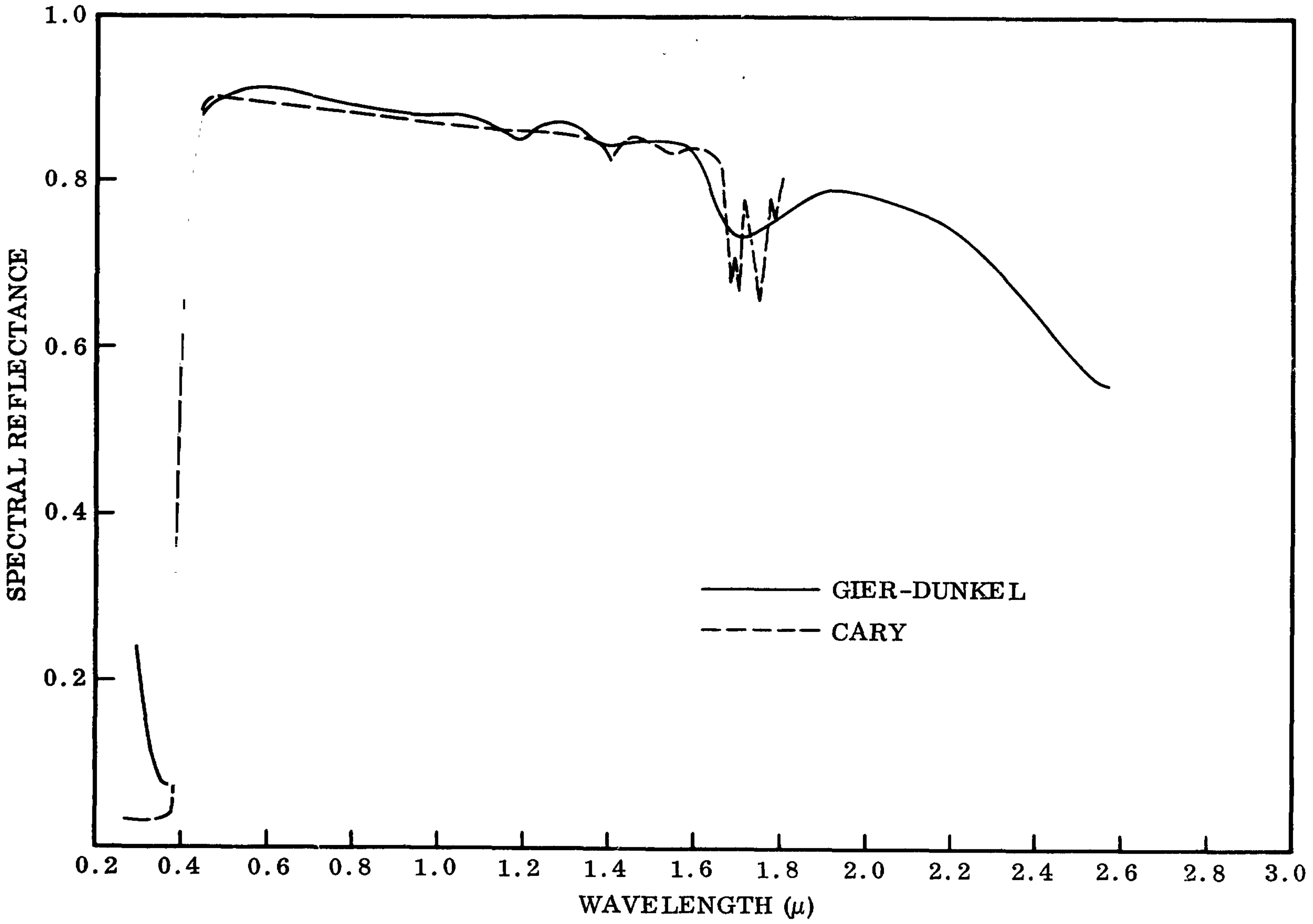

Fig. 3-1 Typical Room Temperature Normal Spectral Reflectance of ZnO/Silicone (S-13G) Coating

Table 3-1

INITIAL ROOM TEMPERATURE OPTICAL PROPERTIES OF ZnO/SILICONE (S-13G) COATING

Sample	Solar Absorptance		Emittance (Optical Surface Comparator)
	Cary	Gier-Dunkle	
43	0.19 ± 0.02	0.20 ± 0.01	0.85 ± 0.03
44	0.20 ± 0.02	0.20 ± 0.01	0.85 ± 0.03
45	0.20 ± 0.02	0.20 ± 0.01	0.85 ± 0.03
46	0.20 ± 0.02	–	0.85 ± 0.03
47	0.19 ± 0.02	–	0.85 ± 0.03
48	0.16 ± 0.02	–	0.86 ± 0.03
49	0.20 ± 0.02	–	0.84 ± 0.03
50	0.19 ± 0.02	–	0.86 ± 0.03

Section 4
EXPERIMENTAL RESULTS

4.1 THERMAL CYCLING TESTS

The ability of the coating systems to withstand thermal stresses was evaluated by repeated temperature cycling of two samples of each coating-substrate combination between their maximum intended operating temperature and 83°K (150°R).

4.1.1 Apparatus

Samples of the coatings were tested in the thermal cycling apparatus shown by Fig. 4-1. The apparatus consisted essentially of a sample holder, vacuum system, temperature control systems for the sample holder, and recorder for continuously monitoring coating substrate temperature. The sample holder was supported on the stainless steel base plate of the vacuum chamber within a glass bell jar. The system was pumped through a liquid nitrogen trap with a 4-in. oil diffusion pump and 5 cfm mechanical pump.

The sample holder, Fig. 4-2, was a machined copper block having six flat surfaces for mounting of the specimens. A 500-W cartridge heater was incorporated into the upper portion of the block, and a copper cooling coil, for passage of liquid nitrogen, was brazed to the lower end of the block. Heater power was controlled with a variable transformer. The cooling rate was controlled using two solenoid valves which regulated the flow of liquid nitrogen to the block by a by-pass arrangement. This permitted maximum flow of liquid at the start of the cycle in order to achieve the required rapid cooling rate and then a reduction in flow to decrease the rate as a function of time.

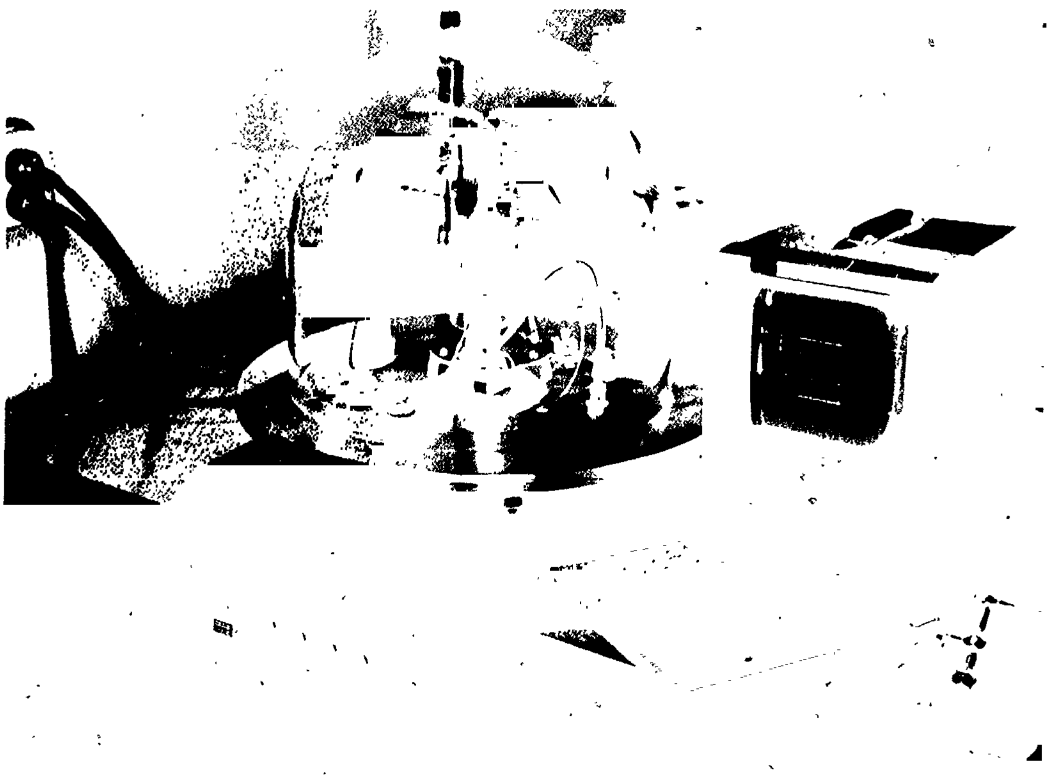

Fig. 4-1 Temperature Cycling Apparatus

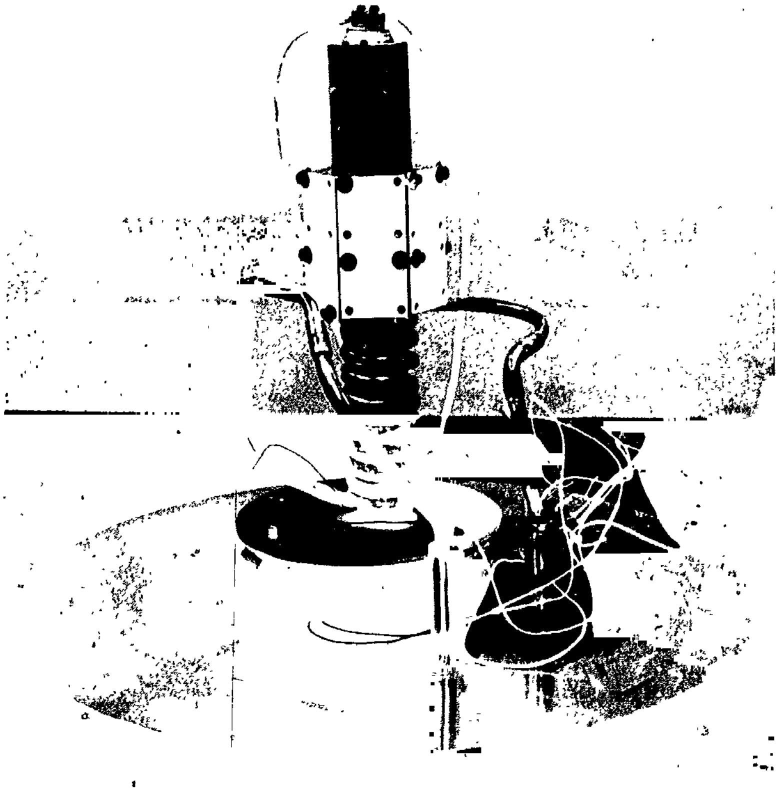

Fig. 4-2 Sample Holder

4.1.2 Sample Preparation

The test samples were coated by the supplier of the coating system using substrates furnished by LMSC. In all cases the substrates were 6061-T6 aluminum alloy discs, nominally 2.54 cm (1 in.) in diameter by 0.127 cm (0.050 in.) thick. The surface to be coated was machined to 30 ±3 rms finish. All substrates were instrumented for temperature measurement prior to coating. This consisted of inserting 0.63-cm (1/4 in.) long, No. 26 gage pins of constantan and chromel into the disc edge approximately 90 deg apart. Pins were inserted 0.32 cm (1/8 in.) and peened in place to insure mechanical and electrical integrity.

4.1.3 Procedure

All coatings were thermally cycled, in duplicate, four times from their maximum intended operating temperature to near liquid nitrogen temperature. Chamber pressure was maintained at 5×10^{-6} Torr or lower during testing. The rate of temperature change during cooling was selected to simulate the cooling of a gas radiator, with the heat source removed, in space when the radiator is in the earth's shadow. Calculations of the cooling curves were based upon a 0.159-cm (1/16 in.) thick 6061-T6 aluminum flat plate radiator having a coating with a 0.85 total hemispherical emittance on both faces. Figures 4-3 and 4-4 show the calculated cooling curves for the 395° K (710° R) and 533°K (960° R) operating temperatures and the actual cooling curves as measured during the testing. The test cycle consisted of holding the sample at test temperature (395° or 533°K) for 1/2 hr, the cooling period for 6 hr, and a 17.5 hr period during which time the sample temperature slowly increased to ambient (300°K).

Specimens were attached to the holder at the edge of the disc at two points, 180 deg apart, using No. 8-32 stainless steel screws and washers. As heating and cooling were by conduction between the copper block and substrate a silicone heat transfer grease (Dow-Corning DC-340) was placed at this interface to minimize thermal contact resistance. Sample temperatures were measured using chromel-constantan thermocouples affixed to the coating substrate. Temperatures were measured using a Minneapolis-Honeywell (SX153X-67) multipoint strip chart recorder.

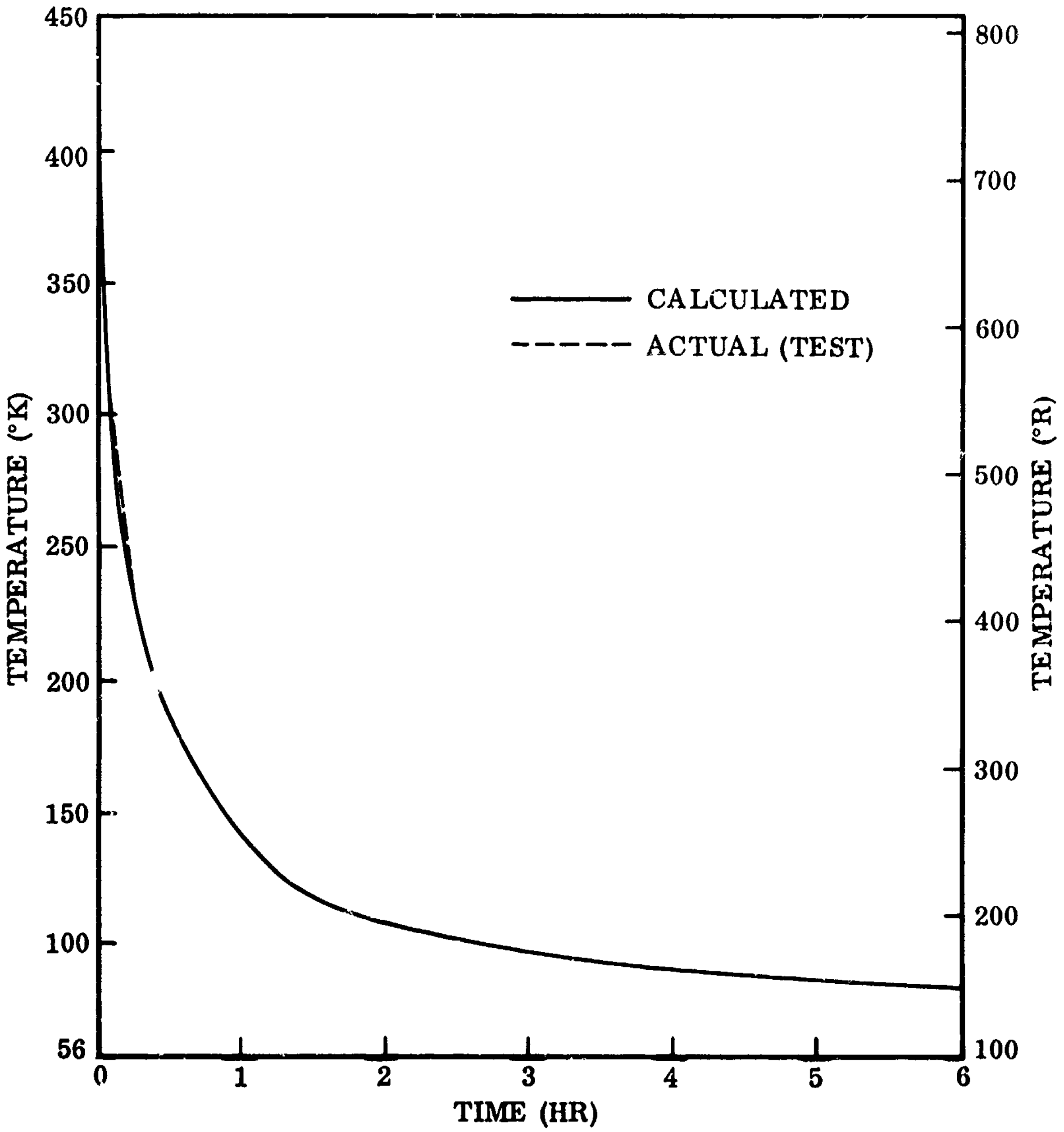

Fig. 4-3 Calculated and Actual Cooling Curve for 395°K Coatings

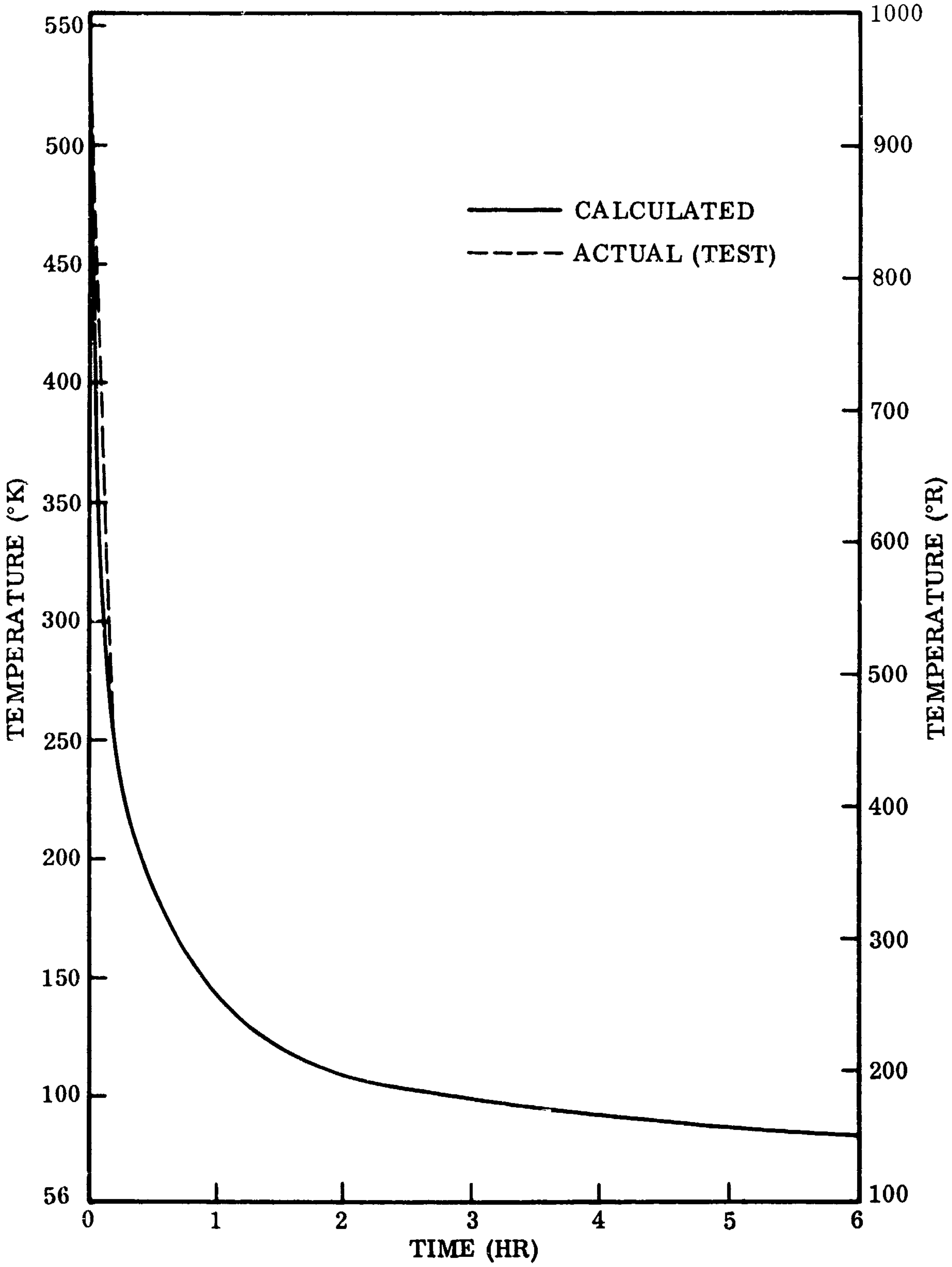

Fig. 4-4 Calculated and Actual Cooling Curve for 533°K Coatings

The test criteria was based upon visual observation of the coatings during and following testing and was no cracking or spalling evident to the unaided eye. Also pre-and post-test photographs were made of each sample at 1× and 100× magnification.

4.1.4 Results

No evidence of cracking or spallation of the coatings was observed by the unaided eye or at 100× magnification. The areas directly adjacent to the mounting screws were disregarded during the examination as clamping pressures could have introduced stresses at these points which are not relevant to the purpose of this test. Both samples of the Al-SiO_4/K_2SiO_4 coating had several areas of a slightly brown appearance during and after testing. One sample had a greater number of discolored brown spots than the other. Room temperature spectral reflectance measurements were performed on these samples after the test. The sample solar absorptances calculated from these data showed an increase of 0.04 for one specimen and 0.07 for the other. No attempt was made to determine the cause of this discoloration. However, it is extremely improbable this could be attributed to contamination by the test system. Four other specimens were tested in the chamber at the same time and they showed no evidence of discoloration.

4.2 EXPOSURE TEST

To evaluate the stability of the solar absorptance and total hemispherical emittance of candidate space power radiator coatings in the space environment, samples are exposed for a period of 500-hr duration to uv radiation at a 1-sun level, defined by the 0.2- to 0.4-μ spectral band, from a xenon source; vacuum of 6×10^{-8} Torr or less; and maximum intended operating temperature level. The result of these screening tests will form the basis for the selection of two coatings to be subjected to long-term exposure testing.

4.2.1 Apparatus

The uv exposure apparatus is described in detail in the First and Second quarterly progress reports of this contract (Refs. 4 and 5).

4.2.2 Sample Preparation

All samples were prepared as described in subsection 4.1.2 of this report and Ref. 5.

4.2.3 Procedure

The test procedure and data reduction are described in detail in the Third quarterly progress report of this contract (Ref. 6). Thermal cycling of specimens during the test was accomplished by removing both xenon and tungsten lamp beams from the specimen for 8 min. This was accomplished by using a shutter for the xenon lamp and opening the tungsten lamp electrical circuit by a switch. Both functions were controlled by a timer. Cycle interval was 4-3/4 hr, and the specimen cooled by radiative transfer to approximately 300°K (540°R) in the 8-min period. The heating from this temperature to 395°K (710°R) occurred in 2 min.

During these tests coating substrate temperatures were continuously monitored with a Leeds and Northrop AZAR W recorder. Specimen temperature control was achieved by utilizing the control slide wire of the AZAR recorder in conjunction with a Leeds and Northrop CAT controller and SCR power supply for the tungsten lamp. This system permitted control of specimen temperature to within 0.5°K of the preset set level. Overall specimen temperature measurement maximum uncertainty was ±3°K. An additional control circuit was used for the tungsten lamp during the period of measurement of the temperature excursions for obtaining absorptance data. An Eppley thermopile was switched into the control circuit in place of the sample thermocouple. This signal fed into the control units maintained a constant lamp output which eliminated perturbations in lamp intensity caused by small voltage changes which were not corrected by the constant voltage power supplies for the lamp circuits.

4.2.4 Results

Based upon the calorimetric in situ measurements the solar absorptance of the TiO_2/silicone coating, Thermatrol 2A-100, at 395°K (710°R) increased from an initial value of 0.18 to 0.32 after 500 hr of exposure to the xenon source at a 1-sun level, 0.20 to 0.40 μ. The total hemispherical emittance remained essentially constant at 0.85 ±0.03 for both samples. The change in solar absorptance $\Delta\alpha_s$ appeared to reach a saturation value of 0.14 after 300 to 400 hr of exposure at this temperature and source intensity.

The in situ absorptance data as a function of exposure time for the first test, sample No. 5, were not valid as the chamber window for the xenon source became contaminated during the run. Examination of this window at the completion of the test showed that its transmission had changed as a function of time in a manner which could not be defined so as to permit reduction of the absorptance data. The contamination appeared as a haze which occurred on the exterior surface of the window. This was readily removed by carefully cleaning the surface with a lint-free optical type cleaning paper. The problem has been resolved by daily inspection and cleaning, if required, of the window. Also, a calorimeter sample was installed directly below the test sample so that comparative intensity measurements can be made during the exposure period. This calorimeter consists of a disc of 6061-T6 aluminum, of the same dimension as the coating substrate, painted with a flat black coating, which is stable under uv exposure and vacuum conditions. By comparison of the initial calorimeter equilibrium temperature with that measured at the same time as the absorptance data is taken any change in total energy into the chamber at the sample position can be determined.

An attempt was made to calculate the change in solar absorptance for Sample No. 5 based upon pre- and post-test room temperature normal reflectance measurements using the Cary instrument (Ref. 4). Post-test measurements were made 7 min and 30 min after exposure of the sample to air (Fig. 4-5). Some recovery was evident during this period as α_s was 0.25 at 7 min and 0.24 at 30 min. A direct extrapolation to zero time of exposure to air is not possible because the time dependancy of recovery

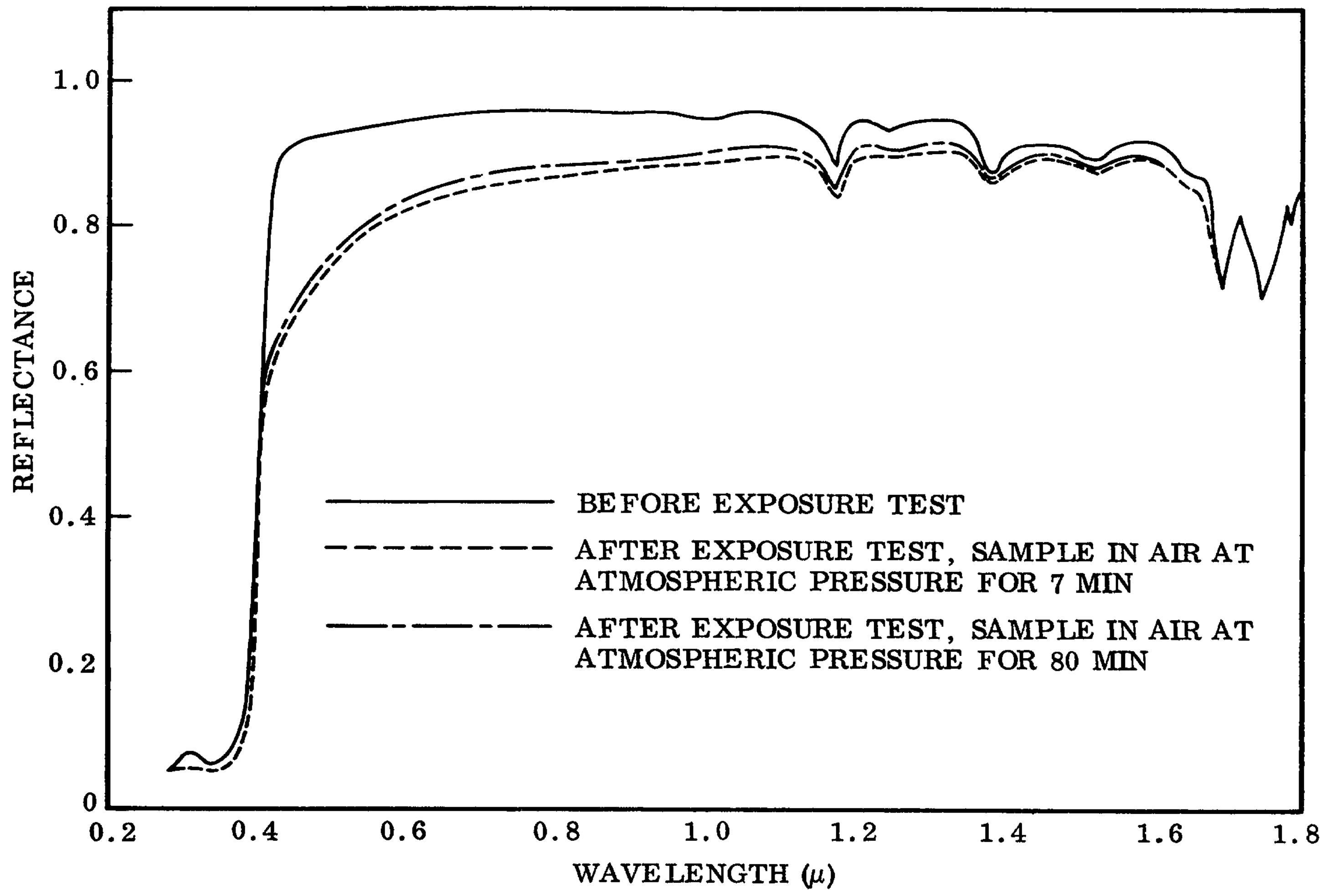

Fig. 4-5 Room Temperature Normal Spectral Reflectance of TiO_2/Silicone Sample No. 5 Before and After 500-hr Exposure Test

effects is not known. The initial value of α_s was determined to be 0.15 from the reflectance data and 0.18 by the in situ method. Sample total hemispherical emittance remained essentially constant at 0.84 ±0.01 during the entire exposure period.

Results of the in situ measurements made during the exposure test on the duplicate TiO_2/silicone Sample No. 1, are tabulated in Table 4-1. The solar absorptance and total hemispherical emittance as a function of time are shown graphically by Figs. 4-6 and 4-7. At 245 hr the electronic pump shut-off and the chamber pressure rose to greater than 20 μ. After restarting of the pump, a set of data was taken immediately upon reaching a pressure of 6×10^{-8} Torr. These data showed a decrease in α_s due principally to the recovery in the near infrared, λ greater than 0.85 μ. This recovery phenomena has previously been reported by McMillan et al. (Ref. 7).

Solar absorptance appears to reach a saturation value of 0.32, $\Delta\alpha_s = 0.14$, after 300 to 400 hr under these exposure conditions. The change in α_s during the 500-hr period is greater than that reported at a 1-sun level (AH-6 lamp) at room temperature for the same time (Ref. 7). However, the room temperature data reached approximately the saturation value.

Pre- and post-test room temperature reflectance data (Cary) are shown in Fig. 4-8. Based upon these data, initial and final values of α_s are 0.15 and 0.26. This compares with values of 0.18 and 0.32 from the in situ measurement, and an initial value of 0.17 as determined using the Gier-Dunkle integrating sphere (Ref. 4).

Table 4-1

CALORIMETRIC TOTAL HEMISPHERICAL EMITTANCE AND IN-SITU ABSORPTANCE DATA FOR TiO_2/SILICONE COATING, SAMPLE NO. 1, AT 395°K

Time (hr)	Vacuum (Torr)	ϵ_{TH}	α_H for Spectral Band, $\lambda^{(a)}$					α_s
			Total	0.20/0.41	0.41/0.60	0.60/0.85	0.85/-	
1/4	5×10^{-8}	0.87	0.18	0.70	0.12	0.07	0.13	0.18
5	5×10^{-8}	0.87	0.21	0.70	0.17	0.07	0.19	0.21
69	5×10^{-8}	0.87	0.29	0.85	0.25	0.16	0.22	0.29
117	5×10^{-8}	0.85	0.30	0.85	0.24	0.18	0.26	0.31
168	5×10^{-8}	0.88	0.31	0.85	0.25	0.18	0.28	0.32
250(c)	5×10^{-8}	0.88	0.29	0.85	0.25	0.17	0.24	0.30
360	5×10^{-8}	0.87	0.29	0.80	0.24	0.17	0.27	0.31
410	5×10^{-8}	0.85	0.30	0.85	0.25	0.18	0.26	0.31
457	5×10^{-8}	0.86	0.30	0.85	0.24	0.18	0.26	0.31
486	5×10^{-8}	0.85	0.31	0.90	0.25	0.18	0.27	0.32
550	5×10^{-8}	0.85	0.31	0.90	0.25	0.19	0.27	0.32
Before Exposure(b)			0.14	0.69	0.08	0.06	0.07	0.15
After Exposure(b)			0.24	0.79	0.27	0.15	0.13	0.26

(a) Absorptance for xenon lamp spectrum

(b) Calculated from room temperature normal reflectance measurement made with the Cary 14 spectrophotometer

(c) Electronic pump shut off at 245 hr chamber pressure increased to $>20\mu$.

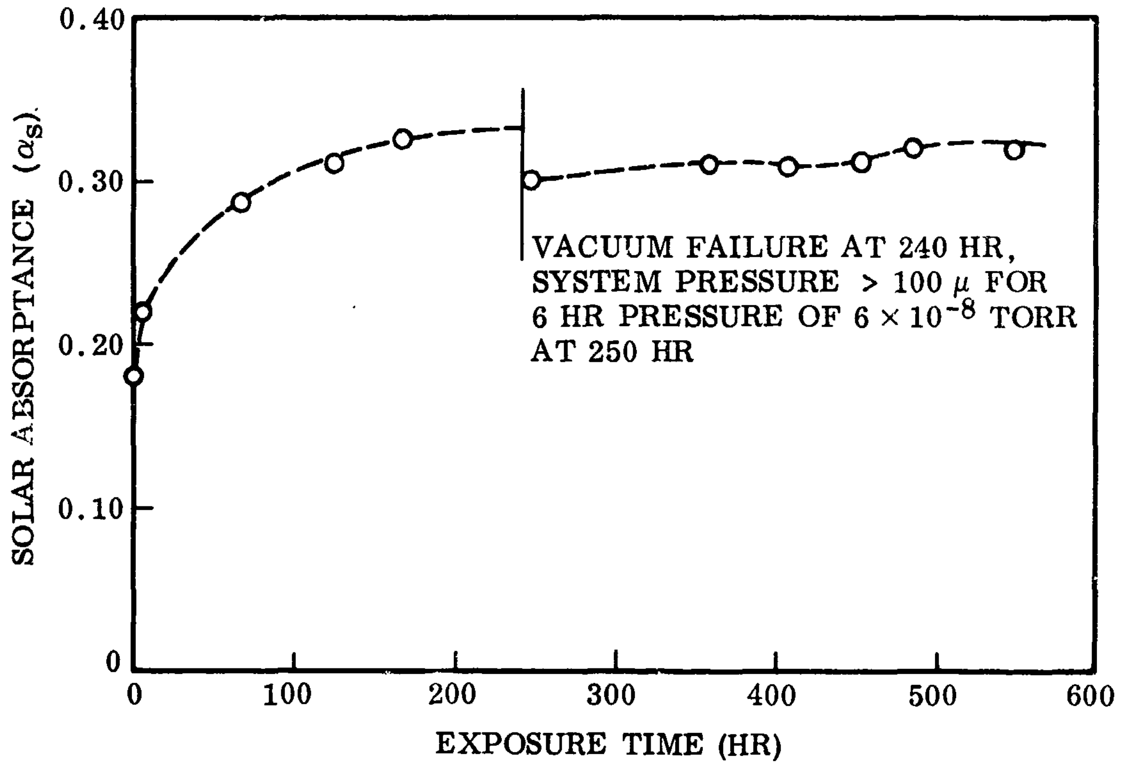

Fig. 4-6 Solar Absorptance of TiO_2/Silicone Coating as a Function of Exposure Time at a 1-Sun Level (0.2 to 0.4 μ, Xenon Source). Chamber pressure 6 × 10^{-8} Torr, Sample Temperature 395°K

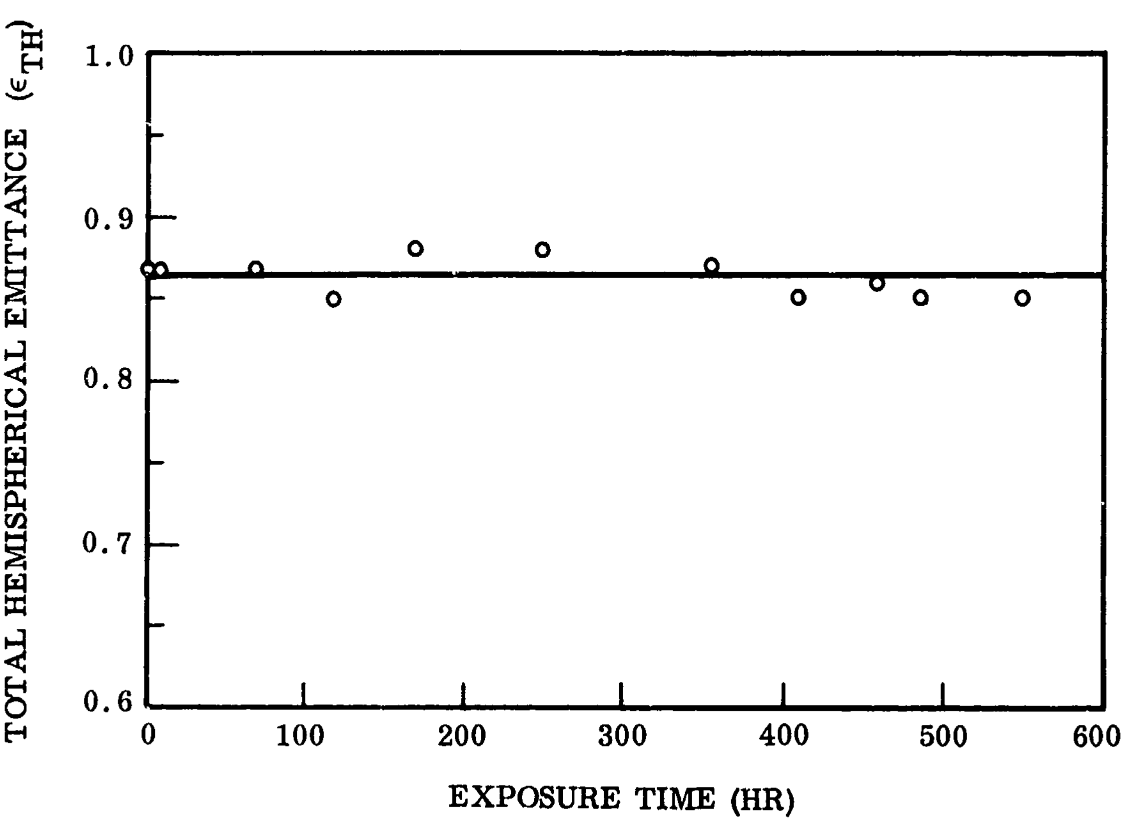

Fig. 4-7 Total Hemispherical Emittance of TiO_2/Silicone Coating as a Function of Exposure Time at 1-Sun Level and 395°K

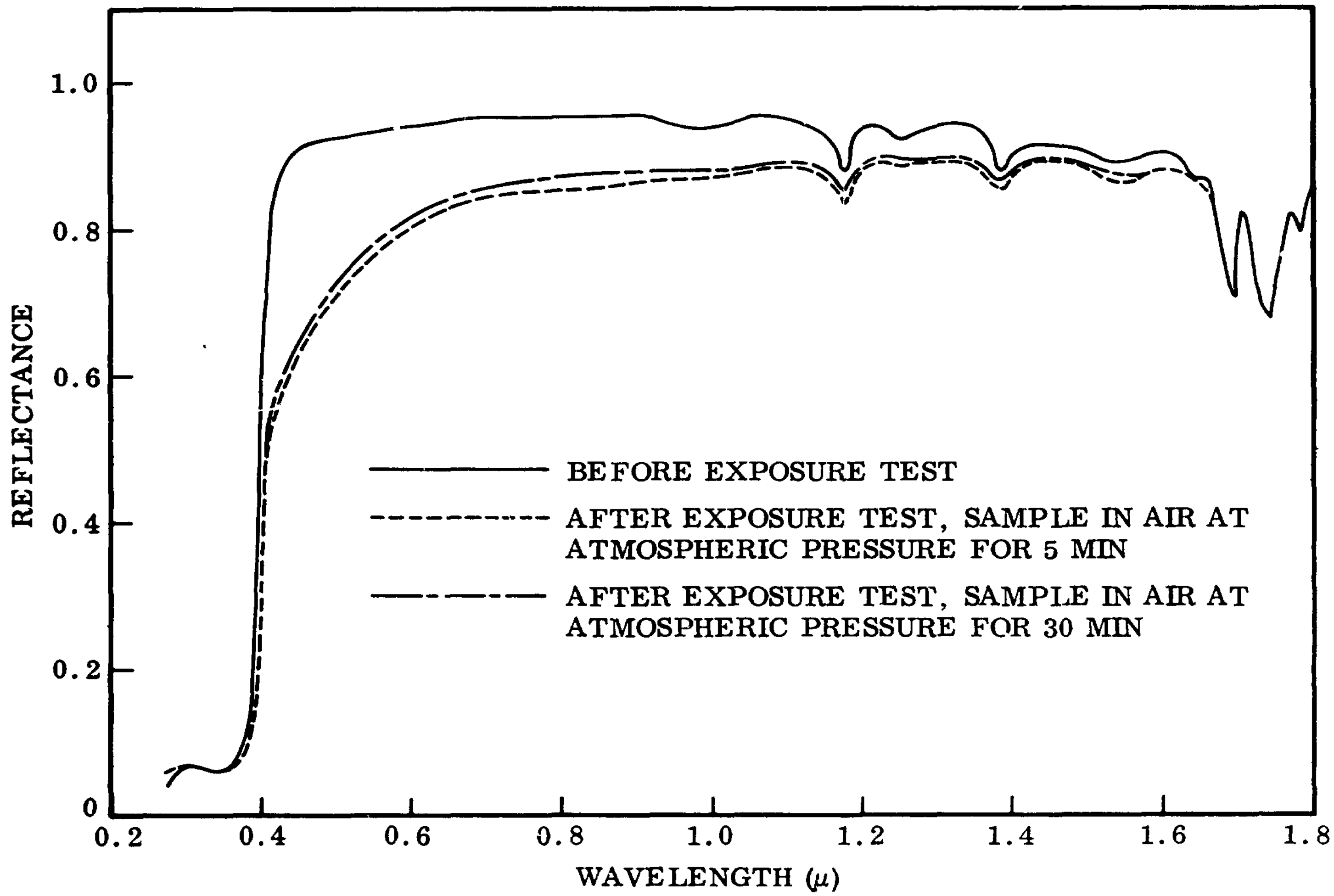

Fig. 4-8 Room Temperature Normal Spectral Reflectance of TiO_2/Silicone Sample No. 1 Before and After 500-hr Exposure Test

Section 5

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