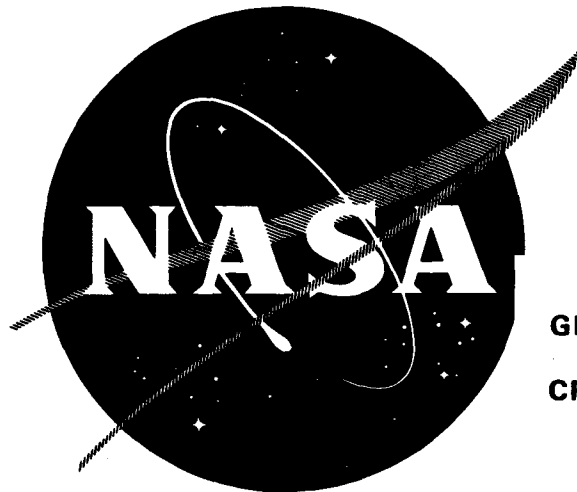


CR-72060

NASA-CR-72060



GPO PRICE \$ _____

CFSTI PRICE(S) \$ _____

Hard copy (HC) 3.00

Microfiche (MF) .65

ff 653 July 65

EMISSION COATINGS FOR LOW-TEMPERATURE SPACE RADIATORS

Quarterly Progress Report No. 3
For Quarter Ending 31 March 1966

CONTRACT NAS-3-7630

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

FACILITY FORM 802

N67 14914	
(ACCESSION NUMBER)	
37	(THRU)
(PAGES)	1
CR-72060	(CODE)
(NASA CR OR TMX OR AD NUMBER)	18
	(CATEGORY)

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

NOTICE

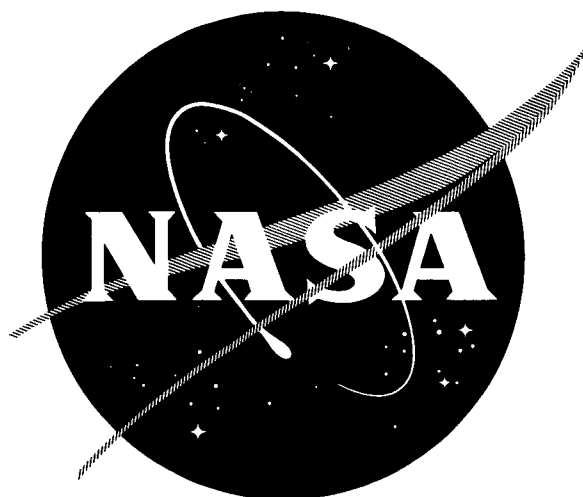
This report was prepared as an account of Government sponsored work. Neither the United States, nor the National Aeronautics and Space Administration (NASA), nor any person acting on behalf of NASA:

- A. Makes any warranty or representation, expressed or implied, with respect to the accuracy, completeness, or usefulness of the information contained in this report, or that the use of any information, apparatus, method, or process disclosed in this report may not infringe privately owned rights; or
- B. Assumes any liabilities with respect to the use of, or for damages resulting from the use of any information, apparatus, method or process disclosed in this report.

As used above, "person acting on behalf of NASA" includes any employee or contractor of NASA, or employee of such contractor, to the extent that such employee or contractor of NASA, or employee of such contractor, has access to, any information pursuant to his employment or contract with NASA, or his employment with such contractor.

Requests for copies of this report
should be referred to:

National Aeronautics and Space Administration
Office of Scientific and Technical Information
Washington 25, D. C.
Attention: AFSS-A



EMISSIVITY COATINGS FOR LOW-TEMPERATURE SPACE RADIATORS

**Quarterly Progress Report No. 3
For Quarter Ending 31 March 1966**

CONTRACT NAS-3-7630

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

**Prepared by:
F. J. Smith and J. G. Grammer
Sr. Thermodynamics Engineers**

**Approved by:
G. R. Cunningham
Project Leader**

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

FOREWORD

The results of the research activities carried out during the third quarterly period (1 January through 31 March 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.

CONTENTS

Section		Page
	FOREWORD	ii
	ILLUSTRATIONS	iv
	TABLES	iv
1	INTRODUCTION AND SUMMARY	1
2	LITERATURE SURVEY	3
3	EXPERIMENTAL RESULTS	5
	3.1 Apparatus	5
	3.2 Operation	7
4	CANDIDATE COATINGS	22
	4.1 Coating Descriptions	22
	4.1.1 S-13 Modification II (ZnO/Silicone)	22
	4.1.2 Z-93 (ZnO/K ₂ SiO ₄)	24
	4.2 Initial Optical Properties	26
5	REFERENCES	30

ILLUSTRATIONS

Figure		Page
3-1	Improved Tungsten Source	6
3-2	Proposed Cooling Curve for Thermal Cycling Test	8
3-3	Enthalpy of 6061T-6	10
3-4	Specific Heat 6061T-6	11
3-5	Spectral Radiant Intensity of Xenon Lamp and Transmission of Filters	17
4-1	Typical Spectral Reflectance of ZnO/Silicone Coating	28
4-2	Typical Spectral Reflectance of ZnO/K ₂ SiO ₄ Coating	29

TABLES

Table		Page
3-1	Comparison of Spectral Irradiance at a Distance of 50 cm	9
4-1	Initial Optical Properties	27

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 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 temperatures, and temperature cycling. During the first phase of the program, candidate materials will be exposed simultaneously to ultraviolet radiation at a 1-sun level, maximum operating temperature, and vacuum for 500-hr periods. The effects of temperature cycling will also be 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.

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

- Continuation of the literature survey
- Establishment of a proposed cooling profile for the temperature cycling test
- Determination of the specific heat of the coating substrate material
- Spectral calibration of a tungsten-iodide lamp
- Updating the exposure apparatus in accordance with findings from the initial operations
- Study of the feasibility of making in situ broad band spectral absorptance measurements

Detailed discussions of activities for this quarter are presented in the following sections.

Section 2

LITERATURE SURVEY

Data relevant to the performance of candidate coatings for this program have become available with the publication of new reports on related contract efforts. Under an Air Force Contract [AF 33(615)-1634] the results of a study of the effects of u-v radiation on the properties of cryogenic temperature thermal control coatings were disclosed (Ref. 1). Of immediate interest to this program was the fact that many of the coatings studied were identical to those examined under this program. The recurrent theme so evident in recent reports, that in situ measurements indicate greater degradation than pre- and post-test optical property measurements, was again apparent. Calorimetric measurements, both at cryogenic temperatures and during thermal cycling provided profiles of the u-v radiation damage kinetics in terms of changes in absorptance.

A series of reports from IITRI confirmed prior LMSC findings on the rapid recovery of u-v damaged zinc oxide/silicone paints (S-13) upon re-exposure to air (Refs. 2 and 3). Attempts to modify the pigment by surface treatment and doping were reported to have met with varying degrees of success. Silicate treatment of the ZnO apparently inhibited the rapid and extensive damage in the infra-red. However, no data on the damage in the visible and u-v regions were reported.

Related studies have also been performed at LMSC but to date have not been released. The results, however, are included since they are of direct interest to this program. In situ reflectance measurements have been performed on ZnO and TiO₂ pigmented silicone paints (S-13 and Thermatrol, respectively) under 1, 10, and 20 sun intensities. The data show conclusively that previous assumptions of reciprocity are not necessarily valid, i.e., that a 1-sun exposure for 10 hr does not produce the same amount of damage as a 10-sun exposure for 1 hr. Not only is the extent of damage (as deduced from α_s) not equivalent, but the coatings exhibit markedly different spectral characteristics under varying intensity. However, after extended exposures (> 1000 equiv. sun-hr) the differences in degradation are minimal.

The pertinent references detailed here tend to reinforce the conclusion that valid engineering data can only be obtained by in situ techniques. While calorimetric measurements performed in vacuum can provide information related to total degradation, $\Delta\alpha_s$, spectral data provides greater insight into the nature of the damage processes.

Section 3

EXPERIMENTAL RESULTS

3.1 APPARATUS

The experimental apparatus described in Refs. 4 and 5 has been improved with a modification of the tungsten source and the addition of a LMSC designed radiometer.

The original energy source consisted of a 750-W tungsten filament spot lamp and associated optics. The nature of the system was such that focusing was critical and the lamp was operating at nearly full power. In the interest of increased reliability and improved control, this system was improved by substitution of a 1000-W tungsten iodide lamp (GE DXW) and the replacement of the optical system with a light pipe (Fig. 3-1). The light pipe system consists of a short, external 1-1/8 in. diameter aluminum tube, polished on the inside, which extends from the lamp to the exterior of the chamber entrance port. The internal light pipe is a 1-1/8 in. diameter copper tube extending from the chamber window to the sample. The tube is supported by attachment to the LN_2 cold wall and is polished on the inside while the outside is coated with a diffuse black paint. The end of the tube adjacent to the sample is fitted with a sapphire window. The optical and thermal properties of the window are such that it appears, to the sample, tube identical to the LN_2 cold wall. This has been verified experimentally in the apparatus by measuring the sample emittance with sample in the normal position then rotating the sample 90 deg and remeasuring the emittance. A comparison of the results indicates no errors are introduced by the tungsten source light pipe.

A spectral radiometer has been built to allow the xenon u-v source lamps to be periodically compared to a NBS traceable standard. This radiometer utilizes a Bausch and Lomb No. 33-86-25 monochromator with four interchangeable gratings which cover

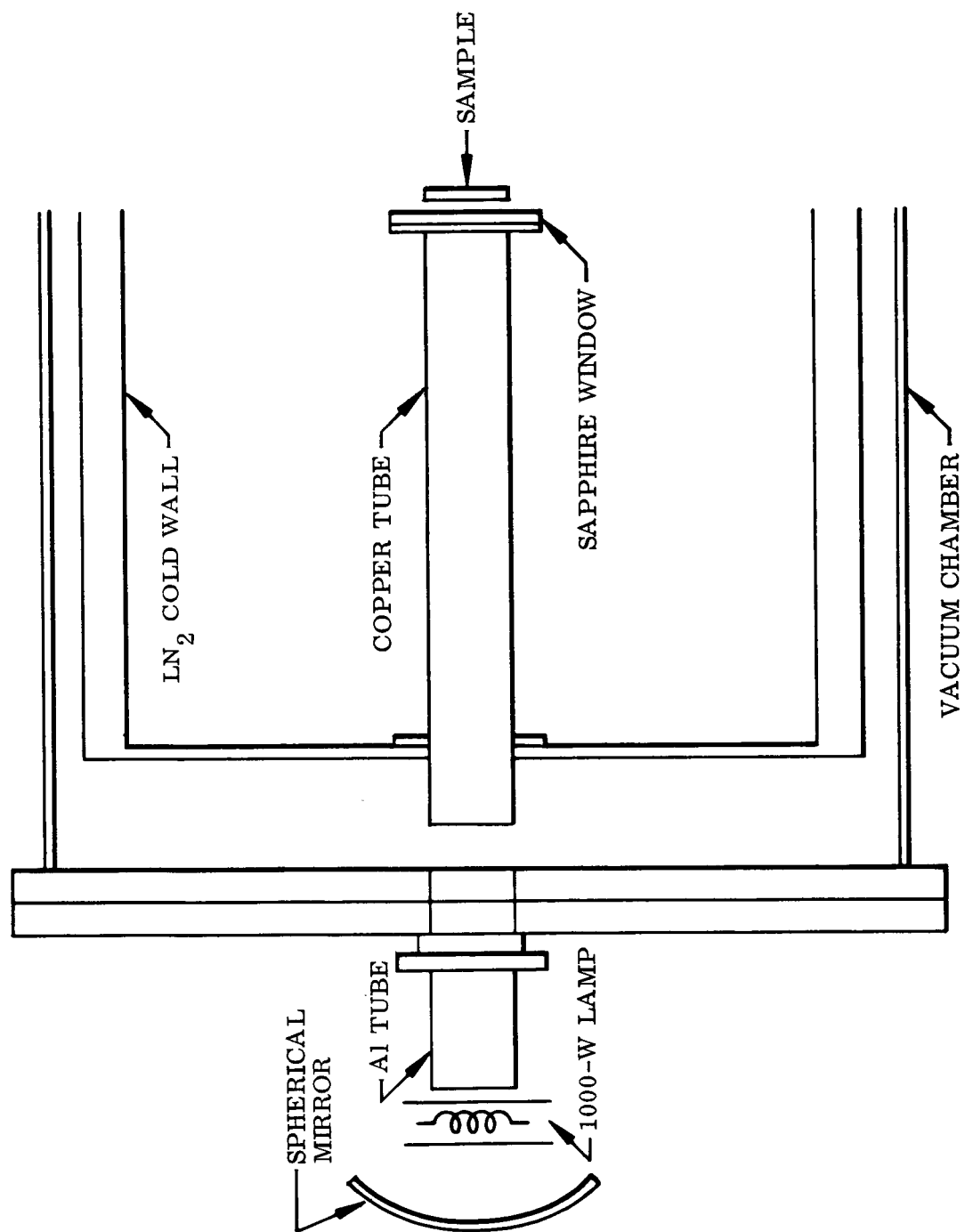


Fig. 3-1 Improved Tungsten Source

the wavelength range 0.2 to 3.2 μ . The incident energy is diffusely reflected off a MgO plate onto the entrance slit. The detector unit houses a 1P-21 photomultiplier detector and a lead sulphide detector, with associated optical focusing and switching.

The unit will be calibrated against a 1000-W tungsten iodide lamp which has been calibrated by comparison to a NBS certified calibrated lamp.

3.2 OPERATION

Four major items have been accomplished during the report period: (1) establishment of a proposed thermal profile for the thermal cycling testing, (2) determination of the specific heat of the sample substrate; (3) spectral calibration of a tungsten iodide lamp, and (4) an investigation of the feasibility of in situ broad band spectral absorptance measurements.

Figure 3-2 shows the proposed cooling curve for the thermal cycling testing. The calculations were based on a 1/16 in. thick 6061 T-6 aluminum substrate coated on both surfaces with a coating having a total hemispherical emittance of 0.85. The test will be started by holding the sample at the test temperature (250 or 500°F) for 1/2 hr. The cooling cycle will be for 6 hr, at which time the LN₂ flow will be stopped and the specimen allowed to approach ambient temperature.

The specific heat of the 6061 T-6 aluminum from which the coating substrates were machined has been determined. The specific heat is required to determine the thermal capacity of the samples in order to compute the optical properties of the candidate coatings from the measured time-temperature histories.

The enthalpy, ΔH_{32} of the material is measured using a drop calorimeter (Ref. 6). The specific heat is determined from the slope of a least square fit of the data to an equation of the form

$$\Delta H_{32} = a_0 + a_1 T^{-1} + a_2 T + a_3 T^2$$

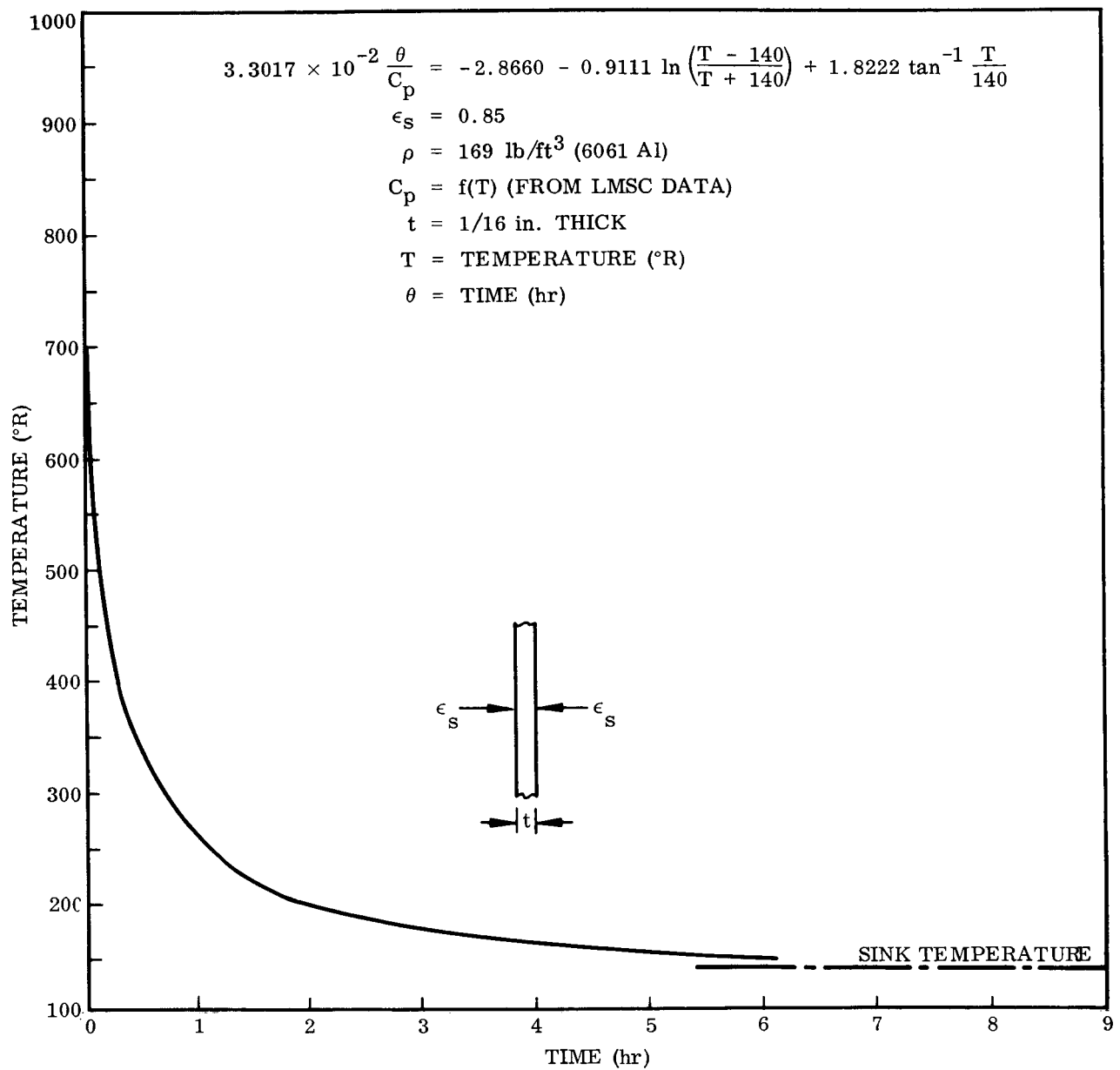


Fig. 3-2 Proposed Cooling Curve for Thermal Cycling Test

The computation was performed using a digital computer. The enthalpy data are presented in Fig. 3-3 and the specific heat data in Fig. 3-4.

A spectral irradiance calibration of an LMSC tungsten iodide lamp (General Electric DX W-1000-Watt) has been obtained. This lamp will serve as a laboratory standard. The calibration was obtained by comparison with a similar lamp at NASA Ames Research Center which has NBS traceability. The comparison was made by using an Engis Equipment Co., Model S-05-02 monochromator with a 1P-28 photomultiplier detector.

The data are presented in Table 3-1.

Table 3-1
COMPARISON OF SPECTRAL IRRADIANCE AT A DISTANCE
OF 50 CM

$\lambda(\mu)$	Irradiance (microwatts/cm ² -nanometer)		
	NASA Ames Lamp EPI-1017	LMSC Lamp	Deviation From Ames Lamp (%)
0.25	0.0199	0.0196	-1.5
0.26	0.0337	0.0330	-2.1
0.27	0.0573	0.0552	-3.8
0.28	0.0950	0.0927	-2.4
0.29	0.146	0.140	-4.1
0.30	0.211	0.200	-5.2
0.32	0.375	0.363	-3.2
0.35	0.848	0.817	-3.7
0.37	1.30	1.24	-4.6
0.40	2.24	2.19	-2.2
0.45	4.52	4.40	-2.6
0.50	7.47	7.33	-1.2

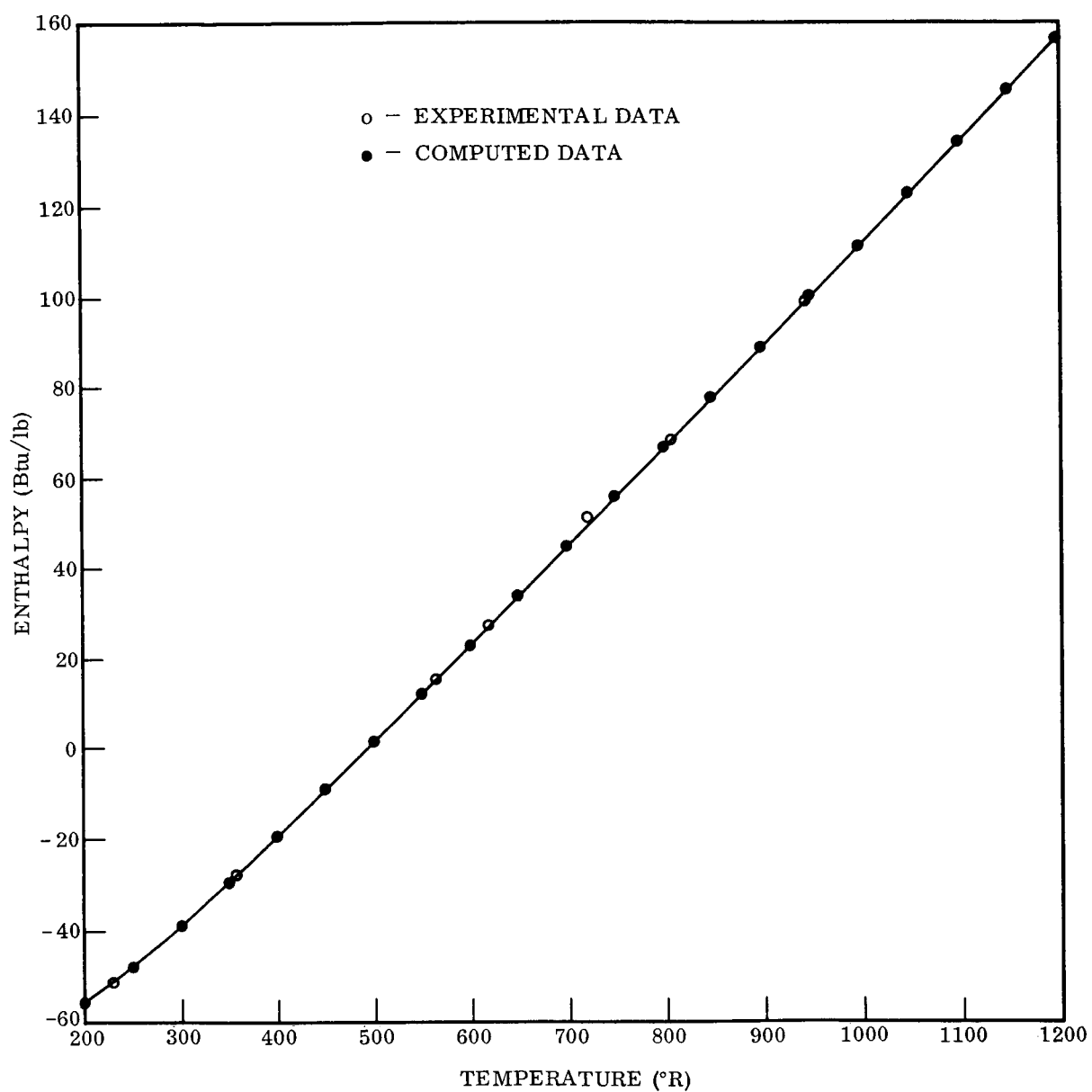


Fig. 3-3 Enthalpy of 6061T-6

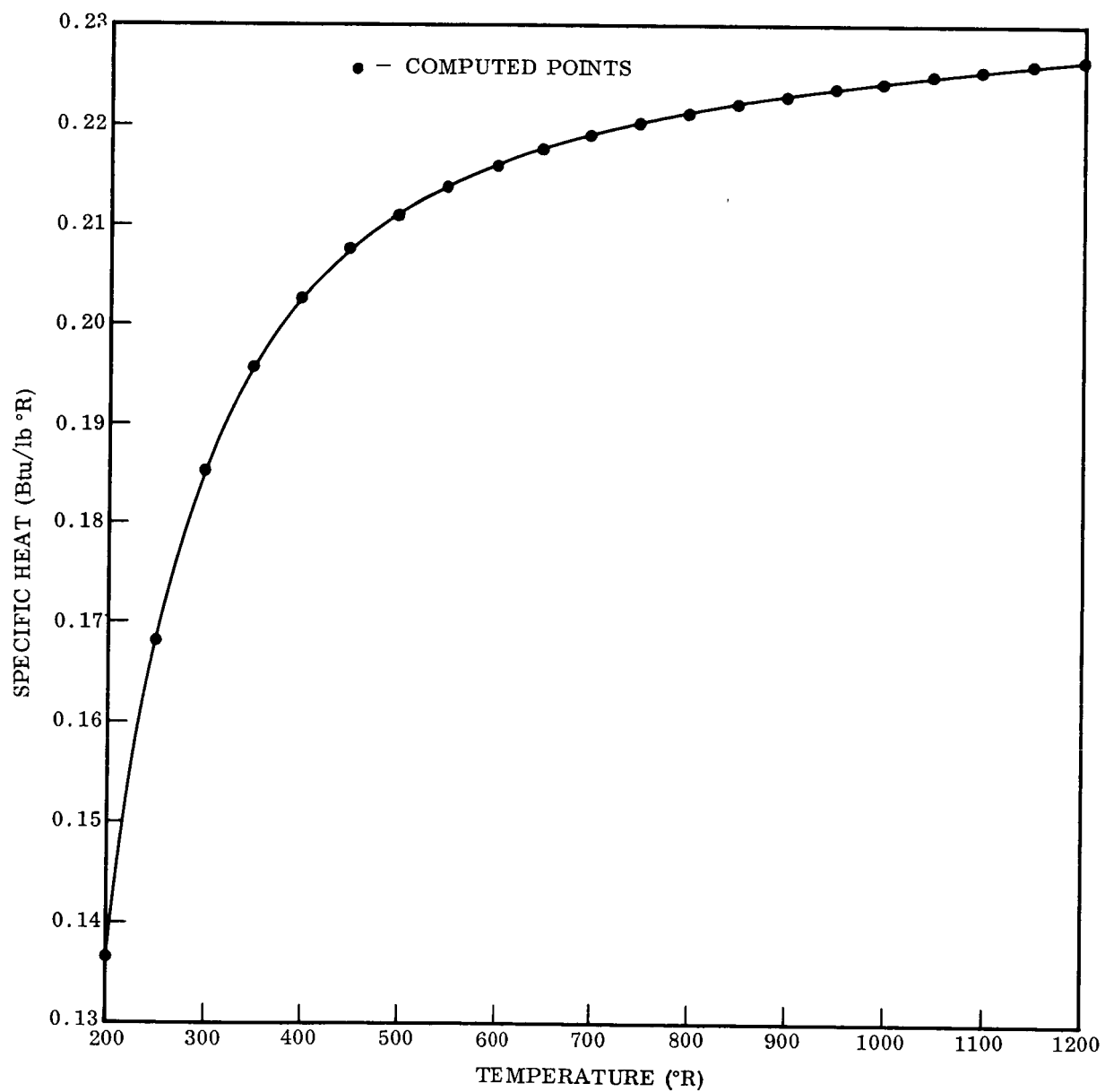


Fig. 3-4 Specific Heat 6061T-6

An investigation of the feasibility of making in situ broad band spectral absorptance measurements using optical filters was undertaken at the request of the NASA project manager. It has been determined that the measurements are feasible and desirable and will provide data that (1) will indicate the spectral ranges in which u-v damage is taking place and (2) will provide in situ spectral data which will aid in converting the measured, source absorptance, α_H , to the desired solar absorptance, α_s . The method including an error analysis is presented below.

Method. For a disk sample having one side painted, while the edge and other side are polished aluminum surfaces, the thermal behavior is described by the following equation:

$$mc_p \frac{dT}{d\theta} = G_H A_H \alpha_H - \epsilon_H A_H \sigma T^4 - (A_e + A_t) \epsilon_t \sigma T^4 - CA_w T + G_t A_t \alpha_t$$

where

mc_p = sensible heat of specimen

$\frac{dT}{d\theta}$ = rate of change of absolute temperature with time

$G_H A_H \alpha_H$ = source energy absorbed by painted surface A_H with source absorptance α_H

$\epsilon_H A_H \sigma T^4$ = energy emitted by painted surface area A_H at temperature T with surface emittance ϵ_H

$(A_e + A_t) \epsilon_t \sigma T^4$ = energy emitted by the back surface area A_t and the edge surface A_e at temperature T with surface emittance ϵ_t

$CA_w T$ = energy transferred from the sample by thermocouple lead wires

$G_t A_t \alpha_t$ = tungsten lamp irradiation G_t , absorbed by sample back surface A_t with absorptance α_t

If the sample is held at temperature by irradiation from the tungsten lamp on the back surface, then exposed to irradiation of the front surface by a solar simulator source, the heating response is

$$mc_p \left(\frac{dT}{d\theta} \right)_h = G_H A_H \alpha_H - \epsilon_H A_H \sigma T^4 - (A_e + A_t) \epsilon_t \sigma T^4 - CA_w T + G_t A_t \alpha_t$$

Then by blanking off the solar simulator source by a shutter, the cooling response is

$$mc_p \left(\frac{dT}{d\theta} \right)_c = -\epsilon_H A_H \sigma T'^4 - (A_c + A_t) \epsilon_t \sigma T'^4 - CA_w T' + G_t A_t \alpha_t$$

The following equation is obtained by subtracting the cooling response from the heating response:

$$mc_p \left[\left(\frac{dT}{d\theta} \right)_h - \left(\frac{dT}{d\theta} \right)_c \right] = G_H A_H \alpha_H \quad \text{for} \quad T' = T$$

Let

$$\left(\frac{dT}{d\theta} \right)_h = R_h \quad \text{and} \quad \left(\frac{dT}{d\theta} \right)_c = R_c,$$

then

$$mc_p (R_h - R_c) = G_H A_H \alpha_H$$

and

$$\alpha_H = \frac{mc_p (R_h - R_c)}{G_H A_H}$$

Values of α_H are obtained for the source being used. From the spectral irradiance, H_λ , incident on the sample and from spectral reflectance data, a multiplicative factor N may be derived which converts α_H to α_s , the solar absorptance.

Consider the value of α_H obtained above, which may be written as

$$\alpha_H = \frac{1}{H} \int_0^\infty \alpha_\lambda H_\lambda d\lambda$$

The desired value is

$$\alpha_s = \frac{1}{s} \int_0^{\infty} \alpha_{\lambda} s_{\lambda} d\lambda$$

and the desired conversion factor is

$$N = \frac{\alpha_s}{\alpha_H} = \frac{H}{s} \frac{\int_0^{\infty} \alpha_{\lambda} s_{\lambda} d\lambda}{\int_0^{\infty} \alpha_{\lambda} H_{\lambda} d\lambda}$$

Thus

$$\alpha_s = \alpha_H N$$

The spectral absorptance of the sample, which is required in the above analysis, may be obtained from measurement of the spectral reflectance:

$$\alpha_{\lambda R} = 1 - \rho_{\lambda R}$$

where the subscript, R, denotes the value was derived from reflectance measurements.

Changes from the values of the measured absorption factors, $\alpha_{\lambda}'s$, may be of two types: a change which is not a function of wavelength, e_o , or one which varies with wavelength, e_{λ} ; thus, the following expression may be written:

$$\alpha_{\lambda} = (1 + e_o) (1 + e_{\lambda}) \alpha_{\lambda R}$$

The conversion factor then is

$$N = \frac{H}{S} \frac{1 + e_0}{1 + e_0} \frac{\int_0^{\infty} \alpha_{\lambda} R (1 + e_{\lambda}) S_{\lambda} d\lambda}{\int_0^{\infty} \alpha_{\lambda} R (1 + e_{\lambda}) H_{\lambda} d\lambda}$$

Thus

$$N = \frac{H}{S} \frac{\int_0^{\infty} \alpha_{\lambda} R (1 + e_{\lambda}) S_{\lambda} d\lambda}{\int_0^{\infty} \alpha_{\lambda} R (1 + e_{\lambda}) H_{\lambda} d\lambda}$$

Thus, the conversion factor is not dependent upon shifts of the total absorption spectrum, but only upon changes which vary with wavelength. If the absorptance change is monitored, then

$$N = \frac{\alpha_S}{\alpha_H}$$

Procedure. Determination of α_S is accomplished by the following procedure: the sample is intermittently heated and cooled by opening and closing a shutter which blocks entry of the simulated solar energy into the chamber. During this time the sample continues to receive energy at a constant level from the tungsten source. The slope of the heating and cooling curves are determined as the sample passes through a given temperature. The source absorptance α_H is then computed from the known slopes, knowledge of mc_p and measurement of $G_H A_H$. The determined α_H is then

converted into a solar absorptance from knowledge of the spectral reflectance and inferred absorptance of the sample surface, and the spectral distribution of the source lamp. Since a knowledge of the spectral absorptance of the sample surface is required for converting from a source absorptance to solar absorptance then the relative changes of the spectral absorptance must be monitored. It is not sufficient to measure the reflectance (and thus the inferred absorptance), before and after and interpolate the changes with time, since many particulate samples recover near their original spectral distribution after removal from vacuum environment. A basis for shifting the spectral distribution of the sample absorptance may be derived from a measurement of broad band absorption. Narrow band absorption measurements are not practical since the thermocouple sensor does not have sufficient temperature resolution for the low energy level.

Sharp cut-off filters may be used to determine broad band absorptances. The source absorptance α_H may be determined as a sum of portions of the total source absorptance as in Fig. 3-5. These portions or wavelength intervals absorptances may be determined by the use of sharp cut-off filters and a knowledge of their absolute transmissions and the spectral distribution of the source.

The heating and cooling response of the sample is measured for the source energy, then as each successive filter is used, an α is calculated for the transmitted beam. These are then subtracted from the total absorptance to determine the band absorptance.

Error Analysis. The total source absorptance is determined from the following equation

$$\alpha_H = \frac{mc_p (R_h - R_c)}{G_H A_H}$$

The maximum probable error of a variable $M = f(w, y, z)$ is given by the relation

$$\Delta M = \left[\left(\frac{\partial M}{\partial x} \right)^2 + \left(\frac{\partial M}{\partial y} \right)^2 + \left(\frac{\partial M}{\partial z} \right)^2 \right]^{1/2}$$

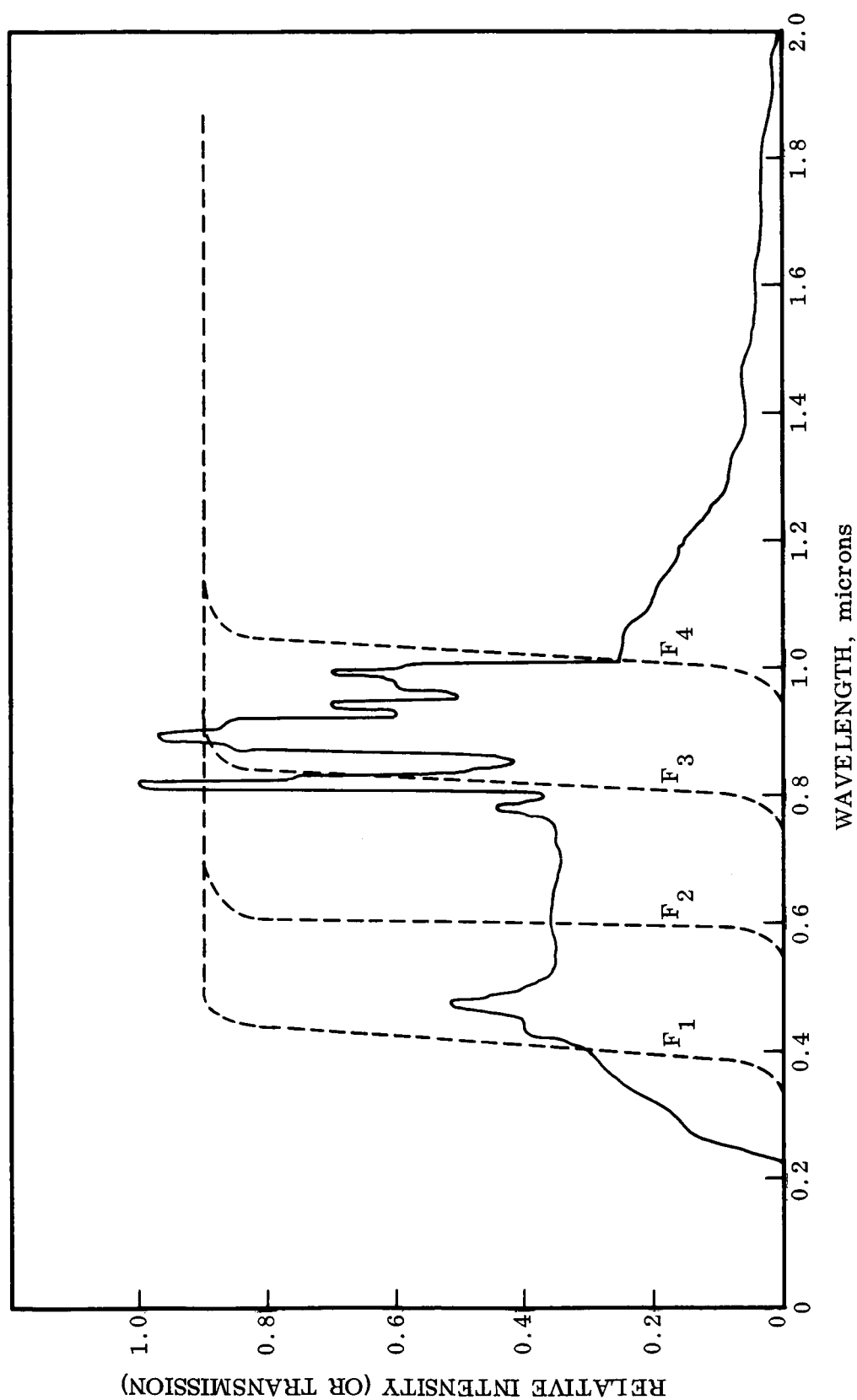


Fig. 3-5 Spectral Radiant Intensity of Xenon Lamp and Transmission of Filters

Thus, for the above equation we have the maximum probable error in α_H given by

$$\frac{\Delta \alpha_H}{\alpha_H} = \left[\left(\frac{\Delta m}{m} \right)^2 + \left(\frac{\Delta c_p}{c_p} \right)^2 + \left(\frac{\Delta G}{G} \right)^2 + \left(\frac{\Delta A}{A} \right)^2 + 2 \left(\frac{\Delta R}{R_h - R_c} \right)^2 \right]^{1/2}$$

Sample calculation:

$$\begin{aligned} m &= 1.70 \text{ gm} & \Delta c_p / c_p &= 0.02 \\ c_p &= 0.84 \text{ J/gm}^\circ\text{C} & \Delta G / G &= 0.03 \\ \alpha_H &= 0.20 & \Delta R &= 5 \times 10^{-3} \text{ }^\circ\text{C/sec} \\ G_H A_H &= 1.0 \text{ W} & \Delta m / m &= 0.001 \\ R_h - R_c &= 7 \times 10^{-1} \text{ }^\circ\text{C/sec} & \Delta A / A &= 0.001 \\ \alpha_H / \alpha_H &= 5.7\% \end{aligned}$$

The total solar absorptance is determined from

$$\alpha_s = \alpha_H N$$

where

$$N = \frac{\alpha_s}{\alpha_H}$$

where

$$\alpha_s = \frac{1}{S} \int_0^\infty \alpha_{\lambda R} (1 + e_\lambda) S_\lambda d\lambda$$

$$\alpha_H = \frac{1}{H} \int_0^\infty \alpha_{\lambda R} (1 + e_\lambda) H_\lambda d\lambda$$

The selected ordinate method is employed to determine the value of the above integrals. In this method intervals of the spectrum of the simulator source and the extra-terrestrial sun are selected that correspond to uniform increments of energy.

The spectral absorptance of the sample is weighted equally in each such interval. The evaluation of the integrals may then be accomplished by a summation of a finite number of selected ordinates of the absorption curve. The values of S_λ and H_λ are known with an uncertainty of approximately 2%. If the absorptance change is monitored, this uncertainty will also be approximately 2%. Thus, a value of 4% is reasonable for the uncertainty in the values of α_s and α_H . Thus, we have

$$\frac{\Delta N}{N} = \left[\left(\frac{\Delta \alpha_{sR}}{\alpha_{sR}} \right)^2 + \left(\frac{\Delta \alpha_{HR}}{\alpha_{HR}} \right)^2 \right]^{1/2} = 5.7\%$$

and from $\alpha_s = N\alpha_H$

$$\frac{\Delta \alpha_s}{\alpha_s} = \left[\left(\frac{\Delta N}{N} \right)^2 + \left(\frac{\Delta \alpha_H}{\alpha_H} \right)^2 \right]^{1/2} = 8.0\%$$

Wide Band Absorption Measurements:

Consider filter which transmits all the source radiant energy above 0.4μ .

Probable error calculation

$m = 1.7 \text{ gm}$	$\Delta m/m = 1 \times 10^{-3}$
$c_p = 8.4 \text{ J/gm}^\circ\text{C}$	$\Delta A/A = 1 \times 10^{-3}$
$\alpha_H = 0.20$	$\Delta G/G = 3 \times 10^{-2}$
$G_H A_H = 0.83 \text{ W}$	$\Delta c_p/c_p = 2 \times 10^{-2}$
$R_h - R_c = 1.2 \times 10^{-1} \text{ }^\circ\text{C/sec}$	$\Delta R = 5 \times 10^{-3} \text{ }^\circ\text{C/sec}$

$$\frac{\Delta\alpha_{H_1}}{\alpha_{H_1}} = \left[\left(\frac{\Delta m}{m}\right)^2 + \left(\frac{\Delta c_p}{c_p}\right)^2 + \left(\frac{\Delta G}{G}\right)^2 + \left(\frac{\Delta A}{A}\right)^2 + \left(\frac{\Delta T}{T}\right)^2 + \left(\frac{2\Delta R}{R_h - R_c}\right)^2 \right]^{1/2}$$

Where T is the transmission of filter and ΔT its uncertainty

$$\frac{\Delta\alpha_{H_1}}{\alpha_{H_1}} = 7.2\%$$

For a filter which transmits all the source radiant energy above 0.6μ the probable error is

$$\frac{\Delta\alpha_{H_2}}{\alpha_{H_2}} = 8.7\%$$

For a filter which transmits all the source radiant energy above 0.8μ the probable error is

$$\frac{\Delta\alpha_{H_3}}{\alpha_{H_3}} = 12\%$$

Conclusions. From the error analysis it appears that wide band source absorption measurements can be made with sufficient accuracy to warrant their inclusion in the measurement program. Using the Corning filters, 3-75, 2-63, and 7-57, it is possible to isolate the spectral regions 0.2 to 0.4μ , 0.4 to 0.6μ , 0.6 to 0.85μ , and 0.85 to 2.0μ . The data obtained will make the conversion from source absorptance to solar

absorptance more accurate; since in the analysis it was shown that the relative spectral character of the absorptance was more important than the total absorptance for determining the conversion factor.

Section 4 CANDIDATE COATINGS

4.1 COATING DESCRIPTIONS

During the report period specimens of two candidate coatings were received from IIT Research Institute. Initial optical properties were measured and are presented in Sec. 4.2. The coating descriptions presented below are based on the information provided by the supplier.

4.1.1 S-13 Modification II (ZnO/Silicone)

Source and Cost. IIT Research Institute; cost not stated by supplier.

Starting Composition.

<u>Materials</u>	<u>Parts by Weight</u>
New Jersey Zinc SP500 zinc oxide	240
General Electric RTV-602 silicone	100
Toluene	170

Formulation — 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-fourth full of grinding stones 0.5 in. in diameter. The paint is ground for 3 hr at approximately 70% critical speed. The critical speed (rpm) is given by $w_{c_s} = 54.2/\sqrt{R}$, where R is the radius of the mill in feet. The basic charge is then removed, and 70 parts of toluene are 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.

Particle shape and size. Particle shape not stated by supplier. The weighted average particle size is 0.9μ .

Substrate. 1-in. diameter disk 0.050 in. thick of 6061 T-6 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 20 parts of toluene per 670 parts of S-13 (as formulated). This concentration represents 0.76% catalyst based on polymer solids. A lower concentration is recommended in order to ensure optimum stability to u-v irradiation in vacuum. A concentration of 0.4% based upon RTV-602 provides optimum stability without greatly sacrificing terminal 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. A catalyst concentration of 0.4% of resin solids corresponds to 1 part SRC-05 per 1275 parts of S-13; 0.5% catalyst requires 1 part catalyst per 1020 parts of S-13. The catalyst should be added as a 20:1 reduction in toluene. The catalyst solution is added only as the paint is used and only to the amount that can be applied in about 30 min. The bulk paint was furnished in 5-gal epoxy-lined metal pails. The paint should be thoroughly stirred before transfer to other containers or before addition of catalyst. Allow the catalyst paint to set for 10 min. before application to the primed surfaces.

Preparation of surfaces for painting — Standard surface cleaning procedures should be used to prepare the surface for application of the S-13 paint. S-13 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 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-13 paint). The primer should be allowed to air-dry for 1 hr before application of the S-13 paint.

The S-13 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, pre-purified nitrogen or prepurified air must be used. The S-13 paint should be allowed to air-cure 16 hr. 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 Inter-chemical 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-13 paint. Damaged or gouged areas can be recoated by making a paste of S-13 in which the bulk of the solvent is omitted. Such a material can be trowelled or brushed over the damaged areas and cures can be tack-free within a few hours.

Coating thickness. 3.5 to 5.5 mils.

Weight loss during vacuum testing. Not stated by supplier.

Final composition. Not stated by supplier.

4.1.2 Z-93 ($\text{ZnO/K}_2\text{SiO}_4$)

Source and cost. IIT Research Institute; cost not stated by supplier.

Starting composition.

Materials — SP500 ZnO is obtained from New Jersey Zinc Co. The vehicle, PS7 potassium silicate, is obtained from Sylvania Electric Products Corp. The pigment is calcined at 600 to 700°C for 16 hr to obtain a mean particle size of $\sim 0.6 \mu$ (heating and cooling rates are not critical).

Formulation — The materials are mixed in a PBR of 4.30 and a solids content of 56.9%. A typical batch is 100 gm of ZnO, 50 cm³ of PS7 (35% solution), and 50 cm³ of distilled water. The ingredients are ball-milled with porcelain balls in a dense alumina mill. The volume ratio of balls to materials is 1:3, and the total charge is < 50%. The milling time of 6 hr at 70% critical speed [rpm = $54.2 \sqrt{\text{mill radius (ft)}}$] yields a satisfactory consistency for spraying and is recommended.

The paint is prepared just before it is to be used. Shelf life for this composition is limited. Actual shelf time should not exceed 24 hr, and the mixture should be shaken occasionally to maintain the pigment in suspension.

Particle shape and size. Particle shape not stated by supplier; mean particle size $\sim 0.6 \mu$.

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

Method of Application.

Application — The formulation is applied by spray-painting. The carrier gas should be clean; prepurified nitrogen is a good source. Aluminum or plastic substrates should be abraded; e.g., with No. 60 Aloxite metal cloth, and thoroughly washed with detergent and water.

The application technique consists of spraying at a distance of 6 to 12 in. until a reflection due to the liquid is apparent. This is followed by air-drying until the gloss is practically gone, at which time the spraying-drying cycle is repeated. A thickness of about 1 mil is achieved per cycle. Coating dimensions can therefore be predictably applied. However, hand-spraying is inherently an art and not a science, and experience must be gained by the individual painter to determine the most satisfactory technique for him.

Reapplication - The porous nature of a cured coating necessitates heavy spraying upon application of a second coat to achieve a satisfactory, finished texture. If the area to be repainted has been contaminated, it should be scrupulously cleaned with detergent and water. If desired, the paint can be removed simply by abrasion, since it is somewhat soft.

Curing - Satisfactory physical properties are obtained by an air-drying cure. Improved hardness is obtained by heat-curing at 140°C. Strict adherence to cleanliness should be observed during this step as in all the other steps. The presence of impurities can greatly decrease the stability of paints to the space environment.

Coating thickness. 4.5 to 6.0 mils.

Weight loss during vacuum testing. Not stated by supplier.

Final composition. Not stated by supplier.

4.2 INITIAL OPTICAL PROPERTIES

The initial optical properties, solar absorptance, α_s , and emittance, ϵ , of the samples (ZnO/silicone and ZnO/K₂SiO₄) received during the report period have been measured at room temperature, and are reported in Table 4-1. The apparatus utilized to obtain the values were the Cary Model 14 spectrophotometer with integrating sphere, the Gier-Dunkel intergrating sphere with a Perkin-Elmer Model 98 monochromator and the Lion Optical Surface Comparator. These instruments are described in Refs. 4 and 5. Typical spectral reflectance curves for the coatings are presented in Fig. 4-1 and 4-2.

Table 4-1
INITIAL OPTICAL PROPERTIES
(Room Temperature)

Sample	Source	Coating	Solar Absorptance (Cary)	Solar Absorptance (Gier-Dunkle)	Emittance (Optical Surface Comparator)
27	IITRI	ZnO/Silicone	0.19 ± 0.02	0.20 ± 0.01	0.79 ± 0.03
28	IITRI	ZnO/Silicone	0.19 ± 0.02	0.20 ± 0.01	0.79 ± 0.03
29	IITRI	ZnO/Silicone	0.19 ± 0.02		0.81 ± 0.03
30	IITRI	ZnO/Silicone	0.19 ± 0.02		0.80 ± 0.03
31	IITRI	ZnO/Silicone	0.19 ± 0.02		0.81 ± 0.03
32	IITRI	ZnO/Silicone	0.19 ± 0.02		0.87 ± 0.03
33	IITRI	ZnO/Silicone	0.18 ± 0.02		0.85 ± 0.03
34	IITRI	ZnO/Silicone	0.19 ± 0.02		0.80 ± 0.03
35	IITRI	ZnO/K ₂ SiO ₄	0.14 ± 0.02	0.15 ± 0.01	0.92 ± 0.03
36	IITRI	ZnO/K ₂ SiO ₄	0.14 ± 0.02	0.14 ± 0.01	0.96 ± 0.03
37	IITRI	ZnO/K ₂ SiO ₄	0.14 ± 0.02		0.96 ± 0.03
38	IITRI	ZnO/K ₂ SiO ₄	0.14 ± 0.02		0.91 ± 0.03
39	IITRI	ZnO/K ₂ SiO ₄	0.14 ± 0.02		0.96 ± 0.03
40	IITRI	ZnO/K ₂ SiO ₄	0.14 ± 0.02		0.96 ± 0.03
41	IITRI	ZnO/K ₂ SiO ₄	0.14 ± 0.02		0.97 ± 0.03
42	IITRI	ZnO/K ₂ SiO ₄	0.14 ± 0.02		0.95 ± 0.03

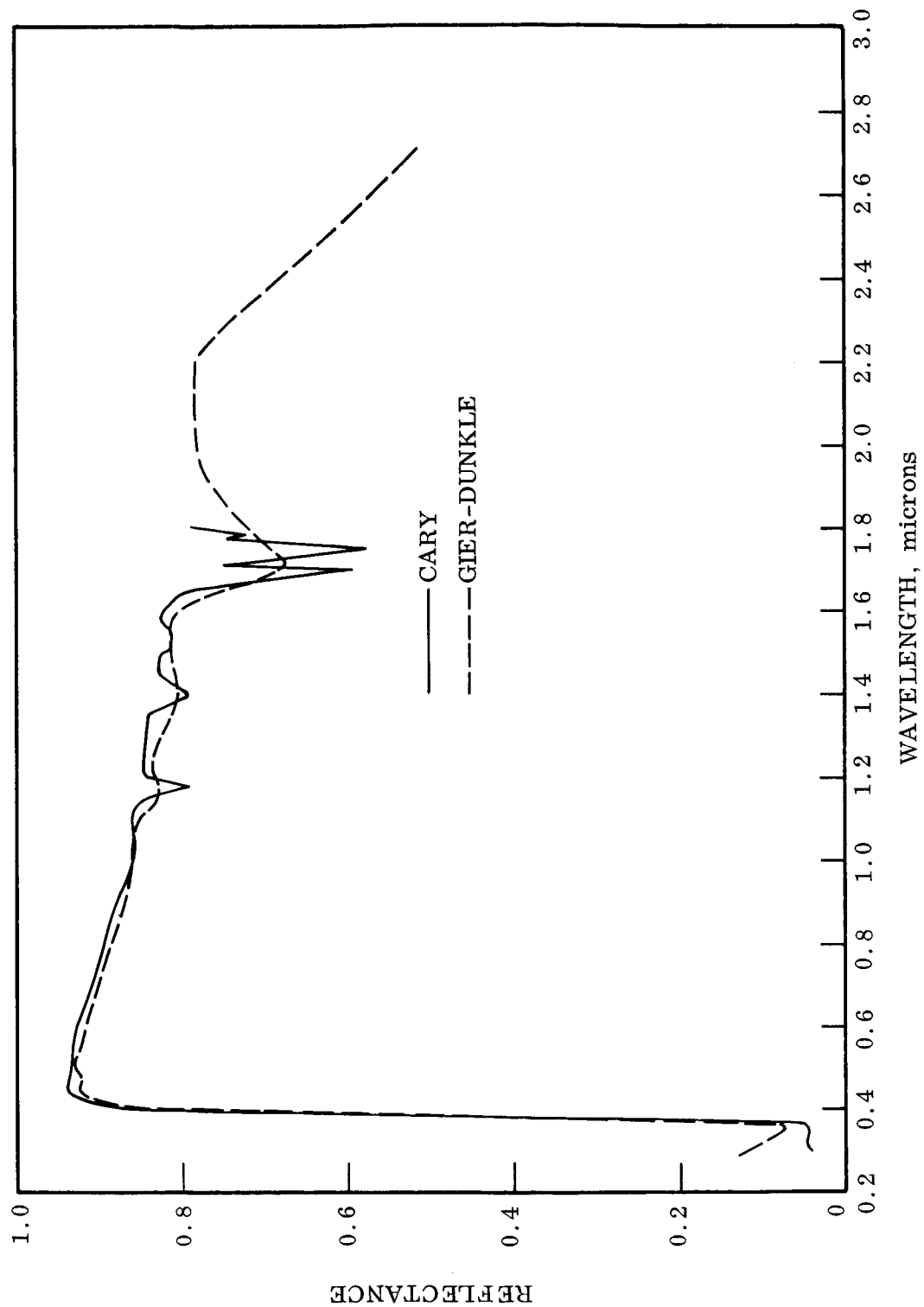


Fig. 4-1 Typical Spectral Reflectance of ZnO/Silicone Coating

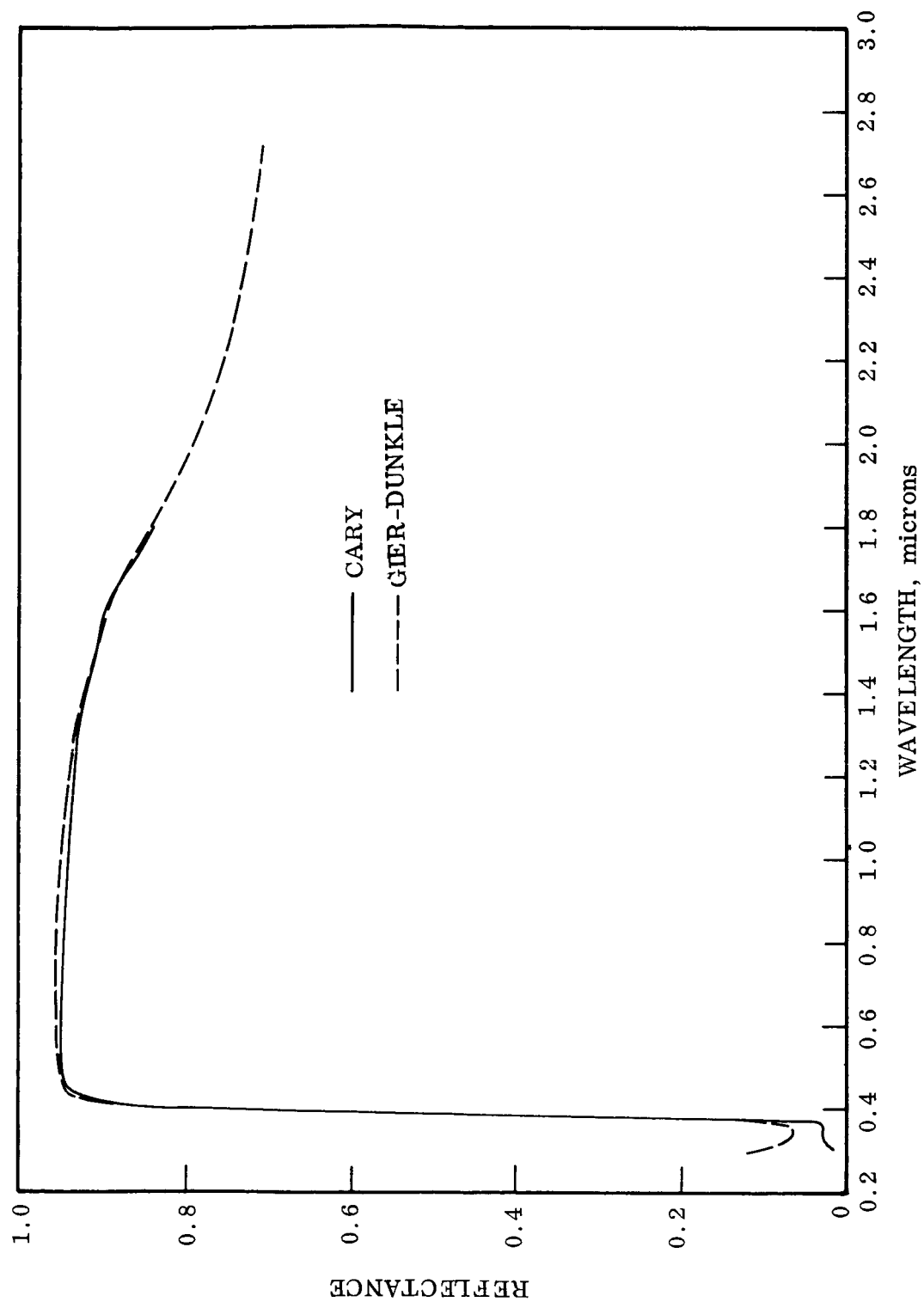


Fig. 4-2 Typical Spectral Reflectance of ZnO/K₂SiO₄ Coating

Section 5
REFERENCES

1. W. F. Schmidt et. al., "Temperature Control Coatings for Cryogenic Temperature Thermal Control Coatings," LMSC M-35-66-6, Lockheed Missiles & Space Company, Sunnyvale, Calif., January 1966.
2. G. A. Zerlaut and G. A. Rubin, "Development of Space Stable Thermal Control Coatings," IITRI Report U6002-36, February 1966.
3. G. E. Gilligan, "Stable White Coatings," IITRI Report U6004-21, February 1966.
4. F. J. Smith, "Emissivity Coatings for Low-Temperature Space Radiators," QPR-1, LMSC NASA-CR-54807, Lockheed Missiles & Space Company, Sunnyvale, Calif.
5. F. J. Smith, "Emissivity Coatings for Low-Temperature Space Radiators," QPR-2, NASA-CR-54807, Lockheed Missiles & Space Company, Sunnyvale, Calif.
6. D. E. Ginnings and R. J. Corruccini, J. Res. NBS, 38, 1947.