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## Formulation of Electrically Conductive Thermal-Control Coatings

Michael C. Shai

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# Formulation of Electrically Conductive Thermal-Control Coatings

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National Aeronautics  
and Space Administration

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## ABSTRACT

The development and formulation of electrically conductive thermal-control coatings was undertaken for use on the International Sun-Earth Explorer (ISEE) spacecraft. The primary effort was to develop a coating with an area resistivity of less than  $1 \times 10^5$  ohm-m<sup>2</sup>, an optical absorptance of approximately 0.55, and a normal emittance of 0.90. The required stability in space called for an area resistivity of less than  $1 \times 10^5$  ohm-m<sup>2</sup>, an absorptance of less than 0.67, and a normal emittance of 0.90 after exposure to approximately  $4 \times 10^{16}$  proton/cm<sup>2</sup> of solar-wind particles and 5300 equivalent Sun-hours. These exposures represent 2 years of ISEE flight conditions.

This paper describes both the unsuccessful formulation efforts and the successful use of oxide pigments fired at 1448 K and discusses problems relative to the reactivity of specific coating vehicles exposed to high humidity. Although the emphasis of this paper is on coating formulation and application techniques, measurement, testing methods and data results are also mentioned. Methods of varying the optical properties as desired and formulations of white, low-absorptance coatings are also described.



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# FORMULATION OF ELECTRICALLY CONDUCTIVE THERMAL-CONTROL COATINGS

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## INTRODUCTION

The Space Environment and Simulation Branch of the Goddard Space Flight Center (GSFC) was assigned the task of developing a coating for the International Sun-Earth Explorer (ISEE) spacecraft that would not only meet the specifications of the thermal design engineers but also be electrically conductive. Electrical conductivity was necessary for preventing a charge buildup on the exterior of the spacecraft that would interfere with the detection of low-energy plasma waves and thermal electrons. The coating specifications called for the following properties:

- Solar absorptance: 0.55 to 0.59
- Thermal emittance: 0.90
- Area resistance: less than  $1 \times 10^5$  ohm-m<sup>2</sup>
- Environmental lifetime: 2 years in the solar-wind environment, with less than 0.07 change in optical absorptance value and no increase in area resistance

## FORMULATION OF COATINGS

During the early stages of development of the conductive coatings, numerous formulations were tried with varying degrees of negative results. The basic initial laboratory efforts were essentially a trial-and-error process. After many false starts, including the addition of metals such as gold and chromium to standard binders, a formulation with fired pigment oxides was developed. The use of fired oxides was suggested by General Electric Company reports\* of Air Force-sponsored programs and stoichiometric considerations. The optical and electrical

\*"Electrically Conductively Coating Materials," General Electric Report, Air Force Materials Laboratory, Technical Report AFMI TR-73-207, Parts I and II, October 1974.

resistivity characteristics were promising. Thereafter, all efforts were directed toward coating formulations based on both sodium and potassium silicate binders containing fired-oxide pigments added in proportions that would optimize the absorptance and conductance (area-resistance product) of the coating.

## **Sodium Silicates**

### *Materials*

The materials used in the sodium silicate formulation included zinc oxide (SP-500) from the New Jersey Zinc Company, aluminum oxide (Type C) from Union Carbide Corporation, and sodium silicate (Star) from the Philadelphia Quartz Company.\*

### *Preparation of Pigments*

The present coating consists of a formulation that includes a pigment of a fired oxide. The pigment is composed primarily of zinc oxide with a small amount of aluminum oxide. The ratio of oxides and dopants were varied to achieve the desired properties. Specific formulations and their resultant properties are shown in tables 1 through 3. In the most preferred composition, the pigment contains about 0.8 to 2.0 weight-percent (wt-%) of aluminum oxide and about 98 to 99.2 wt-% of zinc oxide, based on the weight of the pigment. With these two essential components, the resultant solution is a yellow coating or paint. Because the fired mixture of zinc oxide and aluminum oxide provides the necessary electrical conductivity, the pigment portion of the coating contains these components in their fired form.

In a further composition, the fired-oxide pigment portion may also contain a minor amount of cobalt oxide, also known as cobalt black. The preferable amount of cobalt oxide is in the range of up to about 20 wt-% or, better still, in the range of about 0.25 to 3.0 wt-%, based on the total weight of the pigment. When the pigment includes cobalt oxide, the resultant coating is green.

Another important part of the coating is the vehicle binder. The preferred vehicle binder is an alkali metal silicate or mixture of alkali metal silicates. In general, the vehicle binder is an alkali metal silicate selected from the group consisting of lithium, potassium, and sodium silicates and mixtures thereof. The preferred silicates have an alkali metal oxide to silicon dioxide ratio of about 10 to 30 wt-%. The silicate vehicle binder may be provided by either the alkali metal silicate materials per se or an existing white paint that contains the proper amount of alkali metal silicates.

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\*"Soluble Silicates—Properties and Applications," Philadelphia Quartz Bulletin 17-1, Philadelphia Quartz Company, 1969.



**Table 1**  
**Electrically Conductive Thermal-Control Coatings Formulated**  
**with Sodium Silicate Binder and Fired Oxides**

GSFC Code No.	Pigment (%)			Vehicle Binder			Pigment Concentration	Water (%)	
	Al <sub>2</sub> O <sub>3</sub>	ZnO		Wt-Ratio SiO <sub>2</sub> /Na <sub>2</sub> O	% Na <sub>2</sub> O	% SiO <sub>2</sub>			Weight-Ratio Binder: Pigment
NS43G	Al <sub>2</sub> O <sub>3</sub> 1	ZnO 99		2.50	10.6	26.5	25: 75	5	
NS53B	Al <sub>2</sub> O <sub>3</sub> 1	ZnO 98.75	Co <sub>3</sub> O <sub>4</sub> 0.25	2.50	10.6	26.5	25: 75	5	
NS43E	A		B		Wt-Ratio SiO <sub>2</sub> /Na <sub>2</sub> O	% Na <sub>2</sub> O	% SiO <sub>2</sub>	Weight-Ratio Binder: Pigment	5
	Al <sub>2</sub> O <sub>3</sub> 1	ZnO 98	Co <sub>3</sub> O <sub>4</sub> 1	Al <sub>2</sub> O <sub>3</sub> 1					
	Weight-Ratio				2.50	10.6	26.5		
	A		B						
	50%		50%						

**Table 2**  
**Electrically Conductive Thermal-Control Coatings Formulated**  
**with Potassium Silicate Binder and Fired Oxides**

GSFC Code No.	Pigment (%)			Vehicle Binder			Pigment Concentration	Water (%)	
	Al <sub>2</sub> O <sub>3</sub>	ZnO		Wt-Ratio CCI*/K <sub>2</sub> SiO <sub>3</sub>	% K <sub>2</sub> O	% SiO <sub>2</sub>			Weight-Ratio Binder: Pigment
NS43C	Al <sub>2</sub> O <sub>3</sub> 1	ZnO 99		1:1	11.3	23.7	25: 75	5	
Add one part of above formulation to two parts GSFC-MS74.									
NS55F	A		B		Wt-Ratio SiO <sub>2</sub> /K <sub>2</sub> O	% K <sub>2</sub> O	% SiO <sub>2</sub>	Weight-Ratio Binder: Pigment	5
	Al <sub>2</sub> O <sub>3</sub> 1	ZnO 98	Co <sub>3</sub> O <sub>4</sub> 1	Al <sub>2</sub> O <sub>3</sub> 1					
	Weight-Ratio				1:3.31	11.3	23.7		
	A		B						
	50%		50%						

\*GSFC formulation composed of lithium and potassium silicates.

Table 3  
Summary of Electrical and Thermal Properties of Conductive Coatings

GSFC Code No.	Area Resistance ( $\rho d$ (ohm-m <sup>2</sup> ))*	Absorptance ( $\bar{\alpha}$ )	Emittance (e)
NS43G	$1.7 \times 10^3$	0.38	0.90
NS53B	$1 \times 10^3$	0.52	0.87
NS43E	$2 \times 10^3$ ohm-m <sup>2</sup>	0.57	0.89
NS43C	$1 \times 10^5$ ohm-m <sup>2</sup>	0.20	0.92
NS55F	$6 \times 10^4$ ohm-m <sup>2</sup>	0.57	0.91

\*  $\rho$  = resistivity, d = thickness.

In general, the formulation of the coating contains about 20 to 30 wt-% of the vehicle binder in an admixture with about 70 to 80 wt-% of the pigment. The preferred weight-ratio of the vehicle binder/pigment is 1 to 3.

When added to the pigment as an optional ingredient, the cobalt oxide improves the stability of the optical properties against ultraviolet light and solar-wind degradation without adversely affecting the electrical conductivity of the resulting composition.

When formulating the composition, it is usually necessary to add a small amount of water (up to about 5 wt-%) in order to provide a mixture with the proper flowability and viscosity for application to the substrate.

The relative quantities of ZnO and cobalt oxide in the mixture determine the optical properties of the coating and permit variations in absorptance values ( $\alpha$ ) to meet varying thermal control requirements. A preferred absorptance value of 0.60 is achieved with a mixture of 98.0 wt-% ZnO, 1.0 wt-% Al<sub>2</sub>O<sub>3</sub>, and 1.0 wt-% cobalt oxide. A preferred pigment is obtained by blending this mixture with an equal amount by weight of a mixture of 99.0 wt-% ZnO and 1.0 wt-% Al<sub>2</sub>O<sub>3</sub>. Exemplary methods for preparing this preferred blend of fired-oxide pigments, as well as other blends, follow.

Table 4 shows the effect of varying the percentage of cobalt oxide in the pigment relative to the optical and electrical properties of coatings formulated in accordance with this process. All coatings were formulated with both potassium and sodium silicates, with three parts pigment to one part silicate. All percentages are by weight.

Table 4  
Pigment Percentages Variance Effects

Coating	ZnO (wt-%)	Al <sub>2</sub> O <sub>3</sub> (wt-%)	Cobalt oxide (wt-%)	Absorptance ( $\alpha$ )	Emittance ( $\epsilon$ )	Area Resistance* ( $\rho d$ (ohm-m <sup>2</sup> )) <sup>†</sup>
A	99.0	1	—	0.38	0.90	$1.7 \times 10^3$
B	98.9	1	0.1	0.45	0.89	$1.7 \times 10^3$
C	98.75	1	0.25	0.52	0.87	$<1.7 \times 10^3$
D	98.5	1	0.5	0.58	0.87	$<1.7 \times 10^3$
E	98.4	1	0.6	0.585	0.87	$<1.7 \times 10^3$
F	98.25	1	0.75	0.59	0.87	$<1.7 \times 10^3$
G	98.0	1	1.0	0.63	0.87	$<1.7 \times 10^3$
H	96.0	1	3.0	0.68	0.87	$<1.7 \times 10^3$

\*All measurements were made in a vacuum ( $1 \times 10^{-5}$  torr) after the coatings were exposed to a vacuum for 24 hours.

<sup>†</sup> $\rho$  = resistivity, d = thickness.

For the purposes of this document, a coating that satisfies the following specifications is especially preferred as a satellite or spacecraft coating: absorptance ( $\alpha$ ) of 0.60, emittance ( $\epsilon$ ) of 0.85, and area resistance of  $3 \times 10^3$  ohm-m<sup>2</sup> after being simultaneously exposed to 5300 equivalent Sun-hours (ultraviolet) and  $4 \times 10^{16}$  protons/cm<sup>2</sup> of 5 keV energy. These properties are preferred for providing low thermal absorption, high thermal emittance, and high electrical conductivity.

A preferred formulation for a green coating contains the most preferred pigment detailed previously (that is, a mixture of 98.0 wt-% ZnO, 1.0 wt-% cobalt oxide, and 1.0 wt-% Al<sub>2</sub>O<sub>3</sub> blended with an equal amount by weight of a mixture of 99.0 wt-% ZnO and 1.0 wt-% Al<sub>2</sub>O<sub>3</sub>) from which the fired-oxide pigment is prepared; it also contains, as the vehicle binder, a sodium or potassium silicate and 5-wt-% water, with a weight-ratio of pigment to vehicle binder of about 3 to 1. Although green, white, and yellow are preferred, the color of the coating may be varied as desired.

To prepare the electrically conductive fired-oxide pigment, desired amounts of the pigments are selected, weighed, and stirred together for about 6 hours. Enough water is added to the mixture to produce a creamy consistency during the stirring. The stirred mixture is dried in an oven at approximately 373 K and is then allowed to cool. The dried mixture is broken up with a hand mortar and pestle and placed into OOA Coors porcelain crucibles in 50-gram batches. Each batch is fired at approximately 1448 K for approximately 15 minutes in a

preheated furnace and is then cooled to room temperature. Thereafter, a suitable amount of the fired mixture is placed into the mortar of an electric mortar and pestle and is ground for approximately 52 minutes. Finally, the ground powder is put through a 250-micron sieve. The powder that passes through the sieve is used in formulating the coating.

### *Blending and Application*

The conductive coatings may be blended by either of the following preparative methods.

*Method A*—To one part by weight of a lithium potassium silicate solution (equal parts of a lithium silicate solution with a solids content of 4.0 wt-%  $\text{Li}_2\text{O}$  and 23.0 wt-%  $\text{SiO}_2$ ) and a potassium silicate solution with a solids content of 11.3 wt-%  $\text{K}_2\text{O}$  and 23.7 wt-%  $\text{SiO}_2$ , add three parts by weight of the fired-oxide pigment. Place the mixture in a container, preferably a polyethylene bottle, and roll on a jar-mill roller for approximately 1 hour. To this mixture, add two parts by weight of white silicate paint containing 55.5 wt-%  $\text{K}_2\text{SiO}_3$ , 16.7 wt-%  $\text{ZnO}$ , 11.1 wt-%  $\text{Al}_2\text{O}_3$ , and 16.7 wt-%  $\text{TiO}_2$ , roll for an additional 2 hours, and let the coating stand overnight. Before application, roll the coating for 2 hours.

*Method B*—To one part by weight of a silicate vehicle binder, add three parts by weight of the fired-oxide pigment.

Place the mixture in a container, preferably a polyethylene bottle, roll on a jar-mill roller for approximately 2 hours, and let stand overnight. Before application, roll the coating for 2 hours. Up to 5 wt-% (measured with respect to the total coating prepared) of distilled or deionized water may be added to achieve proper spray viscosity.

The coating can then be sprayed with compressed dry nitrogen on the properly prepared surface. Proper surface preparation is essential to ensure good adhesion. Apply the coating with slow, overlapping strokes with a spray gun held about 15 cm (6 inches) from the surface. Let the first layer dry for several minutes before applying the second coat. Exercise caution when applying all coats to avoid dry spray, which is caused by applying the coating too fast or having a spray that is too thin. Approximately 60 grams of the coating will cover 80 cm<sup>2</sup> (1 ft<sup>2</sup>) of a metallic substrate. After application, air cure for a 24-hour period. Because the coating loses most of its liquid content during applications, the weight increase from the use of the coating is insignificant.

The coatings of this mixture may be applied to substrates, including aluminum, mild steel, stainless steel, copper, and epoxy fiberglass. When applying this coating to aluminum and mild steel, it is preferred that all substrates that have been surface-treated (that is, anodized, alodined, or iridized), be avoided unless stripping is possible; that the surface be sandblasted or abraded; and that the surface then be washed with liquid soap, rinsed thoroughly, and wiped dry. Then, immerse the substrate for about 3 minutes in an acid bath consisting of

11 wt-% nitric acid, 1 wt-% hydrofluoric acid, and 88 wt-% distilled water. Rinse the substrate with distilled water, wipe dry, and apply the coating. When immersion is not practical, this step may be omitted, but a primer of thin paint with 25-percent water by weight should be applied as in applying polish.

When the substrate is stainless steel, sandblast or abrade the surface, rinse with water, and dry before applying the paint.

When the substrate is copper, abrade or sandblast the surface and remove the grit from the surface with soap and a water wash. Then, etch the surface for about 3 minutes with a 5- to 7-percent glacial acetic acid solution or a 5- to 7-percent citric acid solution, rinse with water, and dry before applying the coating.

If the substrate is epoxy fiberglass, abrade the surface until a rough surface that is rich in fiberglass is exposed and remove the debris with soap and water. Apply a primer of thin paint with 25-percent water by weight to the surface by conventional spraying.

### *Optical and Electrical Properties*

A Beckman DK-2 spectrophotometer with a Gier-Dunkle reflectance attachment was used to make absolute reflectance measurements in air both before and after testing of all samples to determine their absorptance. Figure 1 shows three representative samples that were formulated with the sodium silicate binder. The effects of absorption of the fired green oxides are caused by the cobalt oxide. Normal total emittance measurements were also made at room temperature with a DB-100 portable emissometer.

Measurement of coating conductivity and resistance proved difficult. Coated aluminum substrates were used for all measurements. Although specifications on area resistance dictated measurements in a vacuum, the measurements were initially made in air to evaluate various coating formulations. A Keithley Model 6105 was used to obtain sheet and bulk resistance measurements in air. The Keithley Model 6105 employs a biased guard ring to eliminate leakage. The coated electrode contact was enhanced by vacuum-depositing aluminum on the coating sample. With this measurement technique, reproducibility was poor, and results were highly dependent on humidity conditions. However, these measurements permitted rapid comparison of various coatings.

The Materials Applications and Control Branch (MACB) undertook absolute measurement of the area resistance of the coatings in a vacuum. Initially, a gallium anode probe was used to apply voltage to the coated face of an aluminum test piece. Current measured through the coating was used to determine area resistance. Minute cracks in the coating and the irregular surface caused electrical shorting and resulted in poor precision. A contactless

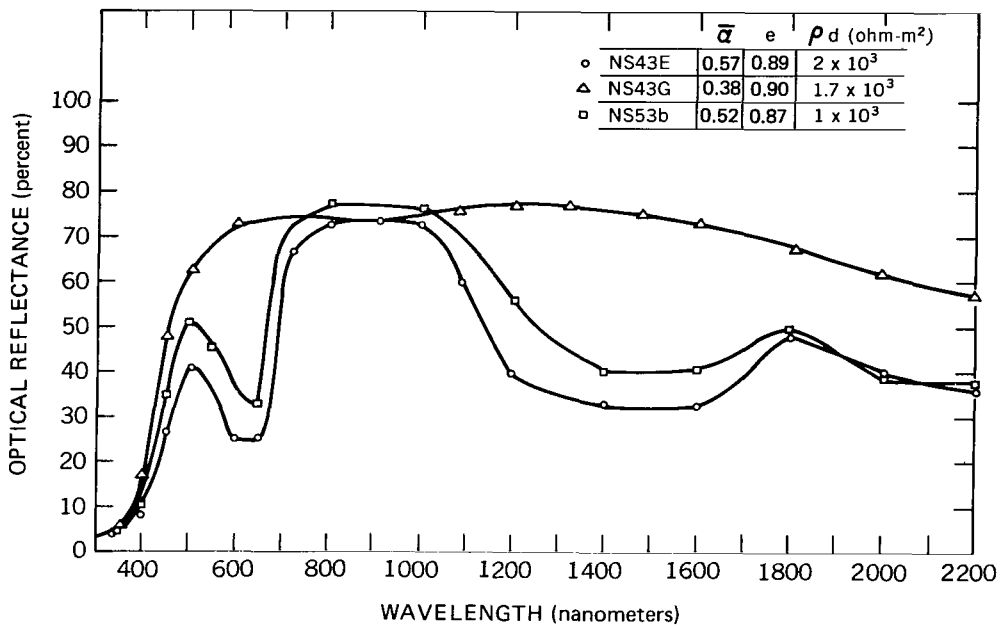


Figure 1. Optical reflectance of electrically conductive thermal-control coatings formulated with sodium silicate binder.

method\* was then investigated. This technique employed a low-energy (0.5 to 2.0 volts) electron beam impinging on the coated face of a test piece. The electron-beam technique was used for determining compliance with coating specifications.†

## Potassium Silicates

### *Formulation of Pigments and Binder*

The formulation of the potassium silicate coatings is basically the same as that of the sodium silicates. Potassium silicate PS-7, purchased from Sylvania, is used as the binder for coatings NS43C and NS55F (table 2). The fired-oxide pigments are prepared as previously described for the sodium silicate coatings.

### *Optical and Electrical Properties*

Figure 2 shows the reflectance of two distinctly different types of electrically conductive coatings. NS43C has excellent reflectance properties and is sufficiently conductive ( $1 \times 10^5$  ohm-m<sup>2</sup>) to fall within the specifications of the ISEE Program.

\*Hans Bentlage, Hans-Peter Spanier, and Walter Wilkens, 1975, "Changes in Electrical Cross-Resistance of Conductive Coatings Due to Contamination By Outgassing Of Silicon Rubber Material Silastic 35," Internal Report 1B 152-75/04 (DFVLR).

†W. Viehman, C. M. Shai, and E. L. Sanford, 1977, "Investigation of Conductive Thermal Control Coatings by a Contactless Method in VACUO," Proceedings of the Spacecraft Charging Technology Conference, p. 687.

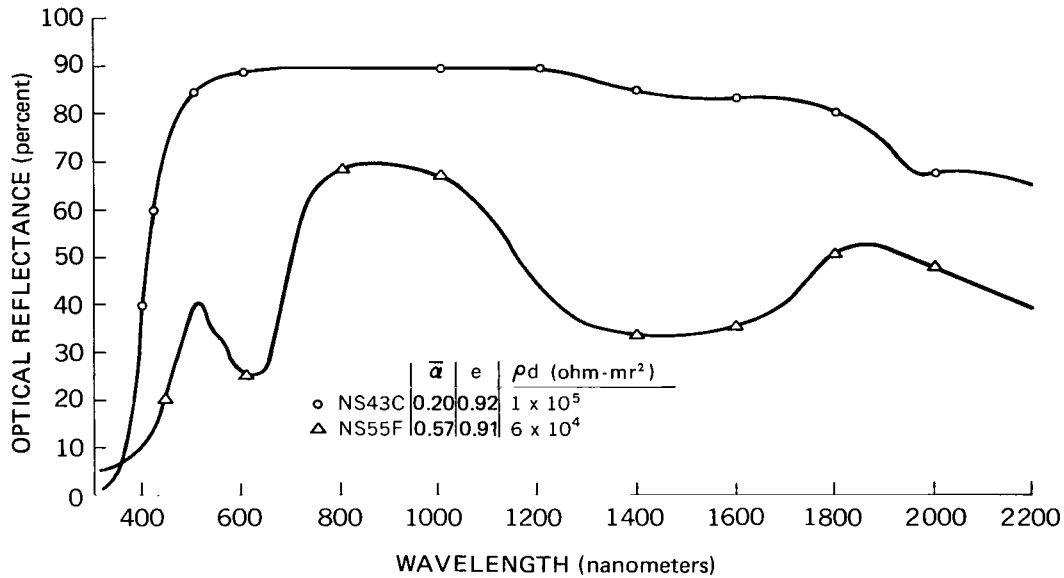


Figure 2. Optical reflectance of electrically conductive thermal-control coatings formulated with potassium silicate binder.

## QUALIFICATION OF ELECTRICALLY CONDUCTIVE COATINGS

### Electrical Resistivity Measurements

A separate phase of the coatings development program has been the measurement of the conductivity (area-resistivity). Because of extensive previous experience in optical measurement and space-environment simulation, no difficulties were encountered. However, measuring area resistance has proved to be more difficult than anticipated. Problems with reproducibility, coating stability, and experimental measurement techniques made it necessary to separate this qualification from the coating formulation efforts.

### Environmental Test Data

#### *Ultraviolet Degradation*

One of the most promising features of this family of fired-oxide coatings is their capability of withstanding high-level ultraviolet (UV) radiation. After exposure to 1000 hours in a high-vacuum environment, samples from each formulation showed virtually no change in optical surface reflectance. In this screening test, the UV source was a low-pressure mercury lamp from which more than 45 percent of the output energy is below 400 nanometers.

Figures 3 and 4 show typical mercury and vapor lamp output spectra, respectively. Table 3 and figure 5 show xenon-lamp and space spectra.

### *Solar Wind*

A principal source of possible damage to the coatings on exterior spacecraft surfaces is bombardment by high fluence levels of low-energy protons contained in the solar wind and solar flares. The relatively constant stream of low-energy protons is continuously emitted from the Sun flux of approximately  $2.5 \times 10^8$  p/cm<sup>2</sup>/s at an average energy of 1 keV with a maximum of 50 keV. The ISEE spacecraft is expected to encounter approximately  $2 \times 10^{16}$  p/cm<sup>2</sup> low-energy protons during a 2-year period. As a consequence, the GSFC Solar Wind Test Facility was used to determine the degradation rates of the conductive coatings. Figure 6 shows the optical degradation of coatings that were selected for use on the spacecraft. The coatings were exposed in a vacuum to 5-keV protons to a fluence of  $4 \times 10^6$  p/cm<sup>2</sup> and were simultaneously irradiated with UV energy. The UV source was a xenon-lamp solar simulator, and samples were exposed to a total of  $4 \times 10^{16}$  protons/cm<sup>2</sup> and 2000 equivalent Sun-hours. Typical exposure rates were  $10^{10}$  p/cm<sup>2</sup>/s and 5 equivalent Sun-hours. Sun equivalence was based on simulator UV output relative to space. Previous test data taken in other UV-degradation test facilities have shown these samples to be highly tolerant of UV sources; therefore, the changes in the optical properties of these coatings have been attributed mainly to the effects of the low-energy protons. After exposure to an equivalent 2-year orbital lifetime, the electrical and optical properties are well within the ISEE specifications.

Optical measurements of the coatings were made at various levels of sample irradiation using a Beckman DK2 spectrophotometer with a Gier-Dunkle reflectance attachment. The spectrophotometer was interfaced with the Solar Wind Test Facility so that measurements could be made without removing the samples from their vacuum-controlled environment.

### *Thermal-Vacuum Cycling*

The conductive coatings were thermal-cycled in a vacuum from 123 to 423 K for a total of 66 cycles of 90-minute duration. No damage was visible on any of the samples, and all of the samples passed a "scotch-tape" adhesion test.

### *Humidity Tests*

Coating samples were subjected to environments of 85-percent relative humidity and 300 K (80° F) for periods as long as 48 hours. This test was prompted by the whitening of several green coatings that were inadvertently subjected to high humidity. Some slight whitening was visible on all coatings that contained sodium silicate binders. No whitening was noted



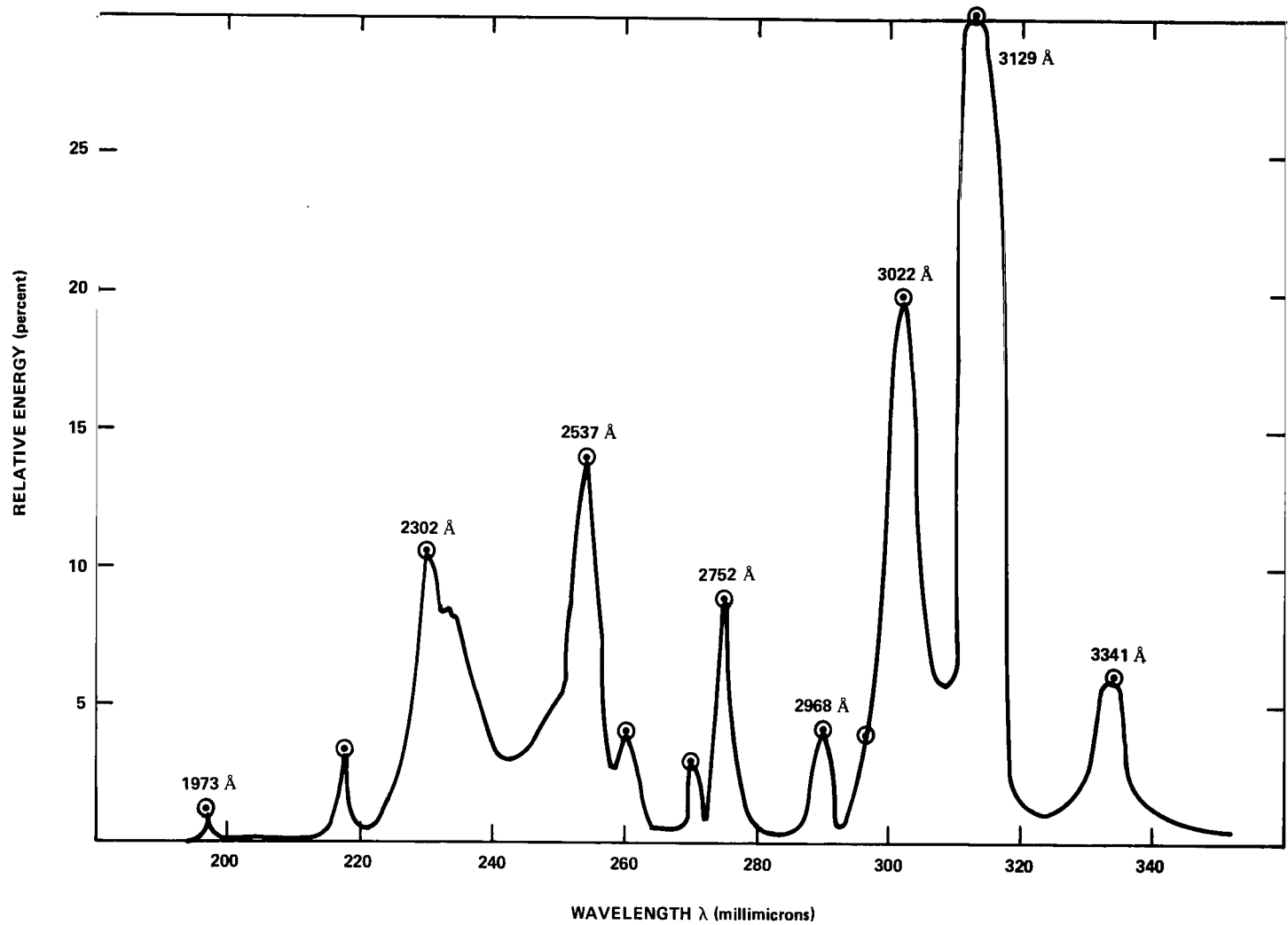


Figure 3. Relative spectral energy distribution (UV), low-pressure mercury lamp (1000 watts).

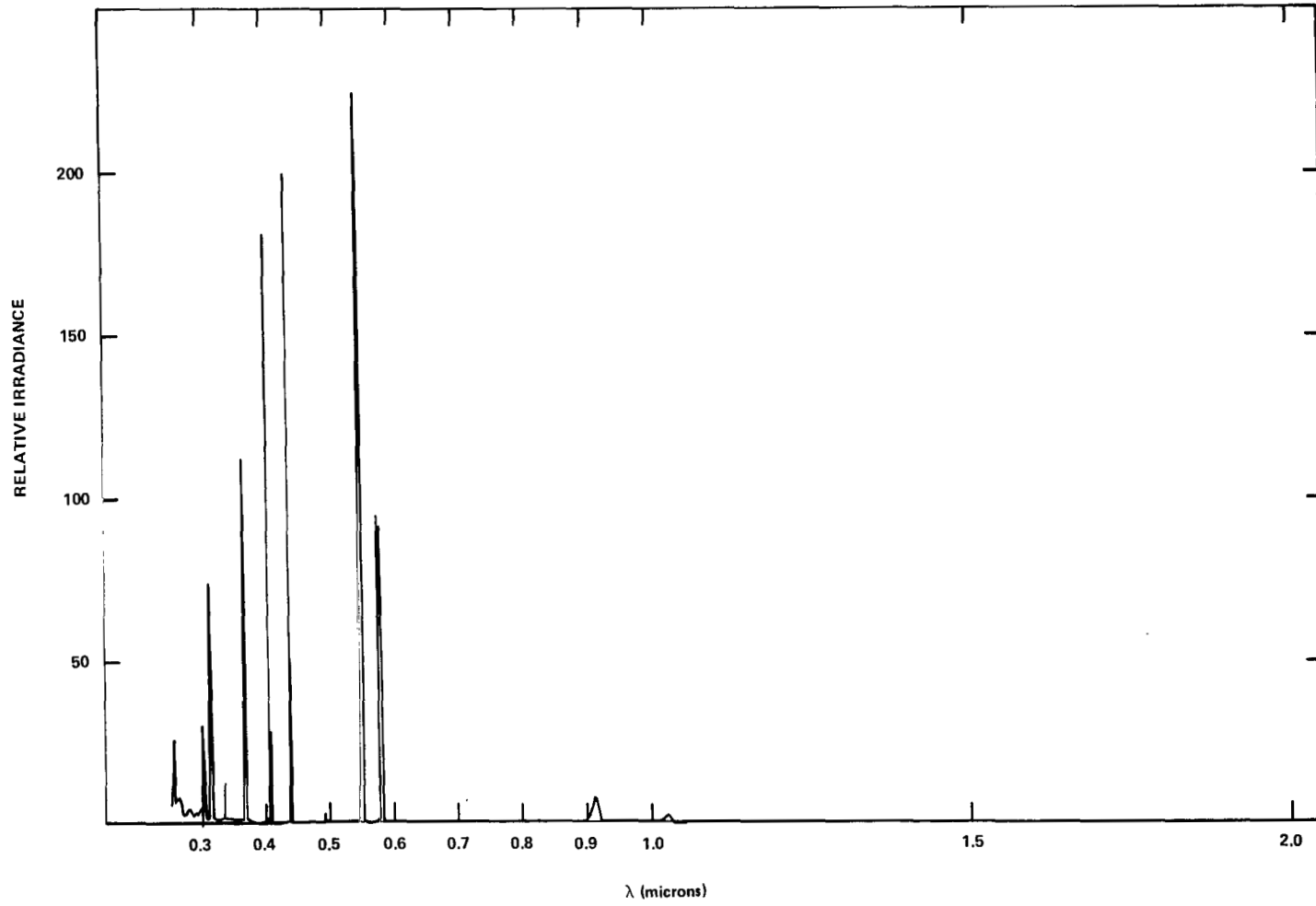


Figure 4. Relative spectral energy distribution (UV) mercury vapor lamp.

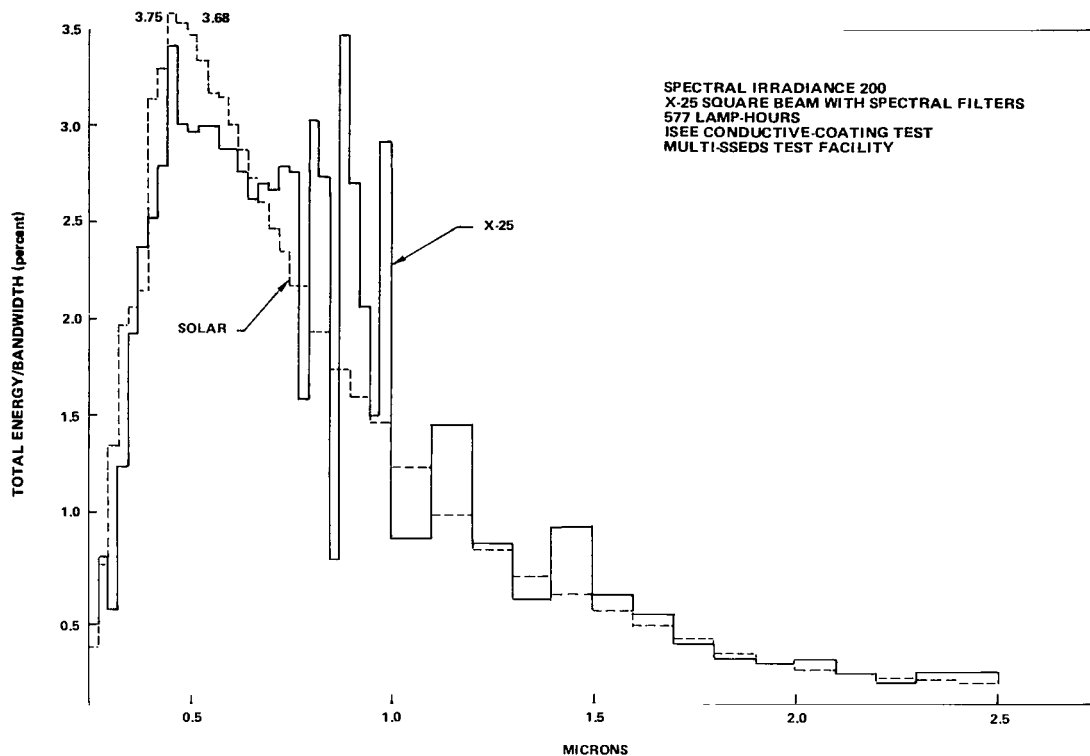


Figure 4. Xenon lamp spectral irradiance.

on potassium silicate based coatings. Analysis by the MACB determined the whitening to be sodium carbonate. The whitening was easily removed by light swabbing with distilled water.

Despite its high degree of visibility, the whitening proved to be significant with regard to measured  $\alpha$  values. Whitened samples, as well as samples with whitening removed by distilled water, were exposed to combined UV and proton (4-keV) irradiation. Degradation results were the same as those encountered with the original coatings that exhibited no whitening.

## CONCLUSIONS

The electrically conductive coatings formulated with potassium- and sodium silicate binders and fired-oxide pigments have met all the specifications required by the ISEE Program. Table 3 summarizes their thermal and electrical properties. As previously reported, these coatings are now flight-qualified after having passed all phases of the environmental testing, including UV, low-energy proton (solar-wind), thermal-vacuum cycling, and humidity tests.

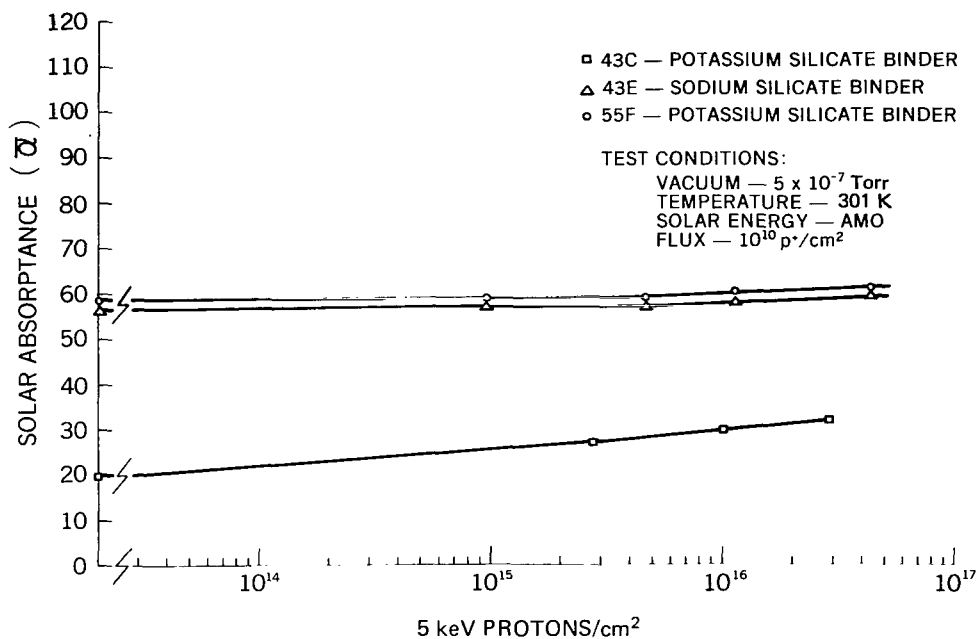


Figure 5. Degradation of coatings exposed to UV and low-energy protons.

The test program indicated greater chemical stability with the potassium silicate binder than with the sodium-silicate binder coatings; however, the sodium silicate coatings exhibited greater stability of optical properties. In addition, the sodium silicate coatings lent themselves to removal of the “white” sodium carbonate. When selecting coatings in the future bear in mind that a sodium-silicate vehicle requires a humidity-controlled environment.

## ACKNOWLEDGMENTS

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