

## Contents

1. Introduction	614
2. Formulation of Coatings	614
3. Qualification of Electrically Conductive Coatings	618
4. Conclusion	619
Acknowledgments	620
References	620

#### 4. Formulation of Electrically Conductive, thermal-Control Coatings

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#### Abstract

Formulation of electrically conductive, thermal-control coatings was undertaken for use on the International Sun Earth Explorer (ISEE) spacecraft. The primary effort was aimed at formulation of a coating with bulk resistivity less than  $1 \times 10^5 \text{ ohm-m}^2$  and optical absorptance and normal emittance of approximately 6.58 and 0.90, respectively. The required stability in space called for built resistivity less than  $1 \times 10^5 \text{ ohm-m}^2$ , absorptance less than 0.87 and normal emittance of 0.90 after exposure to approximately  $4 \times 10^{16} \text{ proton/cm}^2$  of solar wind particles and 6300 equivalent sun hours. These exposures represent two years of the ISEE orbit.

Unsuccessful formulation efforts as well as the successful use of oxide pigments fired at  $1175^\circ\text{C}$  are described. Problems attributed to reactivity of specific coating vehicles exposed to high humidity are discussed.

Measurement and testing methods, including resulting data are mentioned, but the emphasis, in this report, is placed on coating formulation and application techniques. Methods of varying, as desired, optical properties are also described as well as formulations of white, low-absorptance coatings.

## 1. INTRODUCTION ----

The Engineering Applications Branch at the Goddard Space Flight Center was given the task of developing a coating for the International Sun Earth Explorer (ISEE) spacecraft that not only had to meet the specifications of the thermal design engineers, but in addition, the thermal control coating had to be electrically conductive. Electrical conductivity was necessary to prevent charge buildup on the exterior of the spacecraft which would interfere with the detection of low-energy level plasma waves and thermal electrons. Specifically, we were confronted with the task of developing a coating which would have the following properties:

- (1) Solar absorptance of 0.55.
- (2) Thermal emittance of 0.90.
- (3) Area-resistance of less than  $1 \times 10^5$  ohm-m<sup>2</sup>.
- (4) Environmental lifetime of two years in the solar wind environment, with less than 0.07 change in optical absorptance value.

## 2. FORMULATION OF COATINGS

During the early stages of developing the conductive coatings, numerous formulations were tried with varying degrees of negative results. The basic laboratory efforts were more of trial-and-error processes. After many false starts, including the obvious attempt of adding metals such as gold and chromium to standard binders, a formulation with fired pigment oxides was made. The optical and electrical resistivity characteristics were most promising. Thereafter, all attention was directed to coating formulations based upon both sodium and potassium silicate binders containing fired oxide pigments added in such proportions as to optimize the absorptance and conductance (area-resistance product).

### 2.1 Sodium Silicates (See Table 1)

#### 2.1.1 MATERIALS

Sp500 zinc oxide was obtained from New Jersey Zinc Co.; the aluminum oxide (Type C) from Union Carbide; and sodium silicate (Star) from Philadelphia Quartz Co.

#### 2.1.2 PREPARATION OF PIGMENTS

The required amounts of pigment oxides (by weight) are added to distilled water and slowly stirred for five hours. This slurry is initially heated for 48 hr at 90°C and then for an additional 72 hr at 110°C. The pigments are fired at 1175°C for 16 min in 50-g batches. In the final preparation stage, the pigments are ground and sieved to select the desired particle sizes. The desired optical properties, absorptance and emittance, are determined by the relative amounts added to

the binder. For example, NS43E Which has an  $\bar{\alpha}$  of 0.57 to 0.60 and  $\rho d$  of  $2 \times 10^3$  ohm-cm<sup>2</sup>, contains a pigment mixture of fired 98 percent ZnO, 1 percent Co<sub>3</sub>O<sub>4</sub> and 1 percent Al<sub>2</sub>O<sub>3</sub> which is then combined with an equal amount of a pigment composed of fired 99 percent ZnO and 1 percent Al<sub>2</sub>O<sub>3</sub>.

**Table 1.** Formulation of Electrically Conductive, Thermal-Control Coatings with Sodium and Fired Oxides

GSFC CODE #	PIGMENT - %			VEHICLE-BINDER			PIGMENT CONCENTRATION	WATER	
	Al <sub>2</sub> O <sub>3</sub>	ZnO	Co <sub>3</sub> O <sub>4</sub>	WT-RATIO SiO <sub>2</sub> /Na <sub>2</sub> O	% Na <sub>2</sub> O	% SiO <sub>2</sub>			WEIGHT-RATIO BINDER: PIGMENT
NS 43G	1	99		2.50	10.8	26.5	25:76	6%	
NS 53B	1	98.75	0.25	2.50	10.8	26.5	25:75	5%	
NS 43E	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>	ZnO	Co <sub>3</sub> O <sub>4</sub>	Al <sub>2</sub> O <sub>3</sub>					
		WEIGHT-RATIO							
		A	B						
		50%	50%						

### 2.1.3. FORMULATION AND APPLICATION

The conductive pigments are added to the silicate solution (Star) in a binder-pigment Ratio of 25:75 by weight and thinned with distilled or deionized water not exceeding 5 percent of the total of pigment plus binder. The mixture is stirred with a teflon-coated magnet in a 250 ml beaker for approximately 2 hrs, allowed to set for 16 to 12 hr and finally stirred for 2 hrs before application. The coating is then sprayed with compressed, dry nitrogen on the suitably prepared surface. The coating is applied with slow, overlapping strokes with a spray gun held about 8 in. from the surface. The first layer should be allowed to dry for several minutes before the second coat is applied. Caution must be used in applying all coats to avoid dry spray which is caused by applying the coating too fast or having a spray which is too thin.

### 2.1.4 OPTICAL AND ELECTRICAL PROPERTIES

A Beckman DK-2A with a Gler-Dunkle reflectance attachment is used to make absolute reflectance measurements in air before and after testing of oil samples

to determine their absorptance. Shown in Figure 1 are three representative samples formulated with the sodium silicate binder. The absorption effects of the fired green oxides are due to the cobalt oxide.

Normal total emittance measurements are also made at room temperature with a DB-100 Portable Lmissometer.

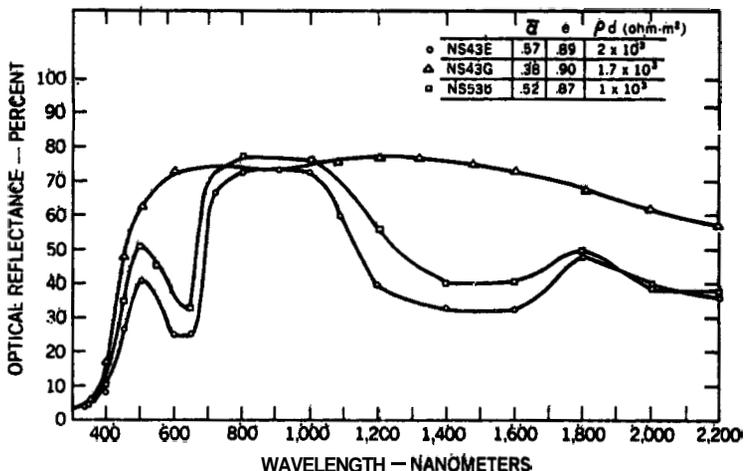


figure 1. Optical Reflectance of Electrically Conductive, Thermal-Control Coatings Formulated with Sodium Silicate Binder

## 2.2 Potassium Silicate

### 2.2.1 FORMULATION OF PIGMENTS AND BINDER

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

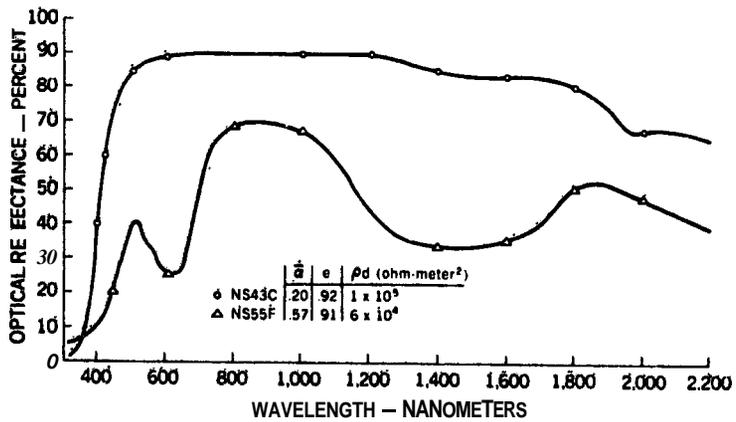
### 2.2.2 OPTICAL AND ELECTRICAL PROPERTIES

The reflectance of two distinctly different types of electrically conductive coatings are shown in Figure 2. 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.

**Table 2. Formulation of Electrically Conductive, Thermal-Control Coatings with Potassium-Silicate and Fired Oxides**

GSFC CODE #	PIGMENT - %					VEHICLE-BINDER			PIGMENT CONCENTRATION	WATER	
NS43C	Al <sub>2</sub> O <sub>3</sub>	ZnO				WT-RATIO	%	%	WEIGHT-RATIO BINDER: PIGMENT	5%	
	1	99				CCl*/K <sub>2</sub> SiO <sub>3</sub>	K <sub>2</sub> O	SiO <sub>2</sub>			25: 76
						1:1	11.3	23.7			
NS 55F	A		B			WT-RATIO	%	%	WEIGHT-RATIO BINDER: PIGMENT	5%	
	Al <sub>2</sub> O <sub>3</sub>	ZnO	Co <sub>3</sub> O <sub>4</sub>	Al <sub>2</sub> O <sub>3</sub>	ZnO						SiO <sub>2</sub> /K <sub>2</sub> O
	1	98	1	1	99	1:3.31	11.3	23.7			25: 75
	WEIGHT-RATIO										
A		B									
50%		50%									

\*GSFC FORMULATION COMPOSED OF LITHIUM AND POTASSIUM SILICATES.



**Figure 2. Optical Reflectance of Electrically Conductive, Thermal-Control Coatings Formulated With Potassium Silicate Binder**

### 3. QUALIFICATION OF ELECTRICALLY CONDUCTIVE COATINGS

#### 3.1 Electrical Resistivity Measurements

A separate phase of the coatings development program and one which we found just as challenging, has been the measurement of the conductivity (area-resistivity). Our previous experience in the optical measurement and space environment simulation has been extensive so no difficulties were encountered. However, the measurement of the area resistance has proven to be more than we bargained for. Problems with reproducibility, coating stability, experimental measurement techniques, to name a few, made it necessary to separate this qualification from our coatings formulation efforts. All area-resistance data is the work of Walter Viehmann who will present in a later paper the details of his contactless resistivity measurements.

#### 3.2 Environmental Test Data

##### 3.2.1 UV DEGRADATION

One of the most promising features of this family of fired-oxide coatings is their capability to withstand extremely high levels of UV radiation. Samples from each formulation after exposure to 1000 hr in a high vacuum environment showed virtually no change in optical surface reflectance. The source in this screening test set-up is a low pressure mercury lamp which has more than 45 percent of the output energy below 400 nm.

##### 3.2.2 SOLAR WIND

The principal source of possible damage to the coatings on the exterior spacecraft surfaces is their 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 continuously emitted from the sun has a flux of approximately  $2.5 \times 10^8$  p/cm<sup>2</sup>/sec and an average energy of 1 keV with a maximum of the order of 50 keV. The ISEE spacecraft is expected to encounter roughly  $2 \times 10^{16}$  p/cm<sup>2</sup> low-energy protons during a two-year period. As a consequence, the GSFC Solar Wind Test Facility was used to determine the degradation rates of the conductive coatings. Shown in Figure 3 are the optical degradation of coatings which have been selected for use on the spacecraft. The coatings were exposed to 5 keV protons to a fluence of  $4 \times 10^{16}$  p/cm<sup>2</sup> and simultaneously irradiated with UV energy. Previous test data taken in Mher UV degradation test facilities have shown these samples to be highly UV tolerant. 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 two-year orbital lifetime the electrical and optical properties are well within the ISEE specifications.

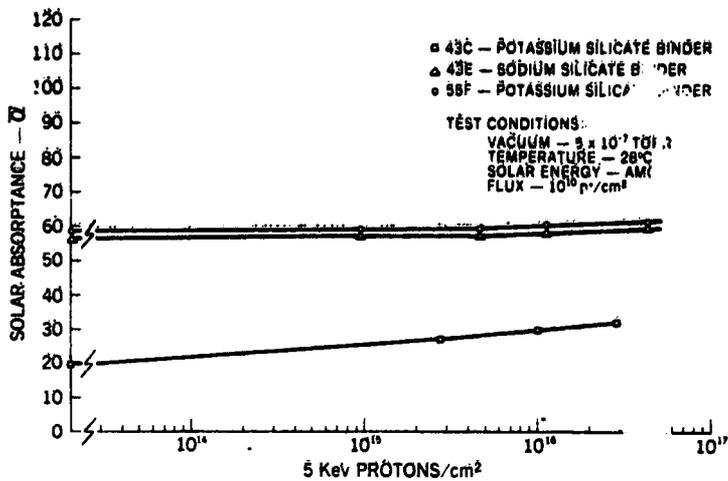


Figure 3. Degradation of Coatings Exposed to UV and Low-Energy Protons

### 3.3.3 THERMAL-VACUUM CYCLING

The conductive coatings have been thermal cycled from  $-150^{\circ}$  to  $+150^{\circ}$ C for a total of 66 cycles of 90-min duration. No damage was visible on any of the samples and all of them passed a "scotch tape" adhesion test.

## 4. CONCLUSION

The electrically conductive coatings formulated with potassium and sodium silicate binders and fired-oxide pigments have met all specifications required by the ISEE Program. A summary of their optical and electrical properties are outlined in Table 3. As we have previously reported, these coatings are now flight qualified after having passed all phases of the environmental testing which included UV, low-energy proton (solar wind), and thermal-vacuum cycling. They are now being applied to portions of the spacecraft.

	AREA RESISTANCE $\rho_d$ (ohm-m <sup>2</sup> )	ABSORPTANCE $\bar{\alpha}$	EMITTANCE $\epsilon$
NS 43G	$1.7 \times 10^3$	.38	.90
NS 53B	$1 \times 10^3$	.52	.87
NS 43E	$2 \times 10^3$ ohm-m <sup>2</sup>	.57	.89
NS 43C	$1 \times 10^6$ ohm-m <sup>2</sup>	.20	.92
NS 55F	$6 \times 10^4$ ohm-m <sup>2</sup>	.57	.91

## Acknowledgments

Formulation and development of the coatings has been the responsibility of Charles Skaf. Qualification and environmental tests were performed by Danny Lester, James Mullins, Lawrence Bromery, Frank Paczkowski, and John Henninger under the supervision of Jule Hirschfeld. Resistivity measurements were made by Walter Viehmann. The entire program was supported by ISEE Project. R. Hoffman and J. Triolo of the Thermal Systems Branch have provided continual input during the qualification stages. Joseph Colony of the Materials Branch provided technical consultation support.

## References

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