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THE DEVELOPMENT AND QUALIFICATION  
OF THERMAL CONTROL COATINGS  
FOR  
SNAP SYSTEMS

*AEC Research and Development Report*

RELEASED FOR ANNOUNCEMENT  
IN NUCLEAR SCIENCE ABSTRACTS



**ATOMICS INTERNATIONAL**

A DIVISION OF NORTH AMERICAN AVIATION, INC.

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THE DEVELOPMENT AND QUALIFICATION  
OF THERMAL CONTROL COATINGS  
FOR  
SNAP SYSTEMS

By  
J.R. CROSBY

**ATOMICS INTERNATIONAL**

A DIVISION OF NORTH AMERICAN AVIATION, INC.  
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## ABSTRACT

Components of the (SNAP 2) Mercury-Rankine and SNAP 10A systems require various radiation control coatings which exhibit particular thermal emittance and solar reflectance properties.

A specialty coating system, designated AI93, has been developed for the SNAP 2 radiator-condenser and the SNAP 10A thermoelectric module and pump radiator fins. This coating exhibits a thermal emittance of 0.91 (400 to 700°F) and a solar absorptance of 0.30. Stability of this coating system in a nuclear and simulated space environment has been demonstrated.

Other coating systems which have been developed and qualified are:

- 1) gold foil and gold on molybdenum foil for use as radiative insulation,
- 2) high-thermal-emittance coatings for use on heat-sensitive components, and
- 3) a mosaic coating pattern for use on the SNAP 10A ejectable heat shield.

Data generated during the development of these coating systems along with the associated equipment descriptions are included herein.

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## I. INTRODUCTION

Two nuclear auxiliary power systems are currently under development for the Atomic Energy Commission (AEC), at Atomics International (AI), a division of North American Aviation (NAA). The 3- to 5-kwe (SNAP 2) Mercury-Rankine power unit is comprised of a 50-kwt, NaK-cooled reactor heat source and a Hg-vapor turboelectric power conversion subsystem (PCS). The 500-w-electrical SNAP 10A power unit is comprised of a 30-kwt, NaK-cooled reactor heat source and a thermoelectric PCS. The special requirements of a reactor operating in a space environment (minimum weight, completely unattended operation in a high-temperature, vacuum environment, etc.), create unique problems in the development and qualification of radiation control coatings.

In February 1961, the Compact Systems Division of AI initiated an effort for development of radiation control surfaces for the SNAP 2 and 10A systems. A major requisite for these coating systems is that they successfully operate for 1 yr in a space and nuclear environment. The environmental requirements for the SNAP system space radiator coating are:

Duration	1 yr
Vacuum	$10^{-11}$ torr
Temperature	650°F maximum
Nuclear irradiation	$10^{18}$ nvt (fast) $10^9$ R
Ultraviolet irradiation	1 yr
Aerodynamic heating	Ambient to 700°F in 140 sec
Mechanical vibration and shock	Launch stress

The various components of the SNAP systems require, basically, four types of radiation control surfaces:

- a) A coating system which is effective in rejecting internal heat and reflecting solar input. The coating must operate at 600°F for 1 yr;
- b) A high-thermal-emittance coating for heat-sensitive components or heat-treated alloys;
- c) A coating which will act as radiative insulation for high-temperature components;
- d) A composite coating pattern for the SNAP 10A ejectable heat shield to control system temperatures during launch and before reactor startup.



A major portion of the effort in the AI coating development and qualification program has been utilized in attaining a successful "tailored coating" for use on the SNAP 2 radiator-condenser (RC), the SNAP 10A thermoelectric module radiator fins, and the SNAP 10A thermoelectric pump radiator fins. The term tailored coating implies a temperature control surface with a high thermal emittance and a low solar absorptance. The desirability of the coating exhibiting a low solar absorptance is due to a distinct gain in thermal effectiveness for a space radiator. For a coating ( $\epsilon = 0.91$ ) operating at the SNAP 2 RC nominal design temperature of 600°F, a 7% gain in thermal effectiveness is attained by utilizing a coating with a  $\alpha_s = 0.30$  as opposed to a  $\alpha_s = 0.90$ . The reference design tailored coating chosen for the SNAP systems is termed AI93. The total hemispherical emittance of AI93 is 0.91 at 600°F; the solar absorptance is 0.30.

Many components of the SNAP 2 and 10A systems require a passive radiation control surface with a high thermal emittance. This coating is to be used on components which are either heat sensitive or utilize a heat-treated alloy as the metallic substrate. Thus, the coating system must be capable of being applied and cured at ambient temperature.

Many industrial enamels were investigated. For SNAP system requirements the best of these high-thermal-emittance finishes was found to be PT404A black enamel (produced by Product Techniques Inc., Los Angeles, California). The total hemispherical emittance of the PT404A enamel is 0.90.

Pure gold foil was chosen as the reference design low-emittance coating for use on SNAP systems. Two methods of attaching foil to the stainless steel (SS) substrate are utilized: spot welding or extremely lightweight clamps. The method used depends on component or piping configuration.

The SNAP 10A ejectable heat shield requires a mosaic pattern of radiation control coatings which will maintain the internal NaK temperature within certain limits. To prevent possible NaK oxide and/or hydride precipitation, the NaK temperature must be greater than 50°F. To prevent premature ejection of the heat shield the NaK temperature should remain less than 300°F.

## II. DISCUSSION OF RESULTS

### A. DEVELOPMENT OF TAILORED COATING (AI93)

#### 1. Thermal Effectiveness

In the determination of the thermal effectiveness of a tailored coating for the SNAP 2 and 10A systems, the heat input to the systems from space must be considered. This input is the sum of:

- a) direct solar radiation,
- b) direct thermal radiation from the earth,
- c) reflected solar radiation from the earth.

The amount of this heat input is a function of the emittance coating used and can be expressed as

$$Q_{\text{space}} = \alpha_s H_s A_{\text{eff}} + \epsilon H_e F_{1-2} A + \alpha_s \gamma_e H_s F_{1-2} \overline{\cos WA}, \quad \dots(1)$$

where

$\alpha_s$  = solar absorptance

$\epsilon$  = thermal emittance

A = area

$F_{1-2}$  = satellite-to-earth view factor

$\gamma_e$  = solar reflectance of earth

$H_s$  = solar constant

W = solar radiation angle of incidence

$H_e$  = earth insolation (Ref. 1)

By far the greatest input is the solar radiation; therefore, a low solar absorptance is highly desirable. A measure of the heat rejection capability of a space radiation surface is its thermal effectiveness (R), which is defined by the relation

$$\epsilon \sigma A \eta T^4 - Q = R \sigma A \eta T^4 \quad \dots(2)$$

where an area (A) receives heat (Q) and radiates at a temperature (T), and  $\eta$  is a parameter depending on surface geometry and temperature.

When measuring the thermal effectiveness of the SNAP 2 RC, the most meaningful calculation is based on maximum total heat input from space. In a constant sun-constant shade orbit, one side of the RC always receives peak solar input and the other side zero solar input. The inputs are given by

$$\frac{Q_{\max}}{(A/2)} = K_3 \alpha_s + K_2 \epsilon, \text{ and} \quad \dots(3)$$

$$\frac{Q_{\min}}{(A/2)} = K_2 \epsilon. \quad \dots(4)$$

The K's are functions of the general RC configuration and orbit parameters. For SNAP 2 conditions they are

$$K_2 = 8.27 \text{ Btu/ft}^2\text{-hr, and}$$

$$K_3 = 343 \text{ Btu/ft}^2\text{-hr (Ref. 2)}$$

From Equations 3 and 4 the value of maximum total heat input is

$$\frac{Q_{\max}}{A_{R/C}} = \frac{K_3}{2} \alpha_s + K_2 \epsilon. \quad \dots(5)$$

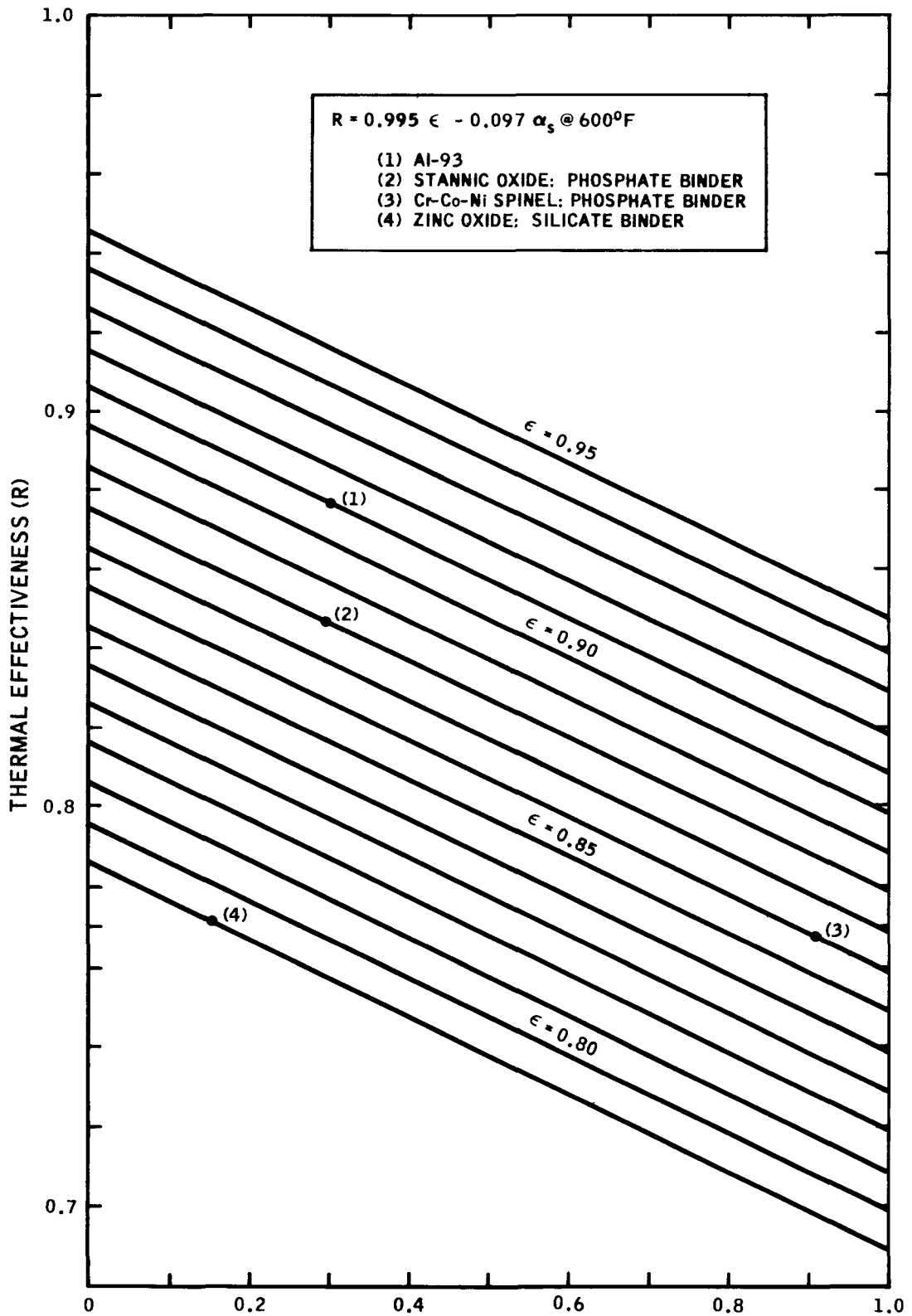
A combination of Equations 2 and 5 gives the thermal effectiveness as

$$R = \epsilon \left( 1 - \frac{K_2}{\sigma \eta T^4} \right) - \left( \frac{K_3}{2 \sigma \eta T^4} \right) \alpha_s \quad (\text{Ref. 2}) \quad \dots(6)$$

For SNAP 2 conditions (i.e., RC temperature = 600 F,  $\eta = 0.83$ )

$$R = 0.995 \epsilon - 0.097 \alpha_s. \quad \dots(7)$$

A plot of Equation 7 as a function of solar absorptance is depicted in Figure 1.



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Figure 1. Thermal Effectiveness of Spacecraft Coatings as a Function of Solar Absorptance

## 2. Coating Evaluation

The initial design requirement for the SNAP systems tailored coating was an  $\epsilon = 0.85$  and an  $\alpha_s = 0.30$ . In 1962, in conjunction with the Los Angeles Division of NAA, AI initiated a development program which lasted 1 yr. Since it was known that the SNAP coatings would operate in high neutron and gamma environments, it was decided not to investigate paints or enamels because of known instabilities in nuclear radiation fields. Additionally, it was determined that primary emphasis would be placed on utilization of two common inorganic binding agents, aluminum phosphate and potassium silicate.

Included in the several hundred coating systems investigated during this endeavor (Ref. 3 and 4) were many of the basic metal oxides, with particular interest devoted to the white oxides. Screening tests were begun with an evaluation of the spectral reflectance and total normal emittance of candidate metallic oxides including the oxides of Ba, Ce, Sb, Si, Sn, Ti, Zn, and Zr. The results of this evaluation are presented in Table 1.

From this study many important factors were determined; the most pertinent of these are:

- a) The thermal emittance at 600°F for coatings with the aluminum phosphate binder was consistently 3 to 5% higher than coatings with the potassium silicate binder.
- b) The solar absorptance for coatings with the aluminum phosphate binder was consistently 10 to 20% higher than coatings with the potassium silicate binder.
- c) The white pigment which exhibited the highest thermal emittance at 600°F was stannic oxide.

In that the optimum thermal effectiveness of the SNAP radiation control coating is determined primarily by the thermal emittance, further coating development effort emphasized use of the stannic oxide pigment and an aluminum phosphate binder. Total hemispherical emittance measurements of the stannic oxide coating system yield a value of  $\epsilon = 0.88$  at 600°F.



TABLE 1  
COATING SYSTEMS ABSORPTANCE AND  
EMITTANCE CHARACTERISTICS

NAA Coating No.	Coating Type	Frit	Pigment	Concentration (wt %)	Spectral Absorptivity (0.23-2.5 $\mu$ )	Total Normal Emittance at 600°F
G-19	Vitreous	J-232 <sup>d</sup>	TiO <sub>2</sub>	40	0.30-0.35	0.80-0.85
G-23	Vitreous	K-944	Co <sub>2</sub> O <sub>3</sub>	15	—	0.80-0.85
			CuO	1.5		
			NiO	2.5		
			Fe <sub>2</sub> O <sub>3</sub>	1.0		
GP-11	Vitreous	K-944 <sup>j</sup>	—			0.75-0.80
GP-13	Vitreous	A1-8 <sup>d</sup>	TiO <sub>2</sub>	15	0.40-0.45	0.85-0.90
GP-24	Vitreous	J-532 <sup>d</sup>	TiO <sub>2</sub>	20	0.25-0.30	0.75-0.80
GP-25	Vitreous	J-232 <sup>k</sup>	TiO <sub>2</sub>	30	0.25-0.30	0.80-0.85
S-1	K <sub>2</sub> SiO <sub>3</sub>	—	SiO <sub>2</sub>	75	0.15-0.20	0.75-0.80
S-2	K <sub>2</sub> SiO <sub>3</sub>	—	ZrO <sub>2</sub>	75	0.10-0.15	0.75-0.80
S-6	K <sub>2</sub> SiO <sub>3</sub>	—	Al <sub>2</sub> O <sub>3</sub>	75	0.10-0.15	0.70-0.75
S-8	K <sub>2</sub> SiO <sub>3</sub>	—	SnO <sub>2</sub>	60	0.15-0.20	0.85-0.90
S-9	K <sub>2</sub> SiO <sub>3</sub>	—	ZnO	60	0.15-0.20	0.80-0.85
S-10	K <sub>2</sub> SiO <sub>3</sub>	—	AlPO <sub>4</sub>	60	0.15-0.20	0.75-0.80
S-11	K <sub>2</sub> SiO <sub>3</sub>	—	CaSiO <sub>3</sub>	60	0.15-0.20	0.75-0.80
S-16	K <sub>2</sub> SiO <sub>3</sub>	—	BN	50	0.20-0.25	0.85-0.90
S-18	K <sub>2</sub> SiO <sub>3</sub>	—	BN	30	0.20-0.25	0.80-0.85
			AlPO <sub>4</sub>	20		
S-19	K <sub>2</sub> SiO <sub>3</sub>	—	Ca <sub>3</sub> (PO <sub>4</sub> ) <sub>2</sub>	50	0.15-0.20	0.85-0.90
S-27	K <sub>2</sub> SiO <sub>3</sub>	—	(Cr, Fe, Ni) Spinel	38	—	0.85-0.90
			SiO <sub>2</sub>	24		
GP-19	K <sub>2</sub> SiO <sub>3</sub>	—	ZrO <sub>2</sub>	33	0.20-0.25	0.85-0.90
			MgO	17		
GP-20	K <sub>2</sub> SiO <sub>3</sub>	—	TiO <sub>2</sub>	50	0.25-0.30	0.85-0.90
GP-22	K <sub>2</sub> SiO <sub>3</sub>	—	MgO	35	0.25-0.30	0.80-0.85
S-12	Alkaphos-C	—	SnO <sub>2</sub>	62.5	0.35-0.40	0.88-0.92

TABLE 1  
COATING SYSTEMS ABSORPTANCE AND  
EMITTANCE CHARACTERISTICS (Continued)

NAA Coating No.	Coating Type	Frit	Pigment	Concentration (wt %)	Spectral Absorptivity (0.23-2.5 $\mu$ )	Total Normal Emittance at 600°F
S-13	Alkaphos-C	-	SnO <sub>2</sub>	31	0.30-0.35	0.85-0.90
			ZrO <sub>2</sub>	19		
			CaSiO <sub>3</sub>	12		
S-24	Alkaphos-C	-	GeO <sub>2</sub>	67	0.30-0.35	0.80-0.85
S-25	Alkaphos-C	-	(Cr, Ni) Spinel	38	0.85-0.90	0.85-0.90
			SiO <sub>2</sub>	24		
S-29	Alkaphos-C	-	(Cr, Fe, Ni) Spinel	38	0.80-0.85	0.80-0.85
			SiO <sub>2</sub>	24		
S-30	Alkaphos-C	-	(Cr, Fe, Ni) Spinel	38	0.80-0.85	0.80-0.85
			SiO <sub>2</sub>	24		
S-31	Alkaphos-C	-	(Cr, Co, Ni) Spinel	38	0.85-0.90	0.85-0.90
			SiO <sub>2</sub>	24		
GP-3	(Alkaphos-C-50) (Colloidal Silica-50)	-	ZrO <sub>2</sub>	60	0.15-0.20	0.75-0.80
GP-15	(Colloidal Silica-50)	-	Lampblack (Carbon)	25	0.90-0.95	0.85-0.90
GP-23	(Colloidal Silica-50)	-	TiO <sub>2</sub>	40	0.35-0.40	0.80-0.85
GP-26	Alkaphos-C	-	AlPO <sub>4</sub>	55	0.25-0.30	0.85-0.90
101A	Vitreous	1941B*	ZrO <sub>2</sub>	10	(5)	(6)
			Zirconia Spinel	10	0.4-0.5	0.7-0.8
101B	Vitreous	1941B*	ZrO <sub>2</sub>	10	0.4-0.5	0.7-0.8
			Zirconia Spinel	20		
101C	Vitreous	1941B*	ZrO <sub>2</sub>	5	0.4-0.5	0.7-0.8
			Zirconia Spinel	25		
102A	Vitreous	5933*	Zirconia Spinel	15	0.7-0.8	0.7-0.8
102B	Vitreous	5933*	Zirconia Spinel	30	0.7-0.8	0.7-0.8
102C	Vitreous	5933*	Zirconia Spinel	45	0.7-0.8	0.7-0.8

**TABLE 1**  
**COATING SYSTEMS ABSORPTANCE AND**  
**EMITTANCE CHARACTERISTICS (Continued)**

NAA Coating No.	Coating Type	Frit	Pigment	Concentration (wt %)	Spectral Absorptivity (0.23-2.5 $\mu$ )	Total Normal Emittance at 600°F
103A	Vitreous	5933 <sup>*</sup>	{ ZrO <sub>2</sub> Zirconia Spinel	20 10	0.5	0.7-0.75
103B	Vitreous	5933 <sup>†</sup>	{ ZrO <sub>2</sub> Zirconia Spinel	20 10	0.5	0.7-0.75
104A	Vitreous	5B <sup>†</sup>	Zirconia Spinel	10	0.45-0.5	—
104B	Vitreous	5B <sup>†</sup>	Superpax	20	0.45-0.5	—
104C	Vitreous	5B <sup>†</sup>	Superpax	30	0.45-0.5	—
104D	Vitreous	5B <sup>†</sup>	{ ZrO <sub>2</sub> Zirconia Spinel	10 20	0.45-0.5	—
105A	Vitreous	74C <sup>†</sup>	Zirconia Spinel	15	—	—
105B	Vitreous	74C <sup>†</sup>	Superpax	25	—	—
106A	Vitreous	J-527+L-389	ZrO <sub>2</sub>	30	0.45-0.75	0.7 -0.75
106B	Vitreous	J-527+L-389 <sup>†</sup>	Superpax	30	0.45-0.75	0.7 -0.75
106C	Vitreous	J-527+L-389 <sup>†</sup>	ZrO <sub>2</sub>	60	0.45-0.75	0.7 -0.75
106D	Vitreous	J-527+L-389	ZrO <sub>2</sub>	50	0.45-0.75	0.7 -0.75
107A	Vitreous	L-388 <sup>*</sup>	ZrO <sub>2</sub>	22	0.45-0.75	0.75
108A	Vitreous	J-405+L-389 <sup>*</sup>	ZrO <sub>2</sub>	20	0.4 -0.6	0.75-0.8
108B	Vitreous	J-405+L-389	ZrO <sub>2</sub>	50	0.4 -0.6	0.75-0.8
108C	Vitreous	J-405+L-389	ZrO <sub>2</sub>	45	0.4 -0.6	0.75-0.8
108D	Vitreous	J-405+L-389 <sup>†</sup>	ZrO <sub>2</sub>	50	0.4 -0.6	0.75-0.8
110A	Vitreous	L-388+L-389 <sup>†</sup>	TiO <sub>2</sub>	10	—	0.7 -0.75
111A	Vitreous	J-405	Superpax	30	—	0.7 -0.75
111B	Vitreous	J-405 <sup>†</sup>	ZrO <sub>2</sub>	28	—	—
111C	Vitreous	J-405	ZrO <sub>2</sub> TiO <sub>2</sub>	15 15	—	0.7 -0.75
111D	Vitreous	J-405 <sup>†</sup>	ZnO	25	—	0.7 -0.75
111E	Vitreous	J-405 <sup>†</sup>	ZnO	12	—	—

**TABLE I**  
**COATING SYSTEMS ABSORPTANCE AND**  
**EMITTANCE CHARACTERISTICS (Continued)**

NAA Coating No.	Coating Type	Frit	Pigment	Concentration (wt %)	Spectral Absorptivity (0.23-2.5μ)	Total Normal Emittance at 600°F
112A	Vitreous	J-527 <sup>†</sup>	ZrO <sub>2</sub>	30	—	—
112B	Vitreous	J-527*	ZrO <sub>2</sub>	37	—	—
112C	Vitreous	J-527*	TiO <sub>2</sub>	30	—	—
113A	Vitreous	1941B*	(ZrO <sub>2</sub> )	(4)	0.35	0.80
113B	Vitreous	1941B*	ZrO <sub>2</sub>	2		
114A	Vitreous	1037A*	(Sb <sub>2</sub> O <sub>3</sub> )		0.35-0.4	0.80
114B	Vitreous	1037A*	Sb <sub>2</sub> O <sub>3</sub>	2	0.35-0.4	—
114C	Vitreous	1037A*	Sb <sub>2</sub> O <sub>3</sub>	7		
			Clay	4	0.35-0.4	—
115A	Vitreous	XT-272*	(TiO <sub>2</sub> )		0.2 -0.3	0.7 -0.8
115B	Vitreous	XT-272B*	(TiO <sub>2</sub> )		0.2 -0.3	0.7 -0.8
115C	Vitreous	XT-272*	(TiO <sub>2</sub> )		0.2 -0.3	0.7 -0.8
115D	Vitreous	XT-272 and L-389 <sup>‡</sup>	(TiO <sub>2</sub> )			
GP-1	Alkaphos-C	—	{ SiO <sub>2</sub>	33	0.15-0.25	0.65-0.75
	Bonded		{ TiO <sub>2</sub>	33		
GP-2	Bonded	—	{ SiO <sub>2</sub>	33	0.15-0.25	0.65-0.75
			{ ZrO <sub>2</sub>	33		
GP-3	Alkaphos-C		{ SiO <sub>2</sub>	20	0.15-0.25	0.65-0.75
			{ ZrO <sub>2</sub>	60		
GP-4	Alkaphos-C	—	{ SiO <sub>2</sub>	60	0.15-0.25	0.65-0.75
			{ ZrO <sub>2</sub>	20		
GP-5	Potassium Silicate	—	MgO	50	0.15-0.25	0.65-0.75
	Bonded					
GP-6	Alkaphos-C	—	{ SiO <sub>2</sub>	25	0.15-0.25	0.65-0.75
	Bonded		{ TiO <sub>2</sub>	25		
			{ ZrO <sub>2</sub>	25		

**TABLE 1**  
**COATING SYSTEMS ABSORPTANCE AND**  
**EMITTANCE CHARACTERISTICS (Continued)**

NAA Coating No.	Coating Type	Frit	Pigment	Concentration (wt %)	Spectral Absorptivity (0.23-2.5 $\mu$ )	Total Normal Emittance at 600°F
61-1	Plasma-Spray	—	{ MgO Al <sub>2</sub> O <sub>3</sub>	34 66	0.3 - 0.4	0.55-0.7
61-2	Plasma-Spray	—	{ MgO ZrO <sub>2</sub>	34 66	0.3 - 0.4	0.55-0.7
61-3	Plasma-Spray	—	{ MgO Al <sub>2</sub> O <sub>3</sub>	66 34	0.3 - 0.4	0.55-0.7
61-4	Plasma-Spray	—	{ Al <sub>2</sub> O <sub>3</sub> NiO	34 66	0.3 - 0.4	0.55-0.7
61-6	Plasma-Spray	—	{ Al <sub>2</sub> O <sub>3</sub> ZrO <sub>2</sub>	50 50	0.3 - 0.4	0.55-0.7
61-11	Plasma-Spray	—	{ Al <sub>2</sub> O <sub>3</sub> ZrO <sub>2</sub>	34 66	0.3 - 0.4	0.55-0.7
61-12	Plasma-Spray	—	{ Al <sub>2</sub> O <sub>3</sub> ZrO <sub>2</sub>	66 34	0.3 - 0.4	0.55-0.7

Commercially Available Frits.

J-232 }  
 J-532 } DuPont Corp.  
 K-944 }

Al-8 - Ferro Co.

5933 - Hammel Corp.

J527 }  
 L-388 }  
 L-389 } DuPont Corp.  
 J-405 }  
 J-527 }

1037 }  
 1941B } Ferro Corp.  
 XT-272 }

NAA Formulated Frits

Pigments.

TiO<sub>2</sub> - Matheson Coleman & Bell (Reagent Grade)

Co<sub>2</sub>O<sub>3</sub> - Matheson Coleman & Bell (Reagent Grade)

Cr<sub>2</sub>O<sub>3</sub> - Matheson Coleman & Bell (Reagent Grade)

MgO - Mallinckrodt (USP)

CA<sub>3</sub>(PO<sub>4</sub>)<sub>2</sub> - Mallinckrodt (USP)

ZrO<sub>2</sub> - Mallinckrodt (USP)

Al<sub>2</sub>O<sub>3</sub> - Mallinckrodt (USP)

SiO<sub>2</sub> - Glasrock Corp. (Reagent)

AlPO<sub>4</sub> - Fisher Co.

BN - National Carbon Co.

CaSiO<sub>3</sub> - G. F. Cabot Co.

CuO - Baker & Adamson (Anal. Reagent)

NiO - Baker & Adamson (Anal. Reagent)

Fe<sub>2</sub>O<sub>3</sub> - Baker & Adamson (Anal. Reagent)

SnO<sub>2</sub> - Baker & Adamson (Anal. Reagent)

ZnO - Baker & Adamson (Anal. Reagent)

Sb<sub>2</sub>O<sub>3</sub> - Baker & Adamson (Anal. Reagent)

Zirconia Spinel (Zirconia Alumina Silicate) - Titania

Alloy Mfg. Co.

Superpax (Zirconia Silicate) - Titania Alloy Mfg. Co.



The thermal emittance of a coating system of finite thickness is not a surface phenomenon, as markedly as is the property of solar absorptance, but is a bulk effect encompassing coating thickness, porosity, surface texture, etc. An effort was made to further increase the emittance of the SnO<sub>2</sub> coating system by analyzing its spectral emittance (emittance vs. wavelength) properties. It was noted that in the near infrared region (1 to 4 μ) the emittance of the SnO<sub>2</sub> coating was depressed below 0.90.

Many black coatings have a high thermal emittance in this short wavelength region. Thus, to further increase the bulk emittance, a composite coating system was devised in an effort to maximize the spectral emittance throughout the infrared region. This was accomplished by utilizing a high thermal emittance, black subcoat and the stannic oxide as a topcoat. The black subcoat is a mixture of the oxides of chromium, cobalt and nickel. Total hemispherical emittance measurements of the two layer composite coating yielded a value of  $\epsilon = 0.91$  at 600 °F.

A chronological appraisal of coating systems, from which the optimum SNAP system coating was chosen, follows (see Figure 1):

<u>Pigment</u>	<u>Binder</u>	<u>Coating Thickness (in.)</u>	<u>Temp. (°F)</u>	<u>Total Hemispherical Emittance</u>
Boron nitride	Potassium	0.002	500	0.83
	Silicate		600	0.80
			700	0.78
Zinc oxide	Potassium	0.002	600	0.79
	Silicate		700	0.77
Stannic oxide	Potassium	0.003	500	0.87
	Silicate		600	0.85
			700	0.84
Stannic oxide	Aluminum	0.003	500	0.88
	Phosphate		600	0.88
			700	0.88

<u>Pigment</u>	<u>Binder</u>	<u>Coating Thickness (in.)</u>	<u>Temp. (°F)</u>	<u>Total Hemispherical Emittance</u>
Cr-Co-Ni spinel	Aluminum	0.002	500	0.86
	Phosphate		600	0.86
			700	0.86
AI93	Aluminum	0.003	500	0.91
Subcoat Cr-Co-Ni spinel	Phosphate		600	0.91
Topcoat stannic oxide			700	0.91

### 3. Radiation Measurements of AI93

The heat-transfer calculations for the SNAP system coating surfaces are based on knowledge of total hemispherical emittance and solar absorptance. Total hemispherical emittance is determined by measuring the power required to maintain an emitting specimen at constant temperature. A description of the total hemispherical emittance measurement technique utilized by AI is given in Section III of this report.

The solar absorptance measurements, which are referred to in this paper, were performed by the Los Angeles Division of NAA. The ultraviolet, visible and near infrared reflectance were measured with a spectrophotometer, Beckman DK-2, having an integrating sphere attachment. All measurements were by comparison to freshly prepared magnesium oxide which is accepted as a reflectance standard in the short wavelength region.

Over 30 distinct AI93 coating samples have undergone total hemispherical emittance measurement. The processing of these samples extended over a 2-yr period and repeatability in emittance values was excellent. A typical long-term, high-temperature emittance record of the AI93 coating is shown in Table 2. A curve of the total hemispherical emittance of AI93 as a function of temperature is given in Figure 2.

In May 1962, the AEC requested that the National Bureau of Standards (NBS) perform emittance measurements on the AI93 coating system. Thus, AI prepared and submitted four coated emittance strips to the Bureau. The NBS subsequently delivered the emittance strips to Pratt and Whitney Aircraft, Hartford,

TABLE 2

## TOTAL HEMISPHERICAL EMITTANCE OF AI93\*

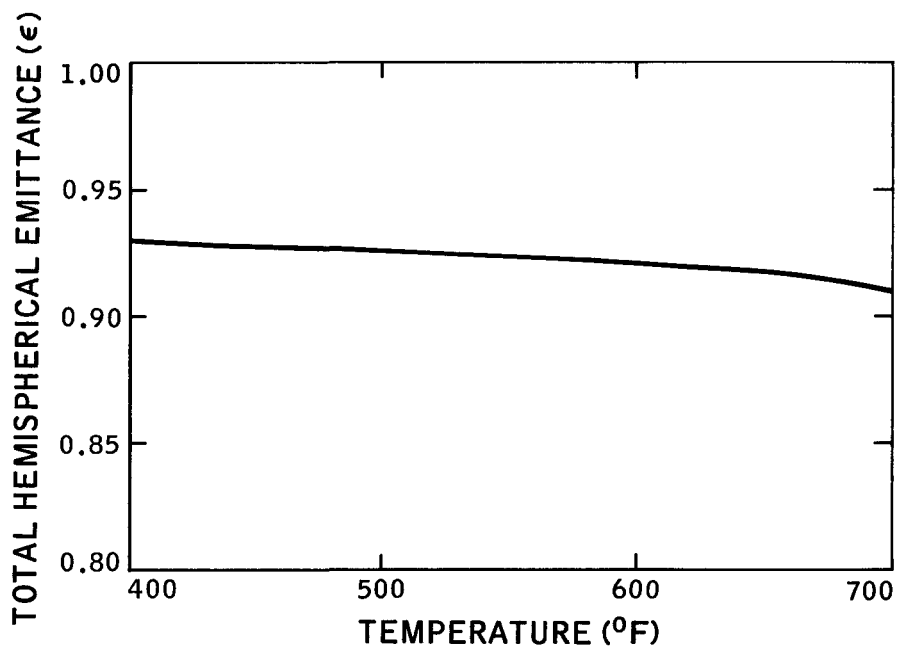
Time (hr)	Temperature (°F)	Vacuum (torr)	Total Hemispherical Emittance
21	598	$1.0 \times 10^{-5}$	0.92
22	399	$1.0 \times 10^{-5}$	0.93
23	500	$1.0 \times 10^{-5}$	0.92
24	700	$9.0 \times 10^{-6}$	0.92
46	705	$1.5 \times 10^{-6}$	0.92
47	600	$1.5 \times 10^{-6}$	0.92
48	402	$1.4 \times 10^{-6}$	0.93
146	604	$4.0 \times 10^{-7}$	0.91
147	404	$4.0 \times 10^{-7}$	0.92
148	710	$4.0 \times 10^{-7}$	0.90

## \*Coating System:

Topcoat - stannic oxide with aluminum phosphate binder

Subcoat - chromium, nickel, cobalt, spinel with aluminum phosphate binder

Composite coating thickness - 0.003 in.



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Figure 2. Emittance of Tailored Coating AI93

Connecticut for measurement. The result of this measurement is shown in Figure 3. Specimen No. 3 was for the AI93 coating on a copper substrate. Note that the Pratt and Whitney data are in excellent accord with results attained by AI.

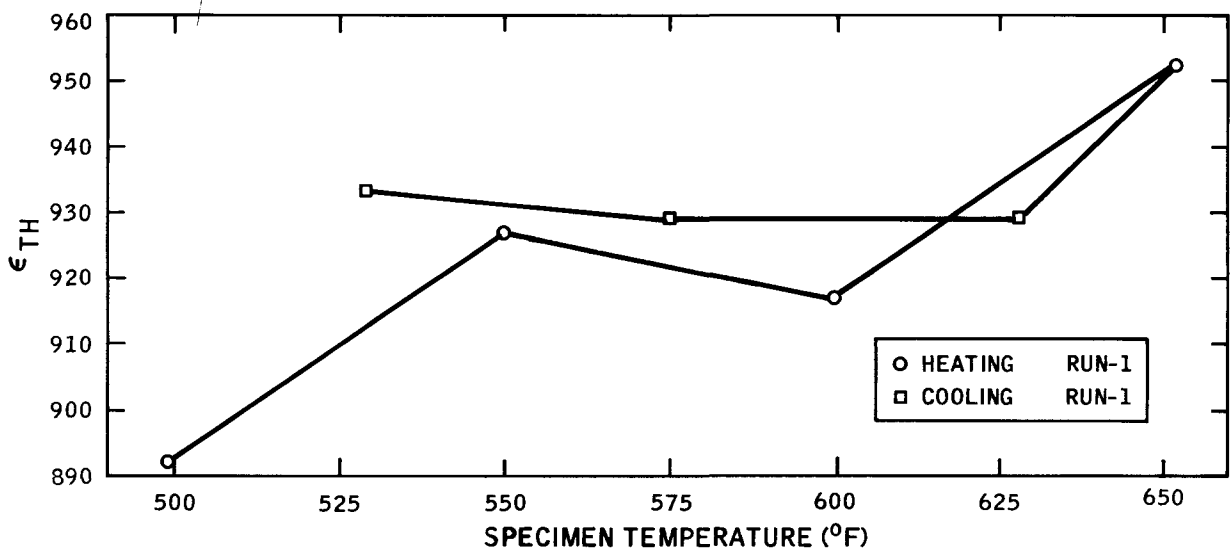
#### 4. Material and Process Requirements

The AI93 coating system is typical of most ceramics presently being used for spacecraft radiation control surfaces. Stringent control must be exercised in the material formulation and process application. However, these tight controls result in consistent adherence and thermal properties being attained.

Use of the AI93 coating at AI is presently made on three substrates: aluminum, copper and titanium alloy. Because each of these base metals presents unique problems in surface preparations, separate AI93 coating specifications have been prepared for substrate application:

NA0109 – 020 Application of a High Emittance Tin Oxide Coating to Aluminum Substrate

NA0109 – 022 Application of a High Emittance Tin Oxide Coating to Tempered Aluminum Alloys



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Figure 3. Total Hemispherical Emittance vs Temperature (AI93, Specimen No. 3, Pratt and Whitney Aircraft)

NA0109 – 023 Application of a High Emittance Tin Oxide Coating to  
Copper Substrate

NA0109 – 014 Application of a High Emittance Tin Oxide Coating to  
Titanium Substrate.

Fullscale processing of components with AI93 has been in effect for 2 yr. The AI93 coating as applied to the SNAP 2, RC-6 is shown in Figure 4. This unit has a copper substrate and is 10 ft high. The coating cure cycle for this particular application was performed in air at 600°F. Fifteen minutes at temperature was sufficient for proper curing of the aluminum phosphate ceramic. The AI93 coating as applied to a SNAP 2 instrument compartment package is shown in Figure 5. The substrate was Aluminum 6061-T6; thus, a minimum temperature cure was required. The coating cure cycle for this component was performed in air at 450°F.

Two major components of the SNAP 10A system are the thermoelectric pump and the thermoelectric converter system. The AI93 coating as processed on the thermoelectric pump aluminum radiator fins is shown in Figure 6. Two thermoelectric module converter leg sections mounted in a liquid metal acceptance test loop are shown in Figure 7. The AI93 coating was processed on the aluminum fins of the converter section subsequent to module fabrication. Both SNAP 10A component processes were performed in high purity argon at 600°F.

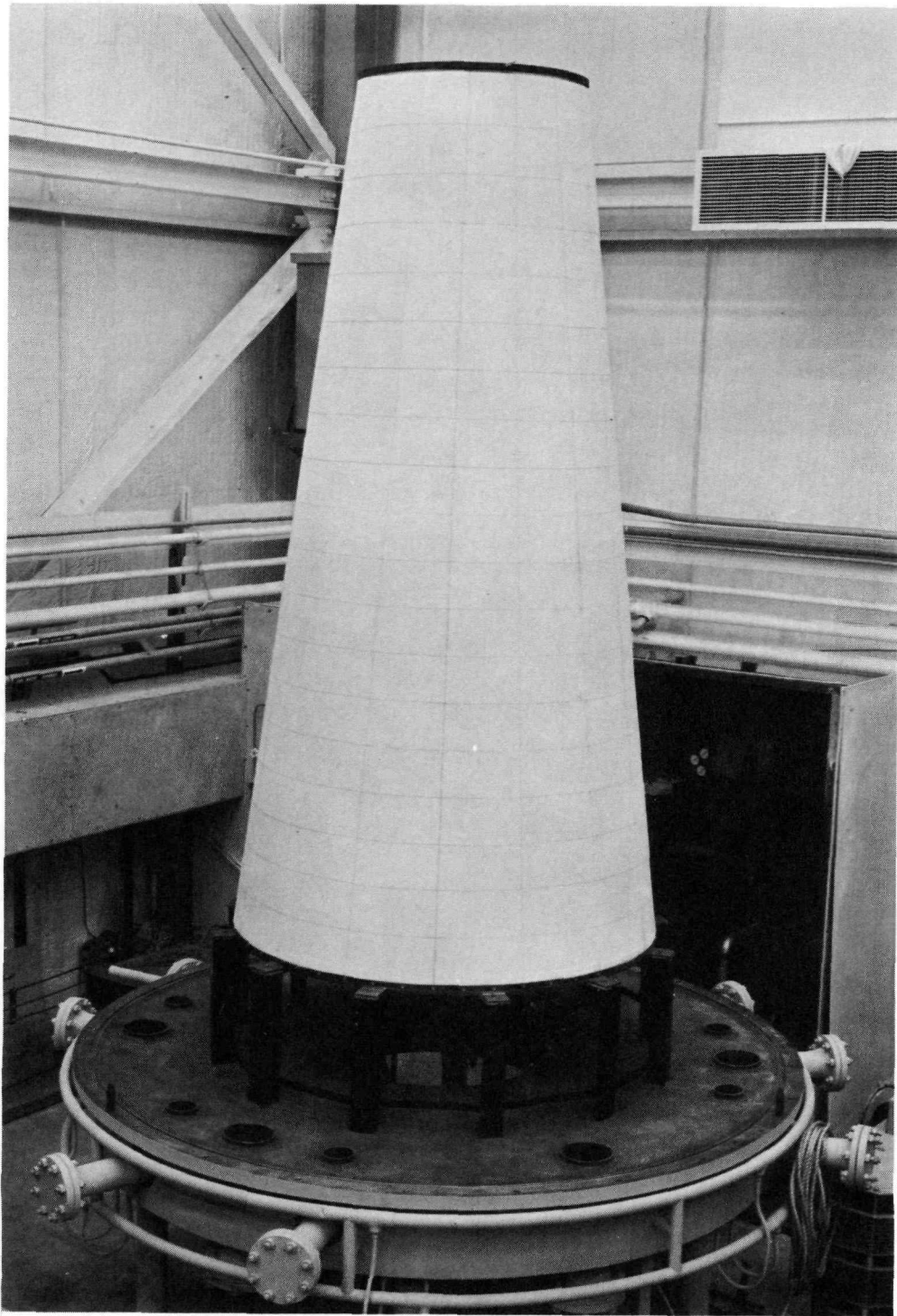
## B. QUALIFICATION OF TAILORED COATING (AI93)

### 1. Long-term Stability of AI93 at 600°F in Vacuum

A qualification requirement performed was long-term emittance stability at 600°F in vacuum. The stability data obtained are:

<u>Coating System</u>	<u>Hours at 600°F 10<sup>-5</sup> torr</u>	<u>Total Hemispherical Emittance</u>
No. 1 Tailored coating AI93	24	0.91
as adaptable to copper	1000	0.91
substrates	2000	0.91
	3000	0.91
	4000	0.93
	5900	0.93



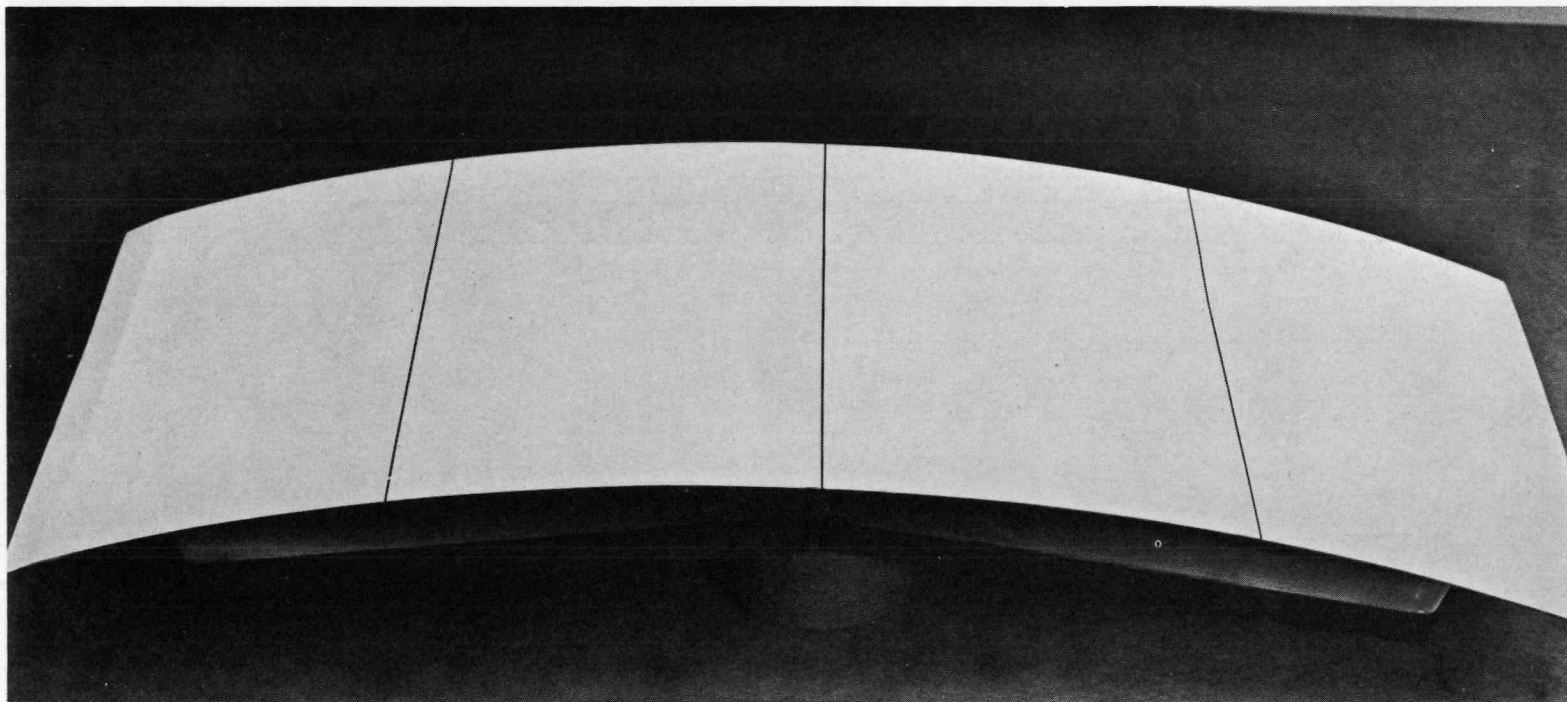


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Figure 4. AI93 Coating on SNAP 2 RC-6

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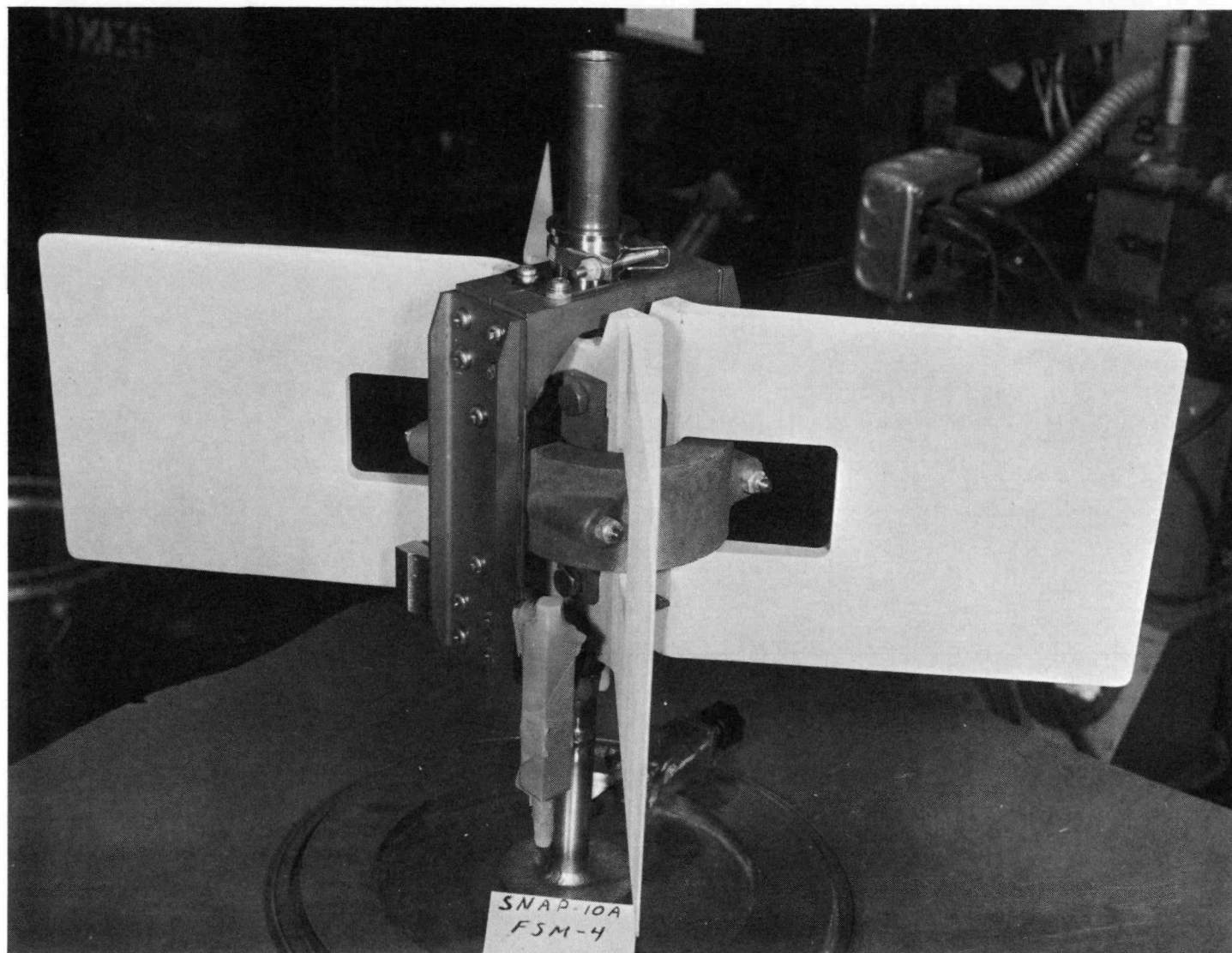


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Figure 5. AI93 Coating on SNAP 2 Instrument Compartment

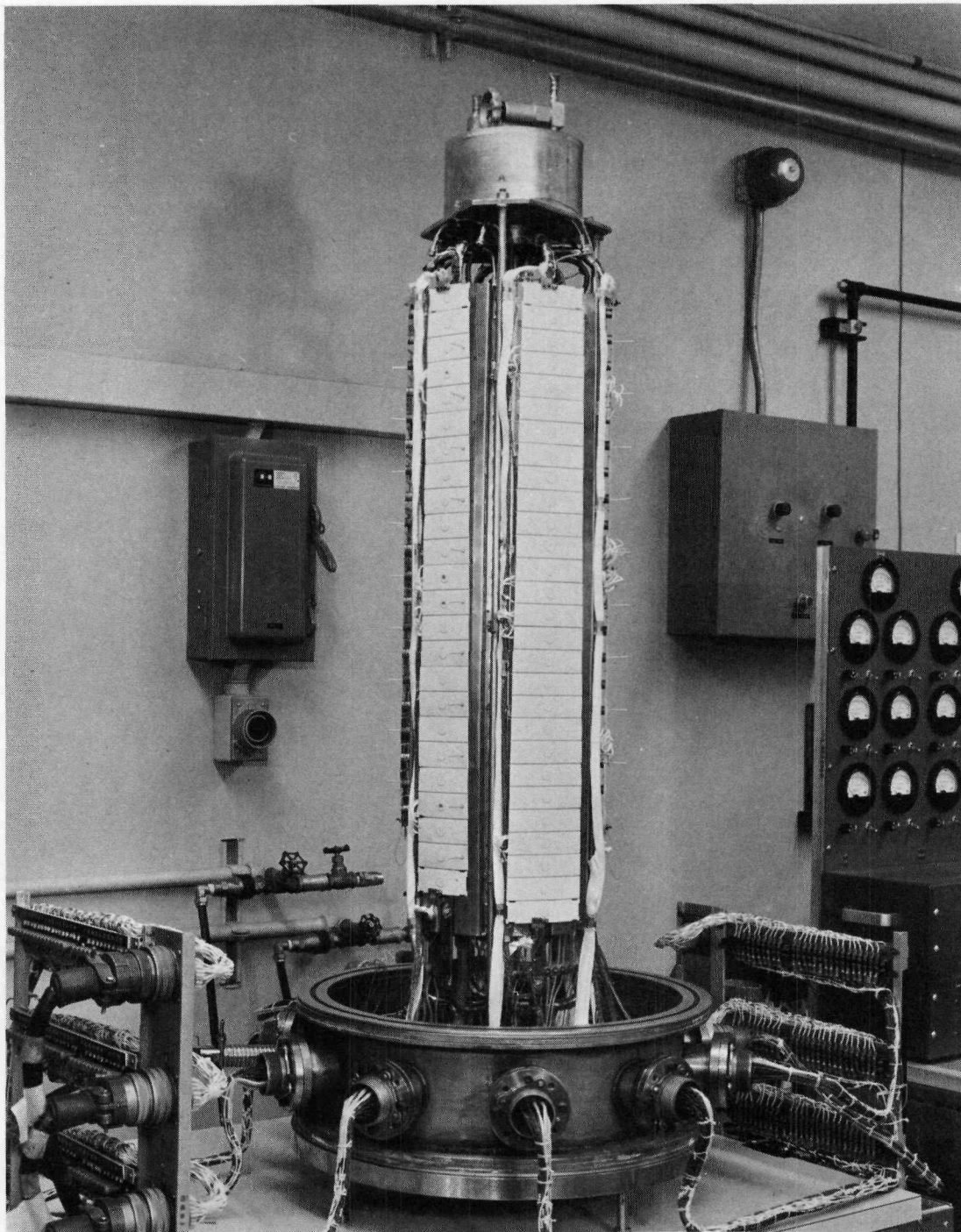
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Figure 6. Al93 Coating on SNAP 10A Thermoelectric Pump



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Figure 7. Al<sub>93</sub> Coating on SNAP 10A Thermoelectric Module



No. 2 Tailored coating AI93	24	0.90
as adaptable to	1000	0.90
aluminum substrates	2000	0.90
	3000	0.92
	4900	0.92

Another long-term stability test of the tailored coating has been completed. During this 2000-hr test, the emittance sample was maintained at 600°F in an ultra-high vacuum ( $10^{-9}$  torr). Prior to the test, the emittance of the sample was 0.90. Subsequent to the 2000-hr test, the emittance of the sample was 0.89.

### 2. Aerodynamic Heating of AI93

An environmental qualification test was performed to determine the adherence qualities of the AI93 coating system. The environment simulated that which would be experienced by the coating during the ascent of the vehicle.

The AI93 coating was applied to 2- by 3-ft panels of aluminum and copper. The ascent heatup rate for the copper panel was ambient to 680°F in 141 sec. The ascent heatup rate for the aluminum panel was ambient to 770°F in 141 sec. Each panel underwent 10 cycles using these heatup rates. No damage to the coating occurred during either test.

### 3. Mechanical Vibration

Mechanical vibration qualification of the AI93 coating system was performed on a honeycomb panel similar in configuration to the SNAP 2 copper face sheet, honeycomb RC. A 2- by 4-ft test panel was used for this test. It was fabricated of 0.005-in. skin, sandwiching 0.001-in. core PH15-7 Mo honeycomb material. A 0.030-in. copper face sheet was brazed to the honeycomb. One Hg tube, which runs lengthwise through the panel, was incorporated in the structure.

The copper face sheet was coated with the high-emittance AI93 coating. Previous testing of the coated panel had included thermal cycle tests from +600°F to -100°F.

The emittance coating successfully withstood strain levels of 2072  $\mu$ in. (approximately 62,000 psi stress) without apparent damage. The 2-min test at the first resonant mode (22 cps) resulted in over 2600 cycles of reversed loading on the coating material. Lesser strain levels occurred during the tests conducted at the second and third resonant modes. The various mode shapes were

determined by use of small sugar crystals as shown in Figure 8. Details of this test are included in Ref. 5.

#### 4. Nuclear Irradiation

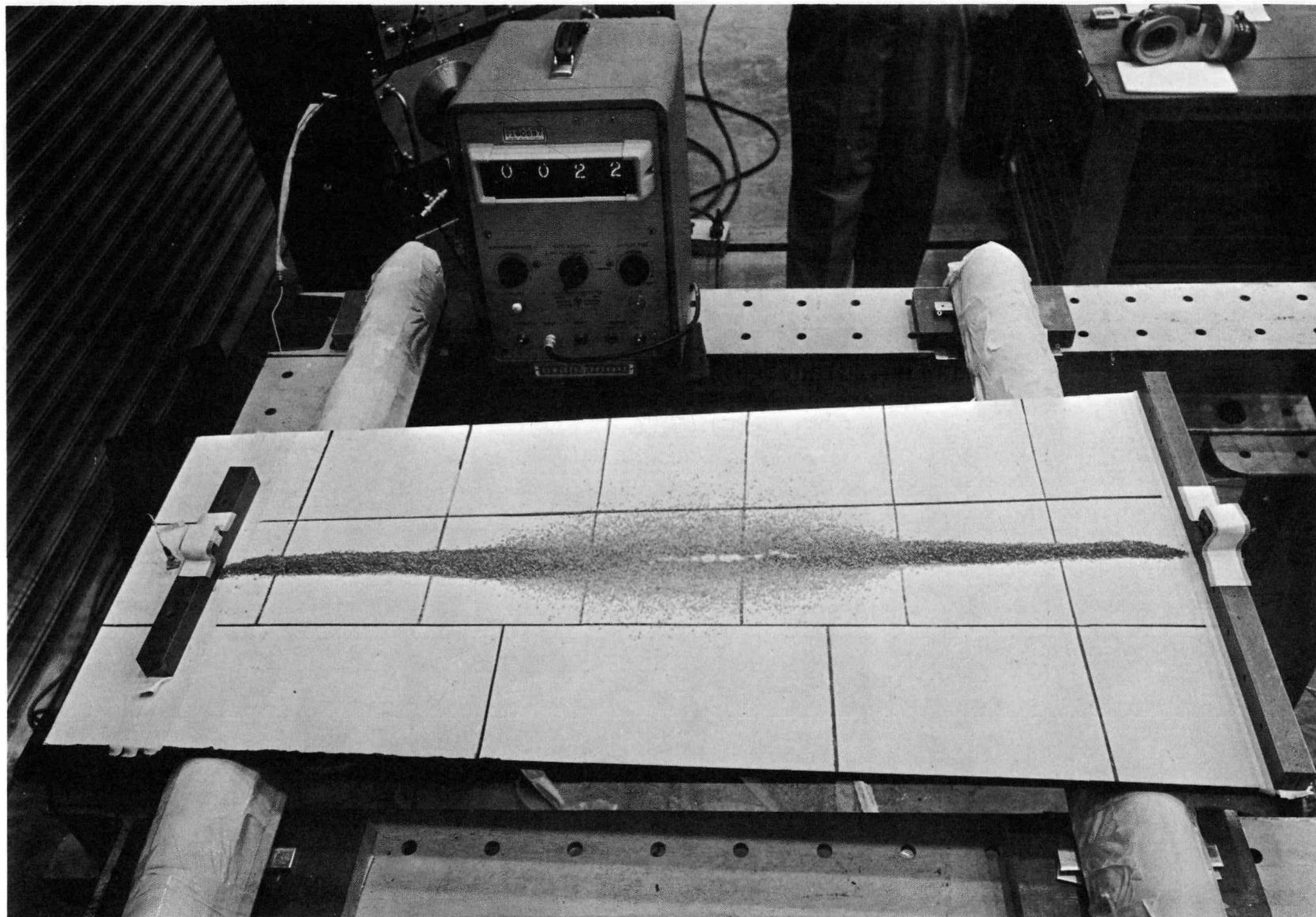
A major requisite for qualification of the AI93 coating for SNAP systems is the emittance stability of the coating in a high density nuclear environment. Depending on component location with respect to the reactor, the coating is required to perform effectively for 1-yr in an isodose region which may range from  $10^{14}$  to  $3 \times 10^{18}$  nvt. To obtain the high irradiation level of  $3 \times 10^{18}$  nvt, a program was formulated to perform this irradiation in the Battelle Memorial Institute (BMI) research reactor at Columbus, Ohio.

The emittance coating stability program could be conducted in either of two ways. One method would be to place the coating in a capsule, irradiate it to a desired total integrated fast neutron dose level, and then perform emittance measurements of the coating in a hot-cell facility. The major disadvantage of this method is that continuous monitoring of the emittance stability as a function of accumulated fast neutron dosage could not be maintained. A second disadvantage is the questionable reliability of sensitive optical emittance measurement equipment in a hot-cell environment.

The second approach utilizes the technique by which total hemispherical emittance measurements are performed at AI. The coating is applied to a high resistivity metallic substrate such as nichrome. The nichrome substrate acts as a resistance heater, and the total hemispherical emittance of the coating is determined by measurement of the Stefan-Boltzmann equation parameters: 1) power input, 2) to a known coated area, and 3) the steady-state temperature of that coated area. Coated specimens, which are instrumented for power and temperature measurements, are placed in an irradiation capsule, and continuous in-pile emittance measurements are performed (Figure 9). In that the data obtained from this type of measurement is the most realistic, this approach was utilized.

Six high-emittance coating specimens were chosen to be tested; the number was limited by two factors. The first limitation was the amount of input power which could be supplied to the irradiation capsule in order to maintain the high-emittance test specimens at  $600^{\circ}\text{F}$ , in vacuum. Secondly, the amount of coating surface area which could be allowed to radiate to the capsule walls had

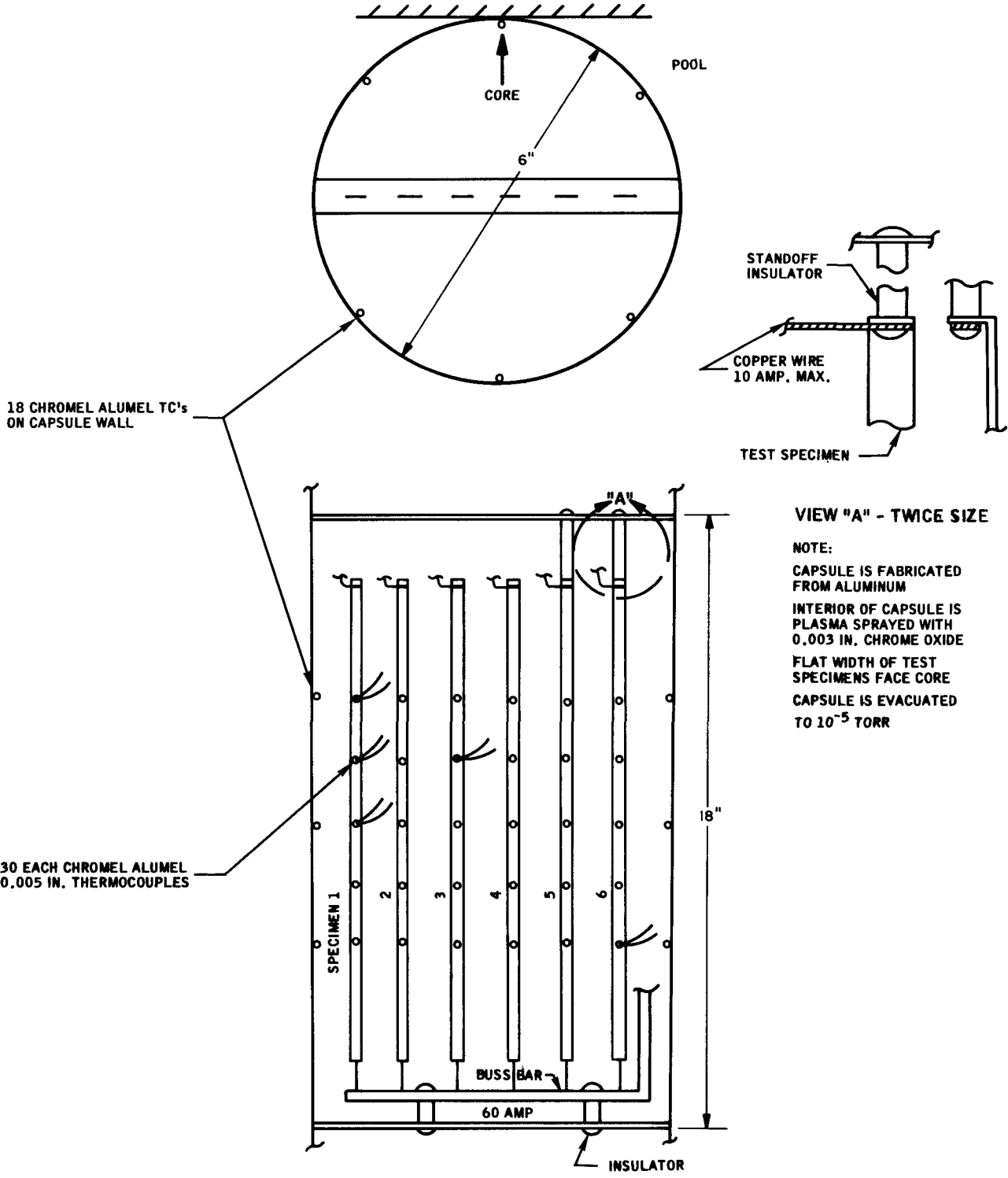
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Figure 8. AI93 Coated Panel Sugar Crystal Pattern at First Bending Mode



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Figure 9. Capsule Loading for Emittance Measurement



to be kept to a minimum. The capsule walls were coated with a high absorptance material (chromic oxide), and operated at the reactor pool temperature, 118°F.

A description of the six coating specimens follows:

Sample No. 1 and No. 2

Subcoat – vitreous ceramic, 0.8 mil thick

Intermediate coat – aluminum phosphate bonded chrome-cobalt-nickel spinel, 1.5 mil thick

Topcoat – aluminum phosphate bonded tin oxide, 0.8 mil thick

Sample No. 3 and No. 4

Subcoat – aluminum phosphate bonded chrome-cobalt-nickel spinel, 1.5 mil thick

Topcoat – aluminum phosphate bonded tin oxide, 0.8 mil thick

Sample No. 5 and No. 6 – Plasmasprayed chromic oxide, 3 mil thick

Although the total hemispherical emittance measurements performed at BMI were absolute measurements, the reliance in the test data was primarily placed on noting relative changes in emittance as a function of time in-pile.

The first step in this test program was to perform total hemispherical emittance measurements of the six coating specimens at AI. Column II, Table 3, includes the results of the absolute total hemispherical emittance measurements performed prior to the nuclear irradiation test. Note that the data is fully corrected for errors inherent in the measurement and that the remaining uncertainty in measurement is 2.0%.

The next step in the emittance stability program was the insertion and instrumentation of the test specimens into the irradiation capsule. The capsule was placed in the reactor pool, the system evacuated to  $10^{-5}$  torr, and the specimens heated to 600°F. An initial emittance measurement (Column III, Table 3) was performed prior to the reactor going to power.

After performing the pre-irradiation emittance measurement in the pool with the reactor at zero power, the power input to the test specimens was turned off, and the specimens were allowed to stabilize to an equilibrium temperature. At this point, the reactor power was turned on and a measurement of the gamma heating was made by noting the temperature of the test specimens. Next, the power input to the coated nichrome strips was adjusted so as to bring the specimen temperature to 600°F. Throughout the following 12-day irradiation period,

TABLE 3

IN-PILE EMITTANCE STABILITY  
DATA FOR AI93 COATING

Column I	Column II	Column III	Column IV	Column V
Specimen No.	Absolute Total Hemispherical Emittance. Uncertainty in measurement is 2.0%	Indicated Emittance Prior to Nuclear Irradiation	Indicated Emittance After Nuclear Irradiation	Change in Emittance
1- Tailored coating applicable to copper substrate	0.90	0.93	0.94	1%
2- Tailored coating applicable to copper substrate	0.90	0.93	0.92	~ 1%
3- Tailored coating applicable to aluminum substrate	0.90	0.99	0.98	~ 1%
4- Tailored coating applicable to aluminum substrate	0.91	0.91	0.89	~ 2%
5- Chromic oxide plasmaspray	0.84	0.90	0.87	~ 3%
6- Chromic oxide plasmaspray	0.84	0.89	0.86	~ 3%

20 emittance measurements were made for each specimen. The emittance was determined from the relationship

$$\epsilon = \frac{P}{\sigma A (T_s^4 - T_\gamma^4)}$$

where

P = electrical power input to the test specimen

$\sigma$  = Stefan-Boltzmann constant

A = area of the test specimen

$\epsilon$  = total hemispherical emittance

$T_s$  = test specimen temperature, absolute

$T_\gamma$  = test specimen temperature due to gamma heating, absolute

The emittance stability data obtained for the six samples is also shown in Table 3. A typical in-pile emittance stability record of an AI93 coating sample is shown in Table 4.

The four AI93 tailored coating samples were irradiated to a neutron flux level of  $3 \times 10^{18}$  nvt. The maximum gamma exposure was  $1.9 \times 10^{10}$  R. During the irradiation test, the samples were maintained at 600°F,  $10^{-5}$  torr, for 288 hr. The change in total hemispherical emittance of three of the AI93 samples was ~1%. The fourth AI93 sample experienced a change in emittance of 2%.

This data indicates that the AI93 coating is quite stable in the nuclear irradiation environment of the SNAP 2 and 10A systems. Vacuum-temperature stability in these environments has also been demonstrated.

##### 5. Electron Irradiation

The initial high-energy-particles bombardment test of the AI93 tailored coating was performed in the AI Statitron. Prior to the electron irradiation, the total hemispherical emittance of the coated specimen was 0.90. During the Statitron test, the coated specimen was maintained at 600°F and received an integrated electron dosage of  $3 \times 10^{18}$ , 1.25-MeV energy.

TABLE 4

EMITTANCE STABILITY OF IRRADIATED SPECIMEN NO. 1  
 (Al93 coating as adaptable to copper substrate)

Reading No.	Date	Time	Total Integrated Fast Neutron Dose (nvt)	Temperature (°F)	Total Hemispherical Emittance
1	10-31-62	1015	0	602	0.93
2	10-31-62	1900	$4.3 \times 10^{16}$	597	0.93
3	11-1	2100	$3.2 \times 10^{17}$	597	0.93
4	11-2	0900	$4.5 \times 10^{17}$	598	0.94
5	11-2	2100	$5.8 \times 10^{17}$	597	0.94
6	11-3	0900	$7.1 \times 10^{17}$	598	0.94
7	11-3	2100	$8.4 \times 10^{17}$	597	0.94
8	11-4	0900	$9.7 \times 10^{17}$	599	0.94
9	11-4	2100	$1.1 \times 10^{18}$	598	0.94
10	11-5	0900	$1.2 \times 10^{18}$	599	0.92
11	11-5	2100	$1.3 \times 10^{18}$	598	0.93
12	11-6	0900	$1.5 \times 10^{18}$	599	0.94
13	11-6	2100	$1.6 \times 10^{18}$	599	0.93
14	11-7	0900	$1.8 \times 10^{18}$	599	0.94
15	11-7	2100	$1.9 \times 10^{18}$	599	0.93
16	11-8	0900	$2.0 \times 10^{18}$	599	0.94
17	11-9	0900	$2.3 \times 10^{18}$	597	0.93
18	11-10	0900	$2.5 \times 10^{18}$	598	0.93
19	11-11	0900	$2.8 \times 10^{18}$	599	0.93
20	11-12	0500	$3.0 \times 10^{18}$	599	0.93
Power off					
21	11-12	1000	$3.0 \times 10^{18}$	594	0.94

After irradiation, visual examination of the specimen showed no damage to, or discoloration of, the coating. Emittance measurements were repeated. The emittance of the coated specimen was 0.88.

Electron irradiation of the coated specimen was again repeated. During this test the temperature of the specimen was maintained at 130°F to ascertain low-temperature irradiation effects. Irradiation conditions were an integrated electron dosage of  $10^{18}$ , 1.25-MeV energy level. No damage to or discoloration of the coating occurred. Emittance measurements were repeated. The emittance of the coated specimen was 0.89. The change in total hemispherical emittance of the coating noted during these tests was within the 2% uncertainty of the emittance measuring device.

#### 6. Ultraviolet Irradiation

The stability tests of the AI93 coating system in a simulated space ultraviolet environment are presently being performed at Hughes Aircraft Company, Culver City, California. The tests being performed are described as follows:

<u>AI93 Sample No.</u>	<u>Temperature (°F)</u>	<u>Solar Factor</u>	<u>Sun Equivalent (hr)</u>
1	150	5	3000
2	150	10	3000
3	600	5	3000
4	600	10	3000

The vacuum attained during test is  $10^{-7}$  torr.

The 3000-hr test is, as yet, incomplete, however initial 1000- and 2000-hr sun-equivalent data shows some degradation occurring, particularly with the high-temperature sample. Initial results obtained from the test are:

<u>Sample No.</u>	<u>Temperature (°F)</u>	<u>Sun Equivalent (hr)</u>	<u>Change in <math>\alpha_s</math></u>
1	150	1000	0.03
—	—	2000	0.05
3	600	1000	0.06

7. Launch Environment Exposure of AI93 at Vandenberg Air Force Base (VAFB)

A qualification requirement for the AI93 radiation control coating is that it undergo exposure at VAFB. This environmental test includes outdoor and indoor exposure for 30 and 90 days respectively.

These tests are complete. The 90-day indoor exposure resulted in no change in thermal emittance or solar absorptance for the AI93 coating system. However, visual inspection indicated that the 30-day outdoor exposure caused severe deterioration of the coating solar absorptance. The  $\alpha_s$  of the exposed coating was estimated to be  $\sim 0.60$ .

The outdoor exposure test clearly indicates that these control surfaces must be protected from the salt air environment. Two approaches to this protection are promising: the use of a stripable tape which can be removed just prior to launch; or the maintenance of a clean atmosphere on the system until just prior to launch.

C. LOW EMITTANCE COATINGS

1. Pure Gold Foil

a. Radiation Properties

The SNAP 2 system utilizes pure gold foil as radiative insulation. The foil is attached to the SS tubing or components by: 1) spotwelding, or 2) light-weight band clamps.

Total hemispherical emittance measurements performed on pure gold foil and Type 300 SS alloys yield the following values:

<u>Material</u>	<u>Temperature (°F)</u>	<u>Total Hemispherical Emittance</u>
Pure Gold Foil	800-1200	0.04
SS Type 300 Series Alloy	800-1200	0.22

If the SNAP systems utilized a composite gold-SS radiation surface, e.g., electroplated gold on SS, radiation transfer calculations would be based on the emittance value,  $\epsilon = 0.04$ . Since the SNAP 2 system radiation surface is a low-emittance foil wrap on SS, a new radiation relationship must be determined.

This relationship is termed "effective emittance." (Ref. 6). Where zero contact exists between tubing and a foil-wrap system, the effective emittance ( $\bar{\epsilon}$ ) is given by

$$\bar{\epsilon} = \frac{\epsilon_{\text{foil}}}{(2 - \epsilon_{\text{foil}}) + \left(\frac{\epsilon_{\text{foil}}}{\epsilon_{\text{tubing}}}\right)} \quad \dots (8)$$

For SNAP system materials and conditions, the effective emittance for the case of zero foil-to-tubing contact would be

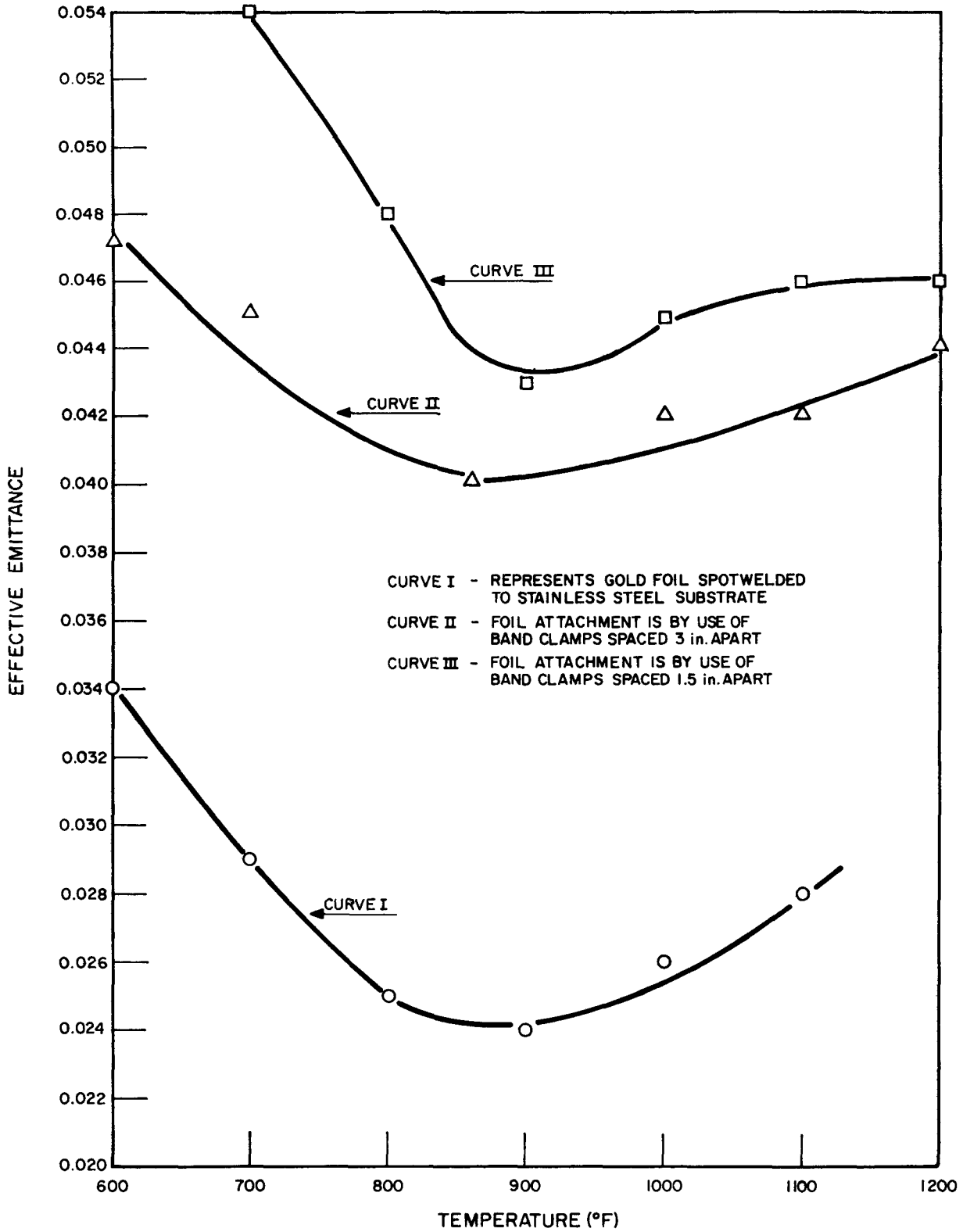
$$\bar{\epsilon} = \frac{\epsilon_{\text{gold}}}{\left(2 - \epsilon_{\text{gold}}\right) + \left(\frac{\epsilon_{\text{gold}}}{\epsilon_{\text{SS}}}\right)} = 0.019 \quad \dots (9)$$

At present, foil is attached by spiral wrapping 1-in.-wide strips of gold foil along the SS tubing. An effort is made to maintain a snug fit between the foil and the tubing. The end product results in some unknown amount of surface contact between the foil and the tubing.

Thus, a test was devised to determine experimentally the effective emittance of the SNAP 2 gold-foil wrap system. The results of this test are presented in Figure 10. Curve I represents the case of spotwelded foil attachment; curves II and III represent cases of band clamp foil attachment. The spacing between clamps is 3 in. for curve II; for curve III the spacing between clamps is 1.5 in.

Figures 11 and 12 represent the test equipment which was utilized to perform the test. Figure 11 is the heater section with nichrome ribbon wrapped spirally along a high purity Alundum tube. Molybdenum baffles are located approximately 3 in. from each end of the heater section. The baffles divide the heater section into three parts, a central 6-in.-section and two guard heater sections. Power input to the three sections can be varied independently.

As shown in Figure 12, a gold-foil-wrapped SS tube is slipped over the heater section. The molybdenum baffles prevent radiation heat transfer from the region of the central heater section to the ends of the tube. Thermocouples

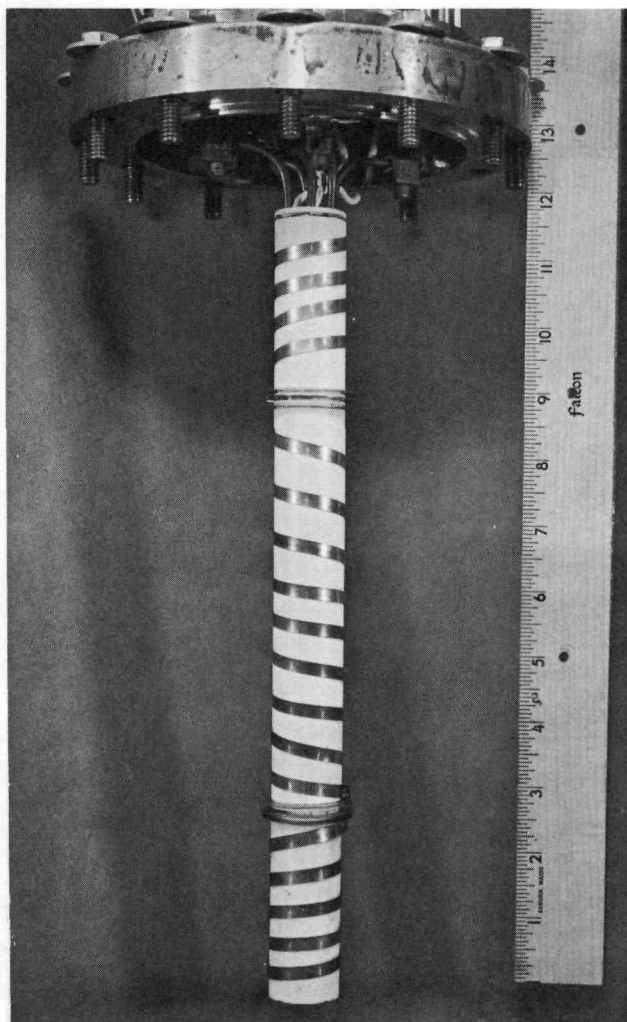


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Figure 10. Effective Emittance of Gold Foil on SS

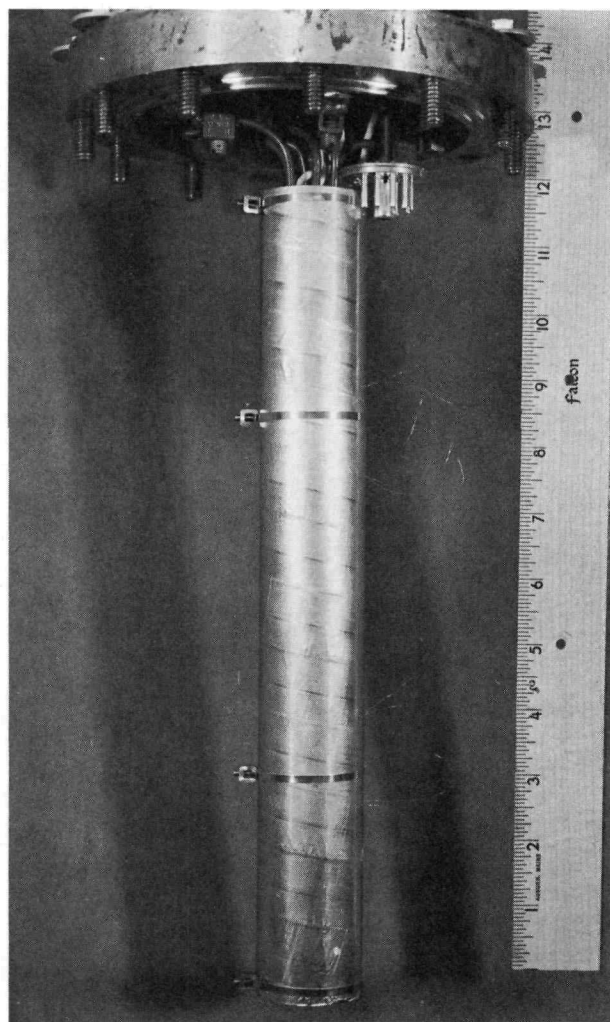




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Figure 11. Effective Emittance Determination, Heater Section



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Figure 12. Effective Emittance Determination, Test Section

are spotwelded to the SS tube on either side of the molybdenum baffles. In testing, a SS cylinder is mounted over the Test Section. The entire external system is immersed in liquid nitrogen and the entire internal system is evacuated.

To minimize test error due to temperature gradients, the effective emittance at any particular temperature is determined when all four thermocouples are at the same temperature. The effective emittance is determined from the relation

$$\bar{\epsilon} = \frac{\text{Power Input (Test Section)}}{\sigma A_{\text{Test Section}} \left( T_{\text{Test Section}}^4 - T_{\text{liq. N}_2}^4 \right)} \quad \dots (10)$$

b. Foil Attachment

The clamps utilized for the foil attachment are made of Type 316 SS. One clamp for use on 1-1/4-in. -O. D. tubing weighs 1.5 grams. The attachment of 3.5 ft<sup>2</sup> of 0.001 in. of pure gold foil to the SNAP 2 PSM-2A primary coolant system is shown in Figure 13. Half of the foil is attached with clamps and the other half is attached with spotwelds.

2. Gold on Molybdenum Foil

a. Radiation Properties

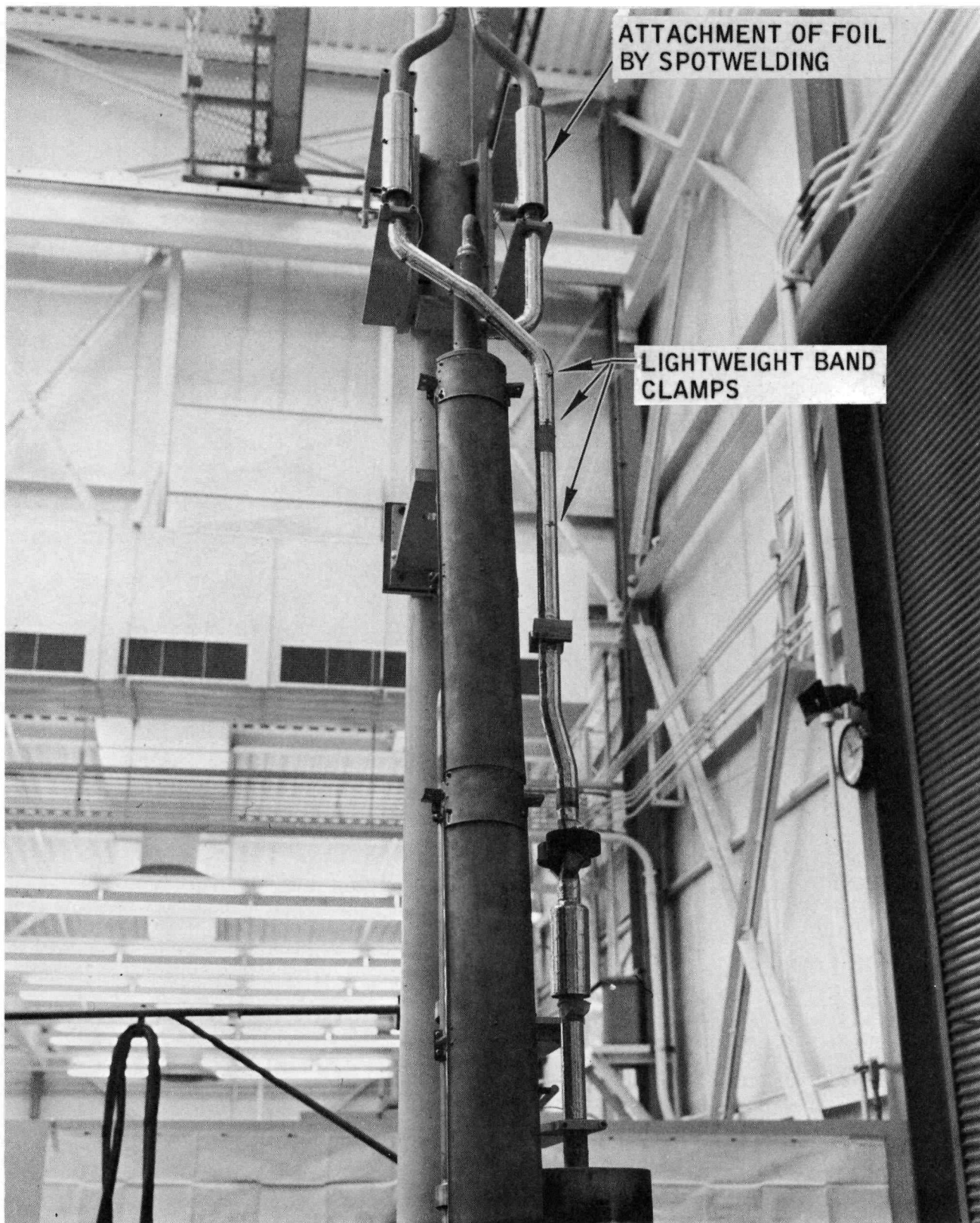
To maintain the integrity of low-emittance coatings, which are required to operate for 1 yr in the temperature range of 850 to 1050°F in a hard vacuum, it is necessary to interpose a diffusion barrier between the SS substrate and the low-emittance (gold or rhodium) coating. The alternative approach is the use of low-emittance foil which can be spotwelded or clamped to the substrate.

For use on the SNAP 10A primary coolant system, where the maximum operating temperatures are 1050°F, it was determined that gold-coated molybdenum foil is a stable low-emittance coating system.

The gold-coated molybdenum foil is prepared with Hanovia Liquid Brite Type 8146 gold. Molybdenum foil, 0.001 in. thick, is coated on one side with two applications of the Liquid Brite solution. The resulting gold film thickness is approximately 10<sup>-5</sup> in. Excellent adherence is attained by subjecting the coated foil to a 1475°F hydrogen cycle. For the SNAP 10A system, the foil is spotwelded to the SS substrate.

The radiation properties are:

<u>Coating System</u>	<u>Temperature (°F)</u>	<u>Total Hemispherical Emittance</u>
Liquid Brite Gold on	800	0.050
0.001-in. molybdenum	900	0.050
foil	1000	0.050
	1100	0.060



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Figure 13. Gold Foil Attachment on SNAP 2 PSM-2A

NAA-SR-9908

b. Stability Tests

Long-term stability tests of the gold molybdenum foil coating system indicate no major emittance degradation. The stability test was performed at 1000°F in vacuum ( $10^{-6}$  torr). The data obtained follows:

<u>Coating System</u>	<u>Time (hr)</u>	<u>Total Hemispherical Emittance</u>
Two coats of Liquid Brite gold on 0.001-in. molybdenum foil	0	0.048
	1000	0.055
	1465	0.055
	2400	0.055
	3000	0.055

Due to the extremely thin layer of gold ( $10^{-5}$  in.) on the molybdenum foil, it was necessary to verify that excessive sublimation of the gold film would not occur in the space environment. Utilizing Langmuir's equation (Ref. 7), calculation shows that the sublimation loss for pure gold operating at elevated temperatures for 1 yr in the SNAP space environment is:

<u>Material</u>	<u>Temperature (°F)</u>	<u>Sublimation (in./yr)</u>
Pure gold	1000	$1.3 \times 10^{-7}$
	1100	$6.7 \times 10^{-7}$
	1200	$5.0 \times 10^{-6}$

Thus, at 1200°F, it would take 200 yr for 0.001-in.-thick, pure gold foil to completely sublime. At 1000°F,  $10^{-5}$  in. of gold would sublime in 77 yr.

To verify Langmuir's theory experimentally, weight loss tests of pure gold foil were performed. This test was made on three samples of pure gold foil, 1 in. by 1 in. by 0.001 in. After careful weighing, the samples were placed in a vacuum retort and were heated at 1200°F for 100 hr. The results follow:

<u>Sample No.</u>	<u>Weight Before Test (grams)</u>	<u>Weight After Test (grams)</u>	<u>Difference</u>
1	0.260734	0.260718	-0.000016
2	0.296904	0.296873	-0.000031
3	0.279790	0.279774	-0.000016

The average weight loss for the three samples was  $21 \times 10^{-6}$  grams; thus the per cent weight loss for the samples during the 100-hr test at 1200°F is 0.007. Under these conditions, it would take 160 yr for the 0.001-in.-thick foil to completely sublime. The experimental test and the theory are in good accord.

### 3. Composite Low-Emittance Coating System

The low-emittance coating ( $\epsilon \leq 0.05$ ) will operate in a space environment for 1 yr in the temperature range of 850 to 1250°F. At these temperatures, gold and rhodium exhibit the lowest emittance of the oxidation resistant metals. When the gold or rhodium is in intimate contact with a metallic substrate such as SS, operation of this composite system at temperatures greater than 850°F results in rapid diffusion of the stainless constituents through the gold or rhodium layer.

Previous discussion has shown that gold or gold-molybdenum foils on SS offer a satisfactory radiation control surface. This approach, while suitable, creates problems in component assembly and handling; thus, a major effort continues in developing an integral, composite low-emittance coating system. Vitreous ceramic diffusion barriers interposed between SS and a Liquid Brite gold film appear effective. This composite low-emittance coating system has demonstrated high-temperature stability. However, the application temperature requirements of the vitreous ceramic to the SS (1700-2000°F) make this approach unattractive for any component or piping process procedure.

In general, metals have much lower emittances than non-metallic compounds such as oxides. At high operating temperatures, the emittance of metallic coatings increases markedly if they have a tendency to oxidize. Since the SNAP system components undergo checkout at design temperatures in soft vacuums, this fact effectively limits the choice of a low-emittance control surface to gold, rhodium, palladium, or platinum.

In the temperature range of 800 to 1250°F, gold has the lowest emittance (0.04 to 0.05) of these oxidation resistant metals. At 1000°F, the emittance of palladium is 0.08 and the emittance of platinum is 0.10. Rhodium ( $\epsilon = 0.06$  at 1000°F) is a secondary choice to gold.

Most data on diffusion of solids take into consideration only the diffusion of ions in ionic crystals or of metals in metals. Comparatively little data is

available on the diffusion of metals into molecular compounds. The mechanisms for solid diffusion (Ref. 8) can be summarized as follows:

- a) The diffusion atom moves through the crystal because of imperfections in the crystal structure. Imperfections include vacancies in the crystal lattice and atoms in interstitial positions (not a normal lattice position). The diffusing atom moves either by pushing crystal atoms into the imperfections or by travelling along the empty spaces created by the imperfections. The vacancy mechanism is considered to be the most likely since, theoretically, it requires the lowest activation energy.
- b) The diffusing atom moves by interchanging positions with atoms of the crystal. This mechanism is considered unlikely because of the relatively high activation energy that is required.
- c) The diffusing atom moves in a liquid region produced by localized melting in the crystal lattice. This mechanism has been proposed to explain the observed correlation between the activation energy and the melting temperature of the solvent.
- d) The diffusing atom moves along grain boundaries and surfaces rather than through the crystal lattice. The rate of diffusion through the crystal, however, increases with increasing temperature, and at high temperatures diffusion through the lattice is the dominant mechanism since only a small fraction of atoms are near the boundaries of the crystal.

It is also noted that:

- a) The rate of diffusion decreases with the increasing melting point of the solvent.
- b) The direction of most rapid diffusion is from the solid with the smaller interatomic distance to that with the larger interatomic distance.

In the work performed on composite SS - gold systems, formation of a surface contaminant on the gold coating is evident after short-term exposure of elevated temperatures. X-ray diffraction analysis of this contaminant indicates the major constituent is iron with small traces of chromium and nickel. Interatomic distances in Angstroms of these metals are: nickel, 2.49; chromium, 2.50; iron, 2.48 and gold, 2.88.

The temperature dependence of the rate of diffusion follows the equation

$$D = D_0 \ell^{-Q/RT}, \quad \dots (11)$$

where

$D$  ( $\text{cm}^2/\text{sec}$ ) = rate of diffusion,

$D_0$  = constant which is a function of the square of the interatomic distance of the solvent,

$Q$  ( $\text{k cal/mol}$ ) = identified with activation energy and is dependent on the melting point of the solvent,

$R$  = gas constant, and

$T$  = absolute temperature.

A material with a low value of  $D_0$  and a high value of  $Q$  should act as a good diffusion barrier. Thus, a material having a high melting temperature and a closely packed crystal structure should be an excellent diffusion barrier.

Results obtained from the effort performed were unsatisfactory based on system objectives. However, a discussion of this work is presented to provide a foundation for future development. Work performed in development of diffusion barriers included the investigation of metallic barriers to interpose between the SS substrate and the low-emittance gold surface. The metallic barriers investigated were chromium, nickel, palladium and rhodium. These metals, especially the palladium system, have much higher melting points than does the gold. Both type 316 and type 405 SS were used as the metallic substrates. Chromium and nickel exhibit similar properties in that complete diffusion between these metals and a gold coating surface occurs within short exposure times,  $< 100$  hr, at elevated temperature,  $1000^\circ\text{F}$ .

Initial effort performed on the palladium and rhodium diffusion barrier systems indicated these materials were successful for high temperature exposures up to 70 hr in duration. However, electrolytic deposition of a palladium or rhodium coating between a SS substrate and a gold surface causes severe blistering of the composite coating system at elevated temperatures. To prevent this blistering, the SS is treated in a high-temperature hydrogen atmosphere to remove all surface oxides and contaminants. This is only partly successful in

the prevention of the blistering. In addition, relatively long-term exposure (>70 hr) of these coating systems results in complete diffusion of the gold coating into the palladium or rhodium barrier.

In summary, it was determined that chromium, nickel, palladium and rhodium are not as effective as diffusion barriers for the gold-SS system as is the vitreous ceramic.

#### D. SNAP 10A EJECTABLE HEAT SHIELD

The SNAP 10A ejectable heat shield requires a mosaic pattern of radiation control coatings which will maintain the internal NaK temperature within certain limits. To prevent possible NaK oxide and/or hydride precipitation, the NaK temperature must be greater than 50°F. To prevent premature firing of the heat shield squibs, the NaK temperature should remain less than 300°F.

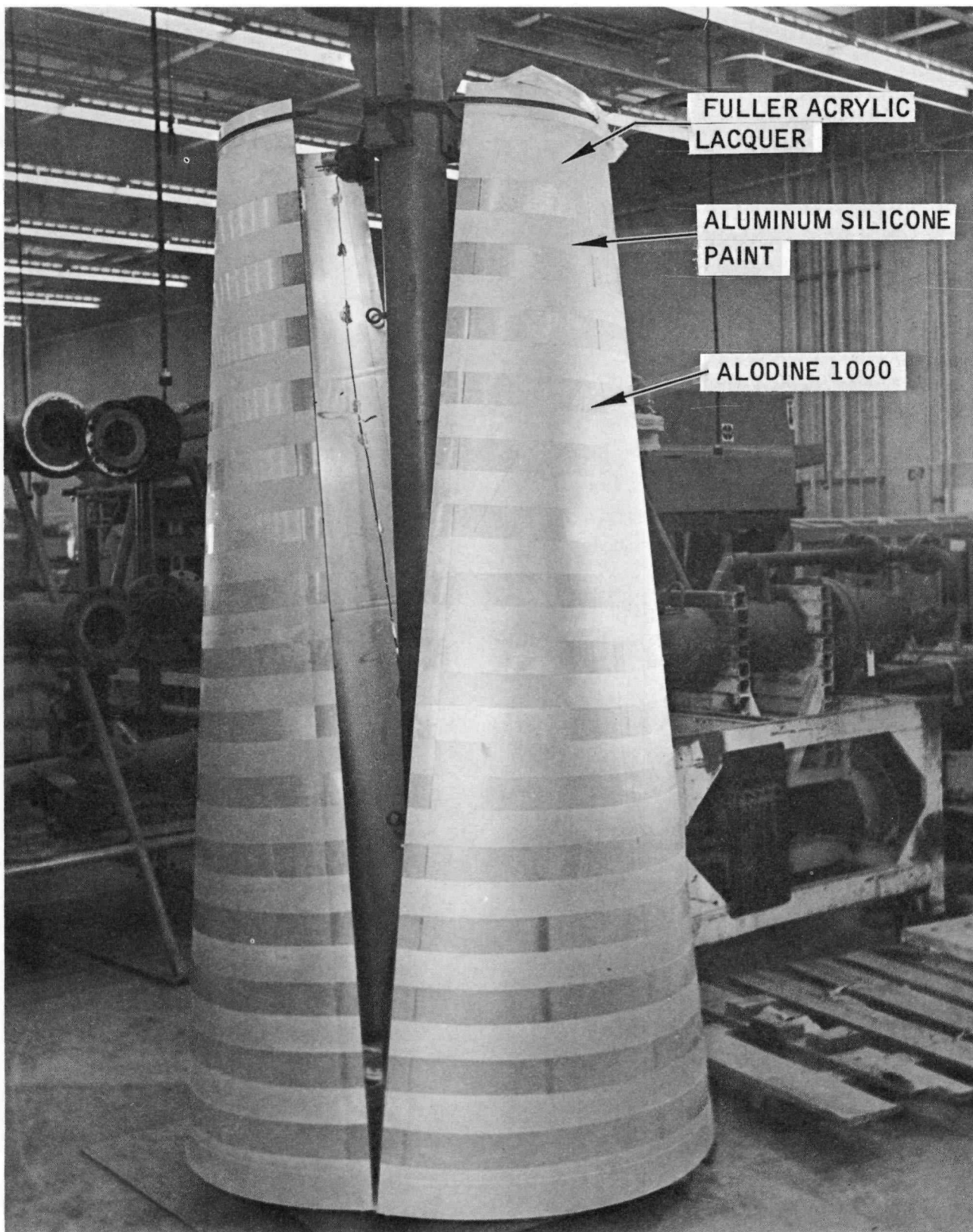
A design analysis was performed on the aluminum honeycomb heat shield to determine the allowable range of  $\epsilon$  and  $\alpha_s / \epsilon$  values which will maintain the internal NaK temperature between 50° to 250°F. The analysis included the necessary parameters resulting from being in either a constant sun-constant shade orbit or a sun-shade circular orbit. From this study, the requisite thermal property limits were defined as: 1) thermal emittance = 0.20 maximum, and 2) the  $\alpha_s / \epsilon$  ratio to be in the range of 1.6 to 2.1.

A successful composite coating system which meets these requirements has been developed and tested. This coating system consists of: 1) Alodine 1000 on aluminum (44% by area), 2) Aluminum Silicone Paint (54% by area), and 3) Fuller Acrylic Lacquer (2% by area). The striped coating pattern utilized on the heat shield is shown in Figure 14. Tests performed on this composite coating system include: a weathering test and a 24 hr, 350°F,  $10^{-6}$  torr stability test.

Initially, in heat-shield coating development progress, a literature search resulted in the choice of a composite coating system which, purportedly, would yield the desired thermal emittance and solar absorptance properties (Ref. 9). The following coating systems were chosen:

- 1) Alodine 1000 (clear chemical film treatment on aluminum honeycomb; the Alodine 1000 film had a thin overcoat of Fuller clear coat #167C70.
- 2) Fuller Acrylic Lacquer #171W560 (white).
- 3) Fuller Aluminum Silicone Paint #171-A-152.





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Figure 14. SNAP 10A Ejectable Heat Shield

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The optical properties of these three coating systems as obtained from the literature were:

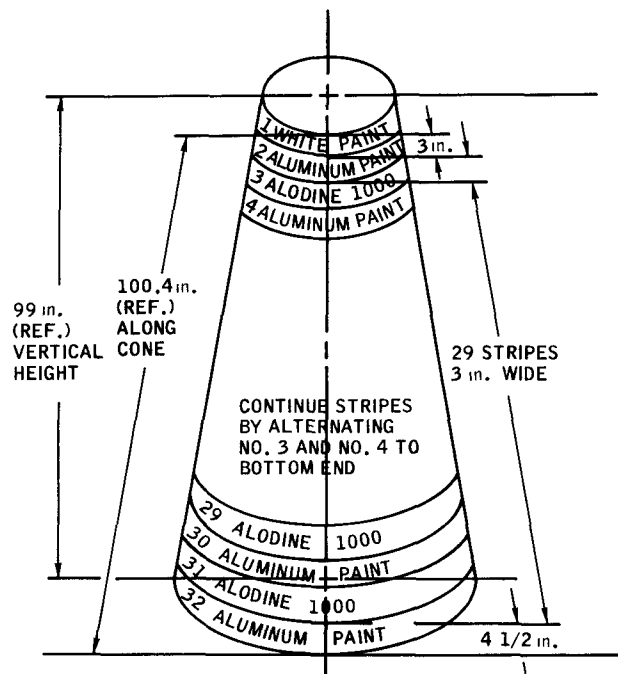
<u>Coating</u>	<u>Solar Absorptance</u>	<u>Thermal Emittance</u>
1) Alodine 1000	0.31	0.05
2) Fuller Acrylic Lacquer	0.30	0.80
3) Fuller Aluminum Paint	0.23	0.20

From this data a composite coating system was devised. The shield pattern chosen is shown in Figure 15.

At this point the optical properties of the individual coatings subsequent to environmental tests were determined. These tests included: 1) weathering, and 2) a 24-hr exposure at 350°F in vacuum. The necessity for the 350°F vacuum-exposure stability test resulted from a study of the temperature profile of the heat shield prior to ejection. This study indicated that portions of the heat shield would approach temperatures of 325°F.

It was determined that the Alodine 1000 film treatment with a thin overcoat of Fuller Clear Coat was exhibiting a much higher emittance than had been indicated in the literature. The purpose of the Fuller Clear Coat was to protect the Alodine 1000 surface from possible corrosion; however the emittance values for the composite surface were consistently measuring 0.30. Thus the protective clear coat requirement was deleted, at which point more desirable emittance values ( $\epsilon = 0.08$ ) were attained.

The test results are tabulated in Tables 5, 6 and 7. The initial measurement of the coating was performed on as-received coated surfaces. The test specimens then were placed in a weathering chamber for 24 hr. The conditions of the weathering chamber were: 1) spray with distilled water for 10 min at each hour of test



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Figure 15. SNAP 10A Heat Shield Pattern

interval, and 2) continuous exposure to a carbon arc which simulates earth sunlight.

TABLE 5

THERMAL EMITTANCE AND SOLAR ABSORPTANCE OF FULLER ALUMINUM SILICONE PAINT ON ALUMINUM HONEYCOMB

Specimen No.	As Received		After Weather		350°F Exposure, 10 <sup>-6</sup> torr, 24 hr	
	ε	α <sub>s</sub>	ε	α <sub>s</sub>	ε	α <sub>s</sub>
1	0.27	0.29	0.25	0.30	0.29	0.32
2	0.27	0.29	0.24	0.27	0.24	0.29
3	0.29	0.37	0.25	0.34	0.25	0.34
4	0.29	0.37	0.25	0.36	0.24	0.33
5	0.30	0.37	0.26	0.31	0.24	0.34
6	0.27	0.29	0.24	0.30	0.23	0.25
7	0.28	0.29	0.24	0.32	0.23	0.30
8	0.29	0.29	0.26	0.33	0.24	0.33
Average	0.28	0.32	0.25	0.32	0.25	0.31

Total hemispherical emittance @150°F = 0.27

Subsequent to the weathering test, solar absorptance and thermal emittance measurements were repeated. The test specimens were then placed in a vacuum oven and subjected to a 24-hr exposure at 350°F, 10<sup>-6</sup> torr. The radiation measurements were again performed.

Table 5 gives the results for the Fuller Aluminum Silicone Paint. The solar absorptance of the coating remained quite constant throughout the tests; however, the thermal emittance of the coating decreased 10% subsequent to the weathering test. At 150°F, the total hemispherical emittance of the as-received silicone paint surface was determined to be 0.27.

Table 6 gives the results for the Alodine 1000 chemical treatment of aluminum. The solar absorptance of this coating surface remained constant throughout the tests; however, extremely erratic results were attained for the thermal emittance. As can be noted in Table 7, the thermal emittance data for the

TABLE 6

THERMAL EMITTANCE AND SOLAR ABSORPTANCE OF  
ALODINE 1000 ON ALUMINUM HONEYCOMB

Specimen No.	As Received		After Weather		350°F Exposure, 10 <sup>-6</sup> torr, 24 hr	
	ε	α <sub>s</sub>	ε	α <sub>s</sub>	ε	α <sub>s</sub>
1	0.08	0.34	0.03	0.38	0.03	0.35
2	0.08	0.33	0.02	0.35	0.03	0.34
3	0.06	0.34	0.02	0.32	—	0.32
4	0.10	0.35	0.02	—	—	—
5	0.10	0.36	0.02	0.37	0.02	0.38
6	0.09	—	0.02	0.32	0.02	0.33
7	0.08	—	0.03	0.34	0.02	0.36
8	0.05	—	0.02	—	—	—
Average	0.08	0.34	0.02	0.34	0.025	0.35

Total hemispherical emittance -0.08

Sample was sprayed with tap water for 4 hr then placed outdoors in sun for 24 hr — emittance 0.07

Sample was maintained at 350°F, 10<sup>-5</sup> torr for 24 hr — emittance 0.07

as-received specimens appeared normal. However, subsequent to the weathering and vacuum temperature test, the emittance dropped from 0.08 to 0.02. This was attributed to the extreme directional nature of the specular Alodine 1000 aluminum surface. Visual examination of this surface showed marked unidirectional streaks which resulted from the rolling process. These measurements were made on a total normal device which behaves erratically for this type of coating.

Thus, it was decided that thermal emittance data reliability for this particular coating would be placed on total hemispherical emittance measurements. This measurement was performed on an as-received Alodine 1000 aluminum sample. The emittance of the coating was determined to be 0.08. The emittance sample was sprayed with tap water for 4 hr, then exposed to sunlight for 24 hr. The resulting total hemispherical emittance was 0.07. Then the sample was

TABLE 7

THERMAL EMITTANCE AND SOLAR ABSORPTANCE OF FULLER  
ACRYLIC LACQUER ON ALUMINUM HONEYCOMB

Specimen No.	As Received		After Weather		350°F Exposure, 10 <sup>-6</sup> torr, 24 hr	
	ε	α <sub>s</sub>	ε	α <sub>s</sub>	ε	α <sub>s</sub>
1	0.90	0.29	0.85	0.28	0.86	0.32
2	0.90	0.28	0.86	0.29	0.87	0.32
3	0.91	0.25	0.87	0.24	0.87	0.26
4	0.91	0.25	0.87	0.24	0.87	0.26
5	0.90	0.26	0.87	0.27	0.85	0.27
6	0.90	0.25	0.86	0.25	0.87	0.27
7	0.90	0.26	0.83	0.27	0.85	0.27
8	0.90	0.25	0.87	0.27	0.86	0.27
Average	0.90	0.26	0.86	0.26	0.86	0.28

Fuller White Acrylic Lacquer #171W560

maintained at 350°F, 10<sup>-5</sup> torr, for 24 hr. The final emittance measurement was determined to be 0.07.

Table 7 gives the results for the Fuller Acrylic Lacquer. The solar absorptance of the coating remained relatively constant throughout the tests. The thermal emittance of the coating decreased 5% subsequent to the weathering test. These data are entirely suitable since the overall balance of the heat shield is only slightly affected by the lacquer. The heat shield surface contains only 2% of the White Lacquer finish. This fact also negates concern that the White Lacquer will degrade in a space ultraviolet environment.

For design considerations, the thermal emittance and solar absorptance values utilized were:

<u>Coating on Aluminum Honeycomb</u>	<u>Thermal Emittance</u>	<u>Solar Absorptance</u>
Alodine 1000	0.07	0.35
Fuller Acrylic Lacquer	0.86	0.28
Fuller Aluminum Silicone Paint	0.25	0.31

The  $\epsilon$  and  $\alpha_s/\epsilon$  relationship for the composite heat shield surface is:

$$\begin{aligned} \epsilon(\text{composite}) &= (0.44 \times \epsilon) \text{ Alodine} \\ &+ (0.54 \times \epsilon) \text{ Silicone} + (0.02 \times \epsilon) \text{ Laquer;} \end{aligned} \quad \dots (12)$$

$$\begin{aligned} \alpha_s(\text{composite}) &= (0.44 \times \alpha_s) \text{ Alodine} \\ &+ (0.54 \times \alpha_s) \text{ Silicone} + (0.02 \times \alpha_s) \text{ Laquer.} \end{aligned} \quad \dots (13)$$

From Equations 12 and 13, the composite  $\epsilon$  and  $\alpha_s/\epsilon$  values are:

<u>Composite Surface Condition</u>	<u>Thermal Emittance</u>	<u>Solar Absorptance To Thermal Emittance Ratio</u>
As-Received Coatings	0.20	1.65
Coatings Subsequent to Weathering	0.18	1.83
Coatings Subsequent to Vacuum, Temperature Stability Test	0.18	1.83

Using these radiation properties and shield areas, an analysis was performed (at 6% NaK flow) to determine the coolant system temperatures. A summary of these results are tabulated as:

<u>Condition</u>	<u>Time (hr)</u>	<u>NaK (Max.) (°F)</u>	<u>NaK (Min.) (°F)</u>	<u>Reactor Outlet Max. (°F)</u>
Sun-shade	15	164		115.2
	1st orbit		61.5	
Constant Sun-constant shade	8	218		160.9
	1st orbit		65.4	

Note, that the NaK temperatures attained are within the required limits.

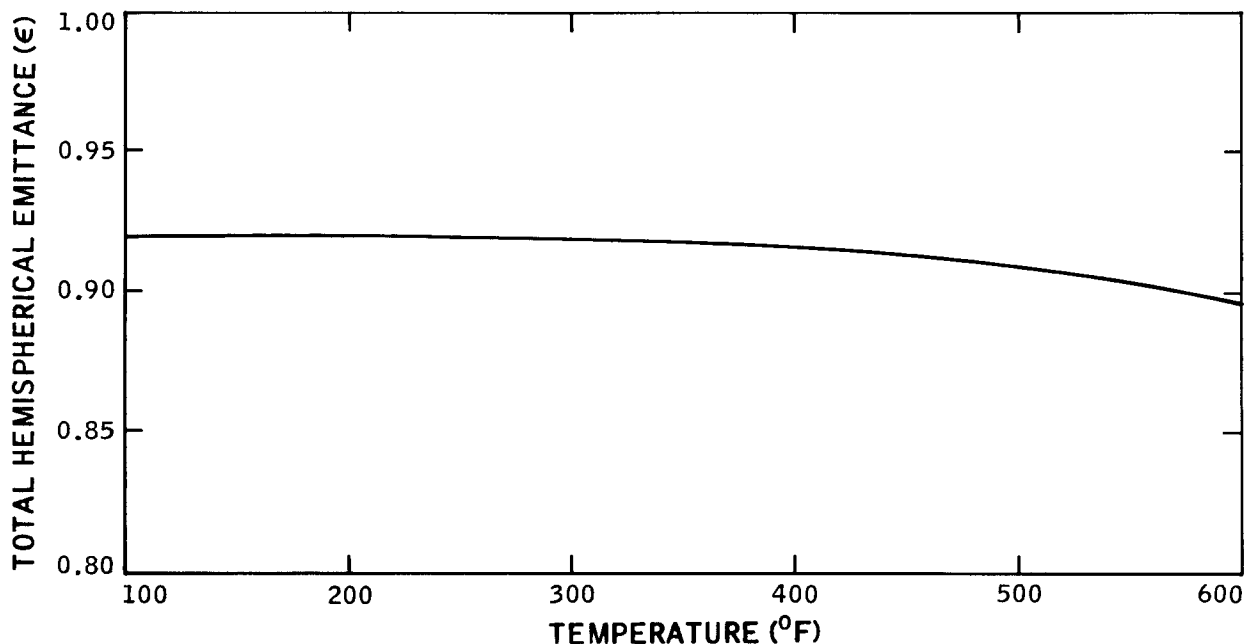
## E. MISCELLANEOUS COATINGS FOR SNAP SYSTEMS

### 1. PT404A High-Emittance Black Enamel

Many components of the SNAP 2 and 10A systems, which require a high-thermal-emittance surface, are either heat sensitive or utilize heat treatable alloys. A high-thermal-emittance coating, which has undergone thorough qualification, is PT404A, a proprietary black enamel marketed by Product Techniques, Inc., Los Angeles, California. For use as a radiation control coating, the enamel can easily be applied by brushing or spraying and can be cured at ambient temperature.

The total hemispherical emittance of PT404A enamel as a function of temperature is shown in Figure 16.

Stability of the PT404A coating in a vacuum-temperature environment is excellent. This coating was applied to the radiator fins of a 16-element SNAP 10A converter module. The module was tested for 3000 hr at  $10^{-3}$  torr with a constant fin temperature of 600°F. Visual examination of the module fins indicated no deterioration of the coating.



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Figure 16. Emittance of PT404A Enamel

Stability of the PT404A black enamel in a nuclear irradiation field is also excellent. Three samples of this coating were included in an irradiation test performed at BMI, Columbus, Ohio (Ref. 10). Conditions of the test were:

Fast neutron dose	$10^{18}$ nvt
Gamma dose	$10^8$ R
Temperature	100°F
Vacuum	$10^{-15}$ torr.

The emittance of the three samples prior to test was 0.92, after irradiation it was 0.90.

Stability of the PT404A when subjected to mechanical vibration has been demonstrated. The coating, as applied to an aluminum 6061-T6 panel, was vibrated at various stress levels up through the point of failure of the aluminum panel. Failure of the coating surface did not occur until the panel exceeded its ultimate stress.

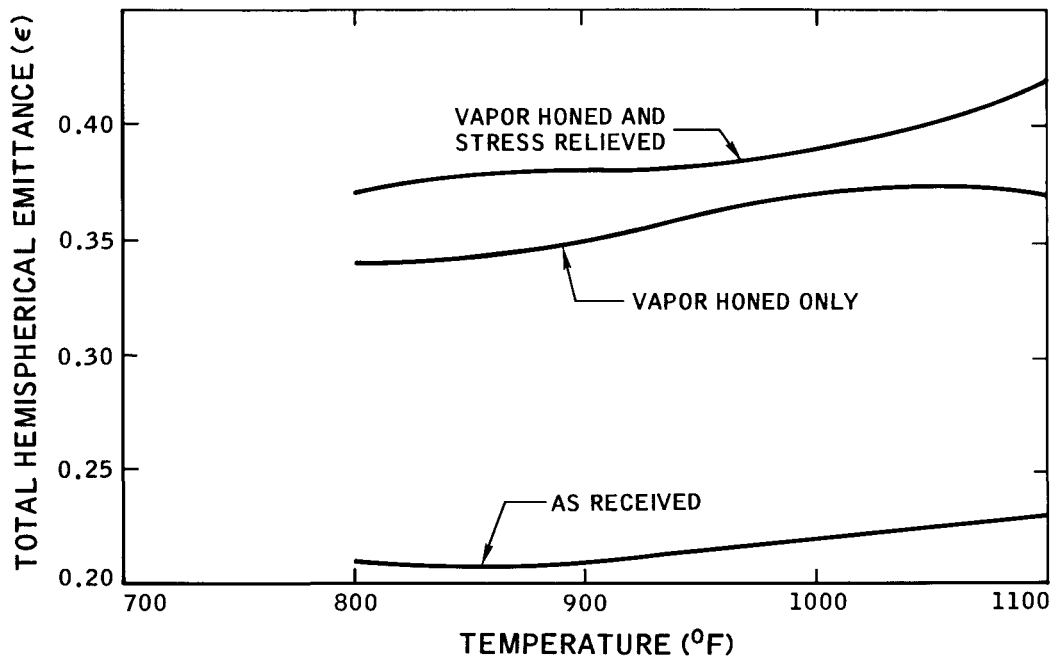
## 2. SNAP Reactor Core Vessel

The SNAP systems SS Type 316 reactor core vessels undergo: (1) a vapor-hone treatment, and (2) a stress relieve heat treatment. The vapor-hone treatment is performed to Specification LA0110-006. The heat treatment cycle is of 6-1/2 hr duration with the vessel maintained at 1000°F for 30 min in vacuum. The effects of these treatments on the emittance characteristics of SS Type 316 were determined as:

<u>Material</u>	<u>Temperature (°F)</u>	<u>Total Hemispherical Emittance</u>
SS	800	0.22
Type 316	900	0.22
As-Received	1000	0.23
	1050	0.24
SS	800	0.35
Type 316 after	900	0.36
Vapor-Hone	1000	0.37
Treatment	1100	0.38
SS	800	0.37
Type 316 after	900	0.38
Vapor Hone and	1000	0.40
Heat Treatment	1100	0.43

(See Figure 17.)





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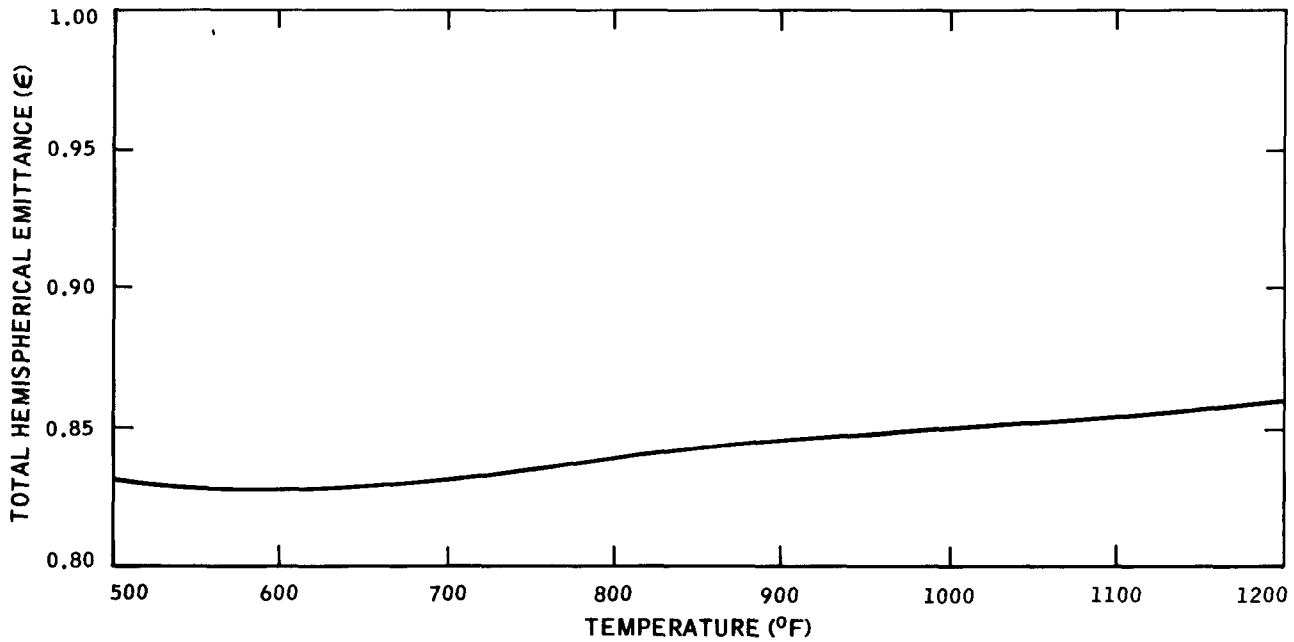
Figure 17. Emittance of Type 316/SS

The sharp increase in emittance subsequent to the vapor-hone treatment is attributed to the large increase in effective surface area which occurs during honing. The slight increase in emittance subsequent to the heat treatment is due to slight oxidation which occurs in soft vacuums at high temperatures.

### 3. Chromic Oxide

Prior to the development of the AI93 coating system, the reference design coating for the SNAP system radiators was chromic oxide ( $\text{Cr}_2\text{O}_3$ ). Presently,  $\text{Cr}_2\text{O}_3$  is utilized as a backup coating system to the AI93.

Shown in Figure 18 is the total hemispherical emittance for a plasma-spray 0.003-in.-thick  $\text{Cr}_2\text{O}_3$  coating on Aluminum 1100 substrate. A 150-hr stability test at 600°F,  $10^{-5}$  torr showed no degradation in emittance for this coating system.



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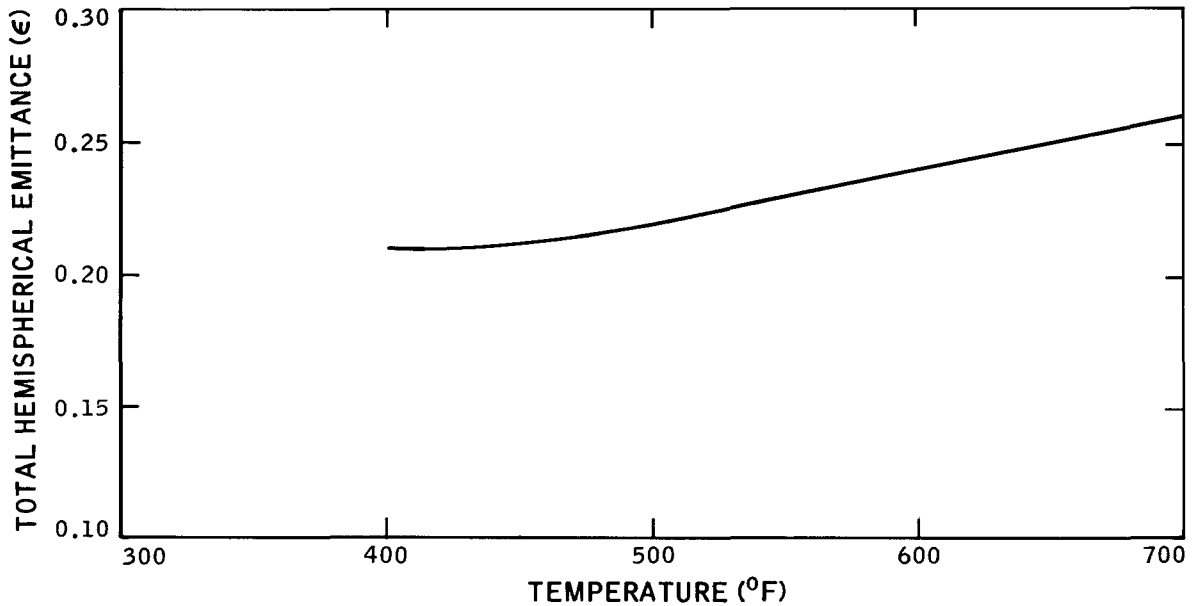
Figure 18. Emittance of  $\text{Cr}_2\text{O}_3$  on Aluminum 1100

Two samples of  $\text{Cr}_2\text{O}_3$  were also included in the nuclear stability test performed at BMI. The results of this test were:

Sample No.	Total Hemispherical Emittance (600°F) Prior to Irradiation	Total Hemispherical Emittance (600°F) After Irradiation
1 $\text{Cr}_2\text{O}_3$ plasmaspray (0.003 in. thick)	0.84	0.81
2 $\text{Cr}_2\text{O}_3$ plasmaspray (0.003 in. thick)	0.84	0.81

#### 4. Commercial Titanium

The structural shell of the SNAP 10A, thermoelectric converter system is fabricated from commercially pure titanium. During operation of the SNAP 10A system, temperatures on the titanium shell range from 700 to 800°F. For the pure titanium, the total hemispherical emittance as a function of temperature was determined and is shown in Figure 19.



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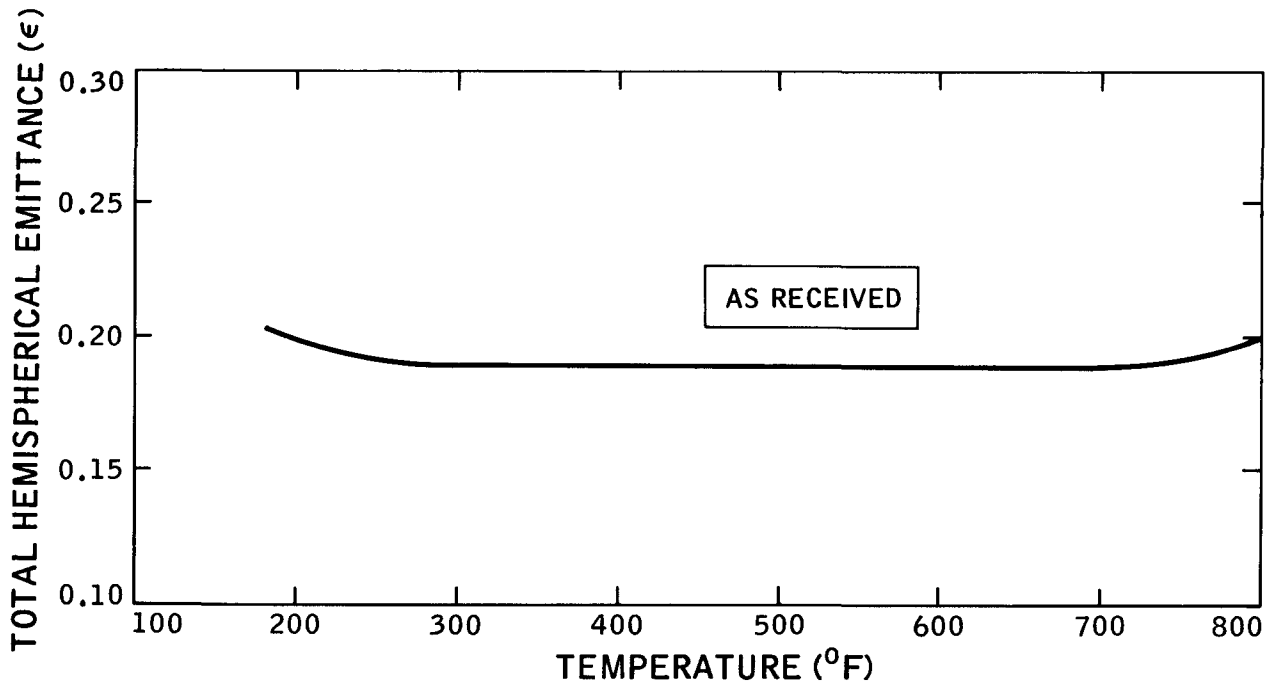
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Figure 19. Emittance of Pure Titanium

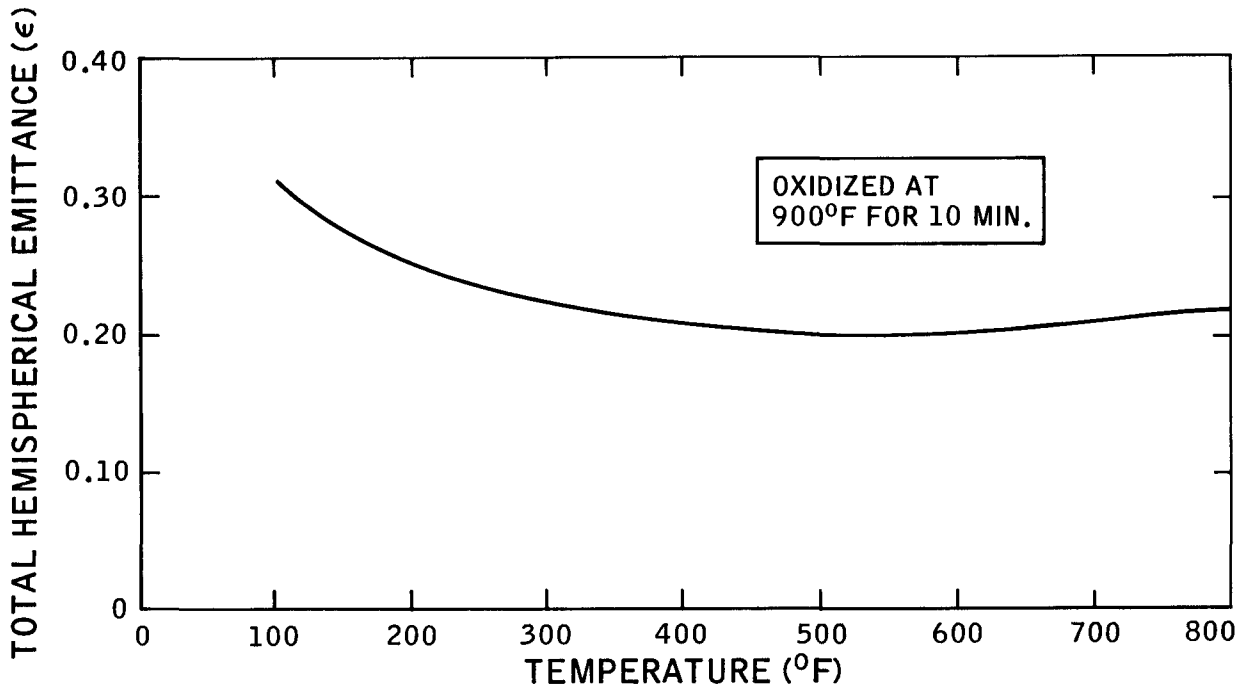
#### 5. Armco PH15-7Mo

The interior radiation control surface of the SNAP 2 RC is PH15-7Mo. Brazing of copper fins to the exterior surface of the PH15-7Mo honeycomb shell is performed in argon at 1800°F. Some contamination of the PH15-7Mo surface does occur during the braze operation. Tests have shown that this contamination can be simulated by deliberately oxidizing as-received PH15-7Mo sheet in air at 900°F for 10 min.

Shown in Figure 20a is the total hemispherical emittance as a function of temperature for as-received Armco PH15-7Mo sheet. The total hemispherical emittance as a function of temperature for Armco PH15-7Mo sheet which has been oxidized in air at 900°F for 10 min is shown in Figure 20b.



20a



20b

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Figure 20. Emittance of Armco PH15-7 Mo

### III. DESCRIPTION OF EQUIPMENT

#### A. TOTAL HEMISPHERICAL EMITTANCE APPARATUS

The total hemispherical emittance is determined by measuring the power required to maintain an emitting specimen at constant temperature. In the measurement technique utilized by AI, a flat strip specimen, with Pt-Pt10%Rd thermocouple leads attached, is enclosed in an evacuated, non-reflecting test cell whose outer walls are cooled by liquid nitrogen. A schematic of this system is shown in Figure 21. Electrical energy is supplied at a constant rate to the specimen which eventually reaches an equilibrium temperature ( $T_s$ ) over a portion (A) of its area. At the steady-state condition, the power to the specimen volume enclosed by A is equal to the radiation from area A. If the space surrounding the specimen is at temperature ( $T_o$ ) the emittance of the sample is determined by

$$\epsilon = \frac{W}{\sigma A T_s^4 - T_o^4} \quad \dots(14)$$

where

- $\epsilon$  = total hemispherical emittance
- W = electrical input to specimen
- $\sigma$  = Stefan-Boltzmann constant
- $T_s$  = steady-state specimen temperature, absolute
- $T_o$  = sink temperature, absolute

The emittance coating specimens tested may be flat strips of pure metals or alloys, or may be surface coatings applied to a metallic resistance strip. The electrical input (W) is determined from: (1) measurement of the current (I) passing through the specimen, and (2) measurement of the voltage drop (E) between thermocouples. The platinum lead of each thermocouple is utilized as a potential tap for performing the voltage drop measurement.

The equipment utilized during the measurement of the necessary emittance relationship parameters is:

- I = specimen current (John Fluke Model #803B AC-DC Voltmeter and Rubicon Model #1166 Standard Resistor)

$E$  = potential drop between thermocouples (John Fluke Model #803B AC-DC Voltmeter)

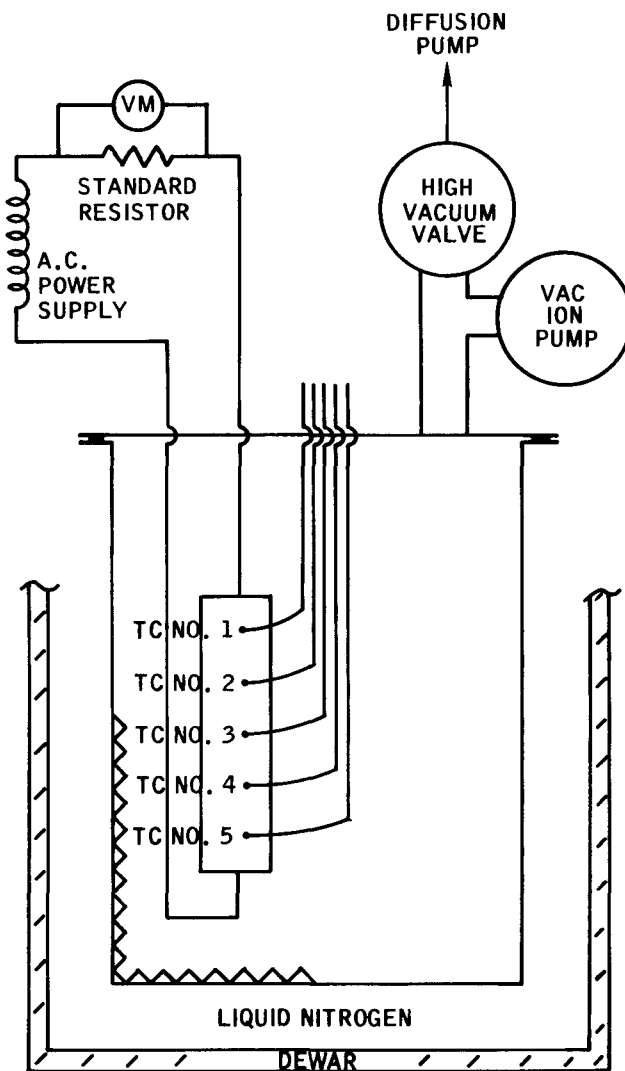
$A$  = specimen area (Swift and Son Model #24036 Traveling Micrometer)

$t_s$  = specimen temperature (Rubicon B).

After instrumentation of the test specimen with thermocouples, the specimen is mounted to a SS head which contains the necessary electrical and thermocouple feed-throughs. A SS cylinder is joined to the head and the system flange is made vacuum tight with a copper crush gasket. The walls of the SS cylinder are kept at a minimum temperature by immersion of the cylinder in a Dewar flask filled with liquid nitrogen. To reduce reflection the inside wall of the cylinder is threaded and coated with a high absorptance enamel.

Evacuation of the system is accomplished with a diffusion pump and a vac-ion pump as depicted in Figure 22. The vacuum maintained during measurement is  $10^{-5}$  torr.

During performance of the total hemispherical emittance measurement several inherent errors exist due to system configuration and the measurement equipment utilized. The magnitude of these errors has been determined and the necessary corrections are applied to the end-product emittance value. In addition, an account of random and non-random uncertainties in the measurement must be made. A detailed discussion of the error and uncertainty analysis is given in Ref. 11. As noted in this reference, the uncertainty in the total hemispherical emittance measurement is 1.9%.

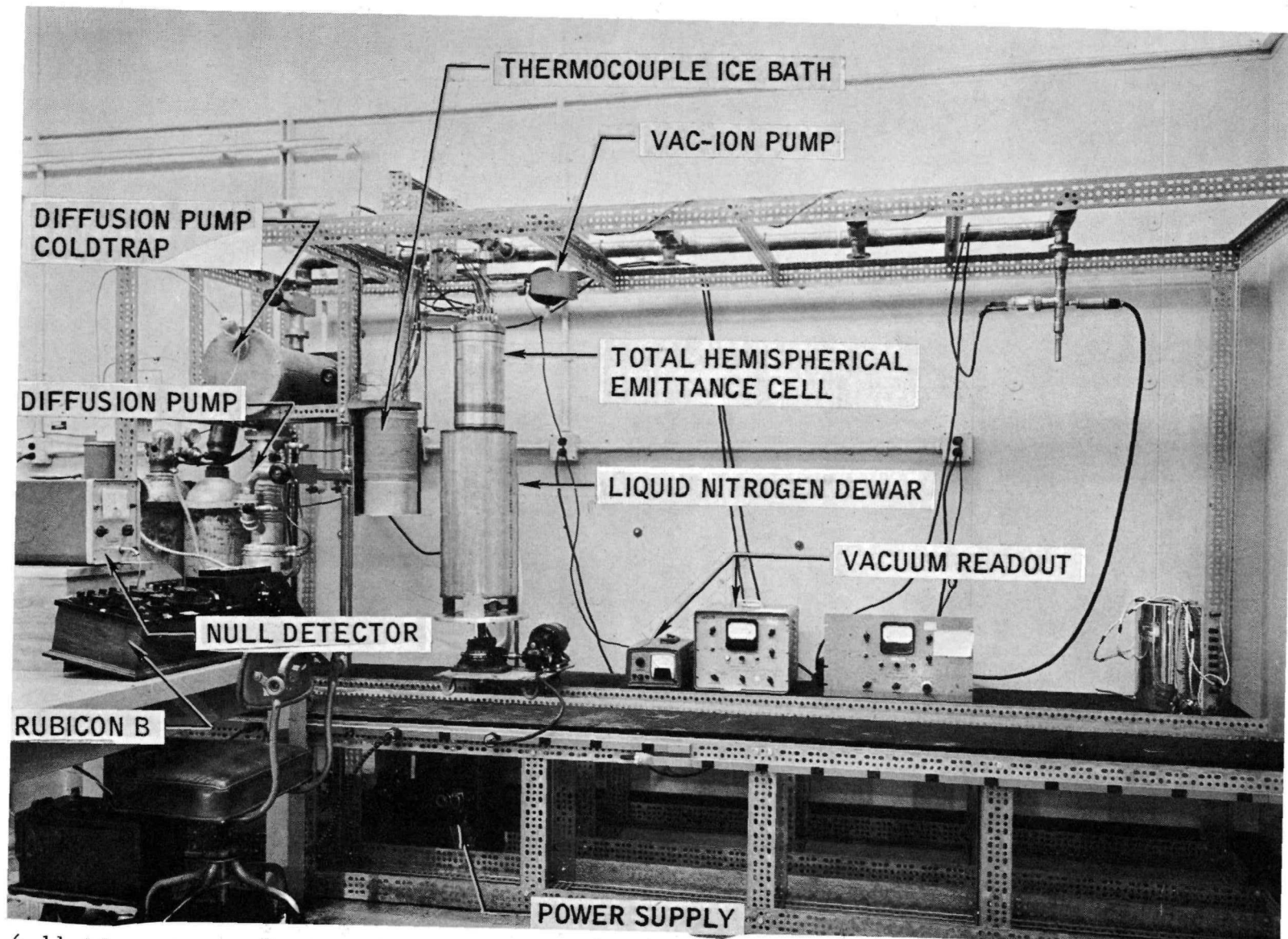


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Figure 21. Schematic of Total Hemispherical Emittance Measurement Device

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Figure 22. Emittance Measurement Test Equipment

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## B. SPECTRAL REFLECTANCE SYSTEM

The spectral reflectance system is designed to measure the relative, room-temperature, hemispherical spectral reflectance of 1-in.-dia, disc-shaped samples illuminated normally with narrow wavelength bands of radiation between 0.2 and 2.5 $\mu$ . The system flow chart is shown in Figure 23. Sample and standard, both positioned at the base of an integrating hemisphere, are normally and alternately illuminated by monochromatic radiation of width  $d\lambda$ (wavelength increment) as produced by a monochromator which automatically scans the spectrum from 0.2 to 2.5 $\mu$ . Alternate illumination is accomplished by a chopper mirror system rotating at 6000 rpm. The radiation reflected by these materials is incident upon a highly reflecting, diffuse coating of MgO which has been smoked on the inner surface of the hemisphere. By multiple reflections, the MgO coating diffuses the specular components of the reflected radiation and ultimately produces uniform illumination of the coating itself. A detector (S-13 or Pb-S) mounted on the side of the hemisphere detects the reflected radiation.

As the monochromator scans from 0.2 to 2.5 $\mu$ , the chopper mirror system alternately blacks-out and transmits radiation into the hemisphere. This results in intermittent signals being received by the detector system. These signals are alternately stored in one of two channels of the detector system. The interval of black-out permits a zero reference for the detector system.

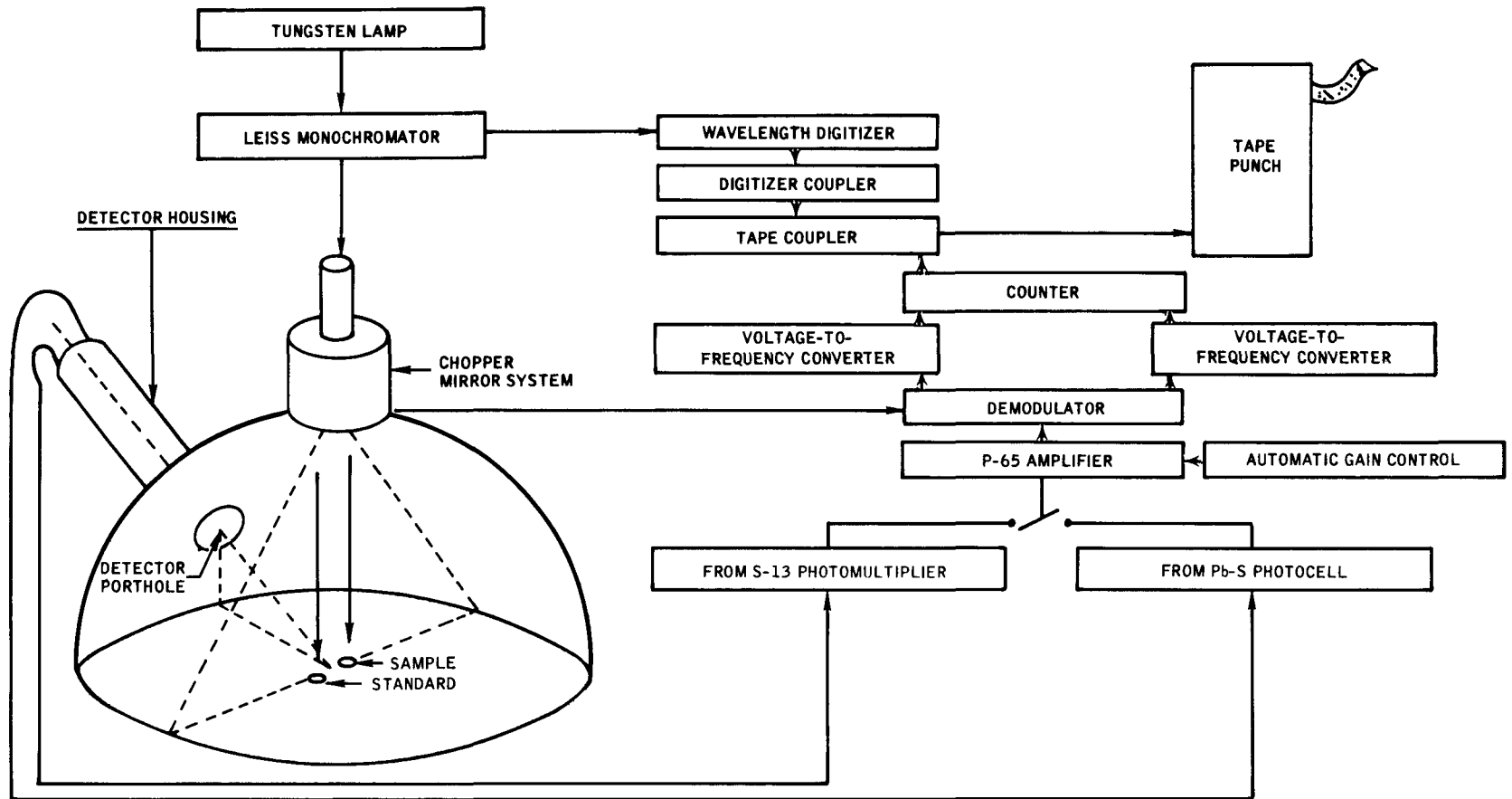
Storage of intermittent signals continues until the monochromator has scanned a small interval from  $\lambda_1$  to  $\lambda_2$ . At this time there occurs separate summations of the signals stored in Channel I and Channel II.

The ratio of the sum in Channel I to the sum in Channel II is equal to the ratio of the reflectance of the sample to the reflectance of the standard for the wavelength interval,  $\lambda_1$  to  $\lambda_2$ . This ratio, along with numbers indicative of wavelength, are produced on punched tape.

Storage and summation of signals in the two channels continues until the monochromator has scanned from 0.2 to 2.5 $\mu$ . The resulting tape and a code for translating from indicative numbers to wavelength are programmed into a computer which automatically results in the desired reflectance versus wavelength curve.



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Figure 23. Schematic of Spectral Reflectance System

### C. SPECTRAL EMITTANCE SYSTEM

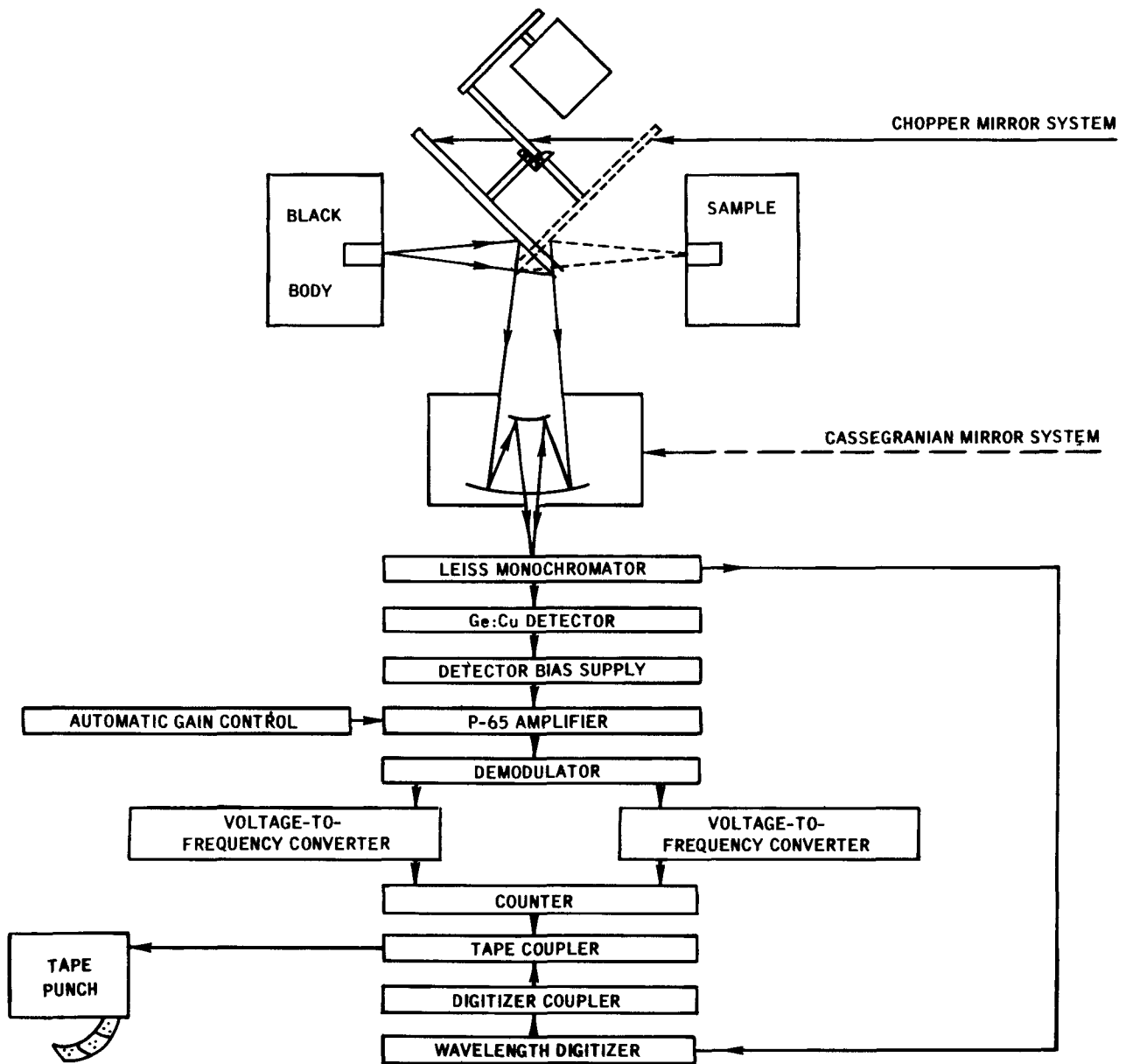
The spectral emittance system is designed to measure the near-normal spectral emittance, at wavelengths between 0.5 to 28 $\mu$ , of samples at temperatures from 400 to 1400°F. The method of measurement is direct. The monochromatic radiation from a sample is compared to radiation at the same wavelength from a black body at the same temperature. The system operation is similar to the spectral reflectance system in that the detector receives intermittent and alternate signals of width  $d\lambda$  from the black body and from the sample as the monochromator slowly scans the wavelength range of interest. The spectral emittance at a specified temperature is obtained as the ratio of the sum of  $n$  signals produced by the sample by radiation of width  $d\lambda$  within the small wavelength interval from  $\lambda_1$  to  $\lambda_2$  to that produced by the black body maintained at the same temperature within the same wavelength interval.

The schematic for the system is shown in Figure 24. The radiation from the sample and the black body is reflected toward the detector system by a sectored-disc, rotating chopper mirror system. The functions of the mirror system are: (1) to allow alternate signals from the sample and black body to be passed through the system, and (2) to chop the radiation signals at a frequency sufficient to permit the detector to operate at highest detectivity (900 cps for a Ge:Cu detector at liquid helium temperatures, and 90 cps for Pb-S at room temperature). This intermittent radiation is incident upon the collector mirror of a Cassegranian mirror system and is then focussed by a second mirror onto the entrance slit of a Leiss double-prism monochromator. Detection, storage, and summation of signals is accomplished as in the spectral reflectance system, with the final output being a computer tape listing emittance values and the corresponding wavelength. The tape is again programmed into a computer, which automatically results in corrected emittance vs wavelength curves for the sample.

### D. MOBILE SOLAR REFLECTOMETER

Components of the SNAP systems are subject to a major amount of handling and exposure in assembly buildings while undergoing vehicle integration. A monitor of the solar reflectance properties of coated components is maintained with a lightweight, portable reflectometer.

The solar reflectometer is a product of Gier Dunkle Instruments, Inc., Santa Monica, California. The reflectometer features: (1) a valid measurement for



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Figure 24. Schematic of Spectral Emittance System

diffuse or specular opaque materials, (2) a capability for band pass and total absolute measurement, and (3) simulation of extraterrestrial sun by selective filtering of a xenon source. Accuracy of a system is  $\pm 0.02$  for grey samples and  $-0.00 + 0.04$  for spectrally selective samples. The detection head is connected to the electronic readout and power supply console with an 8-ft cable; thus, good flexibility for performing measurements on large systems exists. The time for one measurement is 2 min.

#### E. MOBILE EMISSIVITY INSPECTION SYSTEM

A monitor of the thermal emittance properties of coated components is maintained with a lightweight, portable emittance inspection system. This system is a product of Gier Dunkle Instruments, Inc. and is designated EM-2. Accuracy of the system is  $\pm 0.015$  for samples which may be specular or diffuse. The sample temperature is ambient and measurement time is 2 min.

#### IV. CONCLUSIONS

The reference design coating system for the SNAP system high temperature radiators is AI93 ( $\epsilon = 0.91$ ,  $\alpha_s = 0.30$ ). Stability of this coating in a nuclear and simulated space environment has been demonstrated. The composite AI93 coating system consists of: a subcoat of chromium-cobalt-nickel spinel bonded with aluminum phosphate, and a topcoat of stannic oxide bonded with aluminum phosphate. The application temperature range of the phosphate bonded coatings is 450 to 600 °F in an air or argon atmosphere. No limitations on the type of metallic substrates which may be coated have been encountered.

The AI93 coating system has demonstrated the capability of withstanding the severe conditions of shock, vibration and aerodynamic heating imposed upon it during the SNAP system launch. No emittance degradation occurred in 6000-hr coating stability tests at 600 °F, in vacuum. The coating has been exposed to nuclear irradiation ( $3 \times 10^{18}$  n/cm<sup>2</sup>,  $1.9 \times 10^{10}$  R) at 600 °F, in vacuum. The thermal emittance remained constant throughout the test. The coating system was also subjected to electron bombardment without any detrimental effects. During the Statitron test, the coated sample was maintained at 600 °F and received an integrated electron dose of  $3 \times 10^{18}$ , 1.25 MeV energy. Ultraviolet stability tests of 3000-hr duration are presently underway. Initial 1000-hr results indicate a 10 to 15% increase in solar absorptance.

Coating formulation and processing techniques are presently undergoing analysis and development in an effort to further increase the thermal emittance of the basic AI93 coating to a value of  $\epsilon = 0.95$  minimum.

The high-thermal-emittance coating system, which is utilized on heat sensitive components, is PT404A black enamel. Stability of this coating in a vacuum-temperature environment is excellent. Stability tests for 3000 hr at 600 °F,  $10^{-3}$  torr have shown no emittance change. The coating has been exposed to nuclear irradiation ( $10^{18}$  n/cm<sup>2</sup>,  $10^8$  R) at 100 °F, in vacuum. The emittance prior to test was 0.94. The emittance after irradiation was 0.91.

The primary coolant systems of the SNAP 2 and 10A powerplants use a liquid metal, NaK-78. Temperatures at the SNAP 2 reactor outlet are above 1200 °F; thus, to prevent excessive heat losses from the SS components and piping, a need exists for radiative insulation. The optimum low-emittance

surface ( $\epsilon \leq 0.05$ ) for use at high temperature in the space environment was determined to be gold.

Initially, in the low-emittance coating development program an effort was made to utilize an integral, composite coating surface such as electro-deposited gold on SS. Subsequent testing showed that at high temperature a stable, low-emittance coating surface could not be maintained due to interdiffusion.

Thus, the present reference design radiative insulation utilized on the SNAP systems is pure gold foil. The foil is attached to the SS coolant system by spot-welding or by lightweight band clamps.

Development work continues in attaining a composite low-emittance coating system. Diffusion barriers, which are being analyzed, include vitreous ceramics, cerium oxide and nickel oxide. Molybdenum and tantalum reflectors are also being investigated.

A successful composite coating system has been developed and tested for use on the ejectable heat shield. This coating system consists of: 1) Alodine 1000 Aluminum, 2) Aluminum Silicone Paint, and 3) Fuller Acrylic Lacquer. Tests performed on the composite coating system include a weathering test, and a 24-hr, 350°F,  $10^{-6}$  torr stability test.

Further effort being performed in this endeavour includes design and development of a subliming heat shield which will exhibit similar radiation properties to the existing ejectable heat shield.

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