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Quarterly Progress Report
Determination of the Emissivity of Materials
Report No. PWA-2163

Report Period: October 1 through December 31, 1962
Contract NASw-104 with 8 Amendments

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ERRATA

Please make the following corrections to the "Quarterly Progress Report, Determination of Emissivity of Materials," Report No. PWA-2128:

1. Page 30, delete the word "heating" where it appears in the legend.

2. Page 63, change "200°F" to "2000°F" in the legend.

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FOREWORD

This report describes the research activity carried out in fulfillment of contract NASw-104 as modified by Amendments 1 through 8, during the period from October 1 through December 31, 1962.

ABSTRACT

During the three month period covered by this report, work was continued in support of NASA space power systems. An aluminum phosphate bonded mixture of nickel-chrome spinel and silicon dioxide completed 10,720 hours of endurance testing on a SNAP-8 finned-tube radiator segment. Flame-sprayed coatings of titania on SNAP-8 and Sunflower I sections completed 9870 and 9830 hours respectively. A fourth rig containing a SNAP-8 section with an aluminum phosphate-bonded mixture of silicon carbide and silicon dioxide completed 8600 hours of testing.

Emittance measurements are reported on fifteen materials. These fifteen materials are stannic oxide, silicon dioxide, palladium black, nickel oxide, chromic oxide, cobalt oxide, boron carbide, iron-titanium-oxide, iron-titanium-aluminum oxide, oxidized kennametal K-151-A, titania, calcium titanate, strontium titanate, silicon carbide, and manganese oxide. All coatings were bonded to columbium - 1 per cent zirconium substrates except palladium black and oxidized kennametal K-151-A which were bonded to AISI-310 stainless steel. Two grades of titania were tested to determine the effects of purity on emittance. The total hemispherical emittance of all materials were measured over a range of temperatures.

In addition, the spectral normal emittances of boron carbide, iron-titanium-oxide, the higher purity titania, and silicon carbide were measured. Iron-titanium-aluminum oxide, strontium titanate, and silicon carbide were subjected to short term endurance testing. The total hemispherical emittance of strontium titanate was measured in all three emittance rigs for comparison purposes.

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Total Hemispherical Emittance Data

I. COATING ENDURANCE TESTS IN SUPPORT OF NASA SPACE POWER SYSTEMS

Work was continued during this reporting period in support of the SNAP-8 and Sunflower I space power systems. Endurance tests continued on each of four finned-tube radiator segments originally scheduled to run ten thousand hours. A description of the progress of each segment follows.

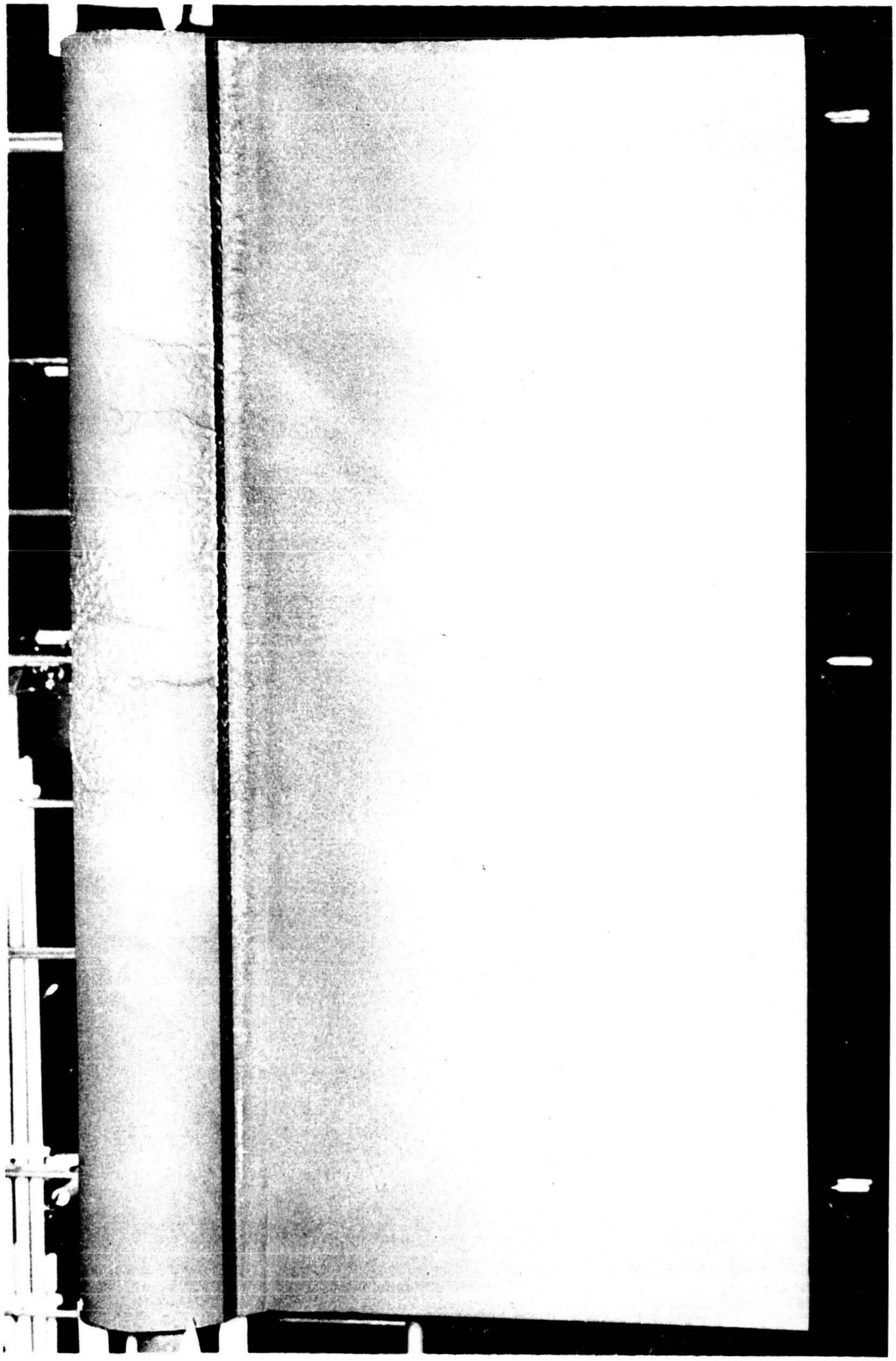
A. Endurance Test No. 1, SNAP-8 Test Section

A mixture of nickel-chrome spinel ($NiO \cdot Cr_2O_3$) and silicon dioxide was aluminum phosphate bonded to this section. The appearance of the section after 10,801 hours is shown in Figure 1. The bubbling of the coating occurred during inadvertent overheating at approximately 2700 hours (PWA-2043). No change in the appearance of the coating has been observed since that time. At the end of this report period the section had accumulated approximately 10,720 hours of endurance testing.

B. Endurance Test No. 2, SNAP-8 Test Section
Endurance Test No. 3, Sunflower I Test Section

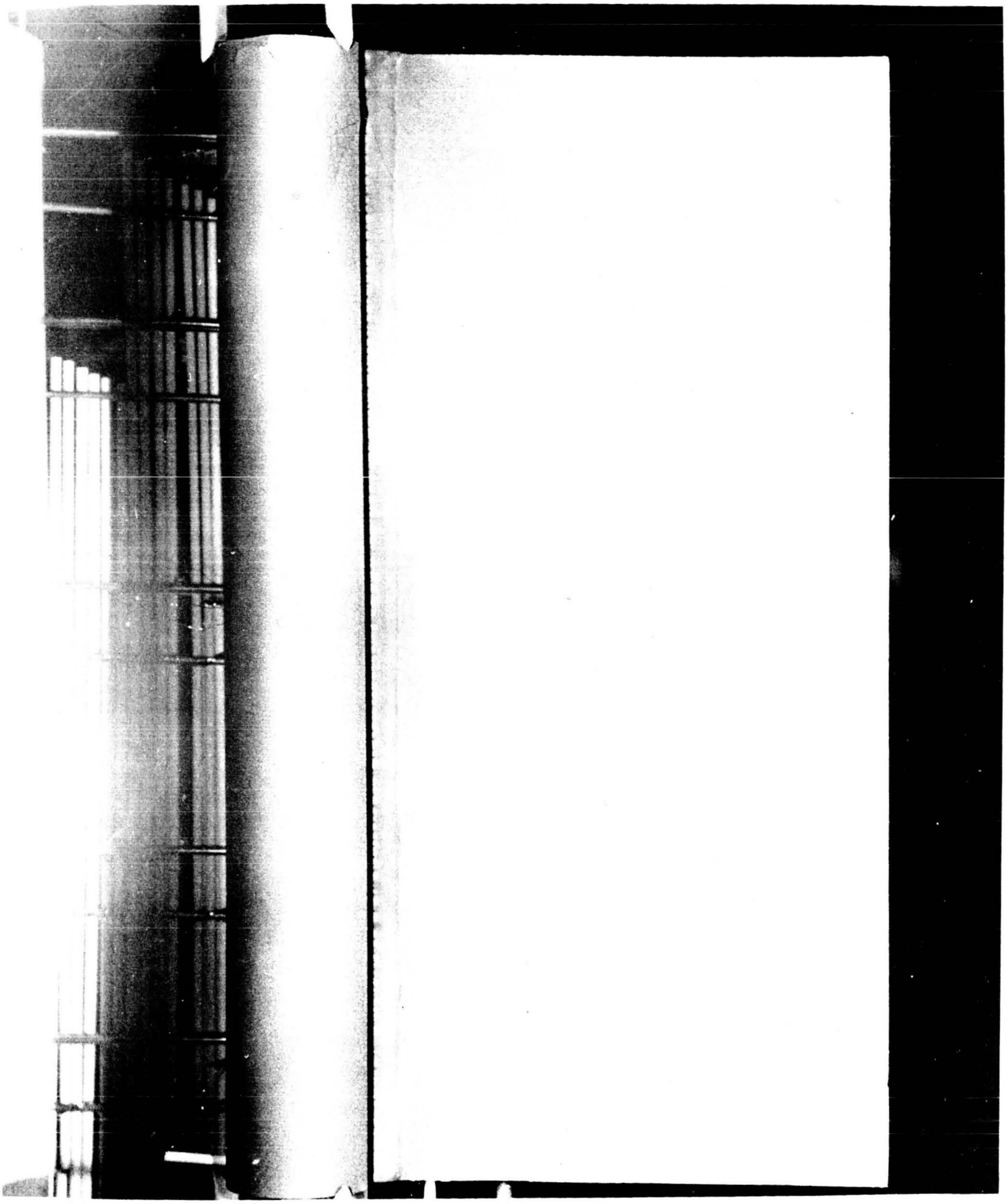
Titania base powder, supplied by the Plasmadyne Corporation, was plasma-arc sprayed onto a SNAP-8 test section and onto a Sunflower I test section at Pratt & Whitney Aircraft with Plasmadyne powder spray equipment. The resulting coating is titanium dioxide with small amounts of other oxides present.

The changing appearance of the titania-base coated SNAP-8 test section between 7811 hours and 9954 hours is shown in Figures 2, 3 and 4. During this period the loss of coating from the tube portion of the segment continued. At approximately 9400 hours flaking of the coating at the tube-fin junction was observed. The severity of the flaking increased through the end of this reporting period. The test section had accumulated approximately 9870 hours of endurance testing at the end of the reporting period.



NICKEL-CHROME SPINEL AND SILICON DIOXIDE COATED SNAP-8 TEST SECTION AFTER 10,801 HOURS OF ENDURANCE TESTING. DEFECTS IN COATING OCCURRED AT 2700 HOURS

Figure 1

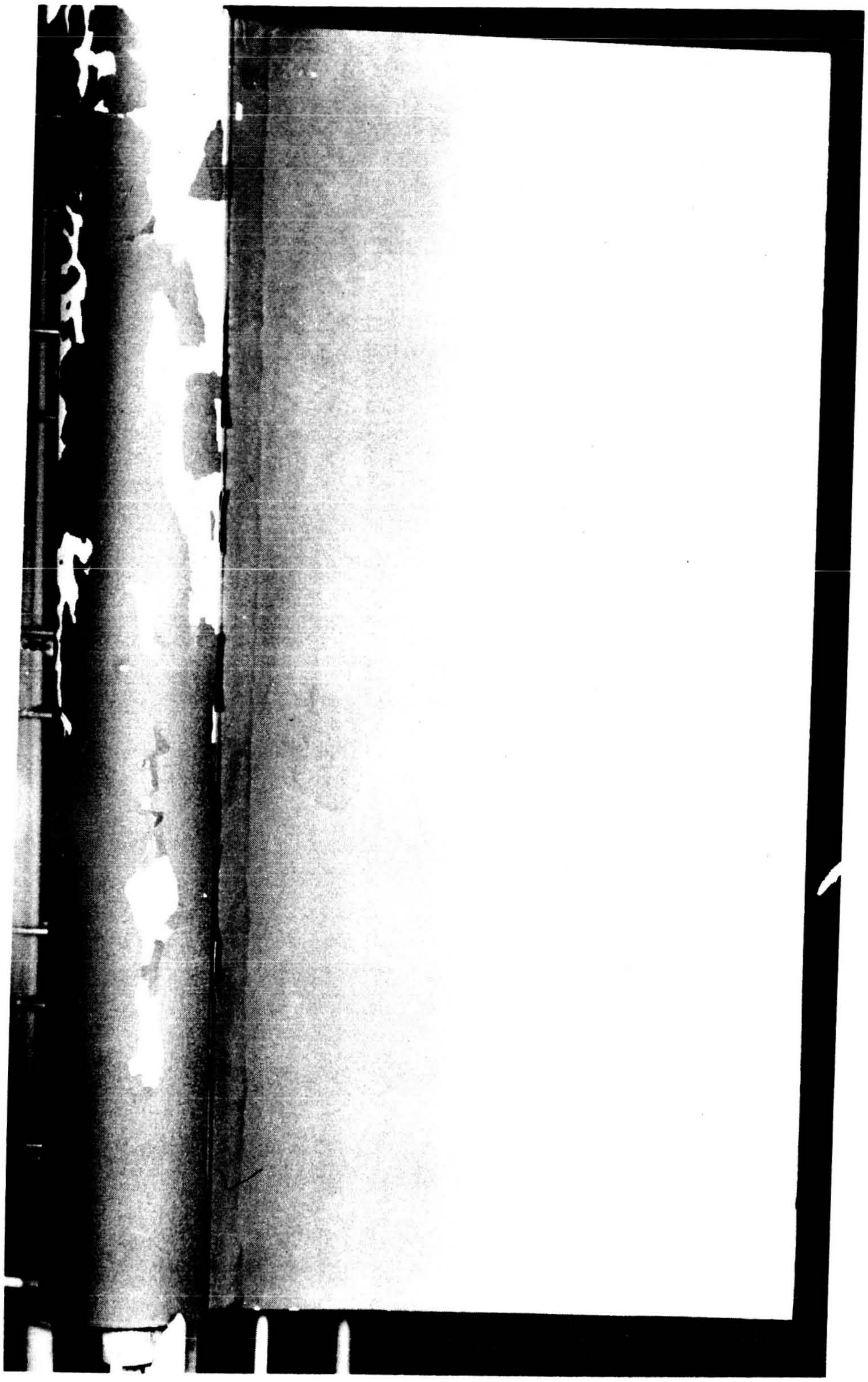


TITANIA-BASE COATED SNAP-8 TEST SECTION AFTER 7811.0 HOURS OF ENDURANCE TESTING. NOTE LOSS OF COATING ON TUBE PORTION OF SPECIMEN

Figure 2

TITANIA-BASE COATED SNAP-8 TEST SECTION AFTER 9424.3 HOURS OF ENDURANCE TESTING. NOTE LOSS OF COATING ON THE TUBE PORTION OF THE SPECIMEN AND FLAKING AT THE TUBE-FIN JUNCTION

Figure 3



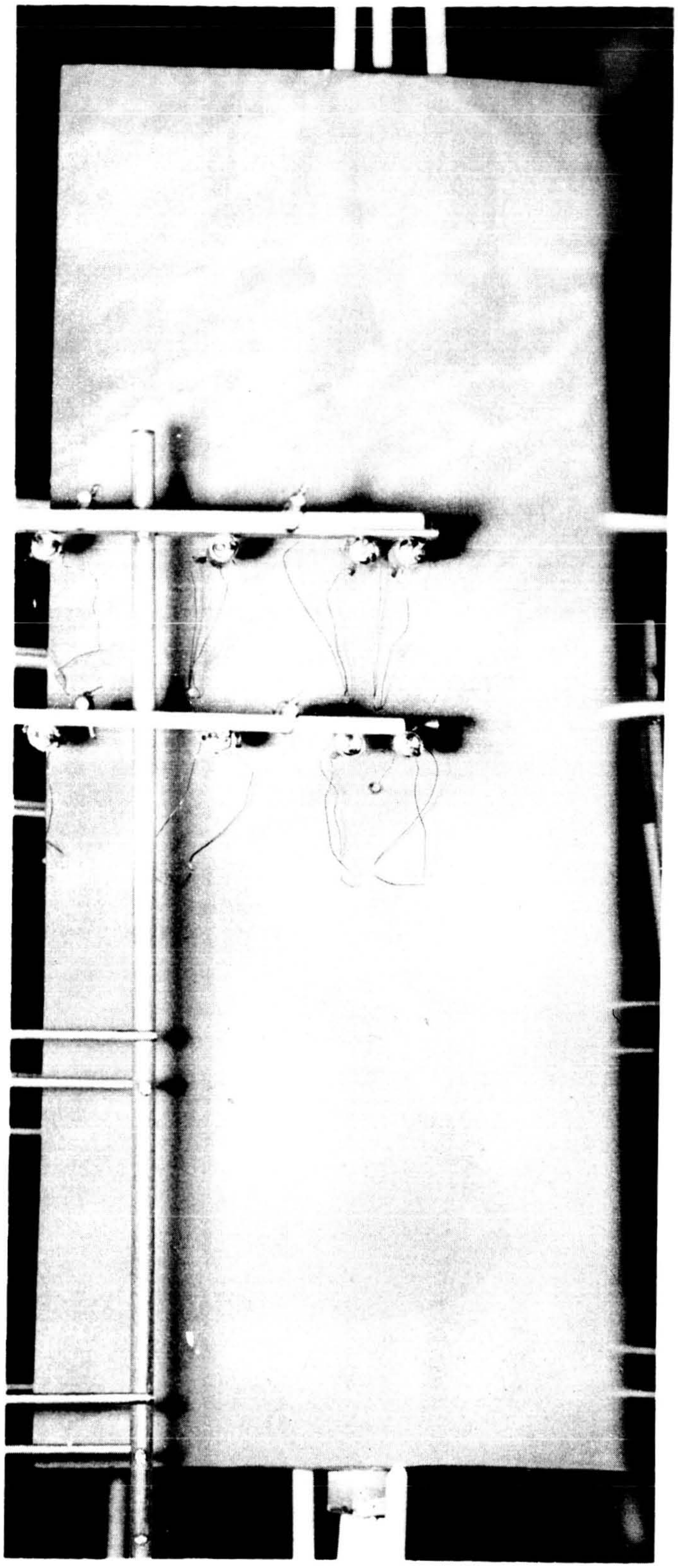
TITANIA-BASE COATED SNAP-8 TEST SECTION AFTER 9954 HOURS OF ENDURANCE TESTING. NOTE GENERAL LOSS OF COATING ON THE TUBE AND FLAKING AT THE TUBE-FIN JUNCTION

Figure 4

The appearance of the titania-base coated Sunflower I test section after 9914 hours is shown in Figure 5. The appearance of the coating has not changed. At the end of this report period this test section had accumulated approximately 9830 hours of endurance testing.

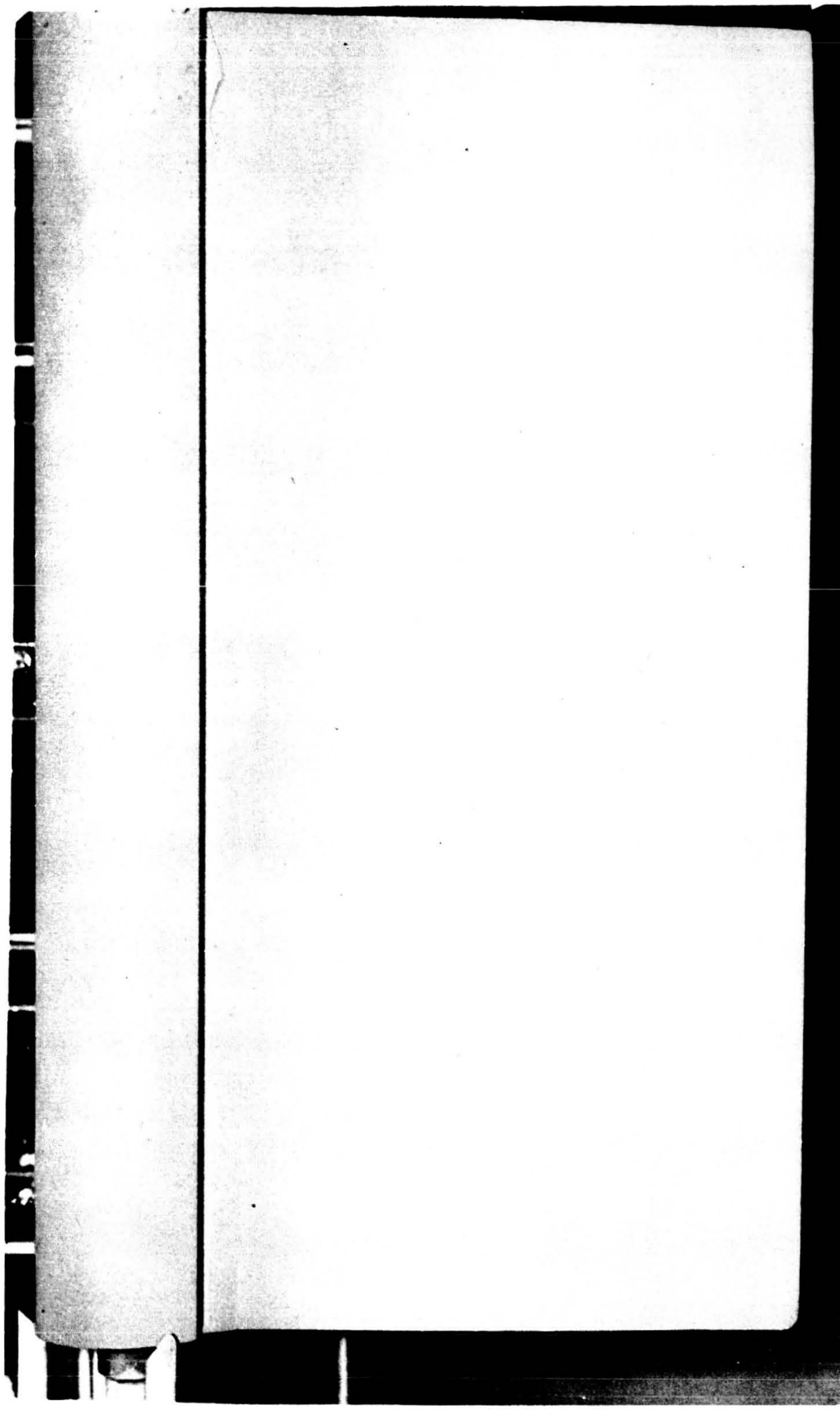
C. Endurance Test No. 4, SNAP-8 Test Section

A mixture of silicon carbide and silicon dioxide was aluminum phosphate bonded to this test section. The appearance of the test section at 8683 hours is shown in Figure 6. No change in the appearance of the coating has occurred. At the end of this report period the section had accumulated approximately 8600 hours of endurance testing.



TITANIA-BASE COATED SUNFLOWER 1 TEST SECTION AFTER 9914 HOURS OF ENDURANCE TESTING

Figure 5



SILICON CARBIDE AND SILICON DIOXIDE COATED SNAP-8
TEST SECTION AFTER 8683 HOURS OF ENDURANCE TESTING

Figure 6

II. EMITTANCE MEASUREMENTS

A. Stannic Oxide

A 4-mil thick coating of stannic oxide (SnO_2) supplied by A. D. MacKay, Incorporated, was aluminum phosphate bonded to a columbium - 1 per cent zirconium substrate. Analysis of the stannic oxide supplied indicated a particle size distribution with 83 per cent less than 10 microns in diameter and 53 per cent less than 3 microns in diameter. The coating was white, soft, and had a matte texture similar to that of 320 grit emery cloth. The coating-substrate bond strength was fair. Because the emittance at high temperatures drops below values useful in space applications, measurements were made only over the temperature range of 300°F to 1000°F. Results from these measurements appear in Table I and Figure 7. The emittance values recorded during cooling were lower than those recorded during heating, indicating that a change in the coating had occurred. Visual inspection of the specimen after testing did not reveal the change and x-ray diffraction analysis indicated both before and after testing that SnO_2 was the only detectable phase. Spectrographic analysis indicated that Sn and Al were the major constituents present before and after testing.

TABLE I

Coating: Stannic Oxide - Aluminum Phosphate Bonded
Substrate: Columbium-1% Zirconium
4.0-Mil Coating

Run Number	Elapsed Time (Hrs.)	Pressure (mm Hg)	Thermocouple Temp (°F)	Thermocouple ϵ_{TH}
1	1.8	4.8×10^{-6}	299	.918
	2.1	4.8×10^{-6}	500	.910
	2.5	6.4×10^{-6}	700	.900
	2.8	5.2×10^{-6}	901	.823
	3.0	9.6×10^{-6}	1000	.786
	3.1	5.2×10^{-6}	800	.803
	3.4	4.5×10^{-6}	600	.878
	4.2	6.2×10^{-6}	400	.871

TOTAL HEMISPHERICAL EMITTANCE vs. TEMPERATURE

COATING: STANNIC OXIDE—ALUMINUM PHOSPHATE BONDED (4-MIL)
SUBSTRATE: COLUMBIUM—1% ZIRCONIUM

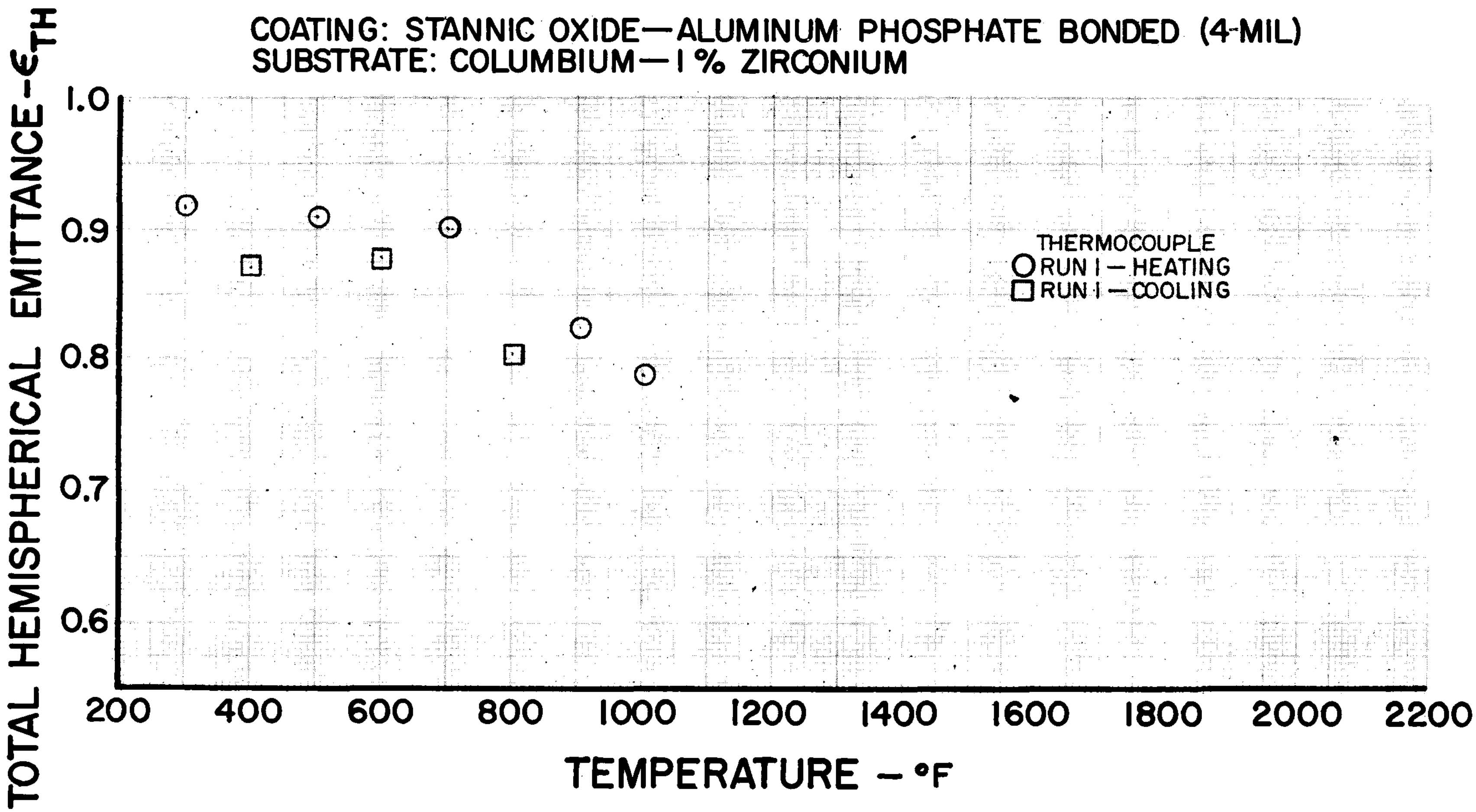


Figure 7
Page 11

B. Silicon Dioxide

A 5-mil thick coating of silicon dioxide (SiO_2) supplied by the Ottawa Silica Company was aluminum phosphate bonded to a columbium - 1 per cent zirconium substrate. Analysis of the powder supplied indicated a particle size distribution with 40 per cent between 15 and 24 microns in diameter and with 79 per cent between 9 and 37 microns in diameter. The coating was white, soft, and had a matte texture similar to 320 grit emery cloth. The coating-substrate bond strength was poor. Similar to stannic oxide, total hemispherial emittances of silicon dioxide were measured only at temperatures lower than 1000°F. Test results are shown in Table II and Figure 8. The emittance decreased from about 0.87 at 300°F to about 0.70 at 1000°F. Emittances during cooling were several per cent lower than during heating, indicating that a change in the coating had occurred.

Although the coating was still white after testing, it was noticed that the color was slightly darker than originally. No other changes were visible. X-ray diffraction analysis before and after testing indicated α SiO_2 to be the only detectable phase present. Spectrographic analysis indicated Si and Al to be the major constituents present before and after testing.

TABLE II

Coating: Silica-Aluminum Phosphate Bonded
Substrate: Columbium-1% Zirconium
5.0-Mil Coating

Run Number	Elapsed Time (Hrs.)	Pressure (mm Hg)	Thermocouple Temp (°F)	ϵ_{TH}
1	2.2	2.9×10^{-7}	300	.867
	3.0	2.8×10^{-7}	500	.855
	3.2	2.9×10^{-7}	698	.807
	3.4	3.9×10^{-7}	904	.725
	3.8	8.1×10^{-7}	1003	.697
	4.1	3.1×10^{-7}	801	.727
	4.4	2.9×10^{-7}	595	.723

Figure 8
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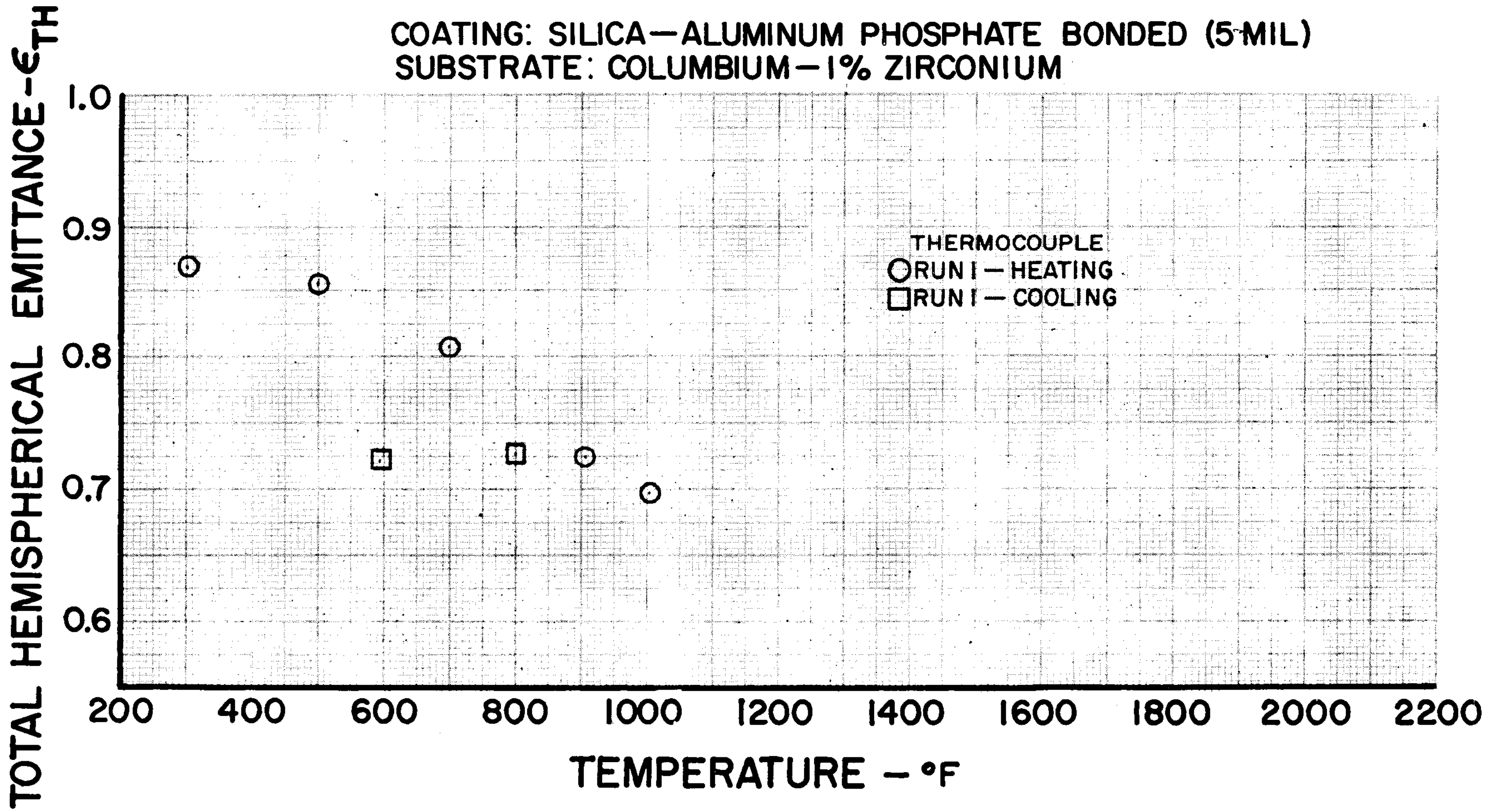
TOTAL HEMISPHERICAL EMITTANCE vs. TEMPERATURE



C. Palladium Black

Palladium black was prepared at Pratt & Whitney Aircraft by dissolving Engelhard alloy sheet stock containing 75 per cent palladium and 25 per cent silver with aqua regia. The solution was than diluted with distilled water to precipitate out all of the silver as silver chloride. The precipitate was removed by filtration and sodium hypophosphite was added to reduce the palladium ions to palladium black. A second filtering removed the palladium black which was given a final rinse with distilled water.

The palladium black was aluminum phosphate bonded to AISI-310 stainless steel and tested in the total hemispherical emittance rig. Before testing the olive drab coating was 1 mil thick, hard, and had a good coating-substrate bond strength. The texture of the coating was similar to that of 320 grit emery cloth and had a glassy appearance. The total hemispherical emittance of the specimen was measured between 300 and 1500°F. The data presented in Table III and in Figure 9 indicate that this 1-mil thick coating was partially transparent at temperatures lower than 700°F since the total emittance increased from about 0.66 at 300°F to approximately 0.86 at 700°F and then leveled off. The emittance remained at about this level up to a temperature of 1300°F, and then began decreasing as the temperature was increased to 1500°F. At 1500°F the total emittance was about 0.80. Between 1400°F and 1500°F the temperature was continuously drifting upwards at a given power setting indicating that the coating properties were changing. As a result of these changes, no measurements were made at higher temperatures. When the temperature was decreased, the emittance values obtained were found to be much lower than those recorded during heating. This further confirmed that a change in the coating properties had occurred. After removal of the specimen from the rig, it was apparent that the coating had separated from the substrate, but no other visible characteristics were found to have changed. Both before and after testing Pd was the only phase detected by x-ray diffraction analysis.

TABLE III

Coating: Palladium Black - Aluminum Phosphate Bonded
Substrate: AISI-310 Stainless Steel
1.0-Mil Coating

Run Number	Elapsed Time (Hrs)	Pressure (mm Hg)	Thermocouple Temp (°F)	Thermocouple ϵ_{TH}	Optical Pyrometer Temp (°F)	Optical Pyrometer ϵ_{TH}
1	0.1	4.6×10^{-7}	300	.658		
	0.3	3.7×10^{-6}	502	.775		
	0.4	2.6×10^{-6}	700	.861		
	0.5	2.3×10^{-6}	901	.870		
	0.7	2.2×10^{-6}	1001	.858		
	0.8	2.5×10^{-6}	1101	.855		
	1.0	2.8×10^{-6}	1202	.853		
	1.1	3.1×10^{-6}	1304	.852		
	1.2	3.2×10^{-6}	1403	.831		
	1.4	3.7×10^{-6}	1502	.802	1505	.797
	1.7	2.7×10^{-6}	1250	.778		
	1.8	2.7×10^{-6}	1150	.765		
	1.9	2.8×10^{-6}	1050	.761		

TOTAL HEMISPHERICAL EMITTANCE vs. TEMPERATURE

COATING: PALLADIUM BLACK—ALUMINUM PHOSPHATE BONDED (1-MIL)
SUBSTRATE: AISI 310 STAINLESS STEEL

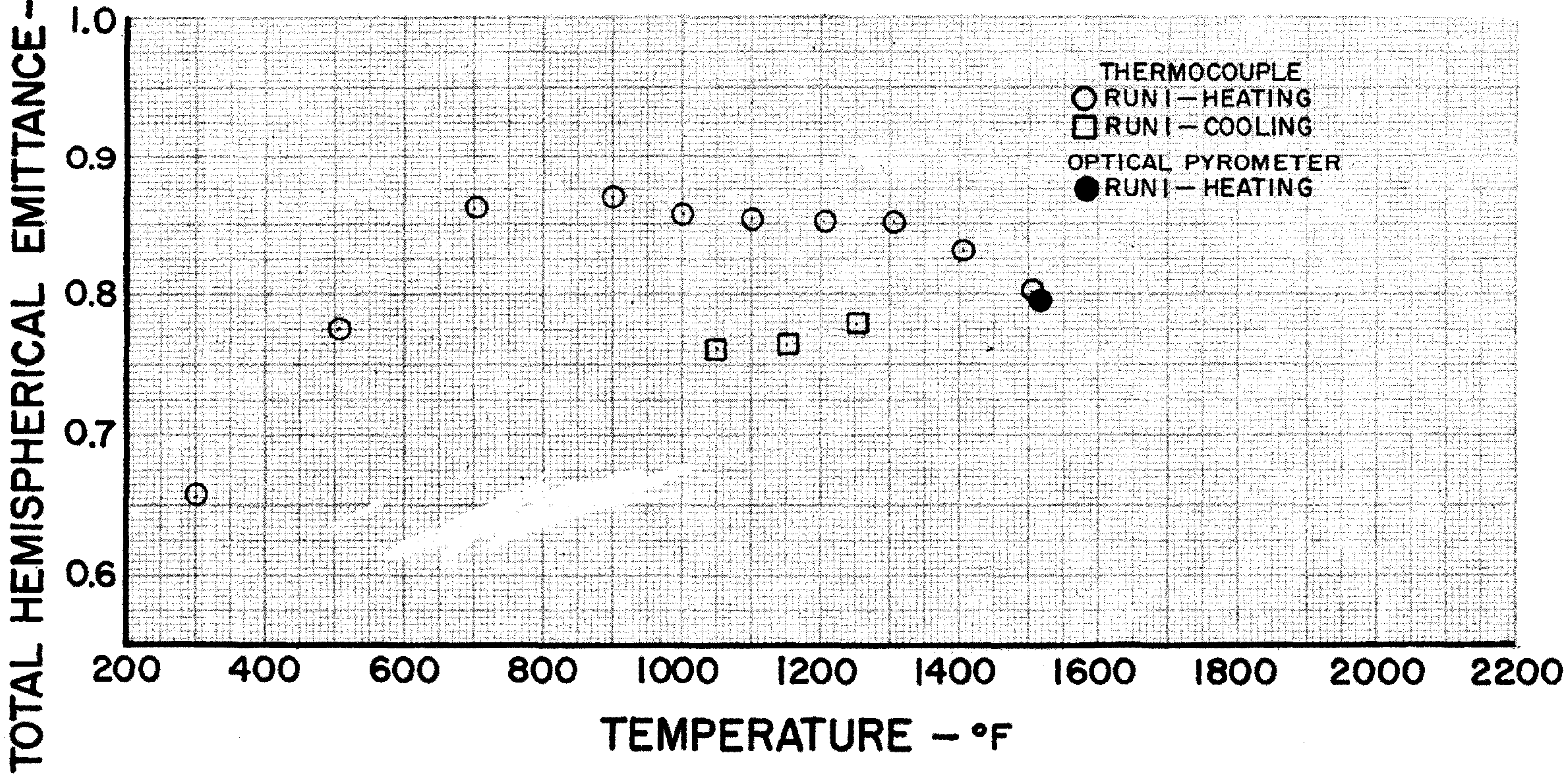


Figure 9
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D. Nickel Oxide

The nickel oxide (NiO) used for this coating was the black type supplied by the Varlacoid Chemical Company. A 3-mil thick coating was plasma-arc sprayed onto a columbium - 1 per cent zirconium substrate. The coating was dark grey, hard, had a good coating-substrate bond strength, and had a matte texture similar to that of 320 grit emergy cloth. The total hemispherical emittance values between 300°F and 2100°F are shown in Table IV and in Figure 10. The total emittance slowly increased from about 0.45 at 300°F to about 0.85 at 1500°F. It remained at this level up to 1800°F and then decreased to about 0.82 at 2100°F. During cooling, the total emittance remained at about 0.80 down to 1550°F, indicating that a change in the coating had occurred. When the specimen was cooled below 1550°F it could be seen that the coating had separated from the substrate, and no further emittance data were taken. The optical pyrometer temperatures appear to be erratic but the platinum-platinum 10 per cent rhodium and the chromel-alumel thermocouples were in good agreement. Emittance values based on platinum-platinum 10 per cent rhodium thermocouple temperatures are therefore the only ones reported. The erratic optical pyrometer readings are believed to have been caused by a partial blocking of the pyrometer lens by one of the window covers.

After testing, the only changes in the coating observed were an increased brittleness and the separation of the coating from the substrate. An x-ray diffraction analysis is presently being conducted on this specimen.

TABLE IV

Coating: Nickel Oxide - Plasma-Arc Sprayed
Substrate: Columbium - 1% Zirconium
3.0-Mil Coating

Run Number	Elapsed Time (Hrs.)	Pressure (mm Hg)	Thermocouple Temp (°F)	ϵ_{TH}
1	0.6	7.2×10^{-7}	300	.447
	1.4	2.2×10^{-6}	500	.588
	2.0	3.1×10^{-6}	700	.667
	2.6	2.9×10^{-6}	897	.725
	2.8	4.8×10^{-6}	998	.724
	2.9	3.3×10^{-6}	1100	.744
	3.1	3.0×10^{-6}	1200	.765
	3.2	2.8×10^{-6}	1299	.793
	3.3	2.7×10^{-6}	1396	.829
	3.5	2.6×10^{-6}	1500	.848
	3.8	2.8×10^{-6}	1601	.853
	4.1	2.2×10^{-6}	1700	.860
	4.5	2.2×10^{-6}	1803	.844
	4.6	2.7×10^{-6}	1902	.837
	4.8	2.1×10^{-6}	2001	.836
	5.0	2.0×10^{-6}	2099	.817
	5.3	1.2×10^{-6}	1851	.805
	5.5	8.7×10^{-7}	1550	.805

Figure 10
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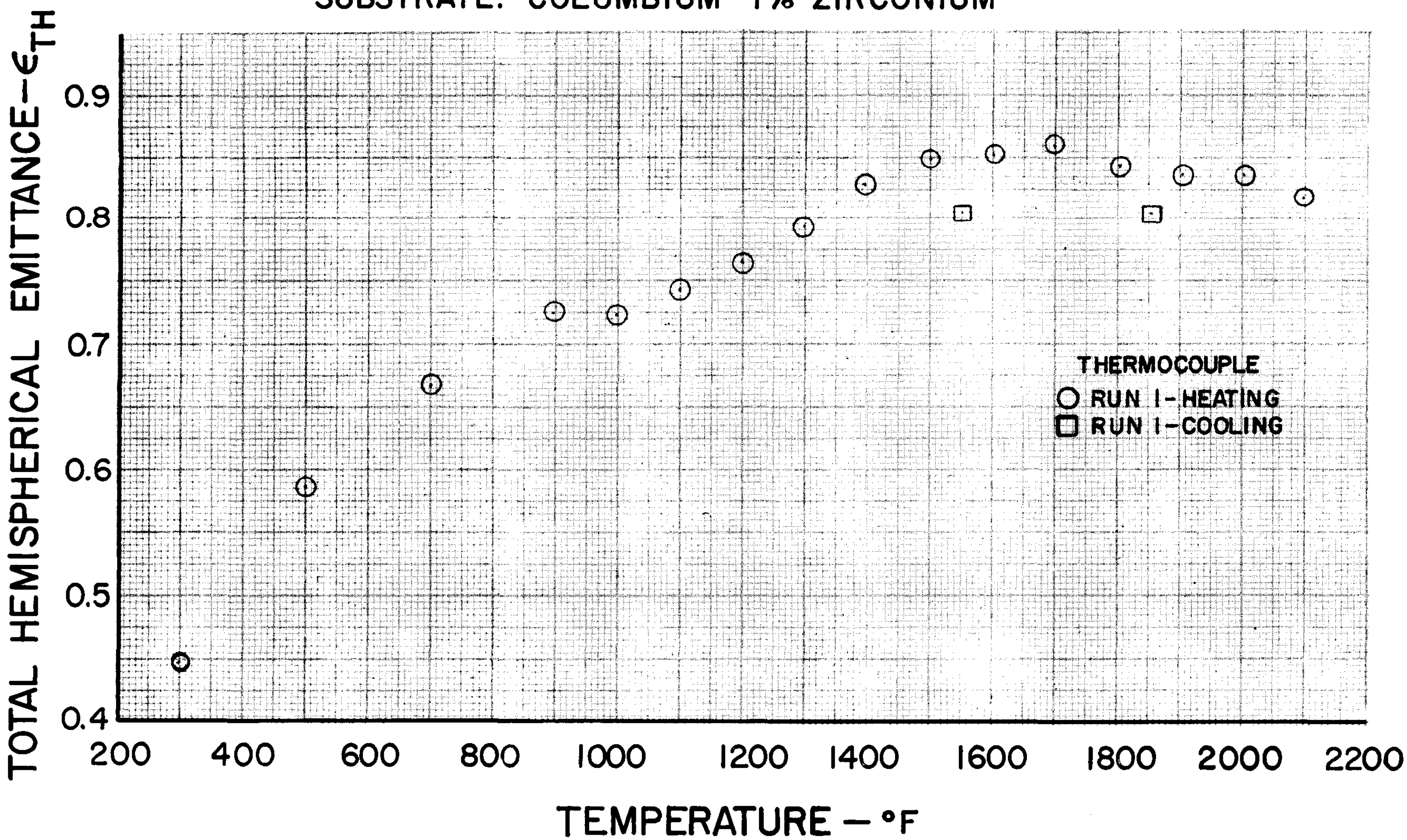
TOTAL HEMISPHERICAL EMITTANCE vs. TEMPERATURE

COATING: NICKEL OXIDE – PLASMA ARC SPRAYED (3-MIL)
SUBSTRATE: COLUMBIUM – 1% ZIRCONIUM



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E. Chromic Oxide

Chromic oxide (Cr_2O_3) obtained from the Plasmadyne Corporation was plasma-arc sprayed onto a columbium - 1 per cent zirconium substrate. The resulting coating was dark grey-green, 4 mils thick, extremely hard, and had a good coating-substrate bond strength. The coating had a matte texture similar to that of 80 grit emery cloth and was tested in the total hemispherical emittance rig over the temperature range of 300°F to 2100°F. As may be seen in Table V and in Figure 11, during heating the total emittance of the coating slowly decreased from about 0.82 at 500°F to 0.70 at 2100°F Up to 1200°F emittance values closely approximated those of the aluminum phosphate-bonded chromic oxide supplied by the Ceramic Color and Chemical Manufacturing Company (PWA-2128, Page 42). At higher temperatures these two curves diverge slightly with the plasma-arc sprayed coating having the higher emittance. During the test, the color of the coating changed to a dark greenish-blue except around the thermocouple wires where it was a bright green. The coating had become brittle but the bond strength remained good. The texture of the coating after testing was about equivalent to that of 320 grit emery cloth. Analysis by x-ray diffraction before and after testing indicated Cr_2O_3 to be the only detectable phase present.

TABLE V
Coating: Chromia - Plasma Arc Sprayed
Substrate: Columbium - 1% Zirconium
4.0 - Mil Coating

Run No.	Elapsed Time (hrs)	Pressure (mm Hg)	Thermocouple Temperature (°F)	ϵ_{TH}	Optical Pyrometer Temperature (°F)	ϵ_{TH}
1	0.7	2.8×10^{-7}	311	.790		
	1.1	4.0×10^{-7}	497	.812		
	1.6	5.7×10^{-7}	698	.819		
	2.3	1.3×10^{-6}	895	.778		
	2.7	4.5×10^{-6}	994	.782		
	2.9	9.1×10^{-7}	1101	.780		
	3.1	6.1×10^{-7}	1200	.780		
	3.3	6.0×10^{-7}	1301	.787		
	3.6	1.0×10^{-7}	1403	.771		
	Heating Current Off; Vacuum Maintained					
2	4.1	1.3×10^{-6}	1502	.761	1510	.749
	4.9	6.4×10^{-7}	1607	.743	1616	.730
	5.4	6.5×10^{-7}	1702	.748	1718	.726
	5.8	7.2×10^{-7}	1804	.746	1821	.699
	6.7	1.9×10^{-6}	2100	.709	2122	.685
	7.4	2.8×10^{-7}	1852	.740	1882	.722
	7.5	1.9×10^{-7}	1552	.818	1585	.767
	7.8	1.8×10^{-7}	1250	.818		
	7.9	1.7×10^{-7}	952	.823		

TOTAL HEMISPHERICAL EMITTANCE vs. TEMPERATURE

COATING: CHROMIA–PLASMA ARC SPRAYED (4-MIL)
SUBSTRATE: COLUMBIUM–1% ZIRCONIUM

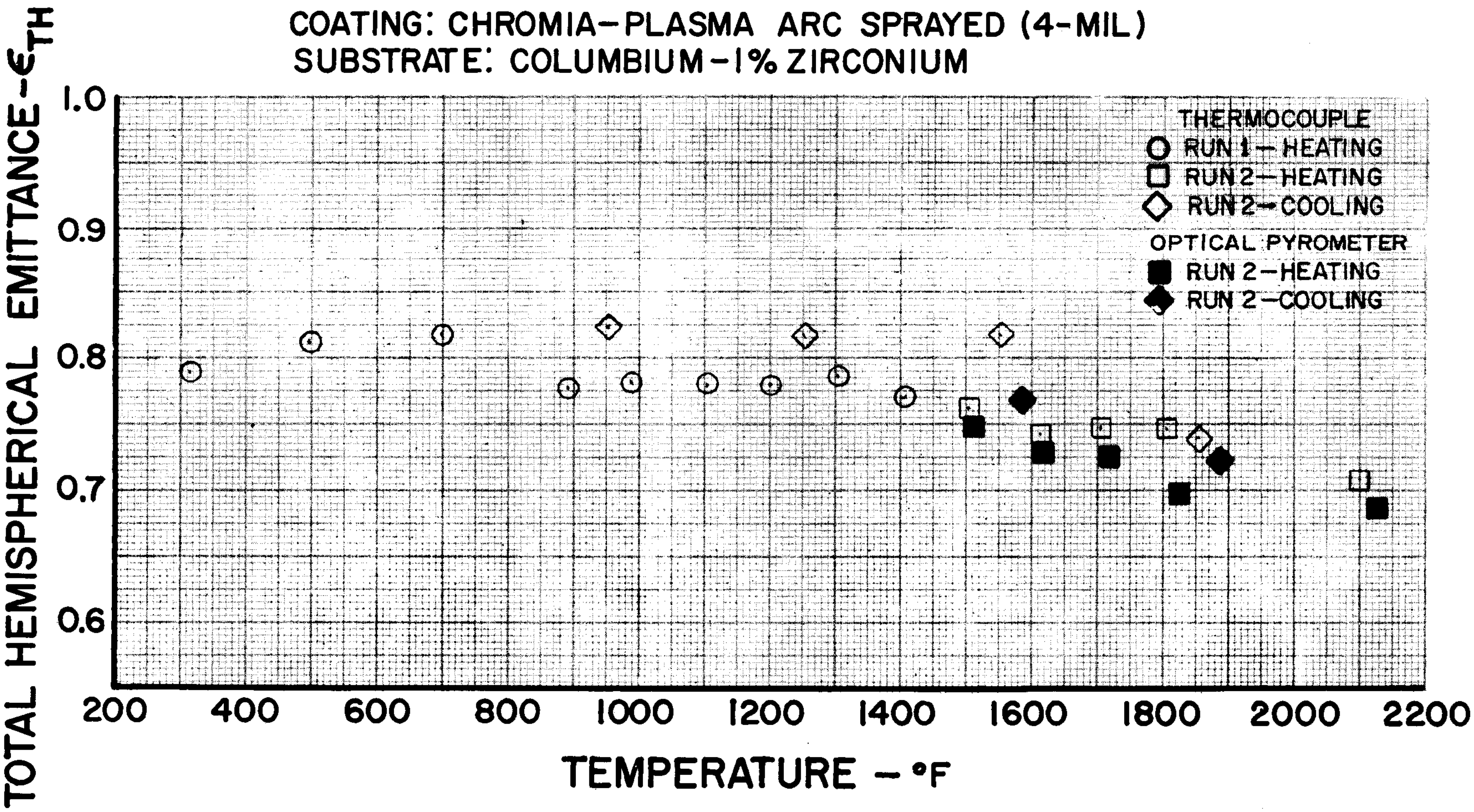

Figure 11
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F. Cobalt Oxide

The Fisher Scientific Company supplied the cobalt oxide (CoO) which was plasma-arc sprayed onto a columbium - 1 per cent zirconium substrate to produce a 4-mil thick coating. The observable characteristics of this coating before testing were recorded as matte blue-black, hard, and with a texture similar to that of 320 grit emery cloth. The coating-substrate bond strength was good. The results of total hemispherical emittance testing from 300°F to 2200°F are shown in Table VI and in Figure 12. The total emittance of this coating during heating was about 0.88 from 300°F to 1100°F and then decreased to about 0.79 at 1500°F. Above 1500°F the total emittance increased until a value of 0.90 was achieved at 2200°F. During cooling, the emittance data substantiated the heating values from 2150 to 1550°F, but then continued decreasing. From this data it appears that the coating underwent two changes during the test, one at about 1100°F, and a second at about 1500°F. When the specimen was removed from the rig, it could be seen that the coating had separated from the substrate at the ends of the specimen, although it remained well bonded in the center portion where data is taken.

It is not believed that the last change in the slope of the emittance curve resulted from the separation of the coating from the substrate. A separation of the coating from the substrate always causes the substrate temperature to increase. If the coating separates from the substrate but remains intact, the decreased thermal conductivity across the coating-substrate gap results in a substrate temperature increase; if the coating does not remain intact but leaves a portion of the substrate exposed, the markedly lower emittance of the substrate will also result in a substrate temperature increase. Emittance is calculated by the use of the following equation:

$$\epsilon_{TH} = \frac{IV_{ts}}{A_{ts}\,\sigma\,(T_m^4 - T_w^4)}$$

TABLE VI

Coating: Cobalt Oxide - Plasma Arc Sprayed
Substrate: Columbium - 1% Zirconium
4.0-Mil Coating

Run Number	Elapsed Time (Hrs)	Pressure (mm Hg)	Thermocouple Temp (°F)	ϵ_{TH}	Optical Pyrometer Temp (°F)	ϵ_{TH}
1	0.8	6.6×10^{-7}	304	.872		
	1.1	2.1×10^{-6}	500	.886		
	1.4	7.7×10^{-7}	700	.897		
	1.8	9.2×10^{-7}	899	.882		
	2.0	1.1×10^{-6}	1000	.878		
	2.1	1.3×10^{-6}	1101	.879		
	3.1	1.4×10^{-6}	1202	.858		
	3.4	1.3×10^{-6}	1306	.824		
	3.6	1.3×10^{-6}	1402	.802		
	3.8	1.3×10^{-6}	1500	.792	1480	.825
	4.0	1.2×10^{-6}	1602	.797	1592	.835
	4.1	1.3×10^{-6}	1701	.806	1690	.823
	4.2	1.3×10^{-6}	1800	.825	1790	.840
	4.4	1.4×10^{-6}	1899	.837	1892	.847
	4.7	1.7×10^{-6}	1999	.851	1975	.885
	4.8	2.1×10^{-6}	2097	.856	2070	.893
	5.1	2.3×10^{-6}	2193	.873	2174	.899
	5.2	1.5×10^{-6}	2147	.884	2150	.880
	5.4	1.5×10^{-6}	1853	.855	1860	.884
	5.6	1.5×10^{-6}	1553	.806	1556	.801
	5.8	1.8×10^{-6}	1250	.775		
	6.1	1.8×10^{-6}	951	.750		

TOTAL HEMISPHERICAL EMITTANCE vs. TEMPERATURE

COATING: COBALT OXIDE—PLASMA ARC SPRAYED (4-MIL)
SUBSTRATE: COLUMBIUM—1% ZIRCONIUM

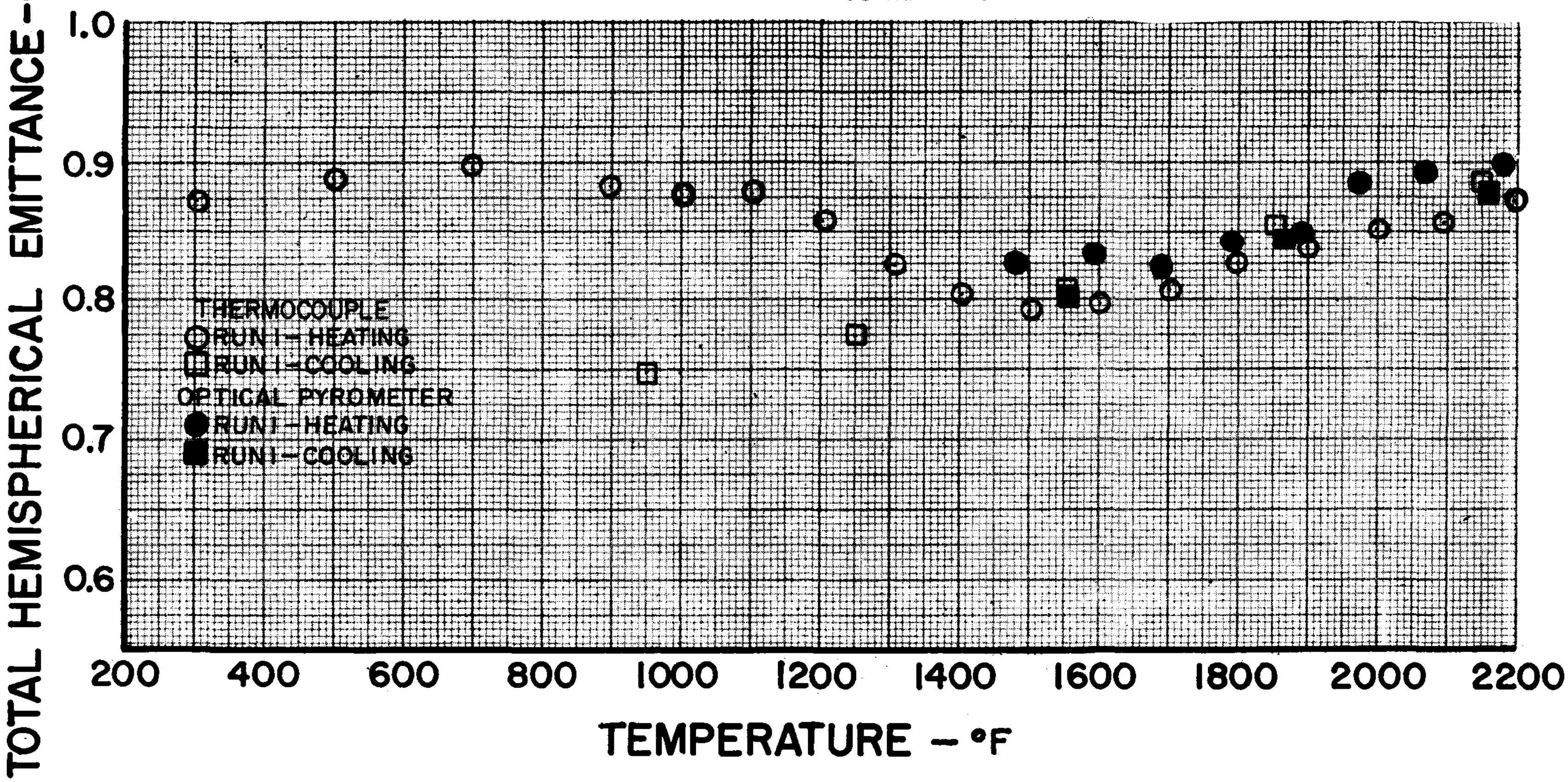

Figure 12
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where:

ϵ_{TH}	is the calculated total hemispherical emittance;
V_{ts}	is the voltage drop across the test section of the specimen;
I	is the current through the specimen;
A_{ts}	is the radiating surface area;
σ	is the Stefan-Boltzman constant;
T_m	is the substrate temperature; and
T_w	is the test-chamber wall temperature.

It may therefore be seen that the increase in substrate temperature which results from coating-substrate separation is reflected as a decrease in the calculated emittance value rather than an increase such as was recorded.

After testing the only observable changes in the characteristics of the coating were the coating-substrate separation and a change in color to a brownish grey. X-ray diffraction analysis before and after testing detected CoO as the only phase present.

G. Boron Carbide

Particle size analysis of the boron carbide (B_4C) obtained from the Carborundum Company indicated that 79 per cent of the particle diameters were between 24 and 37 microns, and that 94 per cent were between 15 and 37 microns. The remaining 6 per cent were smaller than 15 microns. The coating was phosphate bonded on to columbium - 1 per cent zirconium tubes. One of these specimens was tested in the total hemispherical emittance rig and another in the spectral normal emittance rig.

1. Total Hemispherical Emittance Test - The 6-mil thick coating was black with a glassy appearance, hard, and had a texture similar to that of 80 grit emery cloth. The coating-substrate bond strength was fair. Total hemispherical emittance was measured over the temperature range of 300°F to 1400°F. Results of testing appear in Table VII and Figure 13. The emittance level remained at about 0.85 from 300°F to 700°F, then increased to about 0.94 at 1200°F where it remained until the test was terminated at 1400°F. At 1200°F cracks were visible at each end of the test specimen but not in the center section where the emittance data is obtained. At 1400°F thermocouples numbers 2, 3 and 5 (see Figure 4, PWA-2088) fell off. Before 1500°F was reached thermocouple number 4 and the voltage leads fell off, requiring the test to be terminated. The emittance values based on run 2 show an increase in emittance and throughout this run the temperature profile was not flat along the test section, thus introducing possible errors in the results. No change in the coating was observed when the specimen was removed from the rig.

   X-ray diffraction analysis indicated B_4C to be the only phase present before testing, but Cb and B_4C were both present after testing. Spectrographic analysis showed B to be the only major element present both before coating and after testing.

   The total hemispherical emittance of boron carbide in a Synar binder (colloidal silica) was reported in PWA-1877, Page 33, as being less than 0.80 over this same temperature range. The coating reported in PWA-1877, however, was in a different binder, 2.8 mils thick, and was on a molybdenum strip. The binder and perhaps other differences between the two specimens could account for the disagreement.

TABLE VII

Coating: Boron Carbide - Aluminum Phosphate Bonded
Substrate: Columbium-1% Zirconium
6.0-Mil Coating

Run Number	Elapsed Time (Hrs.)	Pressure (mm Hg)	Thermocouple Temp (°F)	Thermocouple ϵ_{TH}
1	0.5	2.6×10^{-7}	300	.841
	0.8	4.2×10^{-7}	500	.850
	1.1	3.9×10^{-7}	700	.845
	Heating current off, vaccum chamber opened to repair thermocouple 4 and replace coolant pump			
2	1.7	5.7×10^{-6}	904	.905
	2.4	8.1×10^{-6}	1000	.936
	2.6	8.4×10^{-6}	1101	.908
	2.7	9.0×10^{-6}	1202	.937
	2.9	9.4×10^{-6}	1304	.939
	3.1	8.0×10^{-6}	1408	.949
	Thermocouple and voltage leads fell off, test terminated			

TOTAL HEMISPHERICAL EMITTANCE vs. TEMPERATURE

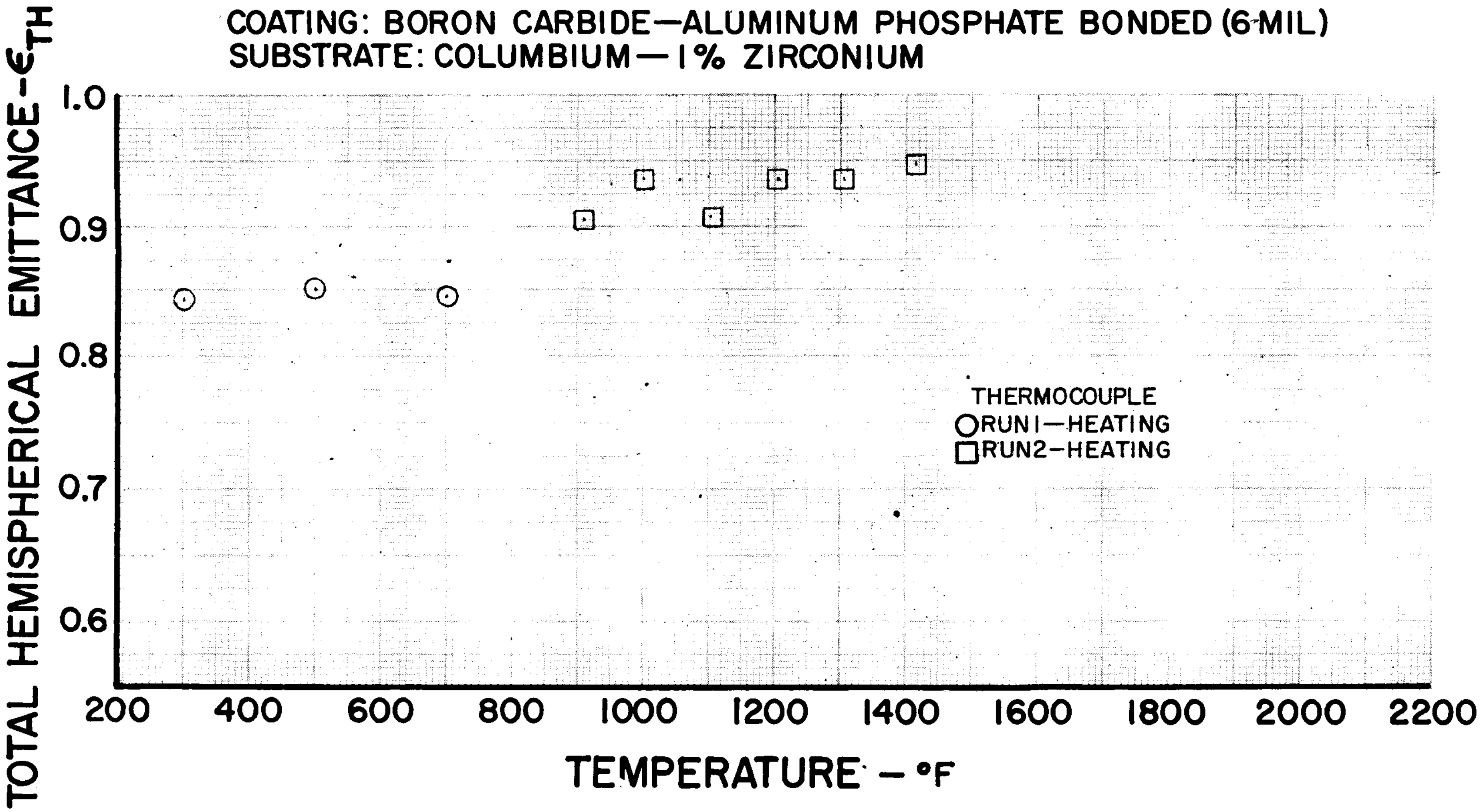

Figure 13
Page 30

2. Spectral Normal Emittance Test - The second specimen from this batch had a blue-black, 5-mil thick coating which was soft and poorly bonded to the substrate. The coating texture was similar to that of 80 grit emery cloth. The results of spectral emittance testing from 1.57 to 12.4 microns at 900°F are shown in Figure 14. When the temperature was increased to 1200°F it was found that the power setting had to be continually changed to maintain the required temperature. The coating had a mottled appearance and seemed to be failing so the test was terminated. The only observable change in the coating upon completion of the test was that its color had changed to black.

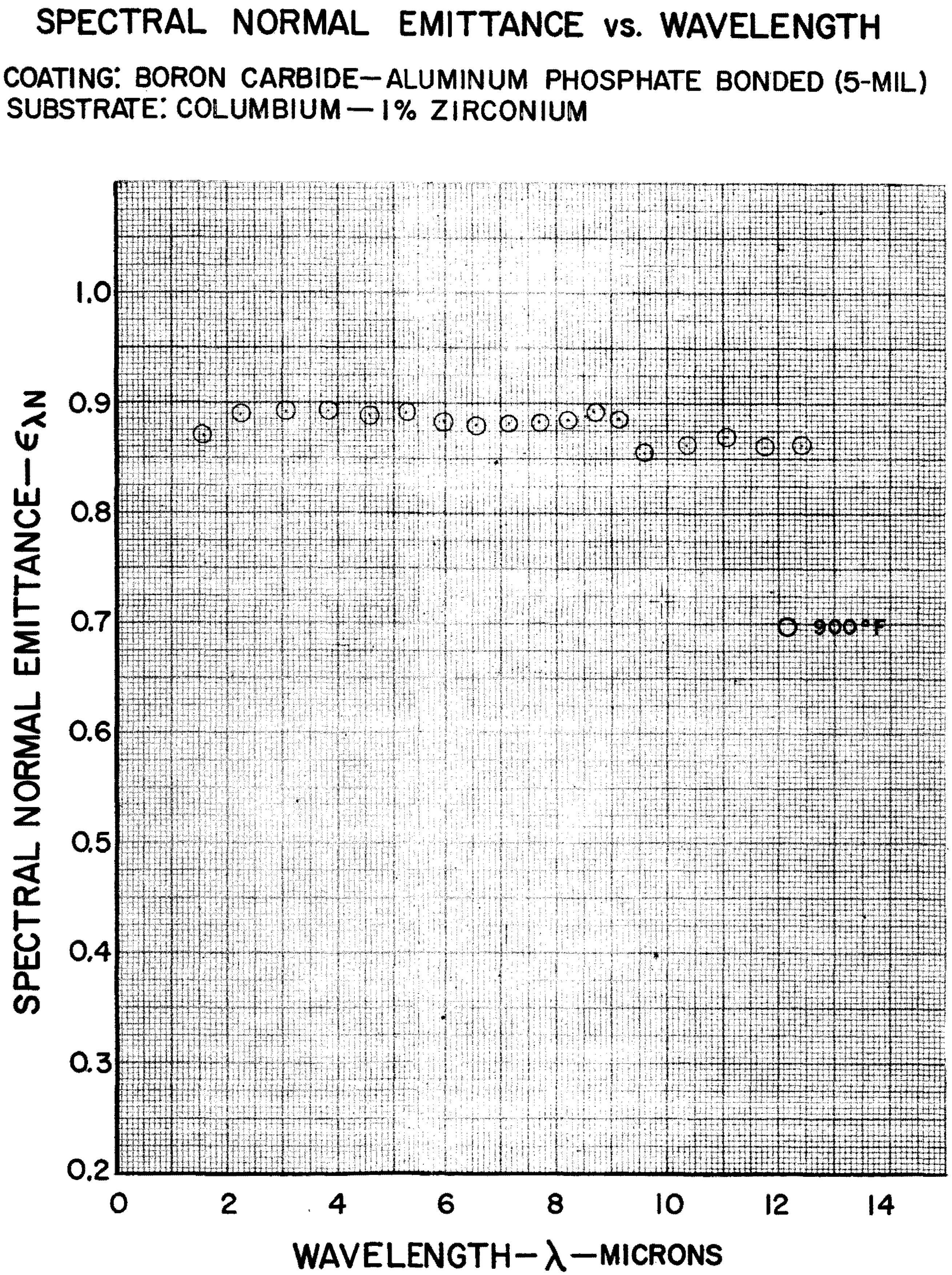
SPECTRAL NORMAL EMITTANCE vs. WAVELENGTH
COATING: BORON CARBIDE—ALUMINUM PHOSPHATE BONDED (5-MIL)
SUBSTRATE: COLUMBIUM—1% ZIRCONIUM
1.0
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
SPECTRAL NORMAL EMITTANCE—$\epsilon_{\lambda N}$
900°F
0
2
4
6
8
10
12
14
WAVELENGTH—λ—MICRONS

Figure 14

H. Iron-Titanium-Oxide

The specimens for which test results are reported in this section are from the same batch of coated tubes for which test results were reported in PWA-2128. The powder used (FCT-11) was obtained from the Continental Coatings Corporation and was plasma-arc sprayed onto columbium - 1 per cent zirconium tubes. Particle size analysis indicated a diameter size distribution with 50 per cent 28 microns or larger. Thirty-two per cent fell between 28 and 35 microns and the remainder of the particles had diameter sizes evenly distributed between 3 and 28 microns.

One of the plasma-arc sprayed specimens had different visible coating characteristics than the other three and therefore this specimen was tested in the total hemispherical emittance rig to determine how its total emittance was effected. Another specimen was tested in the spectral normal emittance rig.

1. Total Hemispherical Emittance Test - This coating was 4 mils thick and had a dark grey background with metallic yellow specks on its surface. This was a very hard coating that was very well bonded to the substrate and had a matte texture similar to that of 40 grit emery cloth. Total hemispherical emittance was measured between 300°F and 1900°F. Table VIII and Figure 15 show that the total hemispherical emittance of the coating increased from about 0.45 at 300°F to about 0.64 at 1500°F. The emittance remained at this level up to 1800°F and then rapidly decreased to about 0.45 at 1900°F where it remained during cooling. After removing the specimen from the rig it was observed that the coating was now light grey but otherwise unchanged in appearance.

2. Spectral Normal Emittance Test - Before testing the coating was 4 mils thick, grey-black, hard, and had a matte texture similar to that of 80 grit emery cloth. The coating-substrate bond strength was fair.

TABLE VIII

Coating: Iron Titanium Oxide - Plasma Arc Sprayed
Substrate: Columbium - 1% Zirconium
4.0-Mil Coating

Run Number	Elapsed Time (Hrs)	Pressure (mm Hg)	Thermocouple Temp (°F)	Thermocouple ϵ_{TH}	Optical Pyrometer Temp (°F)	Optical Pyrometer ϵ_{TH}
1	0.8	1.7×10^{-7}	319	.454		
	1.1	1.3×10^{-6}	501	.475		
	1.6	3.0×10^{-6}	700	.516		
	1.9	5.6×10^{-6}	900	.538		
	3.0	3.0×10^{-4}	995	.543		
	3.2	5.8×10^{-4}	1101	.560		
	3.4	6.6×10^{-4}	1199	.577		
	3.6	6.6×10^{-4}	1299	.588		
Heating Current Maintained; Vacuum Leak Repaired						
2	4.6	1.3×10^{-6}	1001	.580		
	4.9	1.1×10^{-6}	1099	.594		
	5.2	1.2×10^{-6}	1200	.599		
	5.4	1.2×10^{-6}	1300	.612		
	5.6	1.9×10^{-6}	1398	.620		
	5.9	1.5×10^{-6}	1504	.632	1500	.637
	6.3	1.9×10^{-6}	1599	.643	1604	.637
Heating Current Off; Vacuum Maintained						
3	7.9	3.2×10^{-6}	1690	.650	1700	.638
	8.3	1.4×10^{-5}	1801	.644	1810	.634
	9.0	2.4×10^{-6}	1917	.470	1938	.454
	9.3	2.6×10^{-7}	1650	.459	1655	.455
	9.5	1.9×10^{-7}	1551	.456	1540	.466
	9.7	1.5×10^{-7}	1449	.453		
	10.5	2.1×10^{-7}	1251	.454		
	10.7	2.0×10^{-7}	1051	.437		

TOTAL HEMISPHERICAL EMITTANCE vs. TEMPERATURE

COATING: IRON TITANIUM OXIDE—PLASMA ARC SPRAYED (4 MIL)
SUBSTRATE: COLUMBIUM-1%-ZIRCONIUM

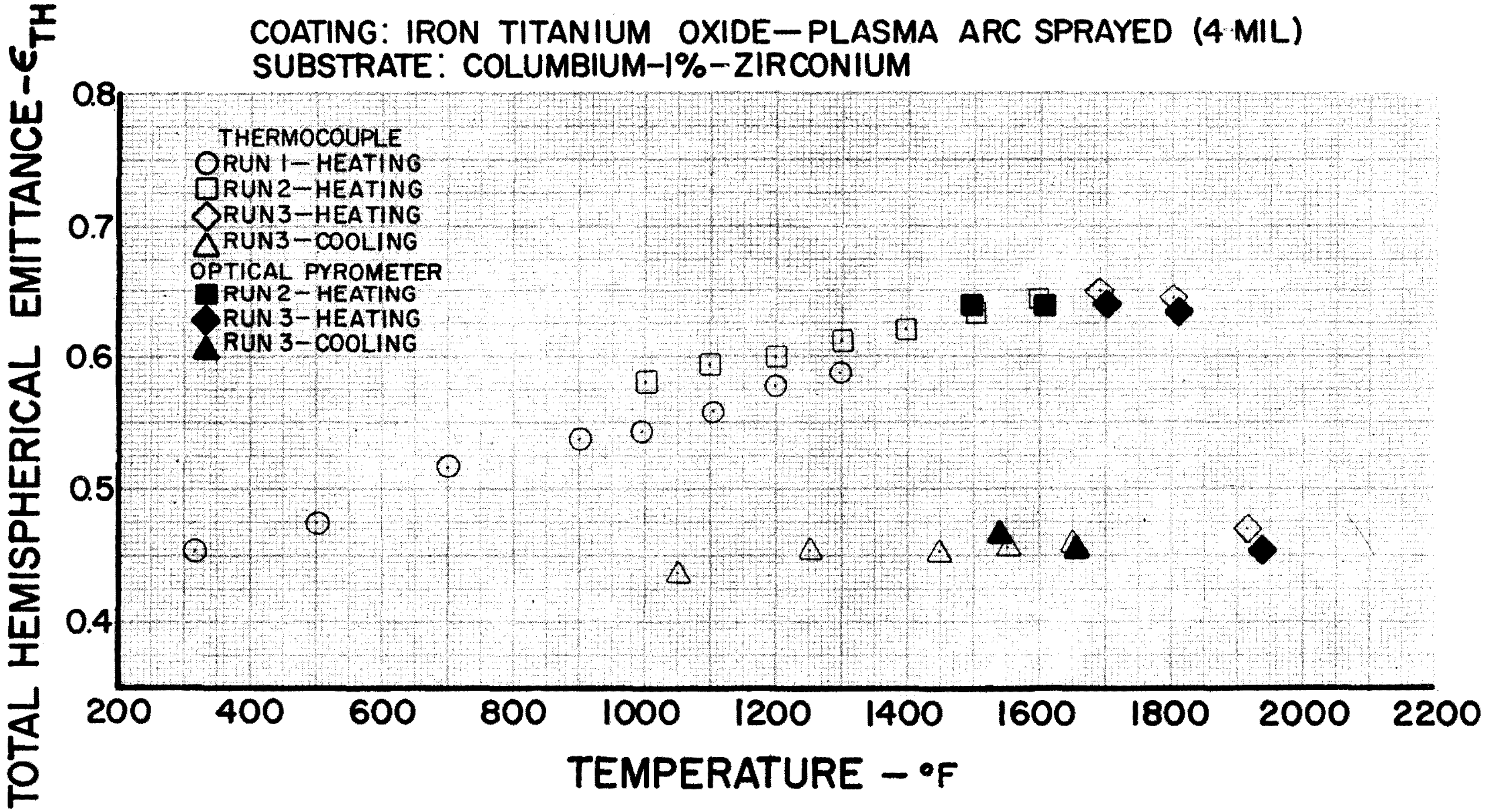

Figure 15
Page 35

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Spectral normal emittance was measured from 1.5 to 12.4 microns at 900°F, from 1.1 to 13.5 microns at 1450°F, and from 0.45 to 13.5 microns at 2000°F. As can be seen in Figure 16, the curve is flat from about 1 to 12.5 microns. The shape and level of emittance values for this curve are very similar to those obtained for iron-titanium-aluminum-oxide reported in PWA-2128, Figure 34. The characteristics of the coating did not change as a result of testing.

3. General Remarks - The specimen which was tested in the total hemispherical emittance rig appears to be an exception for plasma-arc sprayed iron-titanium oxide coatings. While data for this specimen showed no emittance values higher than 0.65, data for a similar specimen reported in PWA-2128, Figures 35, 36 and 37 showed no values lower than 0.80. Further, the change in emittance at temperatures around 1900°F was not observed for the other specimens tested. These emittance differences were expected since the external characteristics of the coatings differed.

X-ray diffraction analysis of the specimen for which test results were reported in PWA-2128 indicated that Fe_2TiO_5 was present both before and after testing. By spectrographic analysis it was found that Fe and Ti were two major constituents present both before coating and after testing Also, Mn was detected as a minor element before coating and after testing.

SPECTRAL NORMAL EMITTANCE vs. WAVELENGTH

COATING: IRON TITANIUM OXIDE-PLASMA ARC SPRAYED (4-MIL)
SUBSTRATE: COLUMBIUM-1% ZIRCONIUM

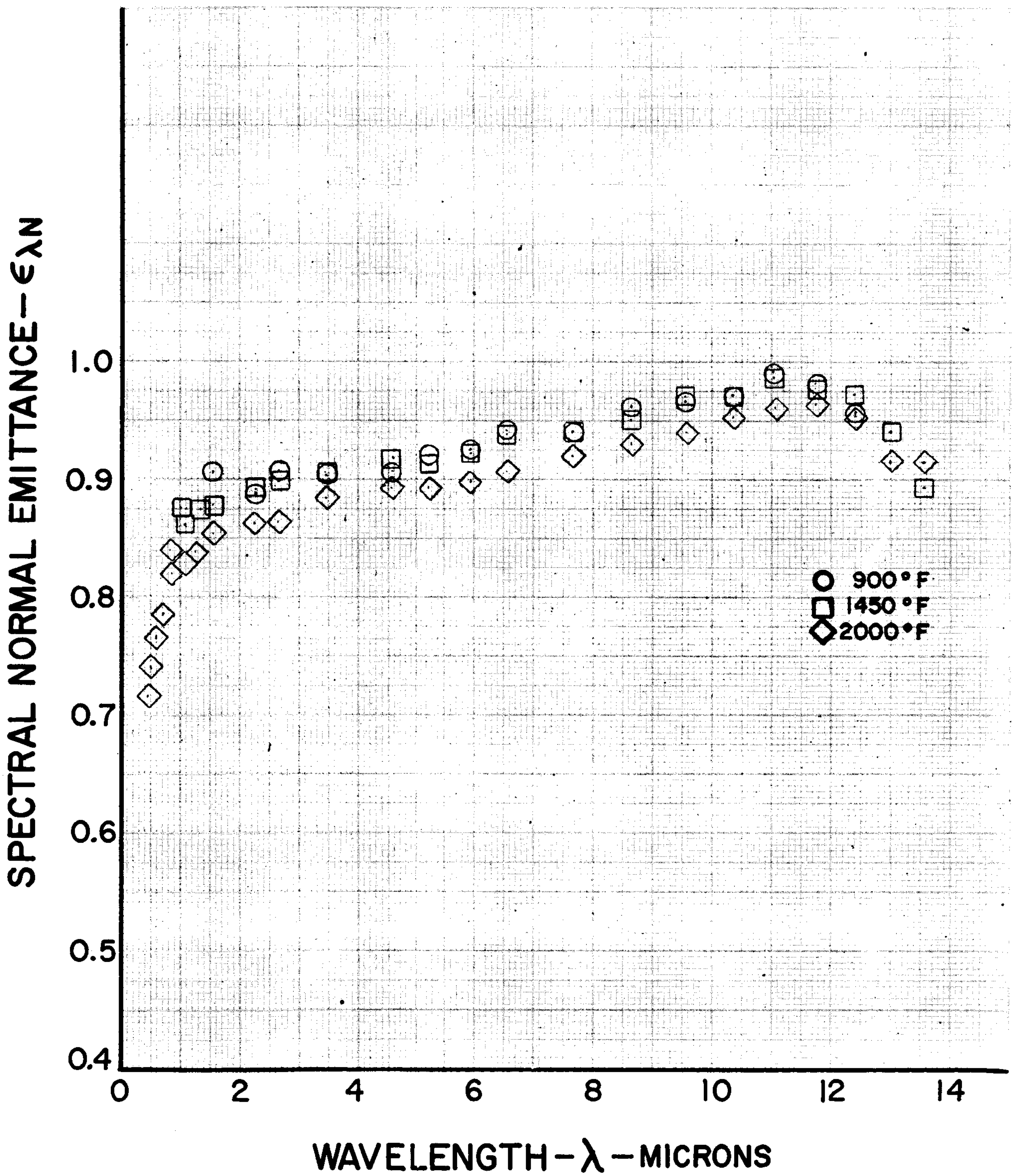

Figure 16

I. Iron-Titanium-Aluminum-Oxide

Particle size analysis of the powder (FCT-12) obtained from Continental Coatings Corporation that 7 per cent of the particles were 69 microns in diameter or greater and 8 per cent were 3 microns in diameter or less. Twenty-six per cent were between 4 and 10 microns in diameter. The remainder had diameters with fairly uniform size distribution over the range of 10 to 69 microns. The coatings were plasma-arc sprayed onto columbium - 1 per cent zirconium tubes and were applied at the same time as those reported in PWA-2128. These specimens were tested in the total hemispherical emittance rig and in the short term endurance rig.

1. Total Hemispherical Emittance Test - The plasma-arc sprayed specimen tested was the same specimen for which total hemispherical emittance data was reported in PWA-2128. The coating was 5 mils thick, medium black, fairly hard, and had a poor coating-substrate bond strength. The matte texture of the coating was about that of 80 grit emery cloth. Since this coating had been previously tested and was being retested primarily to determine the repeatability of the data and to measure changes in emittance at temperatures above 1000°F, the total hemispherical emittance was measured only between 1000°F and 2200°F. Test results appear in Table IX and in Figure 17. Comparison of Figure 17 of this report with Figure 33 of PWA-2128 shows that the emittance was about 0.82 and 0.87 respectively. Above 1500°F the curves coincide at about 0.87 and remain at this level up to 2200°F. During cooling, however, the emittance did not retrace the heating curve but dropped below 0.87 to return to the same level as that measured at the start of the current test.

   After testing the coating was black, and was now hard with a good coating-substrate bond strength. No change in the texture occurred. Further testing of this and other specimens is required before final conclusions may be drawn.

TABLE IX

Coating: Iron - Titanium - Aluminum - Oxide - Plasma Arc Sprayed

Substrate: Columbium - 1% Zirconium

5.0 - Mil Coating

Run No.	Elapsed Time (hrs)	Pressure (mm Hg)	Thermocouple Temperature (°F)	ϵ_{TH}	Optical Pyrometer Temperature (°F)	ϵ_{TH}
1	1.0	4.5×10^{-7}	999	.815		
	1.1	3.3×10^{-7}	1100	.813		
	1.5	2.8×10^{-7}	1196	.826		
	1.8	2.8×10^{-7}	1302	.817		
	2.0	3.4×10^{-7}	1402	.820		
	2.3	3.1×10^{-7}	1497	.819	1508	.817
	2.8	3.1×10^{-7}			1615	.854
	3.0	3.6×10^{-7}			1726	.858
	3.2	4.3×10^{-7}			1821	.873
	3.4	6.4×10^{-7}			1925	.873
	3.6	8.6×10^{-7}			2020	.888
	3.9	8.1×10^{-7}			2134	.867
	4.0	1.4×10^{-6}			2228	.885
	4.2	1.3×10^{-6}			2196	.844
	4.3	3.6×10^{-7}			1887	.789
	4.4	3.6×10^{-7}			1574	.807

TOTAL HEMISPHERICAL EMITTANCE vs. TEMPERATURE

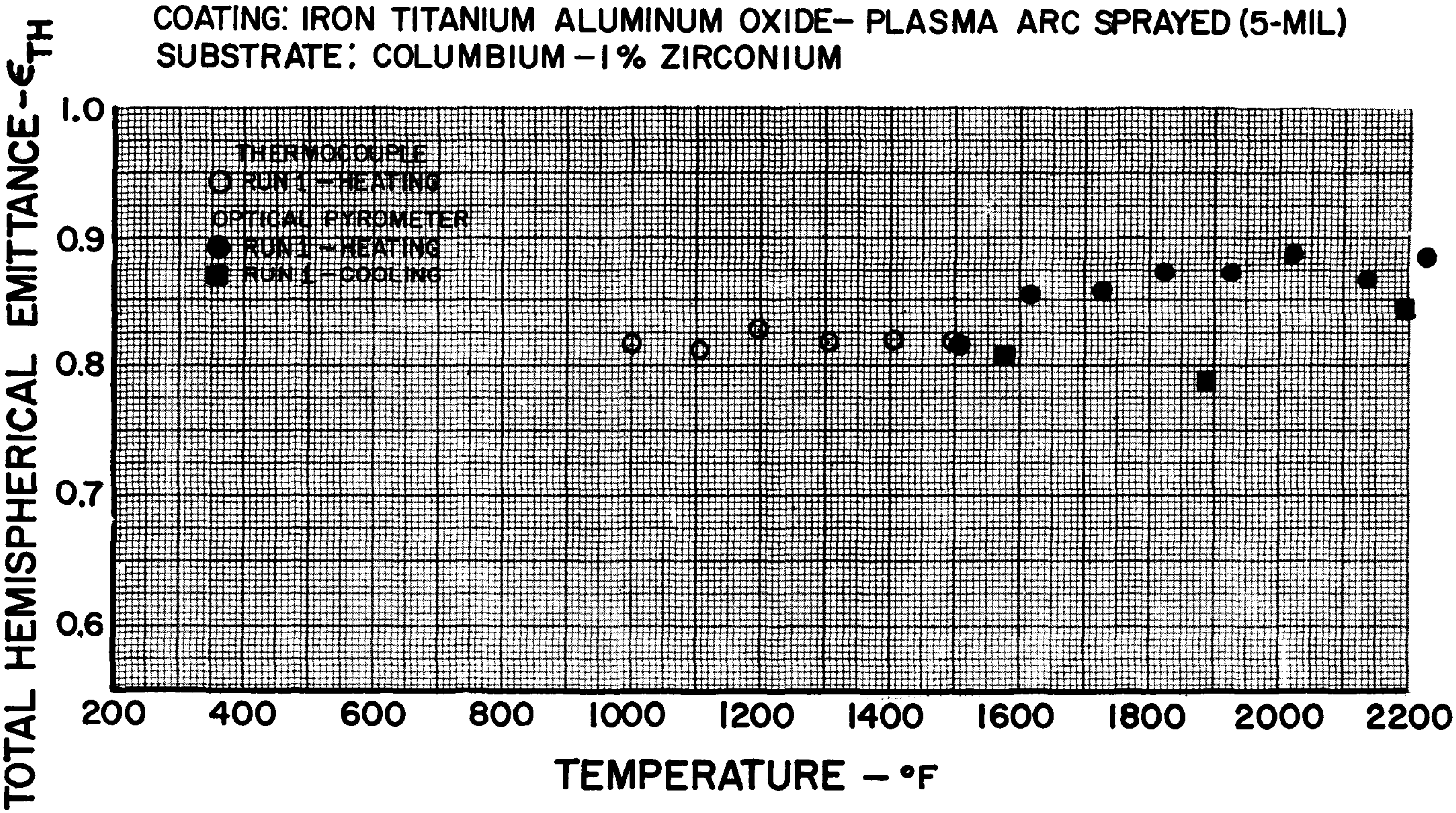

Figure 17

Above 1500°F the thermocouple data are considered unreliable for this particular test and therefore the only results reported are those based on optical pyrometer temperature measurements.

Spectrographic analysis of the powder before coating indicated that Fe, Ti and Al were the major constituents while Mn was one of the minor constituents. After testing Fe, Ti, Al and Mn were found to be the major constituents present. No analysis of this type was run on the specimen after coating but before testing, therefore it is not known at this time when the Mn changed from a minor to a major constituent. X-ray diffraction analysis indicates that before testing the coating consisted of Fe_2TiO_5. There is a possibility that $\eta\,Al_2O_3$ was also present since lines for this phase coincide with those of Fe_2TiO_5. After testing, two phases were definitely present, namely Fe_2TiO_5 and αAl_2O_3. Since Al was found by spectrographic analysis before as well as after testing and since Al_2O_3 was found by x-ray diffraction after testing, it appears probable that $\eta\,Al_2O_3$ was present before testing but could not be detected because of the Fe_2TiO_5.

2. Endurance Test - The 4.0-mil thick plasma-arc sprayed coating was medium black, fairly hard, and had a matte texture similar to that of 80 grit emery cloth. The coating-substrate bond strength was fair. Table X and Figure 18 show the emittance-vs-temperature curve as the specimen was heated from 300°F to 1450°F. The curve indicates that the emittance of this coating is about 0.86 and it varies little with temperature. The results of the endurance test are shown in Table X and in Figure 19. The emittance remained at about 0.87 throughout the 300 hours. The coating did not change as a result of testing.

3. General Remarks - Figures 33 and 34 in PWA-2128 and Figures 17, 18, and 19 in this report indicate that the emittance of iron-titanium-aluminum oxide is reasonably constant over the temperature range 300°F to 2200°F. The most probable values between these temperatures were 0.85 and 0.88. Exposure to high temperatures results in little change aside from the darkening of the coating at temperatures above 1450°F.

TABLE X
Coating: Iron - Titanium - Aluminum - Oxide - Plasma Arc Sprayed
Substrate: Columbium - 1% Zirconium
4-Mil Coating

Run No.	Elapsed Time (hrs)	Pressure (mm Hg)	Thermocouple Temp. (°F)	Thermocouple ϵ_{TH}
1	0.6	3.0×10^{-8}	300	.897
	0.8	1.8×10^{-7}	402	.895
	1.0	1.8×10^{-6}	501	.882
	1.1	3.2×10^{-6}	601	.872
	1.3	2.4×10^{-6}	700	.873
	1.4	4.6×10^{-6}	800	.863
	2.0	1.6×10^{-6}	902	.866
	2.1	3.9×10^{-6}	998	.865
	2.3	3.8×10^{-6}	1097	.853
	2.5	5.1×10^{-6}	1199	.853
	2.6	8.0×10^{-6}	1298	.847
	3.0	9.0×10^{-6}	1400	.845
	3.4	8.0×10^{-6}	1450	.848
		Heating Current Off; Vacuum Maintained		
2	21.0	9.0×10^{-9}	600	.872
	21.3	2.6×10^{-8}	899	.861
	21.6	6.1×10^{-8}	1198	.861
	22.0	1.8×10^{-7}	1401	.859

Endurance Time (hrs)	Pressure (mm Hg)	Thermocouple Temp. (°F)	Thermocouple ϵ_{TH}	Thermocouple Avg. ϵ_{TH}	Optical Pyrometer Temp. (°F)	Optical Pyrometer ϵ_{TH}	Optical Pyrometer Avg. ϵ_{TH}
0.0	3.3×10^{-7}	1449	.867		1449	.867	
3.0	2.2×10^{-7}	1448	.877	.873	1450	.873	.870
5.2	1.7×10^{-7}	1448	.875		1450	.871	
22.3	9.1×10^{-8}	1452	.869		1450	.872	
26.2	8.1×10^{-8}	1453	.865	.867	1450	.871	.871
29.2	6.6×10^{-8}	1450	.866		1448	.870	
45.9	5.0×10^{-8}	1453	.865		1453	.865	
50.1	5.1×10^{-8}	1460	.851	.857	1450	.871	.867
53.0	5.1×10^{-8}	1457	.854		1450	.867	
70.3	4.0×10^{-8}	1455	.856	.855	1448	.869	.866
77.1	4.2×10^{-8}	1454	.853		1448	.864	
143.4	2.4×10^{-8}	1449	.863	.863	1448	.865	.865
148.0	3.0×10^{-8}	1449	.863		1448	.865	
166.0	2.7×10^{-8}	1448	.875	.871	1448	.875	.871
172.5	2.7×10^{-8}	1448	.867		1448	.867	
190.4	2.7×10^{-8}	1449	.870	.867	1449	.870	.869
196.0	2.9×10^{-8}	1450	.865		1448	.868	
214.5	2.7×10^{-8}	1449	.871	.867	1449	.871	.871
220.8	2.7×10^{-8}	1450	.864		1449	.871	
237.4	2.6×10^{-8}	1450	.868		1449	.870	
312.2	2.1×10^{-8}	1449	.876		1449	.876	

TOTAL HEMISPHERICAL EMITTANCE vs. TEMPERATURE

COATING: IRON TITANIUM ALUMINUM OXIDE—PLASMA ARC SPRAYED (4-MIL)
SUBSTRATE: COLUMBIUM—1% ZIRCONIUM

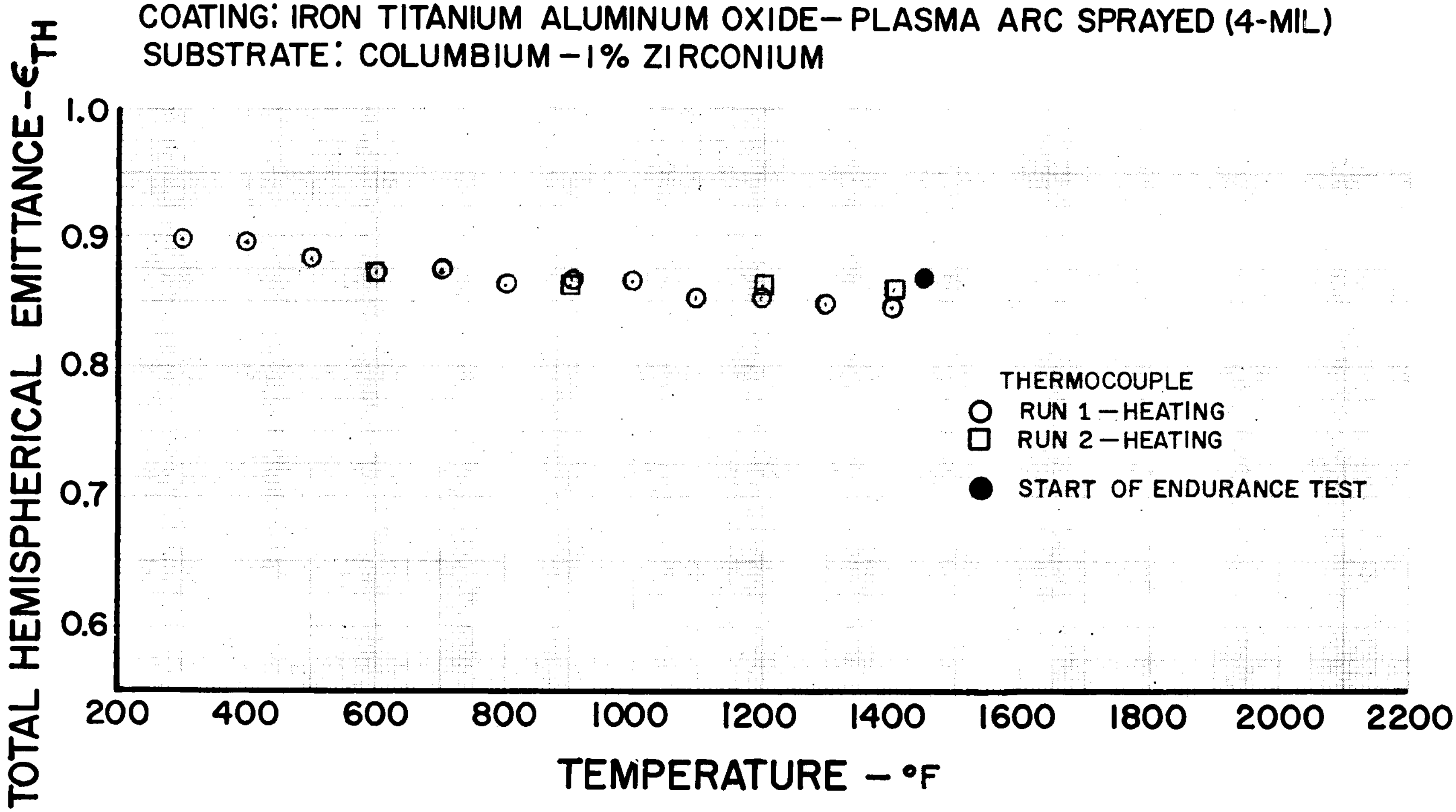

Figure 18

PWA-2163

TOTAL HEMISPHERICAL EMITTANCE vs. TIME

COATING: IRON TITANIUM ALUMINUM OXIDE—PLASMA ARC SPRAYED (4-MIL)
SUBSTRATE: COLUMBIUM—1% ZIRCONIUM

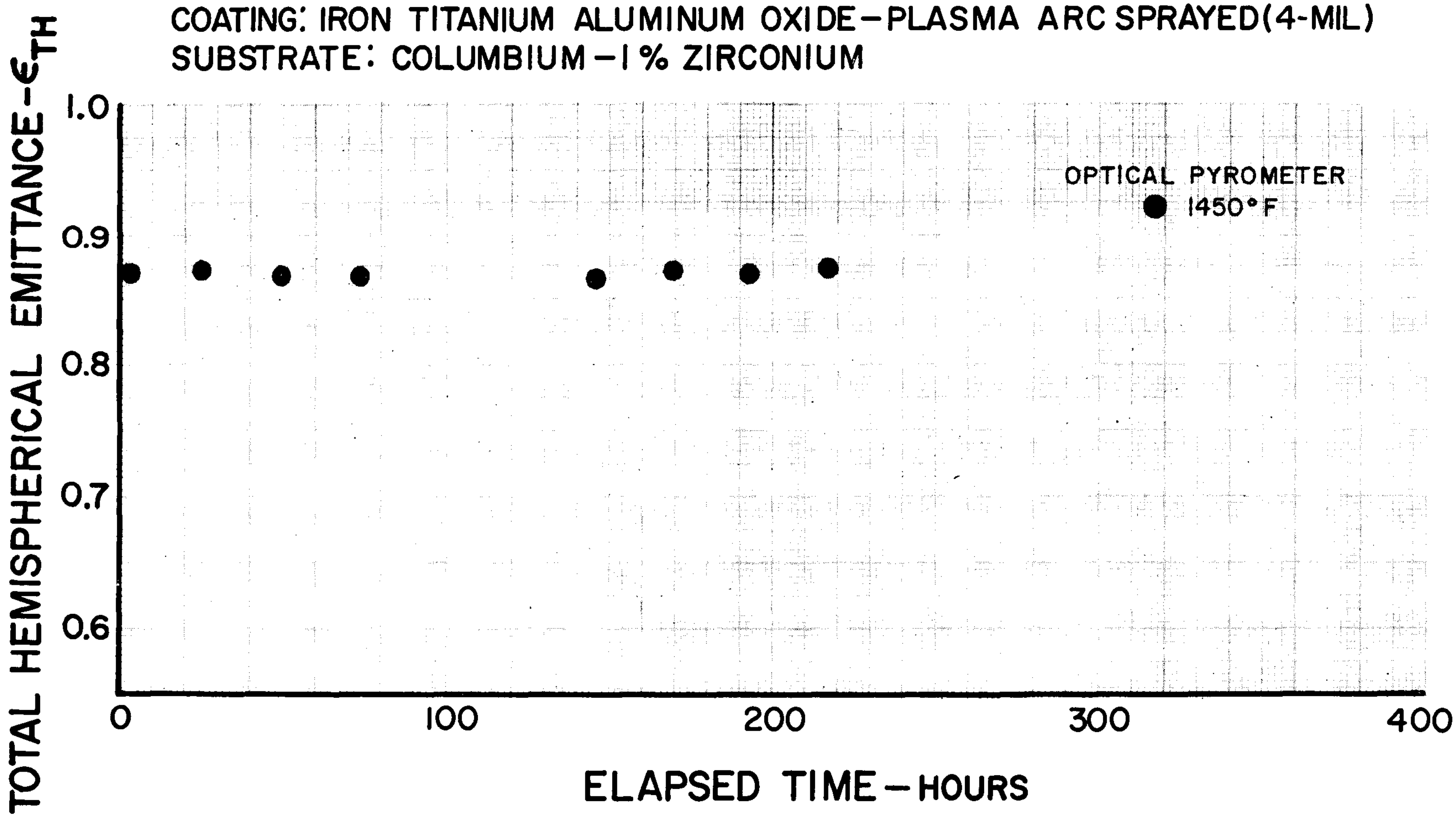

Figure 19

J. Oxidized Kennametal K-151-A

The powder used in this coating was obtained from Kennametal, Inc. and supplied as having particles between 53 and 88 microns in diameter. The powder was oxidized by the supplier for 20 minutes at 1600°F so data could be obtained to compare with work conducted by Wade and Casey* on oxidized sheets of this material. A 4-mil thick coating was plasma-arc sprayed on an AISI-310 stainless steel tube. The coating was dark grey, fairly hard, and had a fair coating-substrate bond strength. Its matte texture was similar to that of 40 grit emery cloth. The total hemispherical emittance of the coating was measured from 700°F to 1600°F in the spectral normal emittance rig. On each successive run the emittance of the coating decreased as may be seen in Table XI and in Figure 20. Between heating run 1 and cooling run 3 the emittance at 700°F decreased from 0.85 to 0.82, but the differences were less at higher temperatures. To determine if any further changes will occur an endurance test should be conducted. The visual characteristics of the coating did not change during testing.

Comparison of the data reported by Wade and Casey with that reported here shows good agreement when the different methods of specimen preparation and test conditions are considered. Wade and Casey prepared their specimen by oxidizing flat sheets of Kennametal rather than by plasma-arc spraying oxidized powder onto a substrate. Further, Wade and Casey measured emittance in air while Pratt & Whitney Aircraft measurements have been in vacuum.

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*W. Wade and F. Casey, "Measurement of Total Hemispherical Emissivity of Several Stably Oxidized Nickel-Titanium Carbide Cemented Hard Metals From 600°F to 1600°F," NASA Memo 5-13-59L, Langley Research Center, Langley Field, Virginia, June 1959.

TABLE XI

Coating: Oxidized Kennametal (K-151A) - Plasma Arc Sprayed
Substrate: AISI-310 Stainless Steel
4-Mil Coating

Run Number	Elapsed Time (Hrs)	Pressure (mm Hg)	Thermocouple Temp (°F)	Thermocouple ϵ_{TH}	Optical Pyrometer Temp (°F)	Optical Pyrometer ϵ_{TH}
1	0.0	7×10^{-7}	700	.852		
	0.3	5.9×10^{-7}	900	.850		
	0.6	6.6×10^{-7}	1000	.854		
	1.0	6.2×10^{-7}	1100	.854		
	1.3	4.2×10^{-7}	1200	.857		

Heating Current Off; Vacuum Maintained

Run Number	Elapsed Time (Hrs)	Pressure (mm Hg)	Thermocouple Temp (°F)	Thermocouple ϵ_{TH}	Optical Pyrometer Temp (°F)	Optical Pyrometer ϵ_{TH}
2	20.3	9.6×10^{-8}	900	.842		
3	21.2	7.2×10^{-8}	700	.834		
	21.4	7.1×10^{-8}	800	.838		
	21.9	7.3×10^{-8}	900	.843		
	22.0	7.5×10^{-8}	1000	.847		
	22.2	8.5×10^{-8}	1100	.852		
	22.4	1.9×10^{-7}	1200	.855		
	22.8	2.7×10^{-7}	1300	.859		
	23.0	6×10^{-7}	1400	.862		
	23.5	1.4×10^{-6}	1500	.864	1519	.831
	23.9	5.2×10^{-6}	1600	.866	1617	.838
	24.2	5.3×10^{-6}	1600	.866	1619	.834
	24.5	9.8×10^{-7}	1500	.862	1515	.835
	24.7	1.1×10^{-7}	1350	.856		
	25.0	5.9×10^{-8}	1250	.851		
	25.3	4.8×10^{-8}	1150	.847		
	25.4	4.6×10^{-8}	1050	.840		
	25.6	4.4×10^{-8}	950	.837		
	25.8	4.1×10^{-8}	850	.832		
	26.0	4.0×10^{-8}	750	.826		
	26.2	4.0×10^{-8}	650	.818		

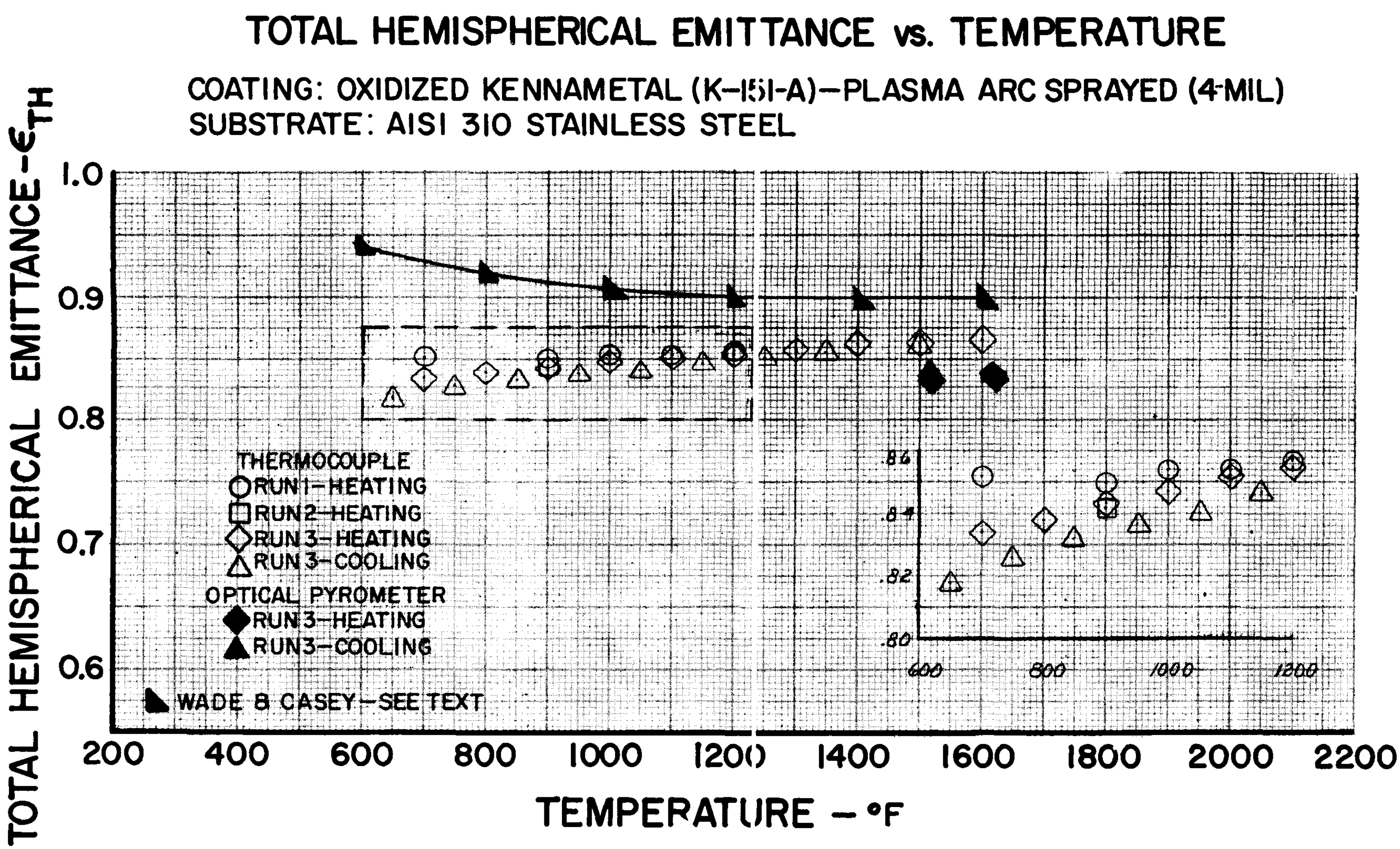

Figure 20
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K. Titania

The titania (TiO_2) used for this series of tests came from two sources. A commercial grade was obtained from Metco, Inc. (XP-1114), and a high purity grade was obtained from E. I. Dupont de Nemours & Company, Inc. (Titania - Pure Rutile (R-510-P). Particle size analysis of the Metco powder indicated that 16 per cent of the particles had diameters of 59 microns or larger. An additional 29 per cent had diameters about 47 microns in diameter. Seventy-nine per cent had diameters of 19 microns or larger, and 12 per cent had diameters between 12 and 19 microns. All of the specimens were prepared by aluminum phosphate bonding the powder to columbium - 1 per cent zirconium tubes. The two coatings using the high purity titania were applied at the same time. One of these was tested in the total hemispherical emittance rig and the other in the spectral normal emittance rig. The Metco coating was applied at another time and was tested in the total hemispherical emittance rig.

1. Total Hemispherical Emittance Test

a. Dupont Coating - The Dupont coating was 1 mil thick, white, fairly hard, and brittle with a matte texture similar to that of 320 grit emery cloth although there were small lumps distributed throughout the coating. The coating-substrate bond strength was fair. Since this was a white coating and its anticipated usefulness as a spacecraft radiator coating would be at temperatures less than 1000°F, thermal emittance values were obtained only between 300°F and 1000°F. Table XII and Figure 21 show the emittance level of this coated tube. The emittance was about 0.82 over the temperature range. The electric current values obtained for the 700°F point appears to be in error and casts doubt on the validity of the corresponding emittance value.

It is believed that the 1-mil thick coating was thick enough so as not to be transparent to the longer wavelengths which constitute the major part of low temperature black body energy. The emittance values were found to be similar

TABLE XII

Coating: Titania - Aluminum Phosphate Bonded
Substrate: Columbium - 1% Zirconium
1.0-Mil Coating

Run No.	Elapsed Time (Hrs.)	Pressure (mm Hg)	Thermocouple Temp. (°F)	ϵ_{TH}
1	1.8	2.4×10^{-6}	299	.806
	2.3	1.4×10^{-5}	500	.833
	2.6	2.1×10^{-5}	699	.882
	2.9	4.5×10^{-5}	902	.826
	3.1	6.3×10^{-5}	1000	.817
	3.3	5.5×10^{-5}	799	.820
	3.6	4.9×10^{-5}	598	.805

TOTAL HEMISPHERICAL EMITTANCE vs. TEMPERATURE

COATING: TITANIA—ALUMINUM PHOSPHATE BONDED (1-MIL)
SUBSTRATE: COLUMBIUM-1% ZIRCONIUM

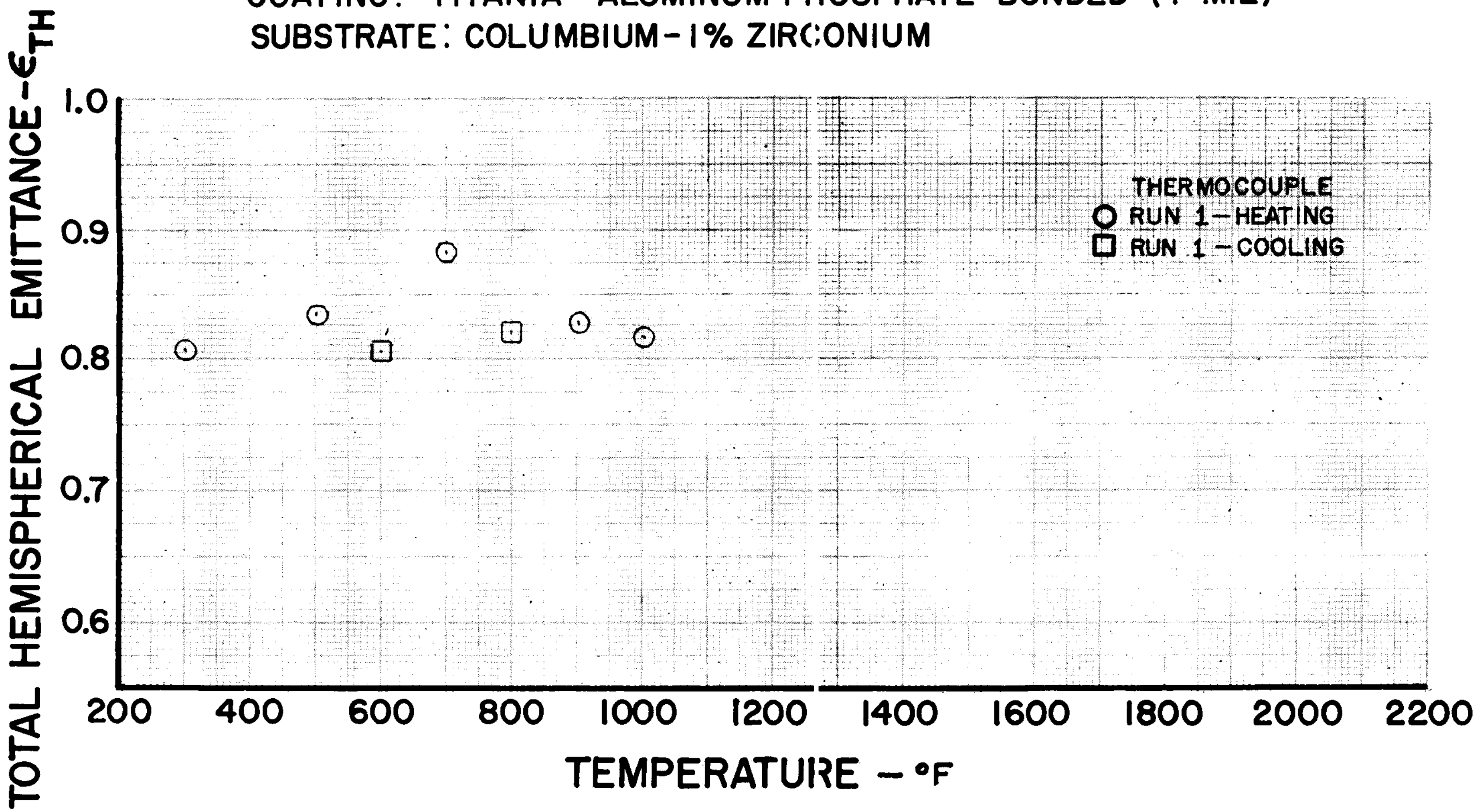


Figure 21

to those of previously tested plasma-arc sprayed titania although the plasma-arc sprayed coating was dark rather than white. Had the coating been partially transparent, emittance from the substrate would have occurred and, unless the substrate had oxidized, would have resulted in somewhat lower emittance values. X-ray diffraction, however, confirmed that TiO_2 was the only oxide present and it may therefore be concluded that the coating had adequate thickness. After testing the coating was greyish, and there were dark areas around the thermocouples which possibly resulted from a loss of the coating in this region when the thermocouples were tack welded. X-ray diffraction analysis before and after testing indicated that rutile TiO_2 was the only detectable phase present. Spectrographic analysis indicated that Ti was the only principle element present after testing. No other spectrographic data is available at the present time.

b. Metco Coating - The Metco coating was 5 mils thick, white, soft, and had a matte texture similar to that of 80 grit emery cloth. The coating-substrate bond strength was fair. It was also observed that the area around the black body holes was a shade lighter than the rest of the specimen which may be attributed to a greater coating thickness in that region. This white specimen was also tested between 300°F and 1000°F.

As shown in Table XIII and in Figure 22 the total hemispherical emittance curve is typical for a white coating. Total hemispherical emittance decreases with increasing temperature. The total emittance decreased from about 0.77 at 300°F to about 0.64 at 1000°F during heating and essentially the same values were obtained during cooling. When the specimen was removed from the rig it was noticed that an area about one-half inch in diameter around the black body holes had changed to a light grey color while the rest of the coating was still white. The coating was slightly harder after testing and still had a fair coating-substrate bond strength. The texture was unchanged. X-ray diffraction analysis before and after testing indicated rutile TiO_2 to be the only detectable phase present. Ti was found to be the only element present in large quantities both before coating and after testing.

TABLE XIII

Coating: Titania - Aluminum Phosphate Bonded
Substrate: Columbium - 1% Zirconium
5.0-Mil Coating

Run No.	Elapsed Time (Hrs)	Pressure (mm Hg)	Thermocouple Temp. (°F)	ϵ_{TH}
1	2.1	3.4×10^{-6}	300	.766
	2.6	3.5×10^{-6}	499	.750
	3.1	4.2×10^{-6}	697	.708
	4.1	2.0×10^{-6}	901	.653
	4.2	3.2×10^{-6}	1002	.636
	4.3	3.1×10^{-6}	701	.717
	4.8	4.3×10^{-6}	499	.755

TOTAL HEMISPHERICAL EMITTANCE vs. TEMPERATURE

COATING: TITANIA—ALUMINUM PHOSPHATE BONDED (5-MIL)
SUBSTRATE: COLUMBIUM-1% ZIRCONIUM

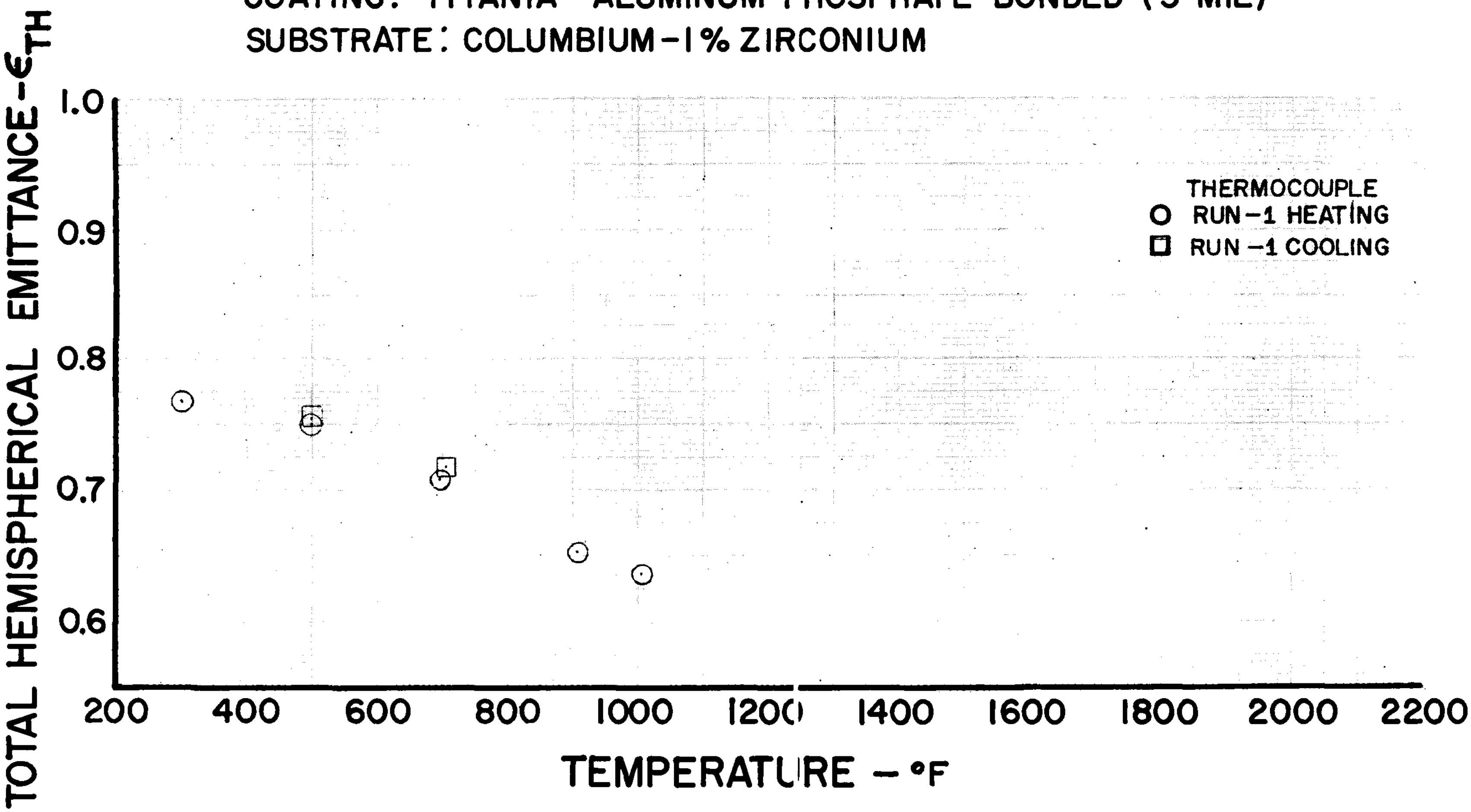


Figure 22

2. Spectral Normal Emittance Test

One of the specimens prepared using the Dupont titania was tested in the spectral emittance rig. This coating, like the one reported in the total hemispherical emittance section, was 1 mil thick, white, fairly hard, but brittle and had a matte texture similar to that of 320 grit emery cloth. The coating-substrate bond strength was fair. Spectral normal emittance was measured over the wavelength range of 1.45 to 12.4 microns at 900°F, and from 1.01 to 13.6 microns at 1450°F. The specimen was held at 1450°F for 17.5 hours. After this interval the spectral normal emittances were measured a second time at 1450°F and at 900°F. As can be seen in Figure 23, the first emittance curve for 900°F was lower than that for 1450° at wavelengths shorter than 7.5 microns. The second curve for 900°F however matches the higher temperature curves. This can probably be accounted for by the change in color of the coating, since it was white before testing and grey after testing. Note that at all temperatures there is a dip in the curve between 8.5 and 11.5 microns. There was no change in the coating-substrate bond strength, coating hardness, or texture as a result of testing.

SPECTRAL NORMAL EMITTANCE vs. WAVELENGTH

COATING: TITANIA-ALUMINUM PHOSPHATE BONDED (1-MIL)
SUBSTRATE: COLUMBIUM-1% ZIRCONIUM

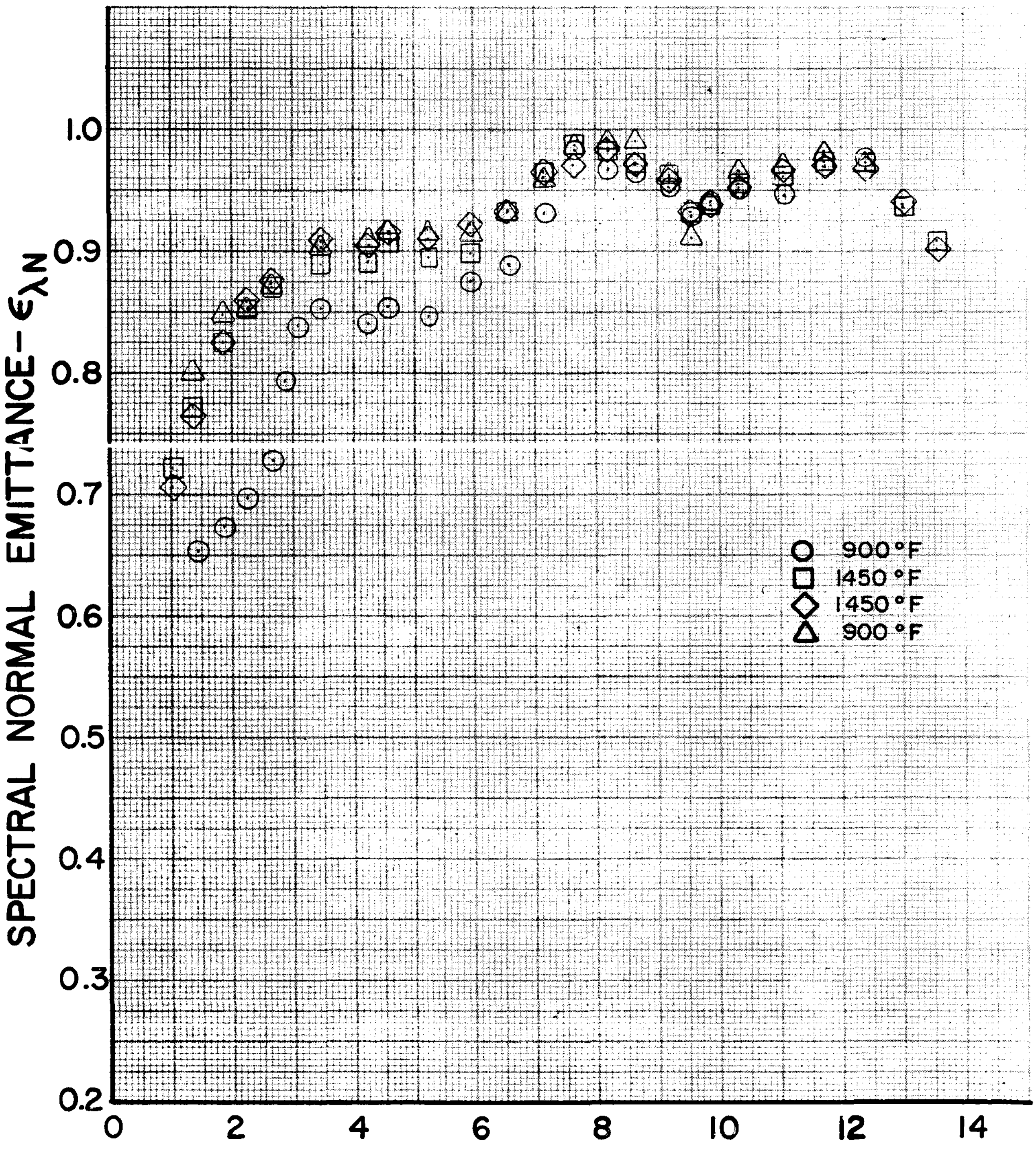


Figure 23

L. Calcium Titanate

Specimens were prepared by plasma-arc spraying high purity calcium titanate ($CaO \cdot TiO_2$) powder, obtained from the Titanium Division of the National Lead Company, onto columbium - 1 per cent zirconium tubes.

1. Total Hemispherical Emittance Tests

a. Short Term Endurance Rig - The total hemispherical emittance of the specimen had originally been measured in the total hemispherical emittance rig. These measurements may be in error, however, as a result of difficulties arising from the volatilization of the Mn_2O_3 specimen described in section II-0 of this report. Results from this test will not be reported until corrective measures have been completed.

The coating, before the first test, was 5 mils thick with white crystals on a blue background. It was hard and the coating-substrate bond strength was good. The matte texture was about the same as that of 40 grit emery cloth. After the first test white crystals were still present, but the background was now grey. No other changes in the characteristics of the coating were observed.

Since the test was originally conducted only to compare the total hemispherical emittance rig with the short term endurance rig, and since changes in the coating had already taken place during initial testing, measurements were taken only during heating to 1500°F and only above 800°F. As shown in Table XIV and in Figure 24, the emittance level remained constant at about 0.90 during heating. This test run was terminated at 1500°F and the specimen was cooled to room temperature with the vacuum maintained. The next day the specimen was reheated to 1500°F and then cooled to 1050°F and the emittance values obtained duplicated those taken the previous day. After testing the coating was uniformly grey.

TABLE XIV

Coating: Calcium Titanate - Plasma Arc Sprayed
Substrate: Columbium - 1% Zirconium
5.0-Mil Coating

Run No.	Elapsed Time (Hrs)	Pressure (mm Hg)	Thermocouple Temp. (°F)	Thermocouple ϵ_{TH}	Optical Pyrometer Temp. (°F)	Optical Pyrometer ϵ_{TH}
1	0.0	5.1×10^{-6}	799	.912		
	0.3	2.7×10^{-6}	901	.907		
	0.5	1.6×10^{-6}	997	.905		
	0.8	1.8×10^{-6}	1098	.904		
	1.0	3.1×10^{-6}	1200	.888		
	1.2	5.6×10^{-6}	1300	.898		
	1.5	6.3×10^{-6}	1400	.885		
	1.8	5.7×10^{-6}	1500	.898	1498	.902
2	2.5	4.7×10^{-8}	801	.916		
	2.7	7.4×10^{-8}	1001	.908		
	3.0	1.6×10^{-7}	1200	.895		
	3.3	1.0×10^{-6}	1400	.889		
	3.7		1500	.898	1508	.883
	4.7	1.3×10^{-7}	1250	.910		
	5.0	5.6×10^{-8}	1049	.894		

TOTAL HEMISPHERICAL EMITTANCE vs. TEMPERATURE

COATING: CALCIUM TITANATE—PLASMA ARC SPRAYED (5-MIL)
SUBSTRATE: COLUMBIUM-1% ZIRCONIUM

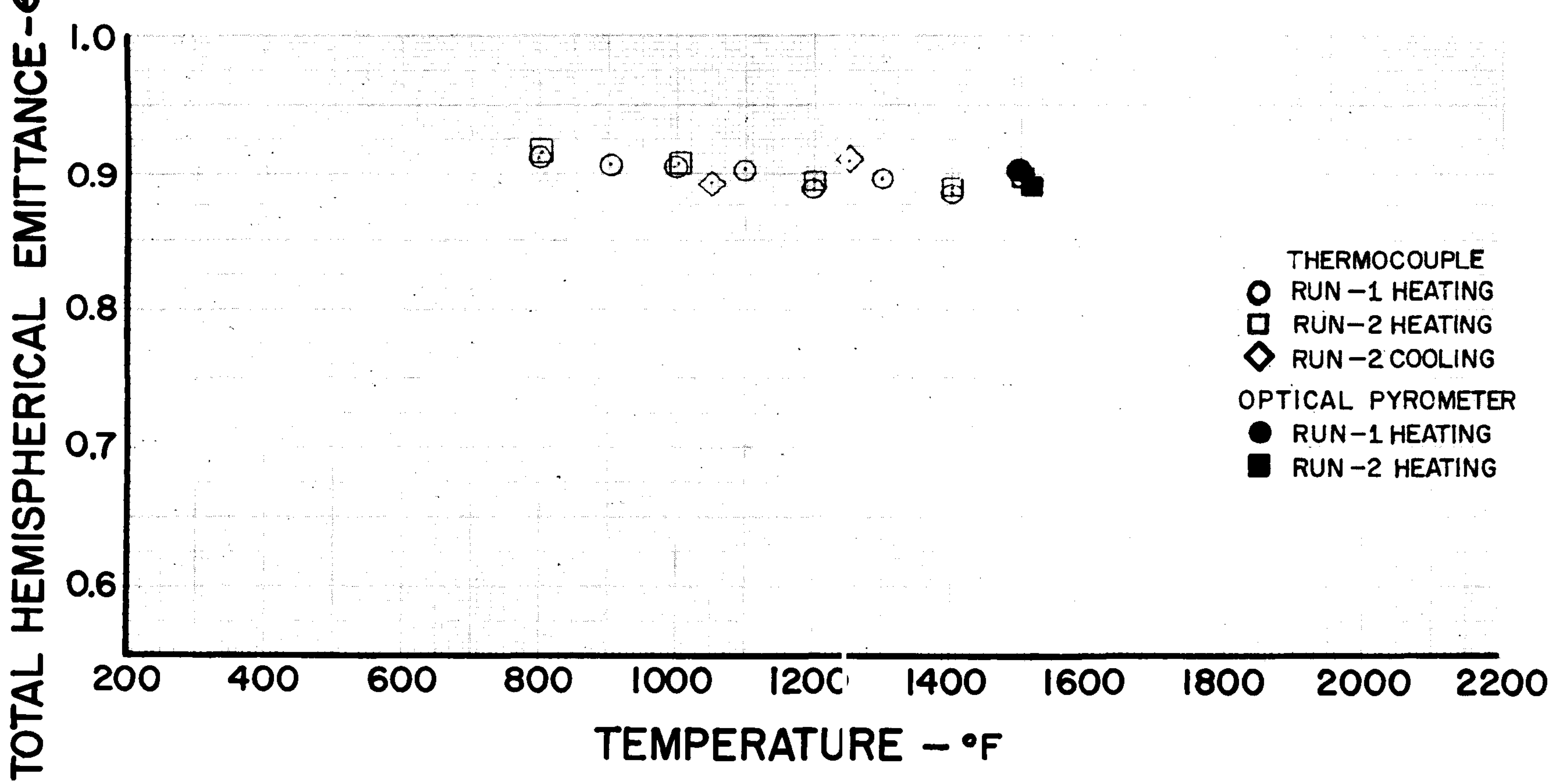


Figure 24

b. Spectral Normal Emittance Rig - This 5-mil thick coating had both white and blue crystals present on a light grey background. It was hard and had a matte texture similar to that of 80 grit emery cloth. The coating-substrate bond strength was good. Table XV and Figure 25 show the total hemispherical emittance values of this coating on two test runs between 800 and 1800°F. The emittance increased from about 0.82 at 800°F to about 0.90 at 1200°F where it remained on subsequent thermal cycling. However, when heated above 1600°F the emittance decreased and then during cooling remained at the lower level. This same phenomenon was also shown in Figure 26 in PWA-2128. After the specimen was removed from the rig the only change in the coating was that the color was now all dark grey.

2. General Remarks

The curves for the plasma-arc sprayed calcium titanate powder obtained from National Lead indicate that the emittance decreases during the first heating cycle from 300°F to 1000°F. At about 1000°F the emittance begins increasing and reaches a maximum at about 1400°F. This higher emittance level is maintained between 800°F and 1600°F on subsequent cycling as long as the coating does not go above 1600°F. No data has been obtained at less than 800°F after the 1400°F temperature has been reached. Above about 1600°F the emittance starts to decrease and during cooling will not return to the higher emittance level.

Further work is required on this coating to determine what is causing this increase and to see if it can be obtained without heating to 1400°F. Also, endurance tests should be run to determine whether or not the emittance changes with time and to determine if the emittance will rise to the higher emittance level if exposed to temperatures less than 1400°F. (See Figure 13, PWA-2012).

TABLE XV

Coating: Calcium Titanate - Plasma Arc Sprayed
Substrate: Columbium - 1% Zirconium
5.0-Mil Coating

Run No.	Elapsed Time (Hrs)	Pressure (mm Hg)	Thermocouple Temp (°F)	Thermocouple ϵ_{TH}	Optical Pyrometer Temp (°F)	Optical Pyrometer ϵ_{TH}
1	41.99	2.8×10^{-7}	800	.818		
	43.26	4.2×10^{-7}	1000	.833		
	44.12	7.6×10^{-7}	1200	.894		
	44.50	1.0×10^{-6}	1400	.912		
	44.64	7.0×10^{-7}	1600	.903	1611	.883
	44.84	4.0×10^{-8}	1250	.898		
	45.37	2.8×10^{-8}	850	.906		
2	45.55	3.3×10^{-8}	1200	.899		
	45.70	6.6×10^{-8}	1500	.896	1509	.879
	45.86	1.3×10^{-7}	1600	.894	1611	.874
	46.10	1.8×10^{-7}	1700	.883	1710	.867
	46.35	2.1×10^{-7}	1800	.870	1813	.850
	46.55	2.0×10^{-8}	1500	.862	1510	.844
	47.09	1.2×10^{-8}	1200	.862		
	47.83	8.0×10^{-9}	900	.865		

TOTAL HEMISPHERICAL EMITTANCE vs. TEMPERATURE

COATING: CALCIUM TITANATE—PLASMA ARC SPRAYED (5-MIL)
SUBSTRATE: COLUMBIUM-1% ZIRCONIUM

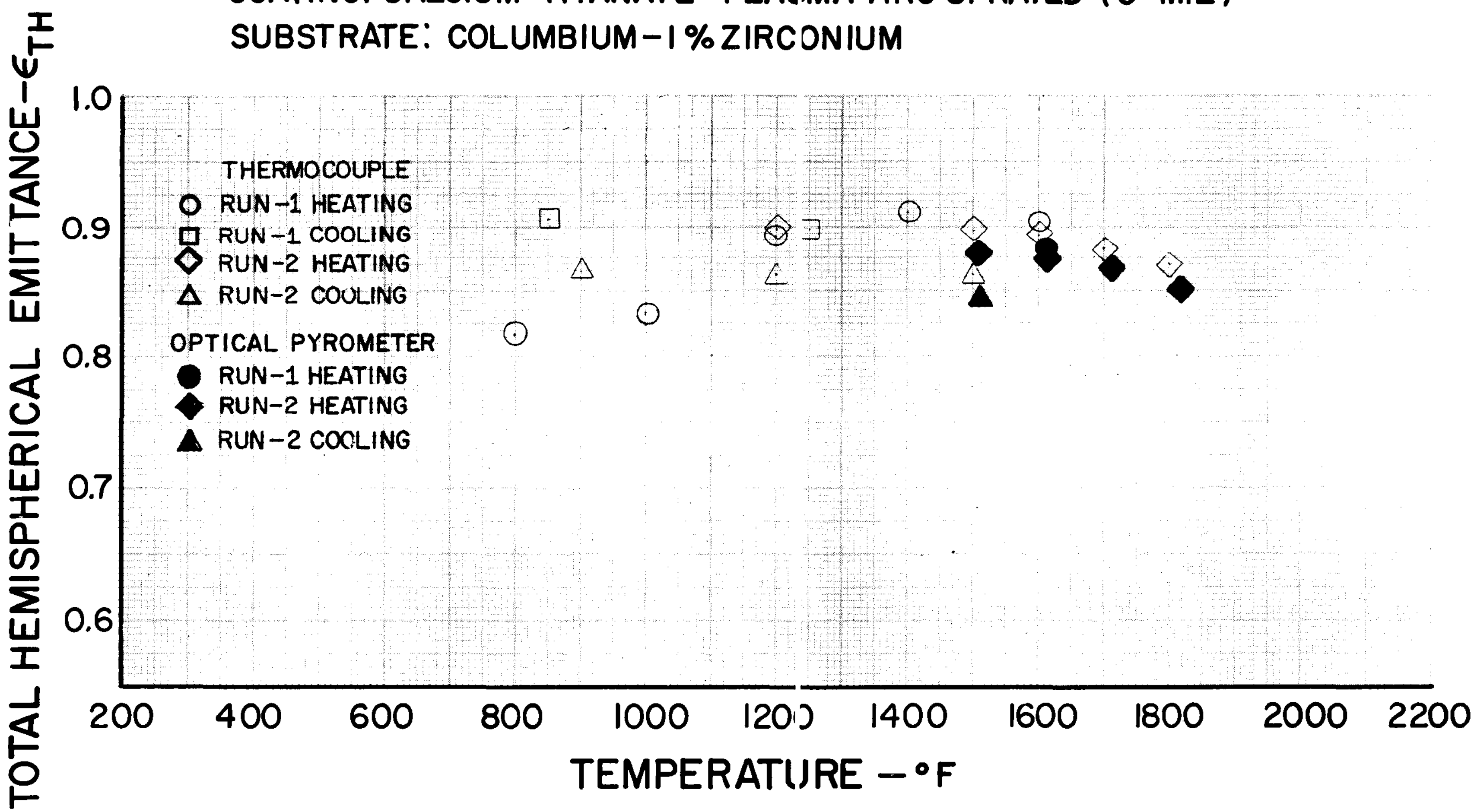


Figure 25

M. Strontium Titanate

1. Recent Measurements - A commercial grade of strontium titanate powder ($SrO \cdot TiO_2$) was obtained from the Plasmadyne Corporation. A particle size analysis of this powder indicated that 87 per cent of the particles had diameter sizes fairly evenly distributed between 10 and 22 microns. Half of the remaining material was larger than 22 microns, and half smaller than 10 microns in diameter. The material was plasma-arc sprayed onto columbium - 1 per cent zirconium tubes. Three specimens were made at the same time and the data obtained from two of these has been reported in PWA-2128, Pages 69 to 73.

   The remaining specimen had a coating 4 mils thick, grey-white, hard, and had a matte texture similar to that of 320 grit emery cloth. The coating-substrate bond strength was good. Short term endurance testing, measuring total hemispherical emittance, was conducted in the spectral normal emittance rig. Total hemispherical emittance values obtained during the heating of the specimen from 300°F to 1450°F appear in Table XVI and in Figure 26. The curve based on thermocouple temperatures in Figure 26 agrees very well with that of the total hemispherical emittance specimen reported in PWA-2128, Figure 31.

   As shown in Table XVII and in Figure 27 the total hemispherical emittance remained at about 0.87 throughout the endurance test. Since the optical pyrometer and window are periodically checked using an optical pyrometer calibrated by the National Bureau of Standards, the emittance values based on optical pyrometer readings are considered the more accurate and therefore these values are the only ones plotted in Figure 27. After testing the specimen was grey-black but no other change was observed.

2. General Remarks - All the data obtained from plasma-arc sprayed strontium titanate this year have resulted in curves which have the same shape as those for calcium titanate specimens. The

TABLE XVI

Coating: Strontium Titanate - Plasma Arc Sprayed
Substrate: Columbium - 1% Zirconium
4.0-Mil Coating

Run Number	Elapsed Time (Hrs.)	Pressure (mm Hg)	Thermocouple Temp (°F)	ϵ_{TH}
1	20.57	1.6×10^{-6}	904	.752
	20.73	2.2×10^{-6}	995	.755
	20.88	2.9×10^{-6}	1080	.774
	21.11	2.9×10^{-6}	1201	.813
	21.29	1.5×10^{-6}	1296	.856
	21.77	4.1×10^{-7}	1396	.887

TOTAL HEMISPHERICAL EMITTANCE vs. TEMPERATURE

COATING: STRONTIUM TITANATE—PLASMA ARC SPRAYED (4-MIL)
SUBSTRATE: COLUMBIUM—1% ZIRCONIUM

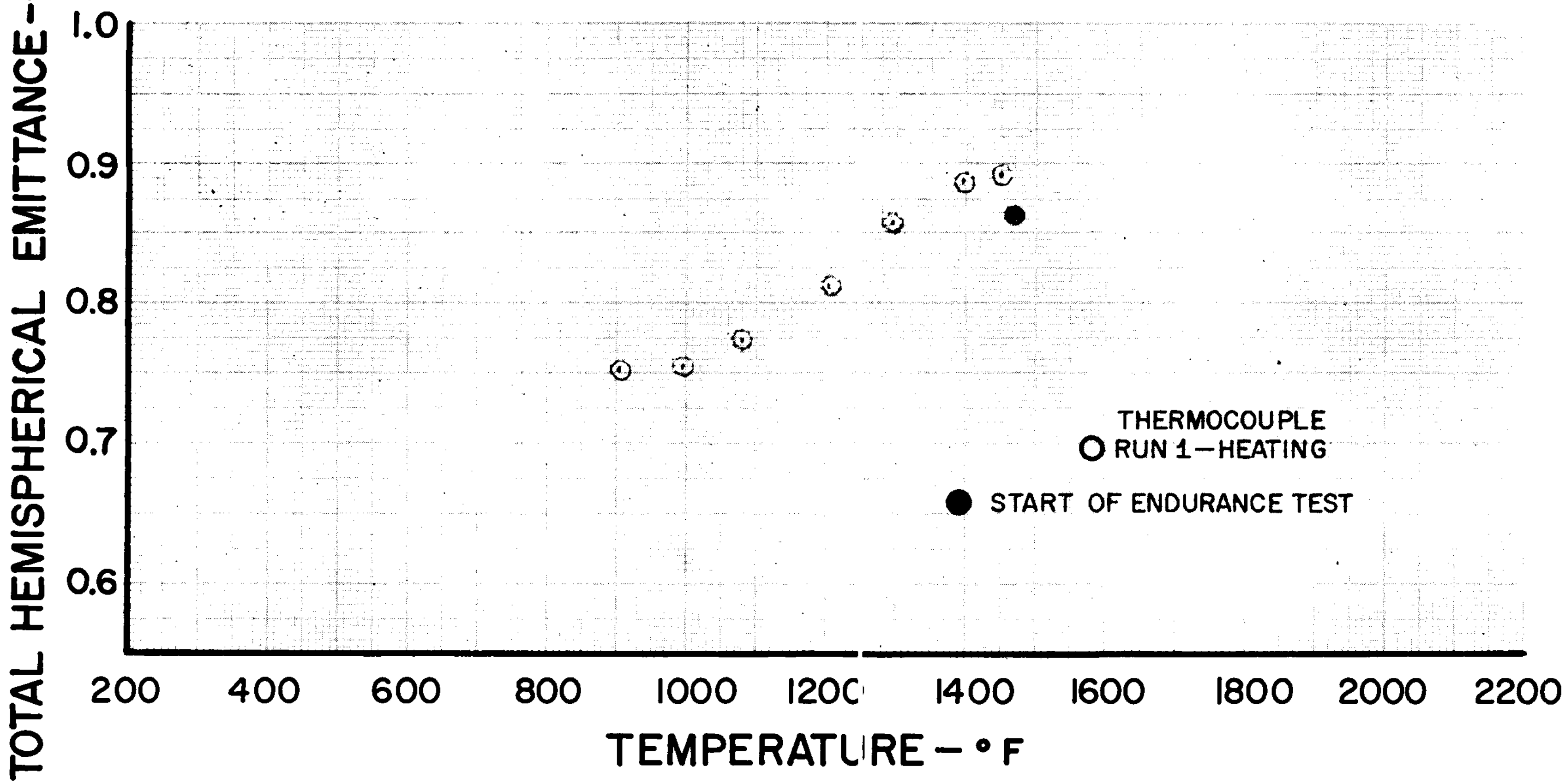


Figure 26

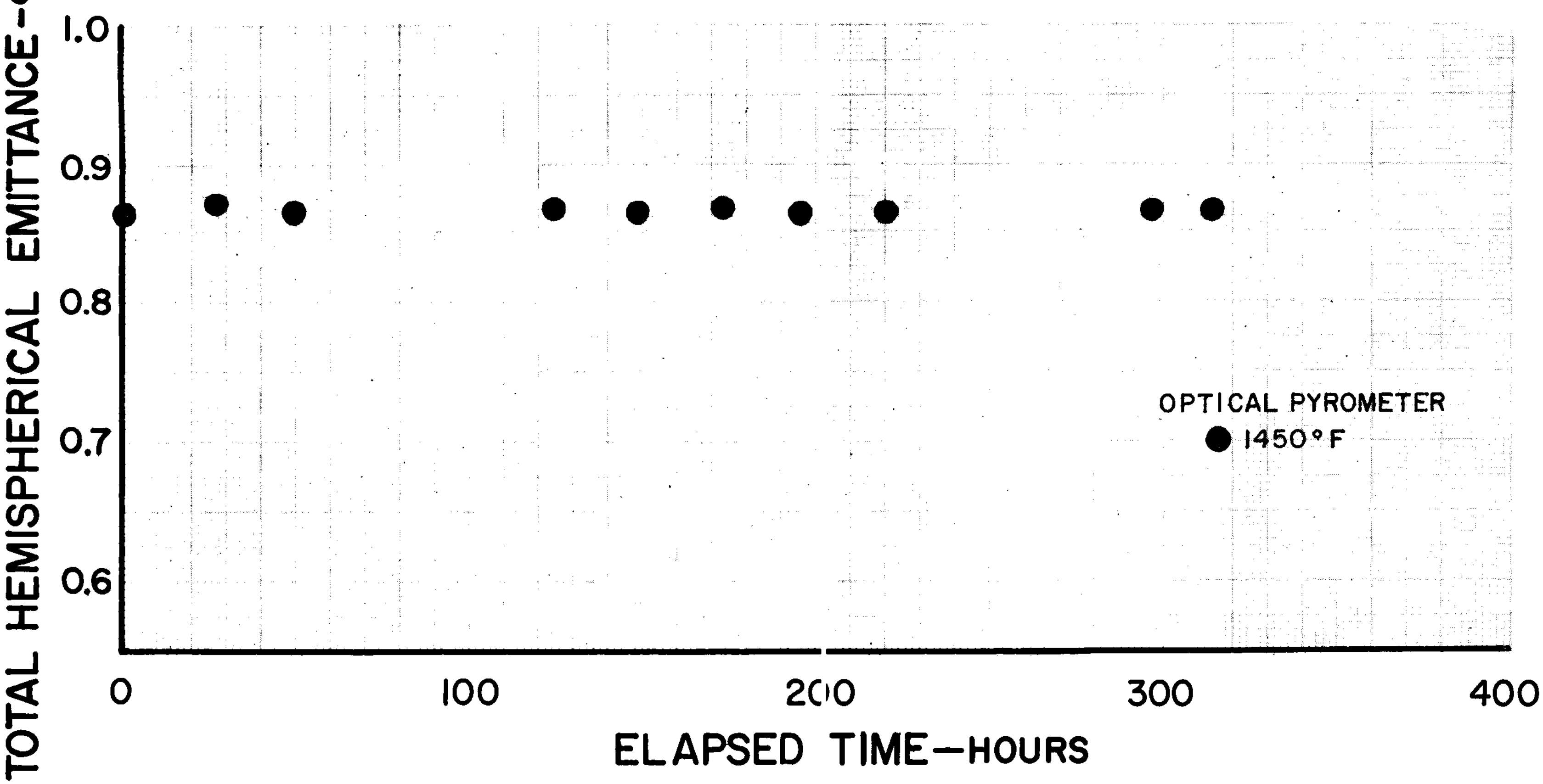
TABLE XVII

Coating: Strontium Titanate - Plasma Arc Sprayed
Substrate: Columbium - 1% Zirconium
4.0-Mil Coating

Endurance Time (Hrs.)	Pressure (mm Hg)	Thermocouple Temp. (°F)	Thermocouple ϵ_{TH}	Thermocouple Avg. ϵ_{TH}	Optical Pyrometer Temp. (°F)	Optical Pyrometer ϵ_{TH}	Optical Pyrometer Avg. ϵ_{TH}
0.0	5.2×10^{-7}	1450	.891		1465	.863	
2.93	1.2×10^{-8}	1449	.893	.894	1468	.858	.867
3.71	9.1×10^{-9}	1450	.892		1454	.878	
5.84	8.3×10^{-9}	1446	.899		1462	.869	
22.58	3.0×10^{-9}	1450	.886		1460	.868	
25.03	2.6×10^{-9}	1450	.887	.887	1459	.870	.869
26.73	2.9×10^{-9}	1451	.884		1460	.868	
30.03	1.9×10^{-9}	1448	.890		1459	.870	
46.51	1.5×10^{-9}	1452	.882		1460	.869	
48.86	1.5×10^{-9}	1451	.886	.884	1463	.863	.864
50.83	1.5×10^{-9}	1451	.885		1464	.861	
119.75	1.4×10^{-9}	1447	.884		1457	.870	
119.94	1.6×10^{-9}	1451	.881		1461	.863	.865
123.89	1.0×10^{-9}	1447	.890	.885	1463	.859	
125.36	1.02×10^{-9}	1449	.885		1460	.866	
143.37	1.3×10^{-9}	1448	.887		1462	.861	
147.39	9.0×10^{-10}	1449	.884	.885	1457	.870	.863
150.73	9.0×10^{-10}	1449	.884		1463	.859	
169.20	7.0×10^{-10}	1448	.885		1457	.868	
171.75	8.0×10^{-10}	1448	.885	.885	1460	.863	.866
174.78	8.0×10^{-10}	1448	.885		1457	.868	
191.26	6.5×10^{-10}	1448	.885		1458	.866	
195.63	7.0×10^{-10}	1447	.887	.885	1460	.863	.863
198.77	7.0×10^{-10}	1448	.884		1461	.860	
214.84	7.5×10^{-10}	1446	.887		1460	.861	
221.03	7.5×10^{-10}	1446	.887	.887	1460	.861	.863
221.79	7.0×10^{-10}	1446	.887		1457	.867	
287.24	8.0×10^{-10}	1449	.884		1457	.870	
292.12	7.5×10^{-10}	1449	.884	.884	1457	.870	.869
294.86	7.0×10^{-10}	1449	.885		1458	.868	
312.17	8.0×10^{-10}	1449	.885		1463	.859	
313.03	6.5×10^{-10}	1447	.888	.886	1460	.864	.862

TOTAL HEMISPHERICAL EMITTANCE vs. TIME

COATING: STRONTIUM TITANATE—PLASMA ARC SPRAYED (4-MIL)
SUBSTRATE: COLUMBIUM—1% ZIRCONIUM



emissivity of strontium titanate, similar to that of calcium titanate, decreases when the specimen is heated above 1600°F.

It remains to determine whether or not the high emittance value achieved at 1400°F will remain while the specimen is cooled, whether the increased emittance can be obtained without heating the specimen to 1400°F, and to determine the effects of endurance testing at temperatures under 1400°F.

N. Silicon Carbide

Silicon carbide (SiC), obtained from the Buehler Corporation, was aluminum phosphate bonded to columbium - 1 per cent zirconium substrates. The resulting coatings were 4 to 5 mils thick, greyish, and soft. The coating-substrate bond strengths ranged from poor to fair. In all, five specimens were tested. Two were tested in the total hemispherical emittance rig, two in the spectral normal emittance rig, and one in the short term endurance rig. Since it is possible to measure both total hemispherical and spectral normal emittance in the spectral normal emittance rig, both of these measurements were made simultaneously for one of the specimens.

1. First Specimen, Total Hemispherical Emittance - The silicon carbide powder used for this specimen was classified by Buehler as 400 mesh. Particle size analysis indicated that 53 per cent of the particles were between 15 and 24 microns in diameter and 80 per cent were between 12 and 30 microns in diameter. Another 11 per cent of the particles were about 9 or 10 microns in diameter.

   The 4-mil thick coating was light grey and had a matte texture similar to that of 320 grit emery cloth. The coating-substrate bond strength was poor. The total hemispherical emittance was measured between 300°F and 1400°F, and results are shown in Table XVIII and in Figure 28. The total emittance increased from about 0.80 at 300°F to about 0.88 at 1400°F with most of the increase occurring below 900°F. Between 1400°F and 1500°F the voltage leads fell off and testing was terminated. After testing the coating had a blue-grey color, was brittle, and was separating from the substrate.

2. Second Specimen, Total Hemispherical Emittance - The powder used for coating this specimen was classified by Buehler as 600 mesh. Particle size analysis indicated that 61 per cent of the particles were between 7 and 10 microns in diameter, and 85 per cent were between 5 and 12 microns in diameter. Another 11 per cent were divided between 4 microns and 15 microns in diameter.

TABLE XVIII
Coating: Silicon Carbide - Aluminum Phosphate Bonded
Substrate: Columbium-1% Zirconium
4.0-Mil Coating

Run No.	Elapsed Time (hrs)	Pressure (mm Hg)	Thermocouple Temperature (°F)	ϵ_{TH}
1	0.4	1.9×10^{-7}	306	.797
	0.8	2.0×10^{-7}	494	.833
	1.3	2.0×10^{-7}	700	.854
	1.6	2.4×10^{-7}	900	.867
	1.8	2.3×10^{-7}	1002	.866
	1.9	3.9×10^{-7}	1097	.868
	2.2	1.4×10^{-6}	1201	.874
	2.4	1.3×10^{-6}	1303	.876
	2.7	1.0×10^{-6}	1398	.882

TOTAL HEMISPHERICAL EMITTANCE vs. TEMPERATURE

COATING: SILICON CARBIDE–ALUMINUM PHOSPHATE BONDED (4-MIL)

SUBSTRATE: COLUMBIUM–1% ZIRCONIUM

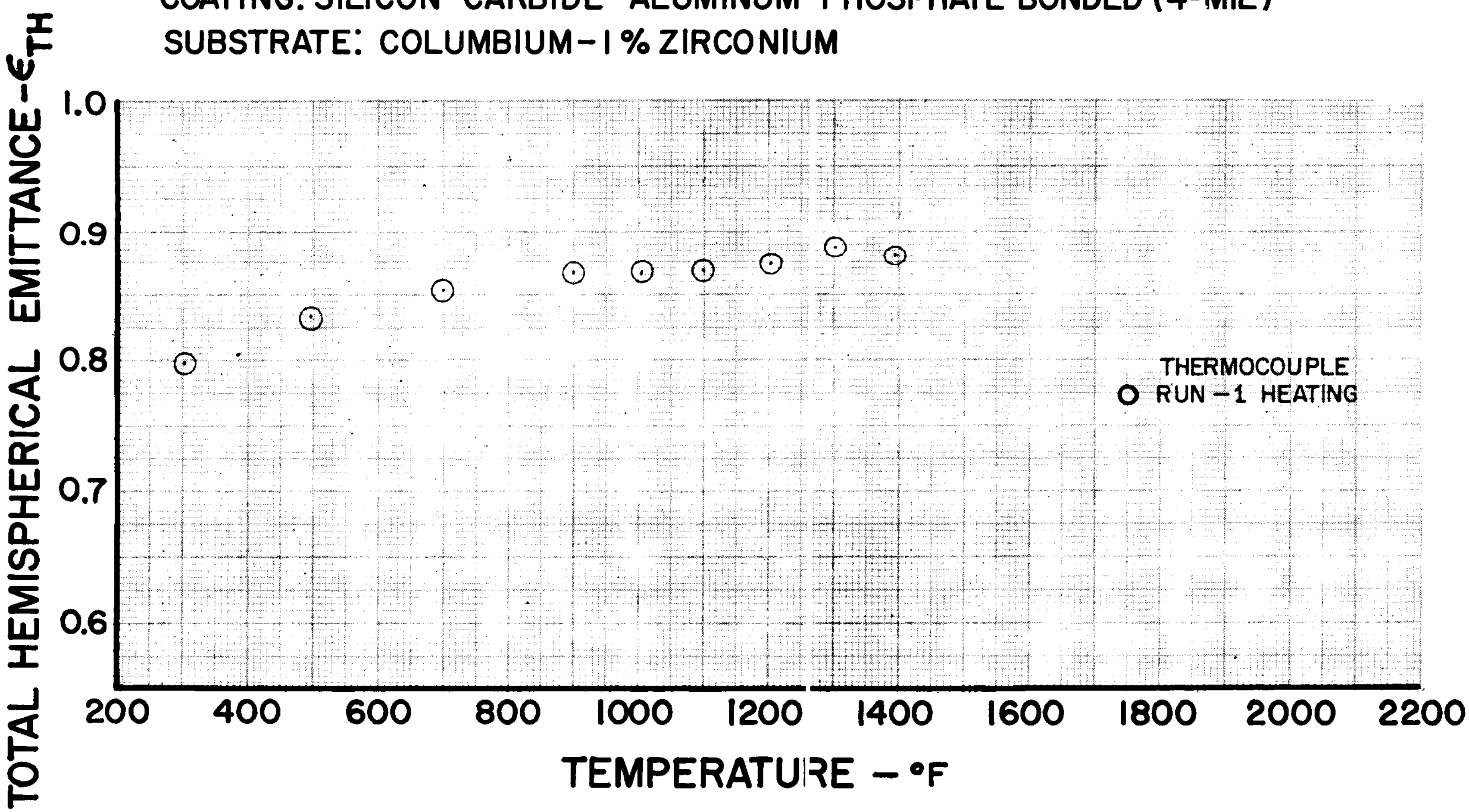


Figure 28

The coating was 4 mils thick, blue-grey and had a matte texture similar to that of 80 grit emery cloth. The coating-substrate bond strength was fair. Total hemispherical emittance was mesured from 300°F to 1700°F and, as shown in Table XIX and in Figure 29, it remained fairly constant over the entire testing temperature range. At 300°F the emittance was about 0.91 and at 1700°F it was about 0.93. At 1800°F the voltage leads failed and the test was terminated. The scatter in data between 1500°F and 1700°F is about 3.5 per cent and this is within the accuracy of the apparatus. When removed from the rig, the coating was light grey but no other changes were observed.

3. Third Specimen, Total Hemispherical and Spectral Normal Emittances - The coating was aluminum phosphate bonded with Alkaphos C, obtained from the Monsanto Chemical Company. (The aluminum phosphate used for all other coatings was prepared at Pratt & Whitney Aircraft from aluminum oxide and phosphoric acid.) The coating was 5 mils thick, grey, and had a matte texture similar to that of 40 grit emergy cloth. The coating-substrate bond strength was fair. Both total hemispherical and spectral normal emittances were measured in the spectral normal emittance rig.

Total hemispherical emittance was measured over the temperature range of 700°F to 1450°F. Emittance data based on thermocouple temperatures were between 0.93 and 0.94 while data based on optical pyrometer temperatures were about 0.90. (See Table XX and Figure 30).

Spectral normal emittance was measured over the wave length range of 1.57 to 12.4 microns at 800°F and at 900°F. The specimen was held at 900°F for 17.6 hours and another set of data over the same wavelength range was taken at 900°F at the end of this interval. Finally, the temperature was increased and the spectral normal emittance was measured over the wavelength range of 1.21 to 13.6 microns at 1300°F and at 1450°F.

TABLE XIX

Coating: Silicon Carbide - Aluminum Phosphate Bonded
Substrate: Columbium - 1% Zirconium
4.0-Mil Coating

Run Number	Elapsed Time (Hrs)	Pressure (mm Hg)	Thermocouple Temp (°F)	Thermocouple ϵ_{TH}	Optical Pyrometer Temp (°F)	Optical Pyrometer ϵ_{TH}
1	0.4	3.5×10^{-7}	303	.908		
	0.6	3.9×10^{-7}	500	.920		
	0.9	5.5×10^{-7}	701	.934		
	1.2	1.6×10^{-6}	900	.916		
	1.4	1.4×10^{-6}	1001	.930		
	1.5	1.6×10^{-6}	1102	.929		
	1.6	2.0×10^{-6}	1204	.918		
	1.8	2.5×10^{-6}	1301	.922		
	1.9	2.3×10^{-6}	1400	.928		
	2.1	2.5×10^{-6}	1499	.930	1507	.915
	2.3	1.7×10^{-6}	1251	.926		
2	2.5	3.3×10^{-6}			1562	.904
	2.6	4.7×10^{-6}			1625	.900
	2.8	5.4×10^{-6}			1668	.932

TOTAL HEMISPHERICAL EMITTANCE vs. TEMPERATURE

COATING: SILICON CARBIDE-ALUMINUM PHOSPHATE BONDED (4-MIL)

SUBSTRATE: COLUMBIUM-1% ZIRCONIUM

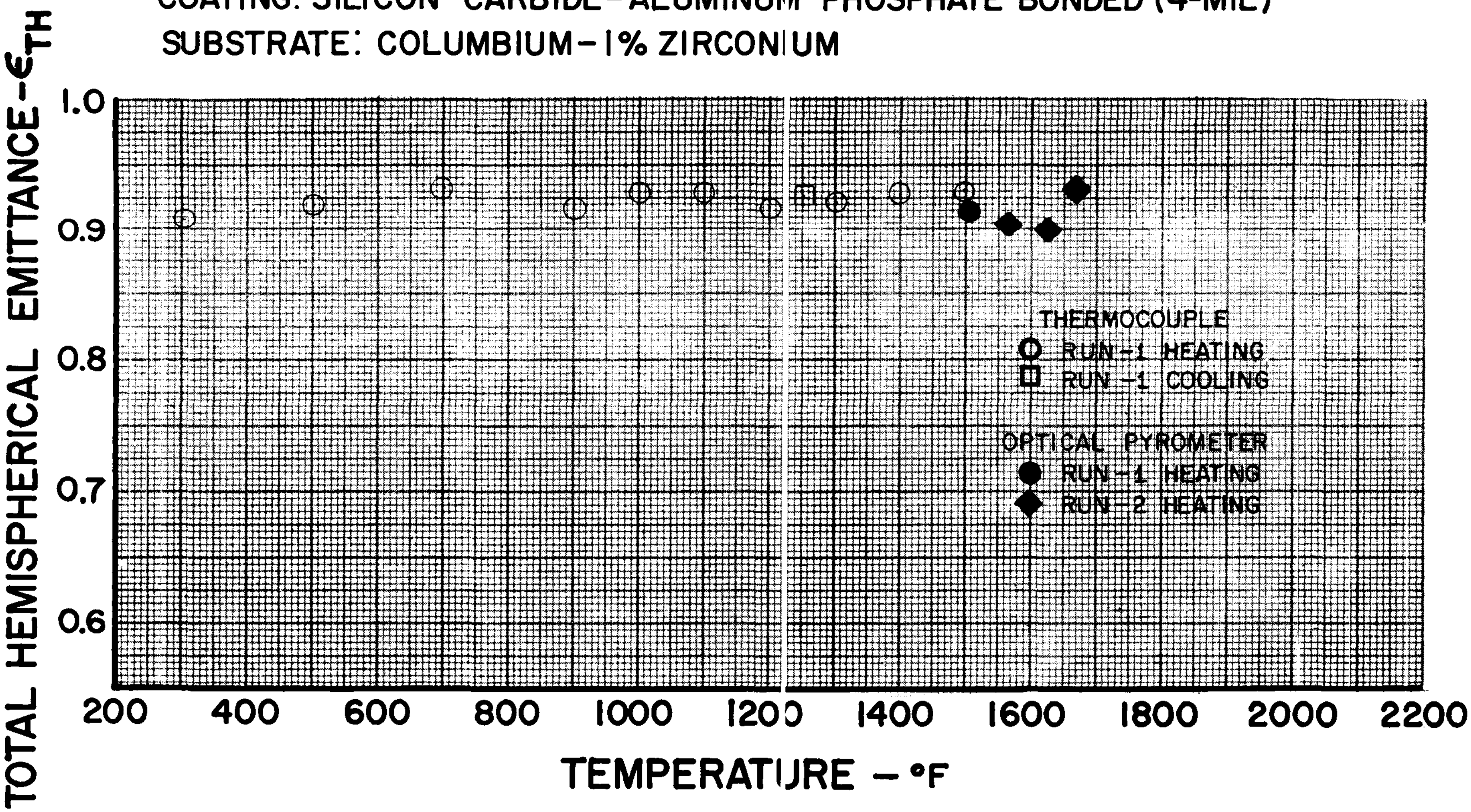


Figure 29

TABLE XX

Coating: Silicon Carbide - Aluminum Phosphate Bonded
Substrate: Columbium - 1% Zirconium
5 - Mil Coating

Run No.	Elapsed Time (hrs)	Pressure (mm Hg)	Thermocouple Temp. (°F)	Thermocouple ϵ_{TH}	Avg. ϵ_{TH}	Optical Pyrometer Temp. (°F)	Optical Pyrometer ϵ_{TH}
1	0.6	3.0×10^{-7}	700	.937	.938		
	2.8	2.5×10^{-7}	700	.939			
	3.4	2.3×10^{-6}	800	.934	.934		
	4.4	1.9×10^{-6}	801	.934			
	4.7	2.7×10^{-5}	900	.944			
	5.5	2.6×10^{-7}	902	.943			
	23.1	2.5×10^{-7}	907	.938	.941		
	24.4	2.6×10^{-7}	907	.937			
	25.1	2.2×10^{-7}	907	.937			
	27.1	$1\ \times10^{-6}$	1000	.935			
	27.3	$5\ \times10^{-6}$	1100	.936			
	27.7	2.2×10^{-6}	1200	.933			
	27.9	$3\ \times10^{-6}$	1300	.932	.936		
	28.8	3.5×10^{-7}	1300	.939			
	29.2	6.5×10^{-7}	1450	.927	.930		
	29.7	3.3×10^{-7}	1452	.932		1468	.902
	30.4	1.9×10^{-8}	1013	.940			
	30.6	1.5×10^{-8}	921	.941			

TOTAL HEMISPHERICAL EMITTANCE vs. TEMPERATURE

COATING: SILICON CARBIDE–ALUMINUM PHOSPHATE BONDED (5-MIL)
SUBSTRATE: COLUMBIUM – 1% ZIRCONIUM

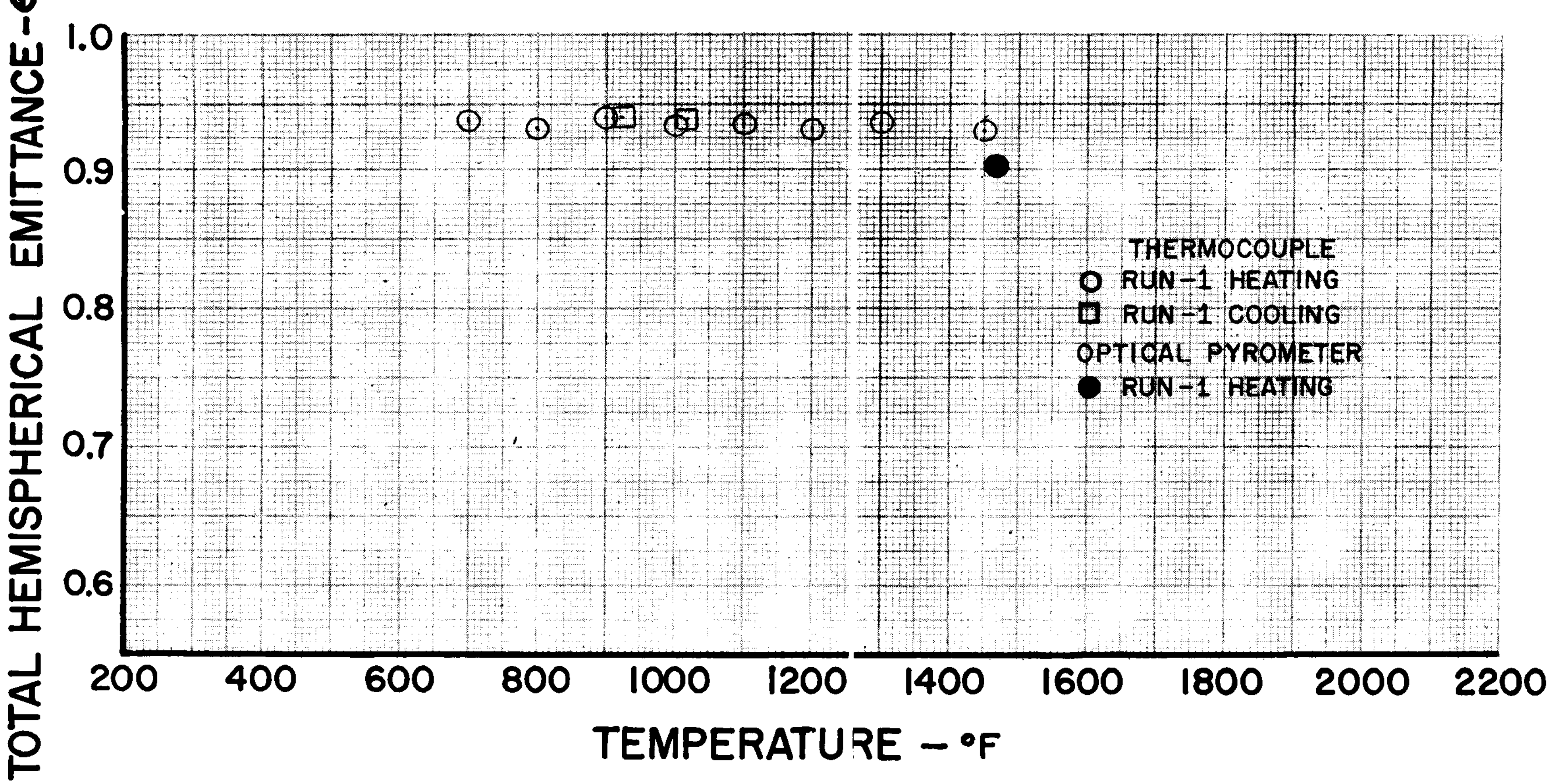


Figure 30

Figure 31 shows results from these tests along with those determined by H. Blau, et al, Arthur D. Little, Inc., in "Infrared Spectral Emittance Properties of Solid Materials, Final Report under Contract AF19(604)-2433," October 1960. Blau's data was obtained from specimens heated in air.

He describes his method as follows:

> Radiation from the heated specimen was directly compared to radiation from a black body at the same temperature. Specimens were embedded in an accurately machined cavity in the surface of a hollow silicon carbide heater element and heated by conduction. A hole in the heater wall provided the source of black body radiation.

It may be seen from Figure 31 that agreement is good. The only change in the coating observed after testing was a separation from the substrate.

4. <u>Fourth Specimen, Spectral Normal Emittance</u> - The coating for this specimen was made from the same silicon carbide powder used for specimen number three and was made at the same time as specimens numbers two and five.

   The resulting coating was 4 mils thick, grey, and had a matte texture similar to that of 80 grit emery cloth. The coating-substrate bond strength was poor. The spectral emittance of the coating was measured between 1.57 and 12.4 microns at 900°F and between 1.01 and 13.0 microns at 1450°F. The results from this test are in very good agreement with those of the preceding spectral normal emittance test, (Figure 32). No visible changes of the coating were observed after testing.

5. <u>Fifth Specimen, Short Term Endurance Test</u> - The last of three specimens prepared at the same time as specimens numbers three and four was placed in the short term endurance rig to determine the effects on the total hemispherical emittance, of exposure to a temperature of 1450°F for 300 hours. The 5-mil thick coating was light grey and had a matte texture similar to

SPECTRAL NORMAL EMITTANCE vs. WAVELENGTH

COATING: SILICON CARBIDE–ALUMINUM PHOSPHATE BONDED (5-MIL)
SUBSTRATE: COLUMBIUM–1% ZIRCONIUM

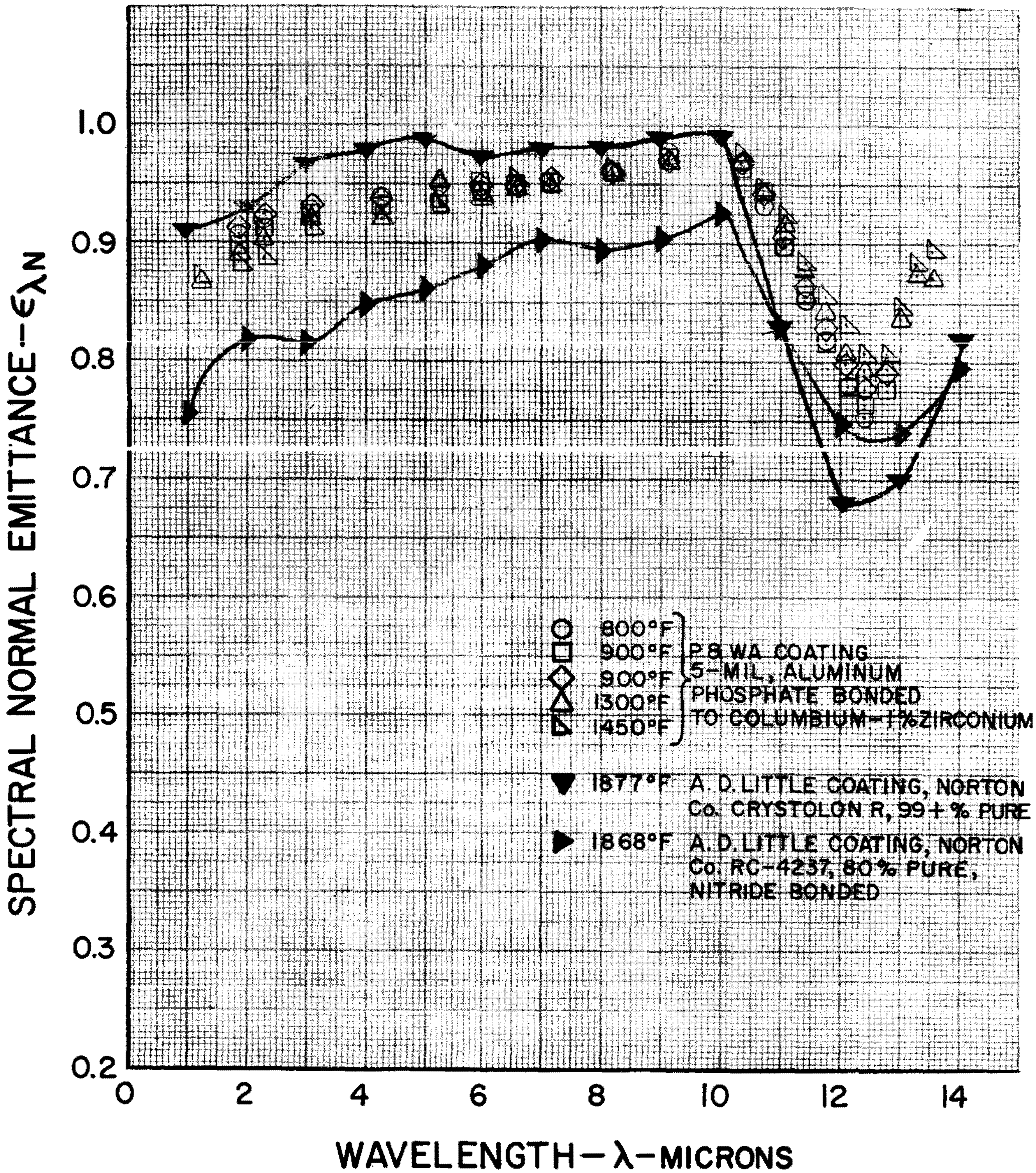


Figure 31

SPECTRAL NORMAL EMITTANCE vs. WAVELENGTH

COATING: SILICON CARBIDE-ALUMINUM PHOSPHATE BONDED (4-MIL)
SUBSTRATE: COLUMBIUM-1% ZIRCONIUM

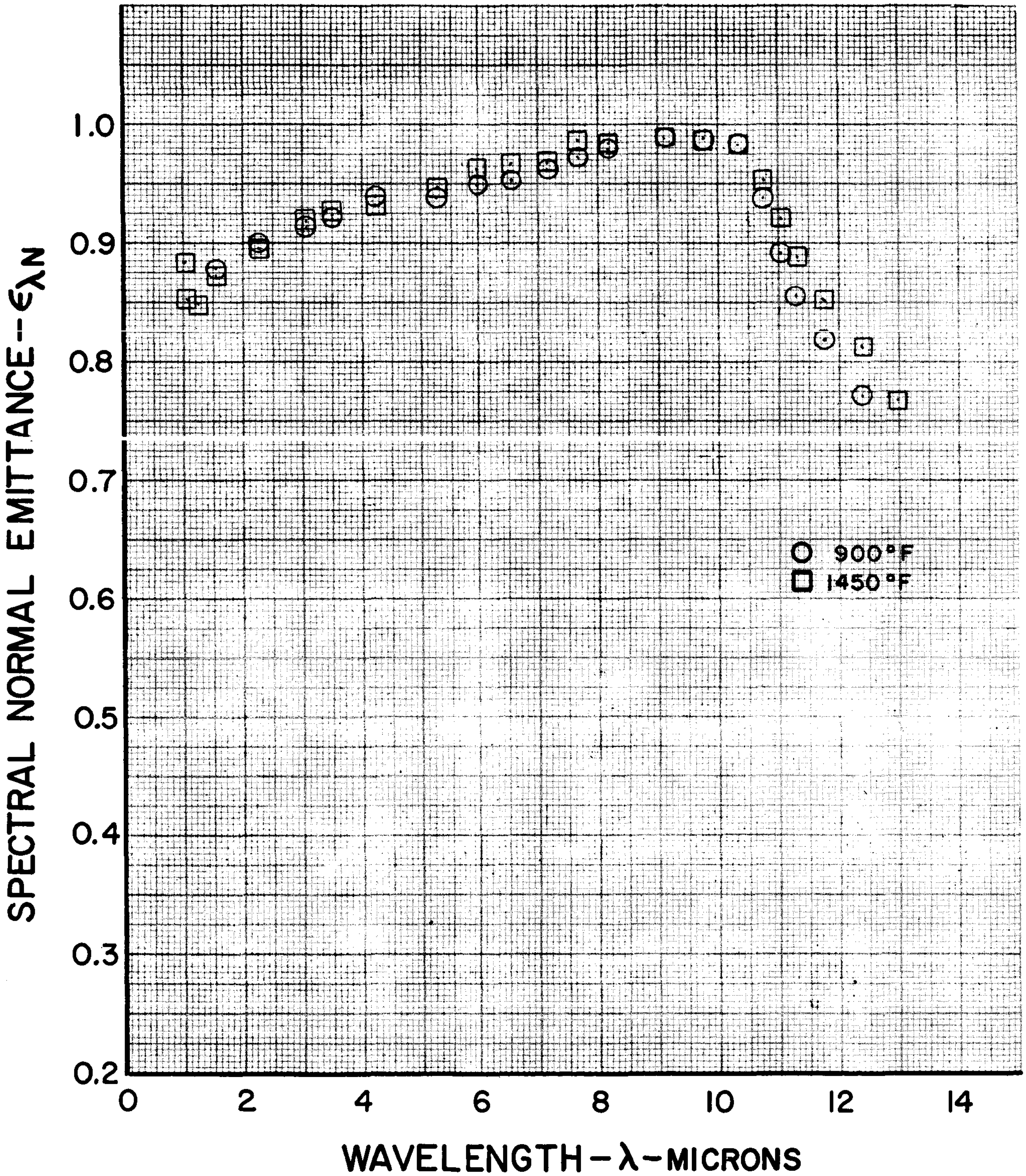


Figure 32

that of 320 grit emery cloth. The coating-substrate bond strength was poor. Table XXI and Figure 33 show the total emittance of the coating as the temperature was increased from 300°F to 1450°F. Over this temperature range the total hemispherical emittance of the specimen decreased from about 0.94 to about 0.91 where it remained throughout the 300 hour endurance test. See Table XXII and Figure 34. The visual characteristics of the coating were unchanged by testing.

6. General Remarks - All of the data presented in this section indicate that the repeatability of the emittance values from one coating to the next using the same silicon carbide falls within the accuracy of the apparatus. In general, it appears that the emittance of the aluminum phosphate bonded Buehler 600 mesh silicon carbide tested lies between 0.90 and 0.93 over the temperature range of 300°F to 1600°F.

X-ray diffraction analysis of specimens numbers one and five before and after testing showed SiC to be the only detectable phase present. Spectrographic analysis of the powders used in both of these specimens indicated that Si was the only major constituent present although Fe and Al were present as semi-major constituents. After testing, both Si and Al were present as the major constituents but Fe was now a minor constituent.

TABLE XXI
Coating: Silicon Carbide - Aluminum Phosphate Bonded
Substrate: Columbium - 1% Zirconium
5 - Mil Coating

Elapsed Time (hrs)	Pressure (mm Hg)	Thermocouple Temp. (°F)	ϵ_{TH}
0.7	5.3×10^{-7}	299	.945
0.9	6.0×10^{-6}	397	.936
1.2	5.4×10^{-6}	498	.930
2.1	5.4×10^{-6}	602	.926
2.4	4.4×10^{-6}	700	.923
2.7	7.0×10^{-6}	800	.917
3.0	6.3×10^{-6}	902	.919
3.2	6.0×10^{-6}	1000	.915
3.5	4.8×10^{-6}	1100	.911
3.7	4.7×10^{-6}	1197	.911
3.9	5.2×10^{-6}	1301	.913
4.2	3.5×10^{-6}	1400	.928

TOTAL HEMISPHERICAL EMITTANCE vs. TEMPERATURE

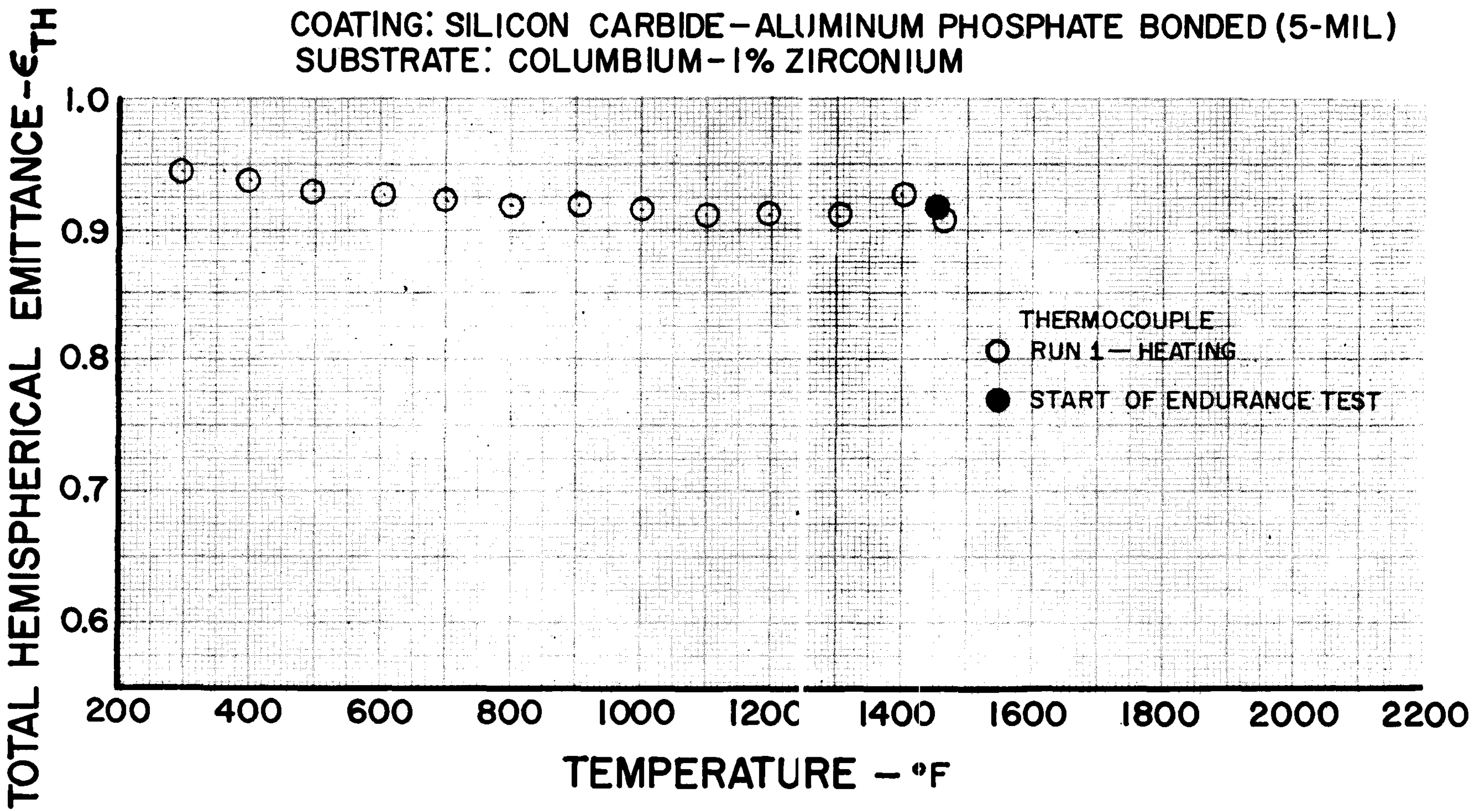
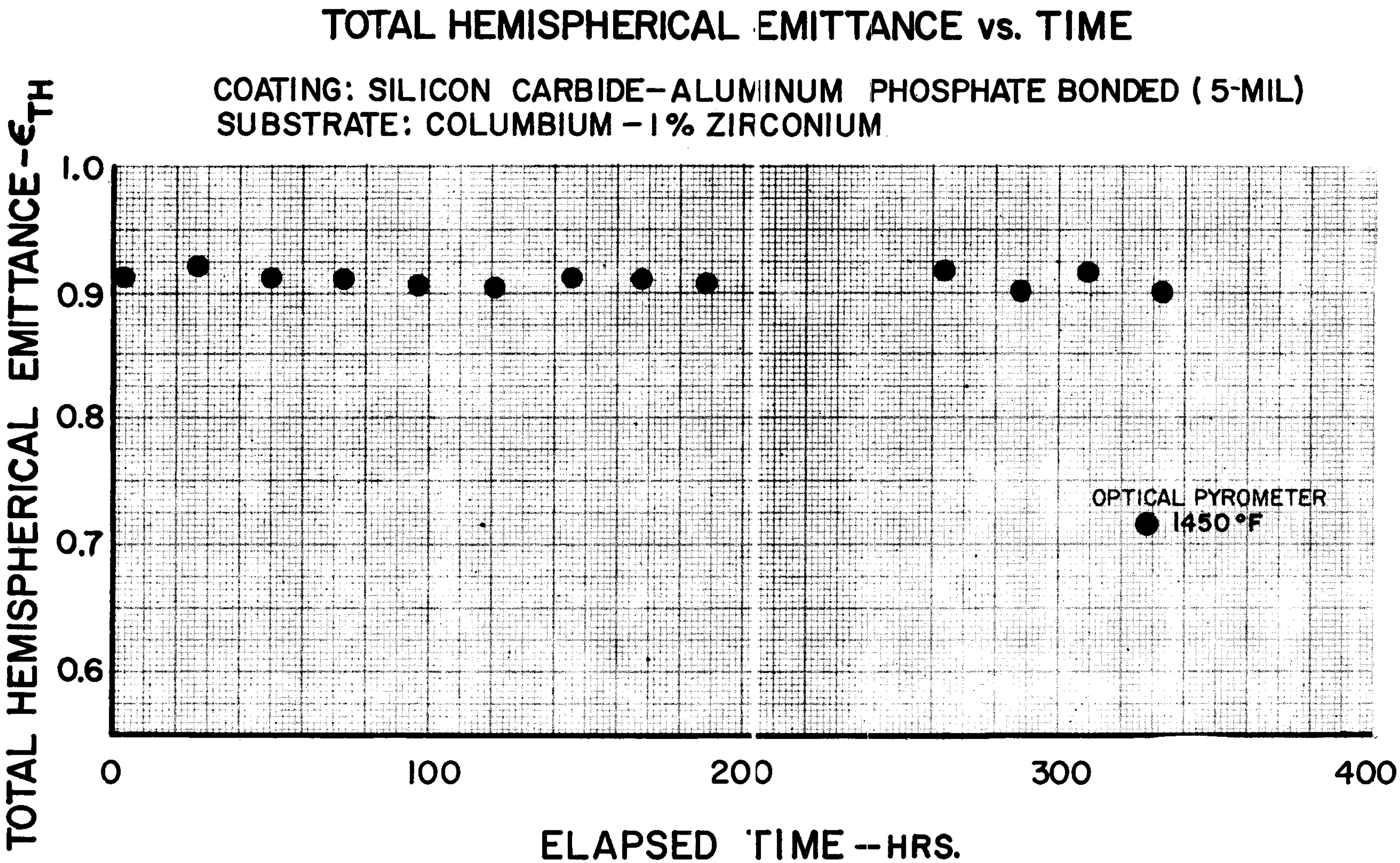


Figure 33

TABLE XXII
Coating: Silicon Carbide - Aluminum Phosphate Bonded
Substrate: Columbium - 1% Zirconium
5 - Mil Coating

Endurance Time (hrs)	Pressure (mm Hg)	Thermocouple Temp. (°F)	Thermocouple ϵ_{TH}	Thermocouple Avg. ϵ_{TH}	Optical Pyrometer Temp. (°F)	Optical Pyrometer ϵ_{TH}	Optical Pyrometer Avg. ϵ_{TH}
0	2.3×10^{-6}	1460	.905	.909	1451	.895	.917
1.3	5.0×10^{-7}	1463	.914		1454	.930	
23.6	9.3×10^{-8}				1449	.937	.923
25.5	6.7×10^{-8}				1454	.908	
47.7	5.5×10^{-8}				1451	.911	
71.0	3.8×10^{-8}				1454	.910	
90.7	3.5×10^{-8}				1454	.905	.906
97.2	3.4×10^{-8}				1454	.907	
115.1	3.3×10^{-8}				1454	.907	.904
121.2	3.3×10^{-8}				1458	.901	
138.5	2.8×10^{-8}				1450	.915	.913
145.5	2.9×10^{-8}				1451	.912	
161.9	2.7×10^{-8}				1451	.913	.912
169.1	2.7×10^{-8}				1451	.912	
186.1	2.5×10^{-8}				1454	.908	.908
193.2	2.4×10^{-8}				1454	.908	
258.6	2.0×10^{-8}				1450	.919	.918
264.8	2.0×10^{-8}				1449	.918	
282.3	2.0×10^{-8}				1458	.901	.901
289.1	2.0×10^{-8}				1456	.901	
306.9	2.2×10^{-8}				1458	.918	
330.6	2.1×10^{-8}				1458	.900	



TOTAL HEMISPHERICAL EMITTANCE vs. TIME
COATING: SILICON CARBIDE–ALUMINUM PHOSPHATE BONDED (5-MIL)
SUBSTRATE: COLUMBIUM – 1% ZIRCONIUM
TOTAL HEMISPHERICAL EMITTANCE – ϵ_{TH}
1.0
0.9
0.8
0.7
0.6
OPTICAL PYROMETER
1450 °F
0
100
200
300
400
ELAPSED TIME -- HRS.

Figure 34

O. Manganese Oxide

The manganese oxide (Mn_2O_3) used for this coating was obtained from A. D. McKay, Incorporated. A 3-mil thick coating was plasma-arc sprayed onto a columbium - 1 per cent zirconium substrate. The coating was black, hard, and had a matte texture similar to that of 80 grit emery cloth. The coating-substrate bond strength was good. Total hemispherical emittance was measured over the temperature range of 300°F to 2150°F. Table XXIII and Figure 35 show that the total emittance increased from about 0.75 at less than 700°F to about 0.88 at 1900°F, and then decreased to about 0.85 at 2000°F. At 2000°F the temperature was increasing rapidly at a constant power setting indicating a rapid reduction in emittance. At 2100°F and at 2150°F, the temperature was decreasing rapidly with a constant power setting requiring a steadily increasing power setting to maintain the temperature long enough to obtain emittance data. At these temperatures the emittance values were increasing. As the specimen was cooled the emittance values remained close to 1.0.

When the chamber was opened it was discovered that the coating had volatilized and that a metallic coating had been deposited on the instrument flange and on other parts of the rig. The reason for the observed rapid decrease in temperature at 2100°F can probably be attributed to the heat of sublimation of the coating and the progressively increasing electrical leakage between thermocouple wires resulting from deposition of metallic material.

When the specimen was removed it was glossy black, extremely hard, and had a texture similar to that of 320 grit emery cloth. The coating-substrate bond strength was extremely good.

The coating inside the chamber had shorted the thermocouple and voltage terminals to ground and it is not believed that the extremely high values of emittance recorded during cooling are accurate. These values therefore are not reported.

TABLE XXIII

Coating: Manganese Oxide - Plasma Arc Sprayed
Substrate: Columbium - 1% Zirconium
3.0-Mil Coating

Run Number	Elapsed Time (Hrs)	Pressure (mm Hg)	Thermocouple Temp (°F)	Thermocouple ϵ_{TH}	Optical Pyrometer Temp (°F)	Optical Pyrometer ϵ_{TH}
1	1.0	6.1×10^{-6}	300	.811		
	2.0	6.2×10^{-6}	501	.778		
	2.6	6.6×10^{-6}	699	.769		
	2.8	7.0×10^{-6}	902	.781		
	3.4	7.6×10^{-6}	1000	.804		
	3.6	6.6×10^{-6}	1101	.822		
	3.7	5.6×10^{-6}	1200	.837		
Heating Current Off; Vacuum Maintained						
2	4.0	1.5×10^{-7}	1000	.800		
	4.2	5.6×10^{-7}	1100	.810		
	4.5	7.8×10^{-7}	1200	.832		
	4.7	1.3×10^{-6}	1300	.849		
	5.0	2.0×10^{-6}	1400	.862		
	5.2	2.1×10^{-6}	1502	.865	1485	.895
	5.5	2.2×10^{-6}	1604	.870	1586	.901
	5.7	1.8×10^{-6}	1700	.874	1700	.874
	5.9	2.2×10^{-6}	1799	.881	1800	.880
	6.1	2.4×10^{-6}	1900	.885	1890	.900
	6.2	2.0×10^{-6}	1999	.850	1990	.863

Coating Volatilized; Test Terminated

TOTAL HEMISPHERICAL EMITTANCE vs. TEMPERATURE

COATING: MANGANESE OXIDE — PLASMA ARC SPRAYED (3-MIL)
SUBSTRATE: COLUMBIUM—1% ZIRCONIUM

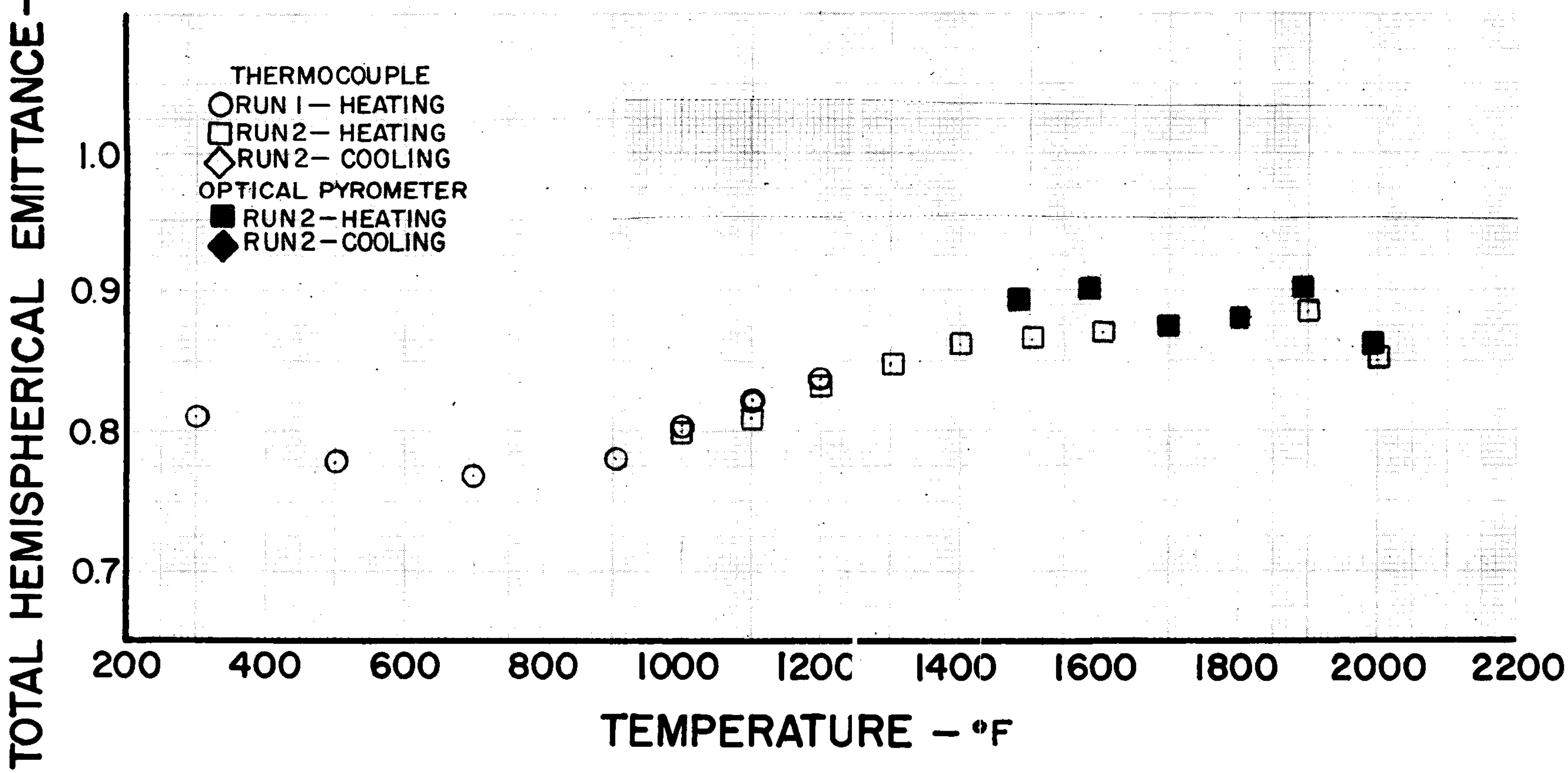


Figure 35
Page 86

III RECONDITIONING OF THE TOTAL HEMISPHERICAL EMITTANCE RIG

The volatilization of the manganese oxide coating in the total hemispherical emittance rig (see Section II 0) necessitated a complete cleaning and recalibration of the rig before further measurements could be made.

The coating was removed from the flange by washing the flange with chromic acid, rinsing with water, and finally rinsing with reagent grade acetone. The ceramic thermocouple standoff insulators on the flange were replaced. At this time the resistance to ground for each of the thermocouple leads was 30 megohms or greater. The resistance to ground for each of the heater leads was 50 megohms or greater. The platinum-platinum 10 per cent rhodium thermocouple leads when shorted showed a maximum variation of 0.004 millivolts, corresponding to one degree fahrenheit. The chromel-alumel thermocouple leads when shorted showed a maximum variation of 0.029 millivolts, corresponding to one degree fahreheit.

An emittance test was run on a tungsten strip and the results indicated that the emittance level at 2000°F was 0.014 higher than that previously reported in PWA-1863. In an attempt to eliminate this error the voltmeter and the current shunts were checked for calibration. The model 803 Fluke voltmeter was calibrated against another Fluke voltmeter and against a Ballantine calibrator. The voltmeter was found to be within the 0.2 per cent accuracy guaranteed by the manufacturer. The current shunts were calibrated against a shunt whose calibration is traceable to the National Bureau of Standards. The deviations found were as follows:

50 amp	0.04%
100 amp	0.08%
200 amp	0.05%

The instrument flange was removed from the rig and cleaned by very fine grit blasting.

The thermocouple stand-offs, both ceramic insulators and wires, were replaced. The hook-up wires from the feed-thrus to the stand-offs were replaced and the insulation on these wires was changed from ceramic beads to teflon tubing. The resistance to ground for each of the thermocouple leads was 50 megohms or greater. The platinum-platinum 10 per cent rhodium thermocouple leads when shorted did not show a variation great enough to be read with the potentiometer. The chromel-alumel thermocouple leads when shorted showed a maximum variation of 0.010 millivolts, corresponding to 0.5 degrees fahrenheit.

An emittance test was run on the tungsten strip and at 2000°F the emittance value was 0.163 which is .005 above the level reported in PWA-1863 and .009 below that obtained before the instrument flange was grit blasted. As a further check, total hemispherical emittance was measured for a tubular tantalum sample in both the total hemispherical emittance rig and in the spectral emittance rig. Agreement was within less than 1 per cent.

Following these low emittance tests, 15 high-emittance coated specimens were tested. Eight of these specimens had plasma-arc sprayed coatings and these were partially oxidized hastelloy C, partially oxidized hastelloy X, oxidized kennametal K-151-A, oxidized kennametal K-162 B, calcium titanate (2 specimens), and strontium titanate (2 specimens). Of the remaining speciments, six had aluminum phosphate-bonded coatings and these were iron-titanium oxide, iron-titanium-aluminum oxide, barium titanate, strontium titanate, and silicone carbide (2 specimens). The last specimen had a silicon carbide coating bonded with alkaphos.

For comparison, several of these specimens were also tested in the spectral normal and the short term endurance rigs. In all cases the emittance values measured in the total hemispherical emittance rig were higher than those measured in the other rigs. It would therefore appear that although the total hemispherical emittance rig produces accurate results for materials with low emittances, it does not produce accurate results for materials with high emittances. For this reason, no data based on measurements made in the total hemispherical emittance rig after the volatilization of the manganese oxide will be reported until such time as corrective measures have been completed.

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