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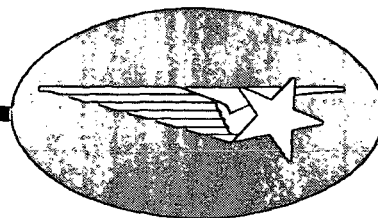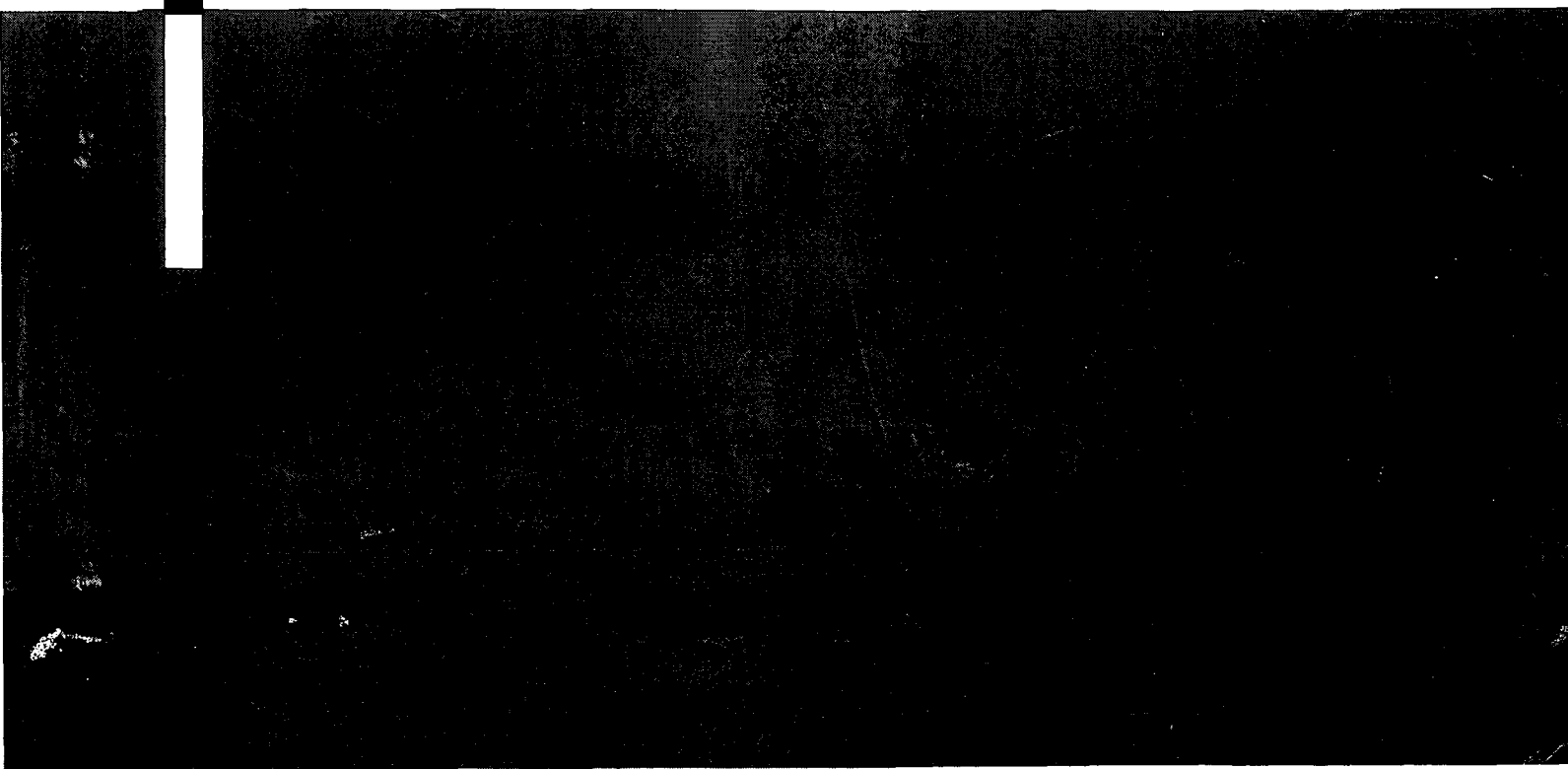
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DEVELOPMENT OF TECHNIQUES
AND ASSOCIATED INSTRUMENTATION
FOR HIGH TEMPERATURE
EMISSIVITY MEASUREMENTS

CONTRACT NAS 8-26304

QUARTERLY PROGRESS REPORT

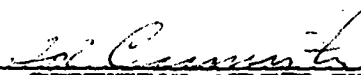FOR PERIOD 29 JUNE 1972 TO 28 SEPTEMBER 1972

PREPARED FOR

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

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FOREWORD

This document was prepared by the Thermophysics
Group of the Infrared Programs Laboratory of the Engineering
Sciences Directorate, Lockheed Palo Alto Research Laboratory,
Lockheed Missiles & Space Company, Inc., for the George C.
Marshall Space Flight Center of the National Aeronautics and
Space Administration. This quarterly report describes the
technical activities carried out under Contract NAS 8-26304
during the period 29 June 1972 to 28 September 1972. The work
was administered under the technical direction of Mr. Roger
Harwell, Materials Division, Astronautics Laboratory, Marshall
Space Flight Center.

ABSTRACT

This report describes the progress on the calibration procedures and specimen testing during the period of 29 June 1972 through 28 September 1972. Test results are given for the NBS Platinum - 13% Rhodium standard (SRM No. 1409), the NBS oxidized Kanthal standard (SRM No. 1427) and oxidized Rene 41 materials.

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Section 1

INTRODUCTION

Design of a radiatively cooled thermal protection system for reusable
entry vehicles is dependent upon accurate emittance values for the exterior
surface coatings or materials under the conditions of the entry environment.
To achieve the optimum in system design, the total hemispherical emittance
must be high at elevated temperatures, and this property should be stable
during entry. To accomplish the multiple reuse goal, the emittance stability
should be maintained for many entry exposures. Because the radiation pro-
perties of candidate surfaces may be dependent upon the elevated temperature
exposure conditions, it is necessary to evaluate their emittance in an environ-
ment which simulates the entry conditions as nearly as is practical within
funding and time limits. During the initial phase of this program, investiga-
tions were made into the potential of using existing facilities to perform emit-
tance   studies for this application. As none met the requirements for simu-
lation together with adequate emittance measurement capability, a new facility
was prepared. This apparatus has the capability for total and spectral emit-
tance measurements on metals and ceramic coatings to $3000^\circ F$ at pressures of
10^{-6} to 760 torr in a static atmosphere or with a flow of air directed onto the
test surface. This air flow may be varied from subsonic to Mach 2.1.

Apparatus checkout has been completed and measurement accuracy verified by
measurement of NBS standards. Oxidized specimens of René 41, HS-188 and
TD Ni Cr alloys have been prepared for emittance testing and initial radiative
properties characterized. Entry simulation tests have been completed on the
René 41 material, and the results show that its total emittance is stable for
five cycles at $1150^\circ K$ ($1600^\circ F$). Calibration procedures and results are des-
cribed in Section 3. Specimen preparation and characterization is discussed in
Section 4, and the entry simulation test results for oxidized René 41 are pre-
sented in Section 5.

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Section 2
SUMMARY

During this reporting period calibration of the radiometric emittance measurement apparatus was completed using NBS standards of Platinum-13 percent Rhodium and oxidized Kanthal (SRM 1409 and SRM 1427 respectively). The results using the Platinum-Rhodium standard showed that the measurement system compared with the NBS data to within 0.02 for both total and spectral normal emittance measurements. The data obtained on the Kanthal strip was consistly higher than NBS values and this is believed to be due to a standard which deviated from the published values. No reason for this difference was found.

Specimens of oxidized René 41, HS-188 and TD Ni Cr have been prepared and characterized for flow testing. Also, emittance measurements have been made on the René 41 material under conditions of no flow and both Mach 1.1 and 2.2 flow conditions. Five typical entry exposure conditions were completed for each flow situation. No significant changes in "as prepared" emittance was observed. The emittance of this material was stable for temperatures to $1150^{\circ}K$ $(1600^{\circ}F)$.

Section 3

RADIOMETRIC MEASUREMENTS STANDARDIZATION

In order to verify the accuracy of the radiometric measurements (Barnes Model 12-511 4-in Research Radiometer) of total and spectral near normal emittance in the set-up for simulated entry test conditions, data were obtained on two NBS Standard Reference Material spectral emittance specimens. These specimens were installed in the test chamber in the configuration for entry testing, and measurements were made only under static air or vacuum (lower temperatures) conditions to avoid possible damage to the specimens or degradation of the standard surfaces. The two standards were Platinum-13 percent Rhodium (NBS SRM No. 1409) and oxidized Kanthal (NBS SRM No. 1427) purchased from NBS in the form of 3/4-inch wide by 10-inch long strips. The majority of testing was conducted in air in accordance with the NBS recommendations for these materials. However, several lower temperature vacuum runs were made on the Kanthal strip to evaluate possible convection effects on thermocouple temperature measurements.

Three Platinum-6 percent Rhodium/Platinum-30 percent Rhodium thermocouples, 3 mil diameter, were spotwelded to the Platinum-13 percent Rhodium specimen on the vertical edge; one at the center of the strip and one each 1/2-inch below and 1/2-inch above the center thermocouple. The edge location was chosen to avoid damage to the central area of the strip which was viewed by the radiometer. This material has a moderately high thermal conductivity and low emittance so unacceptable temperature gradients should not exist between the central area and the edge, as was later verified by an optical pyrometer brightness temperature survey across the width of the specimen. Chromel-alumel thermocouples, 3 mil diameter, were spotwelded to the edge of the Kanthal strip in a similar fashion to the Platinum Rhodium alloy strip with the addition of a thermocouple on the rear surface opposite the central viewing area. This latter thermocouple was used because of relatively large center-to-edge temperature gradients which might exist in this lower thermal conductivity, high emittance material in a convective environment.

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Each standard specimen was installed in the apparatus in a vertical orientation, and the ends were clamped to the water cooled electrode assemblies. Tension on the strip was regulated by a pneumatic cylinder which positioned the upper moveable electrode unit. Electrical power for specimen heating was supplied from a silicon controlled rectifier (SCR) unit operated by a variable set-point, proportional band controller using one specimen thermocouple for the controller set-point deviation input. Radiometer output voltage and attenuator switch position were recorded for each temperature level together with all thermocouple outputs (including control) and electrical power across the test area. Additionally, optical pyrometer brightness temperatures were measured for temperatures of 1100°K and higher.

3.1 Platinum - 13 Percent Rhodium Results

A total of nineteen sets of emittance data were obtained for this material. Measurements were made at four temperatures (800°, 1100°, 1400° and 1600°K). corresponding to those for the NBS spectral data supplied with the standard. Both total and spectral near normal emittance measurements were obtained, the latter through the use of eight spectral filters in the radiometer filter wheel. Band center wavelength, band pass and band transmittance of these filters are given in Table 3-1.

The measured values of E_λ as a function of filter band center wavelength are shown in figures 3-1, 3-2, 3-3 and 3-4 for 800° to 1600°K. The solid curves represent the NBS spectral data at each temperature. These are based upon the arithmetic average of twenty-one measured values at each wavelength. Standard deviations of the average value for each of seven samples measured by NBS are shown by the broken lines of each figure (ref. 1). In general, the spectral data are in good agreement with the NBS values with the exception of the lowest temperature data at the two shortest wavelengths and the 7.96 μm data. The deviations of these data are due to the very low radiometer signal levels caused by the low specimen spectral radiances under these conditions. These resulted in signal uncertainties of 10 to 20 percent from noise at the

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TABLE 3-1

SPECTRAL FILTER CHARACTERISTICS

Filter Position Number	Band Center Wavelength (μm)	Half Band* Width (μm)	Band Transmittance (percent)
1-2	1.42	0.58	75
1-3	2.17	0.20	65
1-4	2.70	0.21	78
1-5	3.39	0.12	75
1-6	4.26	0.27	75
1-7	4.85	0.32	75
1-8	5.46	0.19	58
2-1	7.96	0.22	60
3-1	-	7.84 to 16.6	85

*Transmittance = 50 percent.

very high instrument gain settings required to perform the measurements at these wavelengths. The reproducibility of successive measurements at a given wavelength and temperature, with the exceptions noted earlier, was generally within 0.02 which is considered to be well within acceptable limits for the measurements to be made in this test apparatus.

Measured total near normal emittance (E_{TN}) values are compared with those calculated from the NBS spectral data in Table 3-2. Although data for E_{TN} are not provided by NBS, their spectral values were used to compute E_{TN} for each temperature by numerical integration of the spectral emittance curves for the appropriate blackbody energy distributions for each temperature. Spectral standard deviations were employed to give a deviation for E_{TN} assuming that a high or low standard is consistently high or low at all wavelengths. The measured values of E_{TN} agree with the integrated NBS data to within 0.012 which is within the standard deviation.

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a: Broad Band

O = Case 1
△ = Case 3
□ = Case 8A

Wavelength, μm

ϵ_λ

0.5 0.4 0.3 0.2 0.1 0

0 2 4 6 8 10 12 14 16

Figure 3-1. Spectral Emittance Data for NBS Platinum - 13% Rhodium Emittance
Standard Measured in Air, with Barnes Radiometer.   T = 800°K

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Figure 3-2. Spectral Emittance Data for NBS Platinum - 13% Rhodium Emittance Standard Measured in Air, with Barnes Radiometer. T = 1100°K
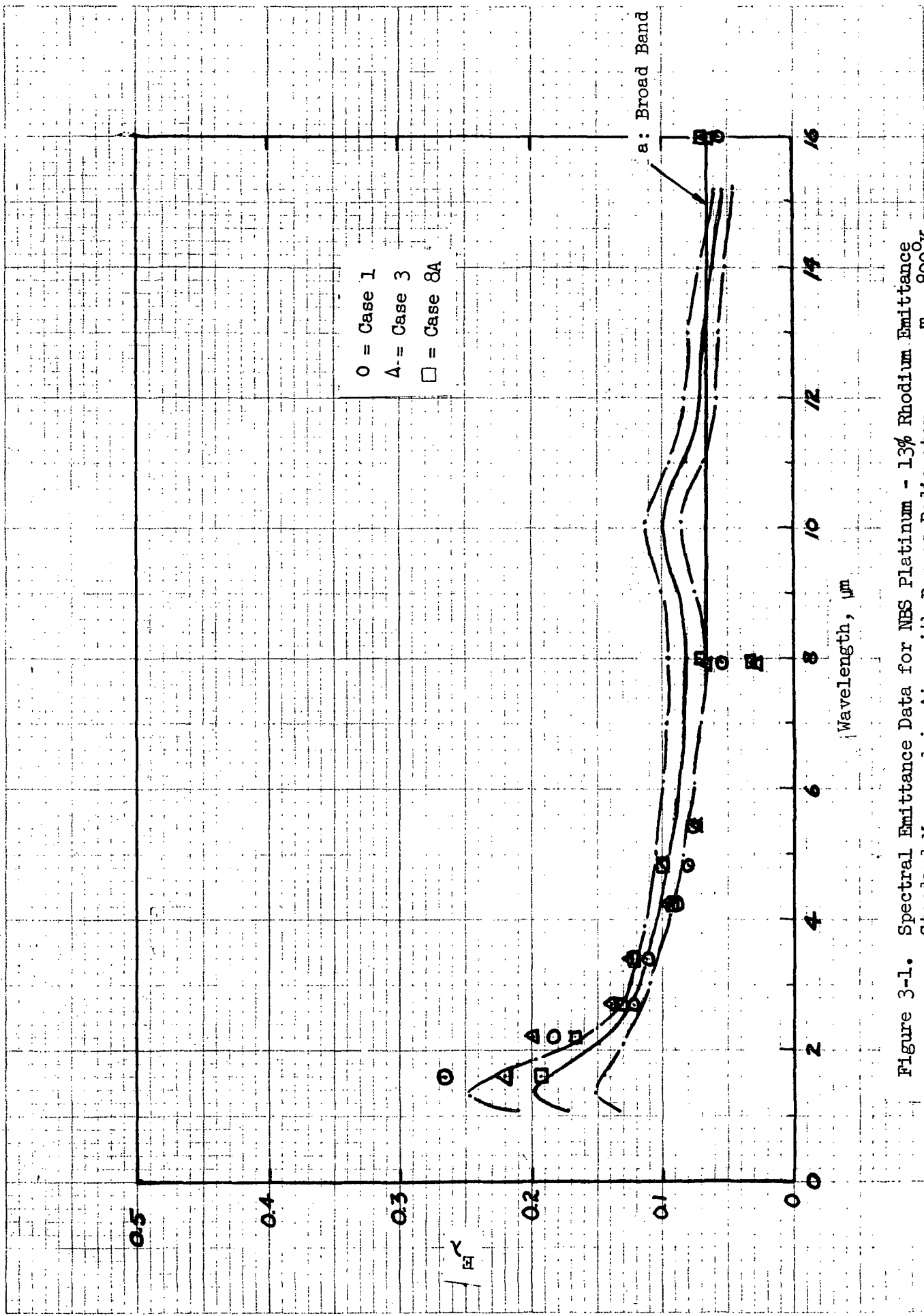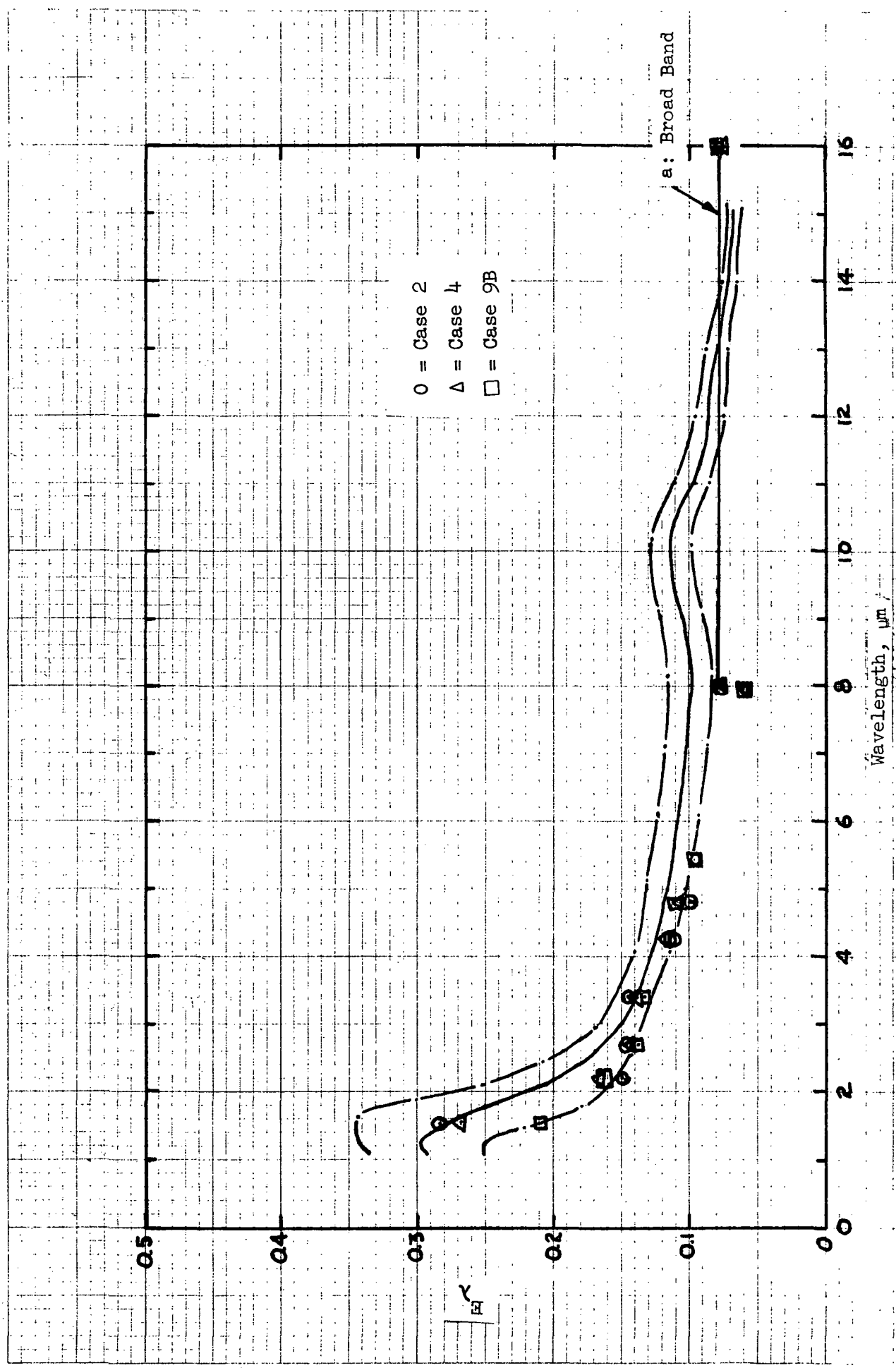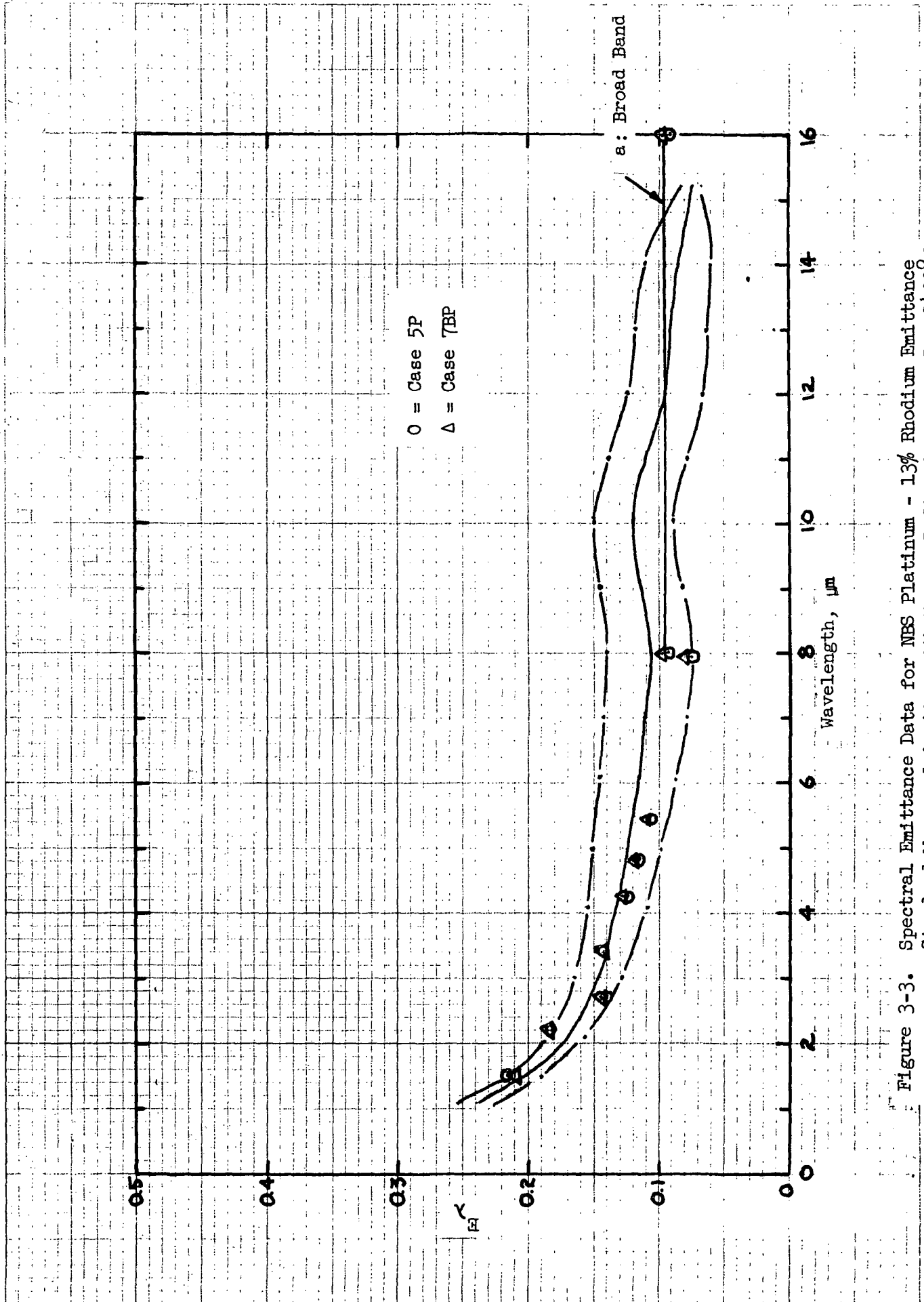
Figure 3-3. Spectral Emittance Data for NBS Platinum – 13% Rhodium Emittance
Standard Measured in Air, with Barnes Radiometer. T = 1400°K

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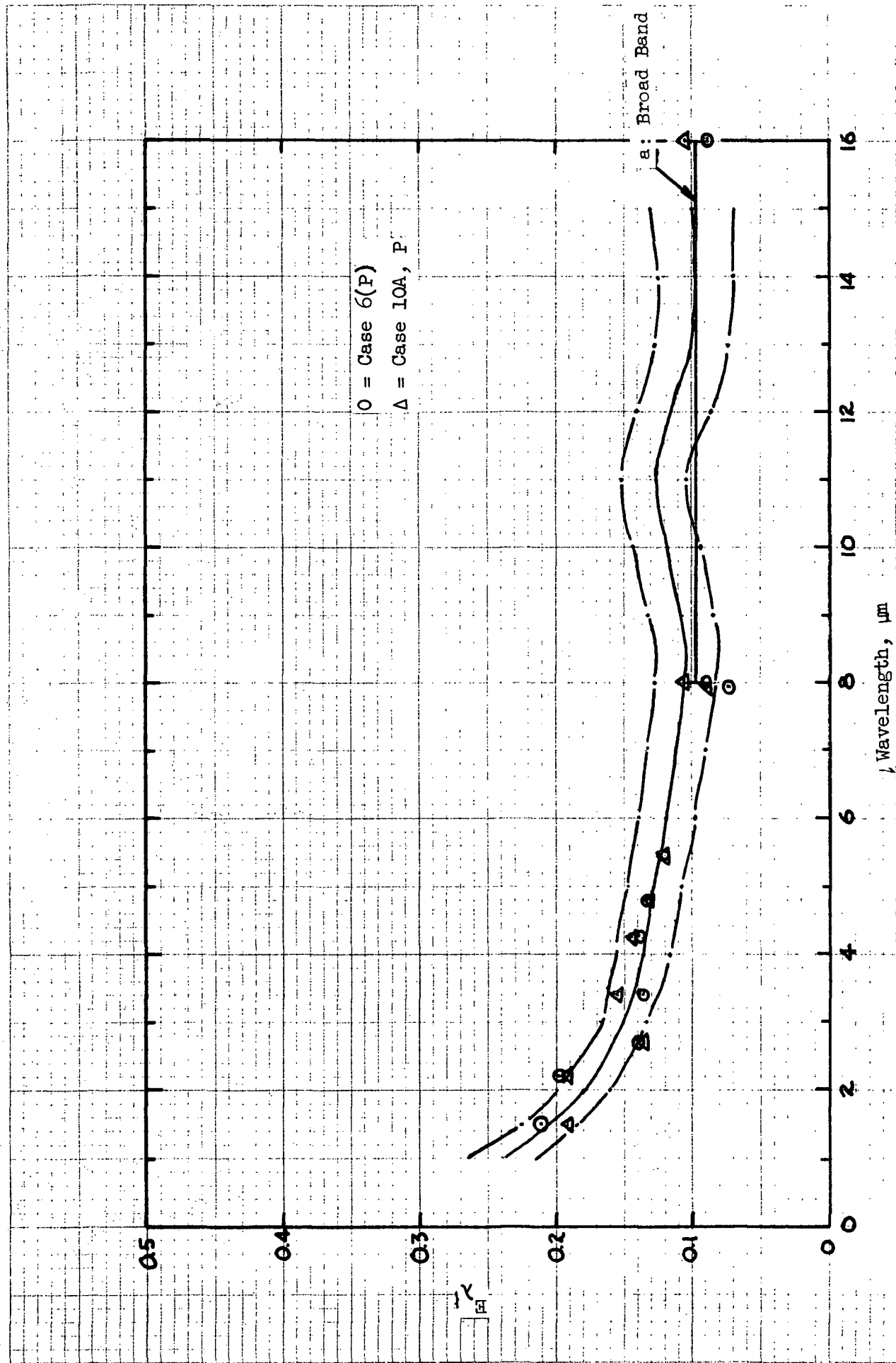
Figure 3-4. Spectral Emittance Data for NBS Platinum - 13% Rhodium Emittance Standard Measured in Air, with Barnes Radiometer. T = 1600°K

Values of E_{TN} computed from thermocouple temperature data are 0.003 higher than those computed using optical pyrometer temperature values at $1100°K$, 0.006 higher at $1400°K$ and 0.013 higher at $1600°K$. True temperatures were calculated from pyrometer brightness temperature using $E_\lambda = 0.32$ and window (KBr) transmittance = 0.91 at 0.65 µm. On this basis it appears that the temperatures measured by the edge thermocouples are $10°$ to $20°K$ low which may be attributed to small gradients at the edge and losses from the leads, both being from convection associated with the cooler boundary layer occurring at atmospheric pressure. Convective losses from the leads are considered to be of more significance than center to edge gradients, due to the higher edge heat transfer coefficient, for the moderately high thermal conductivity Platinum alloy.

3.2 Oxidized Kanthal Results

During the initial measurements on this specimen a temperature controller malfunction caused a momentary (2 to 3 sec) overshoot of power to the strip. This resulted in overheating, and a fracture of the strip occurred approximately 1-1/2-inch from the upper electrode. As there was no visual change in specimen surface appearance, the strip was cut to a 7-inch length and reinstrumented with thermocouples for a new central measuring area. In order to prevent future reoccurrences of this nature a voltage limiter feature of the controller is now used for all tests.

Measured values of E_λ for this material at temperatures of $1100°$ and $1300°K$ are showed in figures 3-5 and 3-6 and are compared with the NBS spectral data for these temperatures. Solid and broken curves represent average and standard deviation values supplied by NBS. The measured values of E_λ are typically 0.03 to 0.05 higher than the upper deviation limit. Temperature measurement error must be considered as one possibility for this behavior; i.e. temperature data from both thermocouple and optical pyrometer readings are lower than the true temperature. A comparison of thermocouple and pyrometer brightness temperatures yields values of E_λ at 0.65 µm of between 0.86 and 0.89 for the $1100°$ and

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TABLE 3-2

TOTAL NORMAL EMITTANCE OF NBS PLATINUM-13%
RHODIUM STANDARD (SRM1409) AS A FUNCTION OF
TEMPERATURE

Nominal Test Temperature ($^\circ$K)	Number of Measurements	Measured E_{TN}	Most Probable Value of E_{TN}*	Calculated E_{TN} From NBS Spectral Data
800	4	$0.0965^{+0.00135}_{-0.0015}$	0.096	0.097 ± 0.013
1100	5	$0.142^{+0.013}_{-0.010}$	0.134	0.146 ± 0.015
1400	6	$0.159^{+0.004}_{-0.003}$	0.156	0.153 ± 0.020
1600	4	$0.174^{+0.005}_{-0.009}$	0.165	0.165 ± 0.020

*For the two lowest temperatures based upon most consistent values for a given number of measurements i.e. for 1100°K three of the five measured values were between 0.132 and 0.135. For the two highest temperatures these are based upon optical pyrometer temperature data.

1300°K temperature conditions. These values are consistent with room temperature values of 0.84 computed from spectral reflectance measurements made on the strip prior to any testing. Spectral emittance data shown in figure 3-5 and 3-6 were computed from pyrometer readings using $0.84 = E_{0.65}$. Thus, the use of the higher value of $E_{0.65}$, from measurements at temperature, would result in a slightly lower temperature and higher emittance.

A second consideration regarding temperature measurement error is the temperature level that would be required to adjust the measurement E_λ's to agree with the NBS data. For example, at the 2.17 μm band of case 6V of figure 2-5 it would have been necessary to use a temperature of 1105°K to obtain the NBS value from the radiometer signal. The temperature actually employed to reduce the signal data to E_λ was 1086°K, and this difference between brightness and true temperature (1105°K for the latter) would require an $E_{0.65}$ of 0.56 which seems unreasonably low for the test temperature level of nearly 1100°K. In

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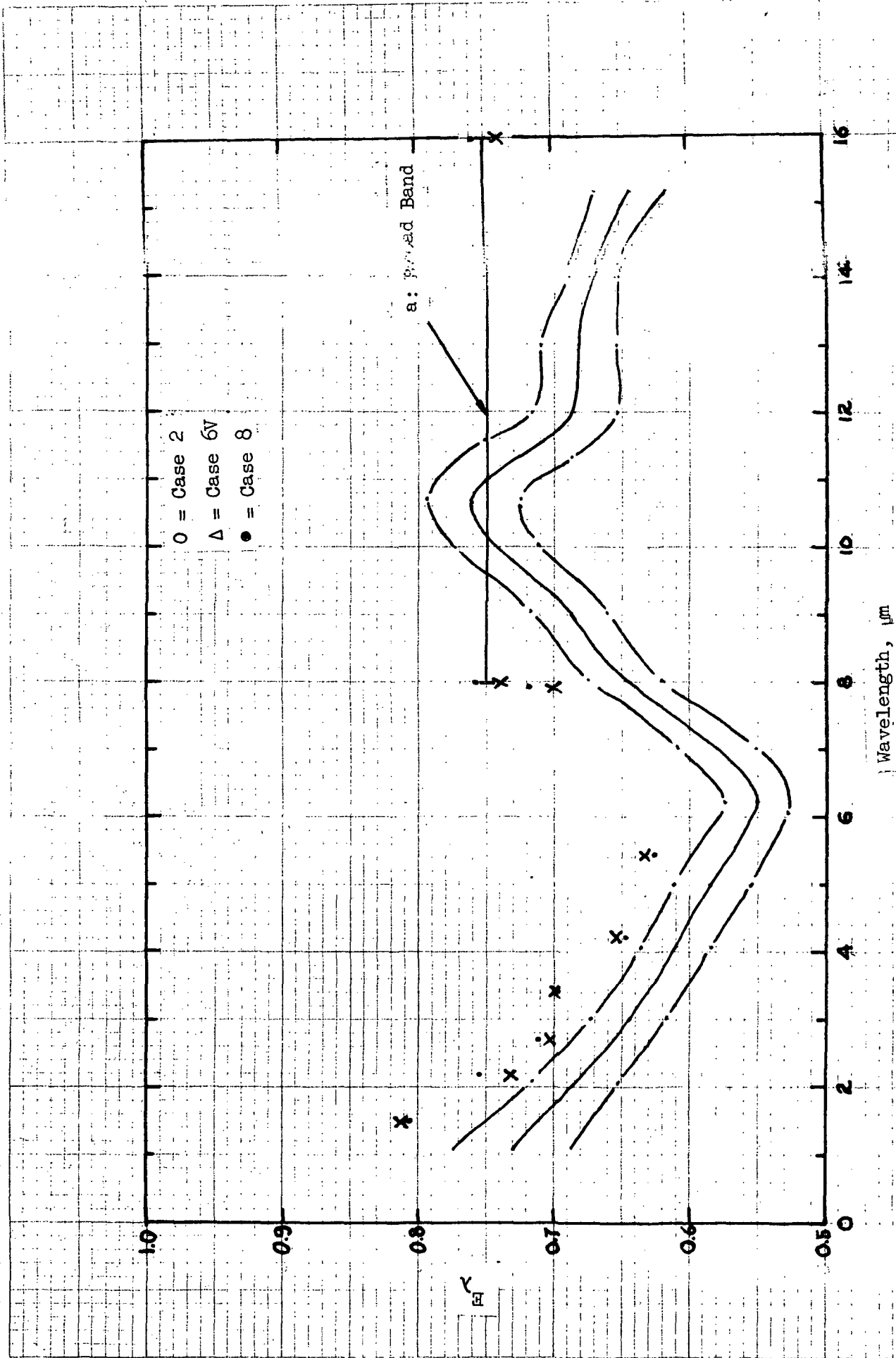
Figure 3-5. Spectral Emittance Data for NBS Oxidized Kanthal Emittance Standard Measured in Air, with Barnes Radiometer. T = 1100°K.

O = Case 2
Δ = Case 6V
● = Case 8

a: Banded Band

Wavelength, μm

E_λ
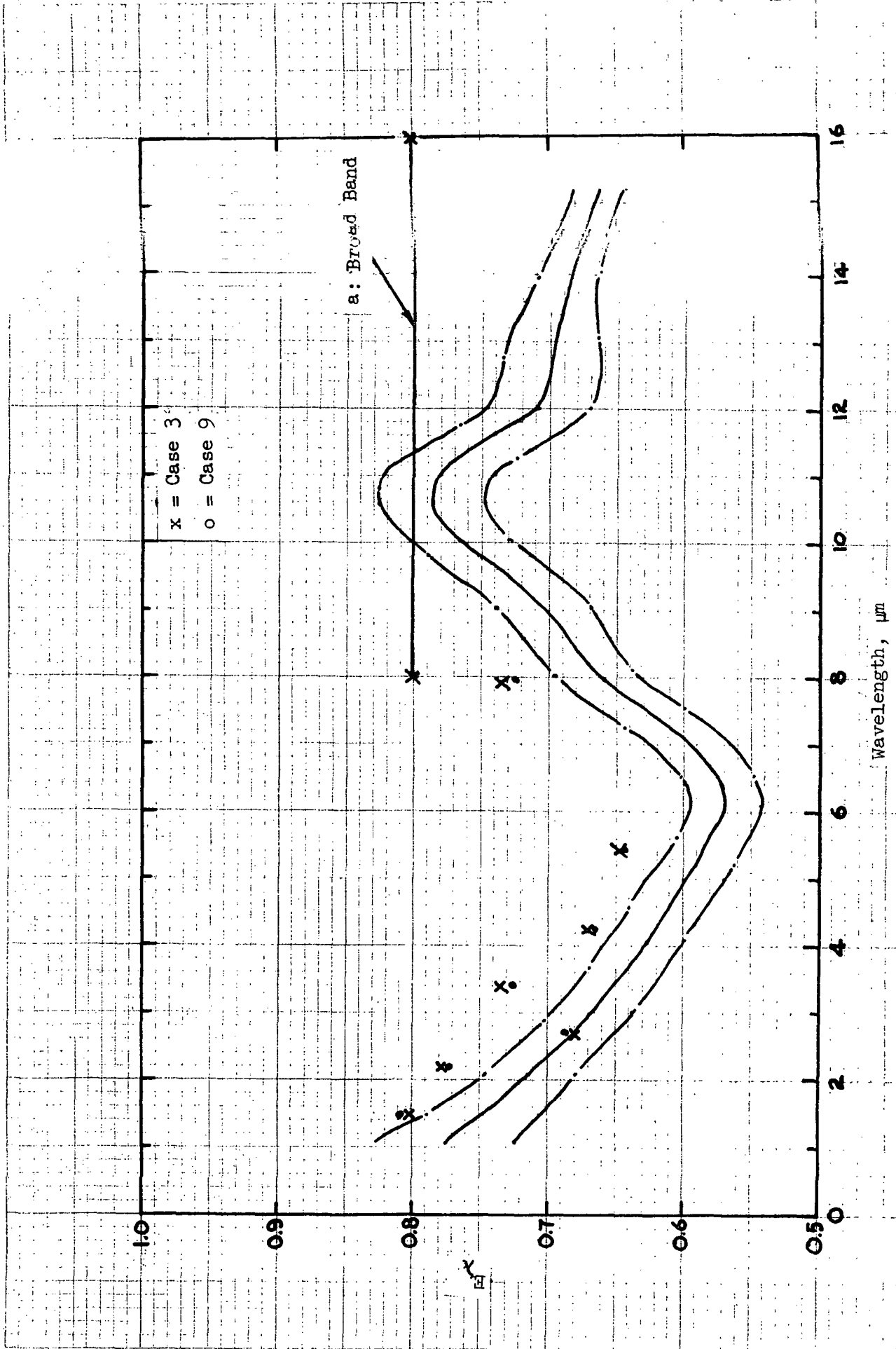
Figure 3-6. Spectral Emittance Data for NBS Oxidized Kanthal Emittance Standard Measured in Air, with Barnes Radiometer. $T = 1300^{\circ}K.$

x = Case 3
o = Case 9

a: Broad Band

Wavelength, μm

E_λ

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a further attempt to resolve the differences in E_λ, room temperature spectral reflectance measurements were performed on a 3/4-inch square segment taken from one end of the standard strip. Spectral emittance data from this method (E_λ = 1 - ρ_λ) are shown in figure 3-7 together with the NBS data at elevated temperatures. The NBS values show a slight increase in E_λ with increasing temperature (as do the radiometer data), and yet the room temperature reflectance measurements yield E_λ greater than the elevated temperature NBS data from 2 to 6.5 μm.

Considering the possible temperature measurement errors and the spectral reflectance data it appears that the spectral emittance of the standard strip is slightly greater than the NBS data supplied with the specimen. The possibility of contamination of the surface during instrumentation and installation in the apparatus must of course be considered, but no evidence to support this was found as care was taken to avoid handling of the strip except at its ends.

Total normal emittances were calculated from the radiometer data at four temperatures, and these are shown in Table 3-3 together with E_{TN} values computed from the NBS spectral data using average values. The standard deviations would add $\pm$ 0.02 to 0.03 to these values. Total hemispherical emittances were determined calorimetrically at three lower temperatures for the vacuum condition, and these values are in reasonable agreement with the total normal data. The higher measured emittances again do not appear to be due to temperature measurement errors but rather are representative of the actual specimen which was measured in this program, E_{TN} 0.05 to 0.07 greater than computed from the NBS spectral data.

From the results obtained on the Platinum-Rhodium specimen the values of E_λ and E_{TN} determined in the test apparatus should be within 0.02 to 0.03 of the true surface properties. Thus, this method is considered to be of sufficient accuracy for evaluation of the shuttle materials.

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TABLE 3-3

TOTAL EMITTANCE OF NBS OXIDIZED KANTHAL
STANDARD (SRM 1427) AS A FUNCTION OF
TEMPERATURE

Test Temperature ($^{\circ}$K)	Test No.*	Measured Value		Calculated E_{TN} from NBS Spectral Data	Remarks
		E_{TN}	E_{TH}		
645	4V	0.648	0.667	–	In vacuum
805	5V	0.661	0.636	{ 0.60	In vacuum
797	7	0.697	–		Air
1100	2	0.731	–	0.64	Air
1086	6V	0.728	0.711	{	Vacuum
1093	8	0.723	–		Air
1320	3	0.724	–	{ 0.67	Air
1296	9	0.723	–		Air

*Denotes sequence of testing, i.e. 2, 3, 4V, etc.

Figure 3-7. Spectral Emittance of NBS Oxidized Kanthal Standard No. 1427.
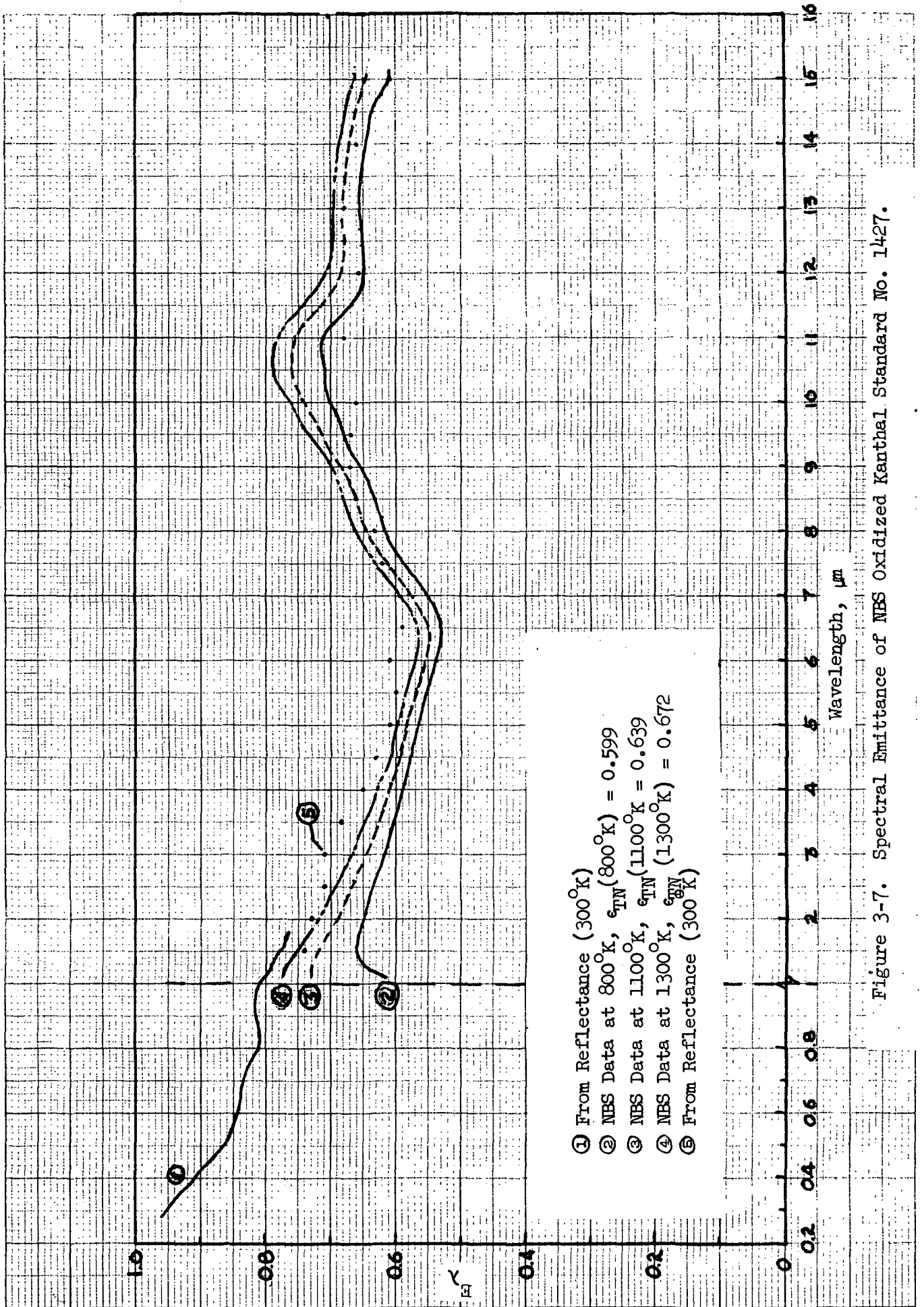
① From Reflectance (300°K)
② NBS Data at 800°K, $\varepsilon_{TN}(800°K) = 0.599$
③ NBS Data at 1100°K, $\varepsilon_{TN}(1100°K) = 0.639$
④ NBS Data at 1300°K, $\varepsilon_{TN}(1300°K) = 0.672$
⑤ From Reflectance (300°K)

Wavelength, μm

ε_λ

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Section 4

SAMPLE PREPARATION METHODS
AND
CHARACTERISTICS

During the past three months sample preparation procedures have been investigated and evaluated in terms of the emittance characteristics obtained. Oxidation procedures for the Rene 41, HS-188 and TD Ni-Cr alloys were selected and oxidized test strips were prepared for the emittance-stability tests to be performed in this program. These procedures and the initial sample characteristics are described in this section of the report.

The high-temperature/high-strength alloys selected for testing in this program are listed in Table 4-1 along with pertinent information provided by the supplier on the as-received condition of each alloy. Samples of the two columbium alloys are to be coated with commercial, oxidation-resistant coatings and their initial emittance characteristics will be described in a later report. Samples of the remaining three alloys were oxidized in the laboratory using a high temperature tubular furnace in conjunction with an air-drying system. A description of the furnace and of the oxidation exposures and results for each alloy is given in the following subsections.

4.1 Oxidation Apparatus

A schematic of the high temperature furnace and air-drying system is shown in Figure 4-1. Pressurized air was regulated to a nominal flow rate of 7 CFH through the system as indicated by a gas flow meter. Excess moisture was removed from the air by passing it first through a cooled (dry-ice/alcohol) flask and then through a 12-inch column of molecular-sieve drying agent. The dry air was then introduced into a 3-ft. long by 1 3/4-in. ID quartz tube which ran through the center of the furnace and extended 6 inches from both ends of the furnace. This tube served as a clean housing for each sample during its oxidation exposure and was plugged at each end with unfired lava plugs with air feed-thru tubes. A wad of glass wool was stuffed into the air-entry end of the tube to aid in diffusing the air flow through the tube.

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TABLE 4-1

High Temperature Alloys for Emittance Tests

| Alloy	Supplier	Heat No.	Strip Dimensions	As Received Condition
Haynes Alloy R-41 (Rene 41)	Stellite Division Cabot Corp.	2490-1-8218	12"x1.08" x 0.040"	Smooth dull grey surface.
Haynes Alloy No. 188 (HS-188)	Stellite Division Cabot Corp.	1880-0-0159	12"x1.03"x0.041"	Smooth bright surface. Annealed and cooled in dry hydrogen atmosphere.
Ta Ni-Cr Alloy (Ni-20% Cr-2% ThO$_2$)	Fansteel Inc.	TC 3277	12"x1.06"x0.043"	Bright clean surface. Rolling direction $\perp$ to length of strips. Stress relieved.
Columbium Alloy Cb-752 (Cb-9.5%W- 2.6% Zr)	Teledyne Wah Chang Albany	770036	12"x1.01"x0.041"	Smooth clean surface. Annealed. 100% recrystallized. Avg. ASTM. Grain size No. 8
Columbium Alloy C-129Y (Cb-9%W-10% Hf- 0.1%Y)	Teledyne Wah Chang Albany	572068	12"x1.01"x0.041"	Smooth, clean surface. Annealed. 100% recrystallized. Avg. ASTM. Grain size No. 9

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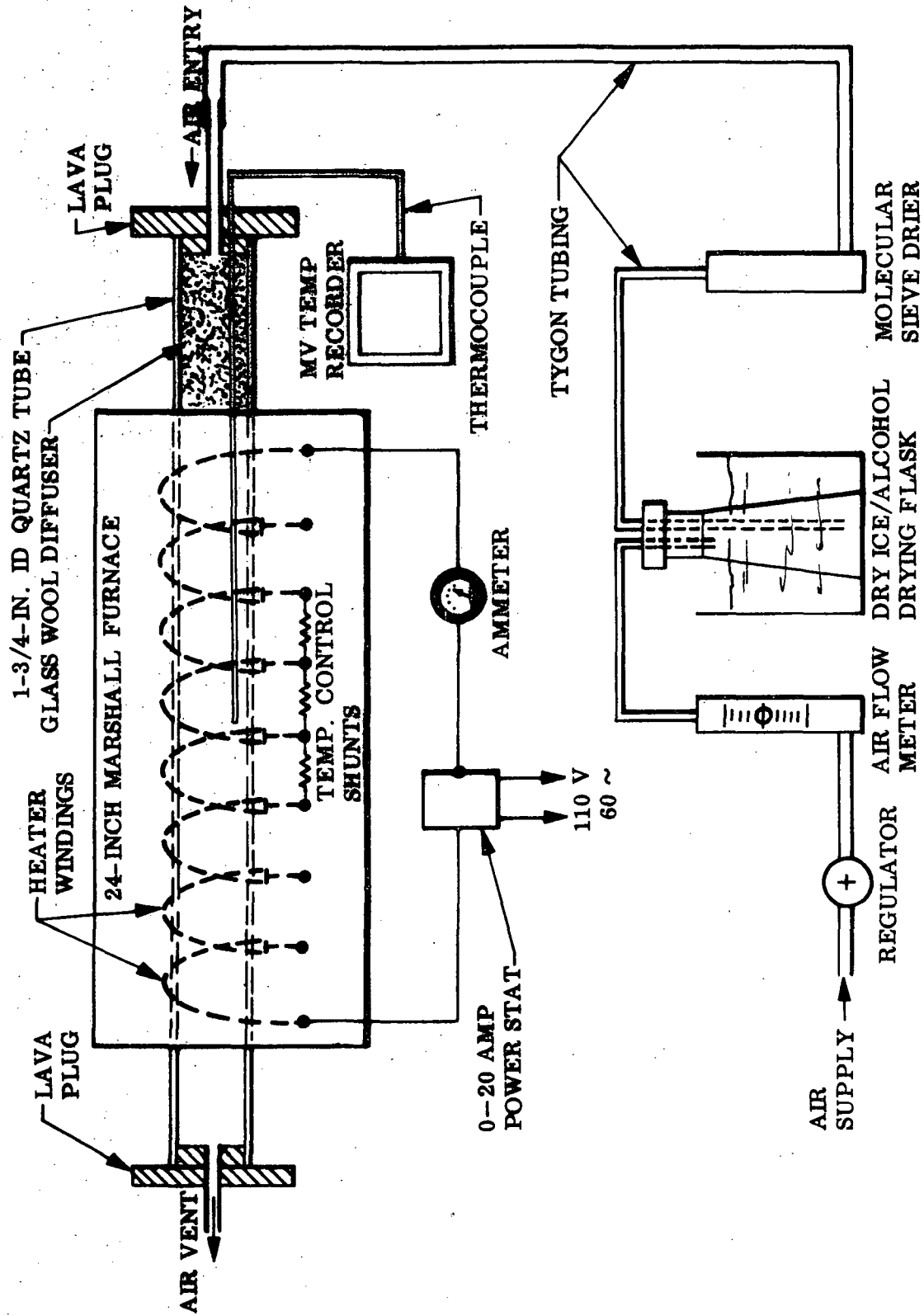
Figure 4-1. Schematic of Sample Oxidation Apparatus

Samples strips or disks were introduced into the tube by removing the air-vent plug, sliding the samples to the hot central region of the furnace with a quartz rod, and then replacing the vent plug. Disk samples were supported horizontally on a small boat fashioned from quartz and high purity alumina tubing which was tied together with platinum wire. Strip samples were supported horizontally by tying each end of the strip to a small quartz boat with platinum wire. These supports were of such size as to position the top surface of each sample near the longitudinal center-line of the furnace. At the end of each oxidation exposure, the air-vent plug was removed and the samples were dragged out of the furnace by catching a quartz hooked-rod around one of the quartz support boats. The samples were then allowed to cool in ambient room air.

The Marshall furnace is a 24-in. long by 10-in. diameter insulated furnace with a 2-in. diameter tubular hole through its center. The heater windings are heavy Kanthal A-1 strips with a maximum temperature rating of $2400^{\circ}F$. External shunt posts are accessible along the length of the furnace which permit localized control of the temperature gradient along the length of the furnace. For these exposures, 2-ohm   shunts were connected in parallel with the heater windings around the center 6 inch region of the furnace in order to provide a longer uniform temperature zone for the 12-inch long sample strips. Electrical power to the furnace was controlled manually with a 0 to 20 ampere Powerstat and an ammeter in series with the heater windings.

Furnace temperatures were monitored with an Pt/Pt-13% Rh thermocouple which could be positioned anywhere along the length of the furnace inside the quartz support tube. The thermocouple leads were protected with 1/8-inch diameter, two-hole, alumina sleeving with three flexible joints along its 30-inch length to facilitate sliding the thermocouple through a small hole in the lava plug at the air-entry end of the furnace. The thermocouple junction was an exposed bead at the end of the sleeving which provided a fast response ($< 1/2$ min.) to changes in temperature along the length of the furnace. The normal location of the thermocouple was at the center of the furnace and the temperature at this point was controlled to the nominal oxidation temperature used for each sample. Temperature-time records for each sample exposure were recorded with a calibrated millivolt strip chart recorder.   20

4.2 Methods

Due to the large mass of the furnace and its slow response to input power changes, several hours were required to bring the system to a desired steady-state temperature. For each operational temperature the furnace was brought to steady state and maintained there for at least 1 hour before starting a sample oxidation exposure. During this period the proper input power (i.e. current) level was determined and only minor adjustments were required thereafter to maintain a constant furnace temperature. The introduction of a strip sample into the furnace resulted in a short-duration temperature drop of from 50 to $100^{\circ}F$ at the center of the furnace, but the steady-state condition was recovered within a 3 to 5 minute period.

The 12-inch long strip samples were oxidized one at a time due to the limited working volume within the furnace, but 1-inch diameter disk samples were oxidized four or five at a time. Disk samples were used to evaluate different exposure-time effects on emittance and to determine the sample-to-sample emittance variations for a particular exposure condition. The disks were punched from the same sheet of material that the strips were cut from, and after oxidation are assumed to be representative of the center 6 to 8-inch region of each strip sample with the same oxidation exposure.

Exposure temperatures, time durations, and atmosphere conditions for each alloy were selected on the basis of recommendations from MSFL which were as follows:

o For René 41: a) Resolution heat treat at $2050^{\circ}F$ for 1/2 hr. in a controlled atmosphere of Argon or vacuum, then cool rapidly.

b) Age at $1650^{\circ}F$ for 2 hrs. in a controlled argon or vacuum atmosphere, then

c) Oxidize at $1650^{\circ}F$ for 2 hrs. in air.

o For HS-188: Oxidize at $1900^{\circ}F$ for a short time in dry air.

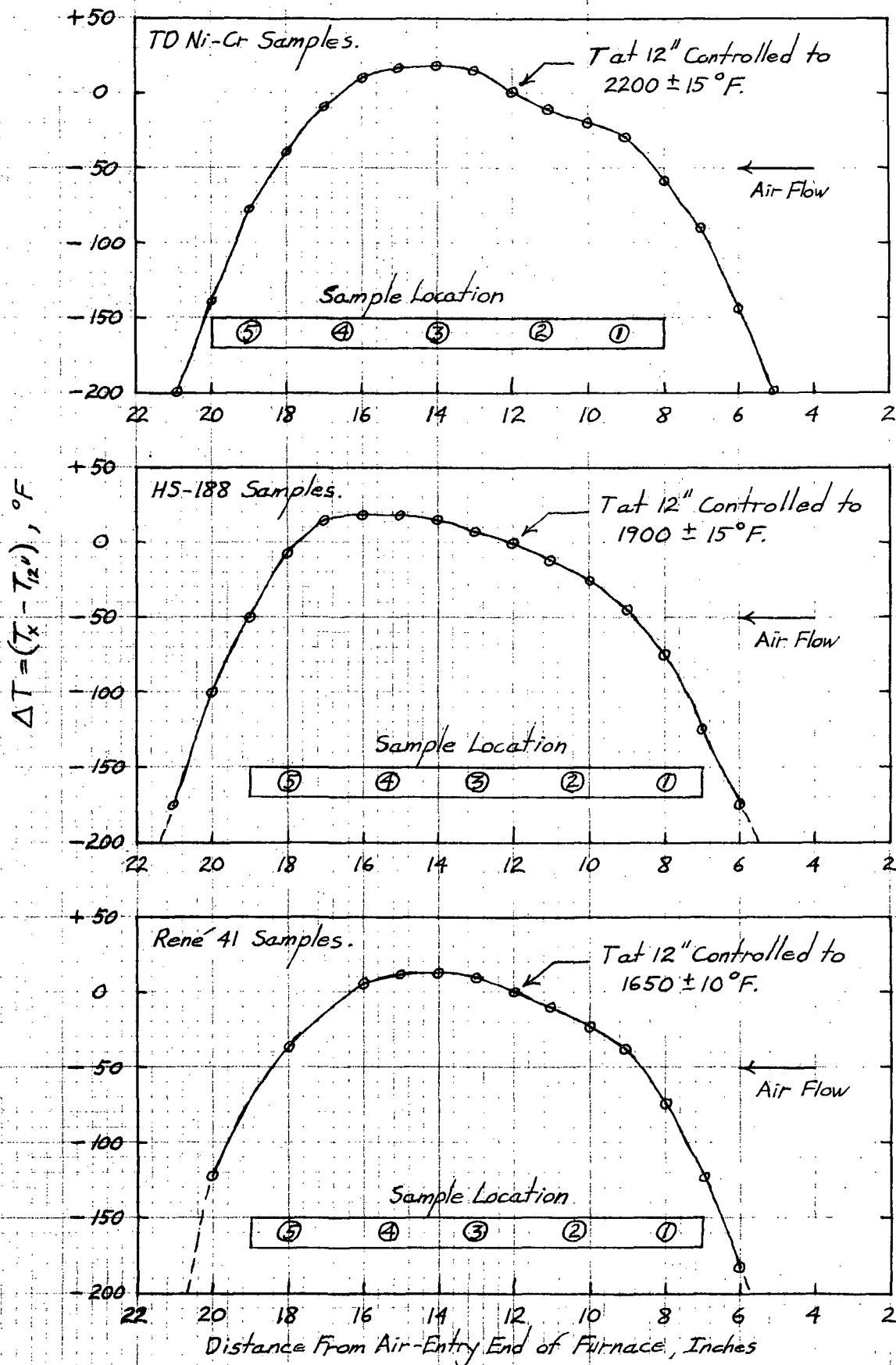o For TD Ni-Cr: Oxidize at $2200^{\circ}F$ for 1/2 to 1 hr. in air.

Since specific oxidation exposure times were not stated for the HS-188 and TD Ni-Cr alloys, times of 1, 2 and 3 hours were evaluated for the HS-188 alloy and times of 1/2, 1 and 1-1/2 hrs were evaluated for the TD Ni-Cr alloy. On the basis of spectral reflectance results for disk samples with these exposures, the 3 hr. exposure time was selected for the HS-188 alloy and a 1 hr. exposure time was selected for the TD Ni-Cr alloy. These results are presented in the next subsection.

Sample cleaning procedures were kept simple in order to obtain results which would be representative of the as received condition of each alloy. The use of strong pickling acids or caustic washes was avoided so as to not alter the surface chemistry of the materials. Also, the use of surface roughening methods to improve the initial emittance characteristics of the materials was not investigated. Prior to oxidation, each sample was lightly scrubbed with steel wool, Boraxo and warm water, rinsed, and then wiped with an alcohol-saturated Kimwipe. This procedure resulted in uniformly clean, grease-free surfaces for each sample.

Resolution heat treatment of the René 41 samples at 2050°F was accomplished using the same system as described in Figure 4-1 with the exception that argon instead of compressed air was connected to the inlet regulator. The argon flow rate was maintained at 5 CFH during this treatment and after 1/2 hr., the samples were 'quick-cooled' in air by rapidly removing them from the furnace with a pair of tongs. This treatment results in the formation of a light oxide film on the surface of the samples which was strongly adherent to the substrate, therefore no attempt was made to reclean the surface before the aging and oxidation exposure. No pre-oxidation heat treatments were given to the HS-188 and TD Ni-Cr alloys except those performed by the suppliers and noted in Table 4-1.

Typical temperature gradients along the length of the furnace for each oxidation exposure temperature are shown in Figure 4-2. These temperature differences were determined from thermocouple readings of the temperature distribution inside the quartz support tube during actual sample oxidation exposures.

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Figure 4-2. Furnace Temperature Gradients During Oxidation of Test-Strip Samples

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Sample locations within the furnace are also shown on this figure. The sample
strips were positioned 1 or 2 inches towards the air-vent end of the furnace
because the air flow shifted the maximum temperature region inside the quartz
support tube towards that end. The figure shows that temperatures were uniform
to within $\pm 25°F$ of the nominal control temperature over the central 6-inch
region of each strip sample. The circled numbers on each sample location sketch
indicate areas where total reflectance measurements were made after the sample
was oxidized to indicate the uniformity of the oxide coating from end-to-end
and on both sides of each sample.

4.3 Results

Five test strip samples and three witness disk samples of each alloy have
been prepared by the methods just described. The disk samples have been used
to obtain spectral reflectance measurements which indicate the initial emittance
characteristics of each sample. The visual appearance of the oxidized strip
samples is as follows:

o Rene 41: Uniform smooth, dull-slate-grey colored oxide on both sides
   and from end-to-end with a slight blush of lighter, cream-colored oxide
   at each end.
o HS-188: Uniform smooth, dark-blue-grey colored oxide on both sides and
   from end-to-end
o TD Ni-Cr: Uniform smooth, dark-blue-grey colored oxide on both sides
   and from end-to-end with a few, randomly scattered black specks on the
   order of 0.025-in. in diameter.

All of the oxides appear to be tough (i.e., abrasion resistant) and strongly
adherent to the subsrate metal.

Total room-temperature emittance values for the oxidized strip samples at
the five location points indicated previously in Figure 4-2 are shown in Table
4-2. These values were determined from total reflectance measurements with a
Gier-Dunkle Model DC-100 reflectometer. The emittance values are average values

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TABLE 4-2

Variations in Room Temperature Total Emittance for Oxidized Sample Strips

Sample/Side	Location Point Along Strip[*]				
	①	②	③	④	⑤
Rene 41					
Top	0.58 ± .08	0.59 ± .04	0.62 ± .03	0.56 ± .05	0.54 ± .04
Bottom	0.54 ± .05	0.56 ± .04	0.57 ± .04	0.56 ± .04	0.53 ± .05
HS-188					
Top	0.37 ± .03	0.45 ± .02	0.47 ± .01	0.47 ± .02	0.44 ± .02
Bottom	0.38 ± .02	0.45 ± .02	0.47 ± .01	0.47 ± .01	0.44 ± .03
TD Ni-Cr					
Top	0.43 ± .03	0.42 ± .01	0.42 ± .01	0.42 ± .01	0.43 ± .01
Bottom	0.43 ± .01	0.43 ± .01	0.43 ± .01	0.43 ± .01	0.44 ± .01

[*]See Figure 4-2.

at each location for the five sample strips of each alloy that were oxidized. The variability values indicate the range between maximum and minimum readings obtained at that location for each set of five samples. The measurements indicate that the TD Ni-Cr samples have the most uniform and least variable emittance, and the emittance of the Rene 41 samples is the least uniform with the highest variability between samples.

Results of room temperature spectral reflectance measurements for the oxidized disk samples are shown in Figures 4-3 through 4-7. These measurements were made with a Cary Model 14 spectroreflectometer at wavelengths between 0.28μ and 1.8μ; and with a Gier-Dunkle Model HC-300 heated cavity reflecto-meter at wavelengths between 2μ and 24μ. Predicted values of total normal emittance at five temperatures up to 2000°F, determined by numerical integra-tion of the room temperature spectral curves shown in the figures, are listed in Table 4-3. These predicted values are based on the assumption that the spectral emittance characteristics of the sample surface do not change with temperature. For these materials they should be approximately correct up to the temperatures at which the oxides were formed, and are probably somewhat low since the E_λ values for most oxides tend to increase slightly with increasing temperature. The above remarks apply only to the initial characteristics of the samples and become invalid as soon as changes in the thickness or chemical composition of the surface oxide occurs.

Figure 4-3 shows the variations in spectral emittance obtained for two of the heat treated, aged and oxidized Rene 41 disk samples. The emittance char-acteristics of the unoxidized as received material is also shown for comparison. The total normal emittance values in Table 4-3 for the oxidized Rene 41 samples are average values for two curves shown in Figure 4-3. With these spectral characteristics, the total emittance of the oxidized Rene 41 samples should in-crease with temperature from about 0.56 at room temperature to about 0.75 at 1600°F.

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Figure 4-3. Room Temperature Spectral Reflectance of as Received and Oxidized Rene 41 Samples

① As Received.
② #R2: Oxidized 2 hrs, 1650°F.
③ #R3: Oxidized 2 hrs, 1650°F.

Wavelength, μ

R_λ

27
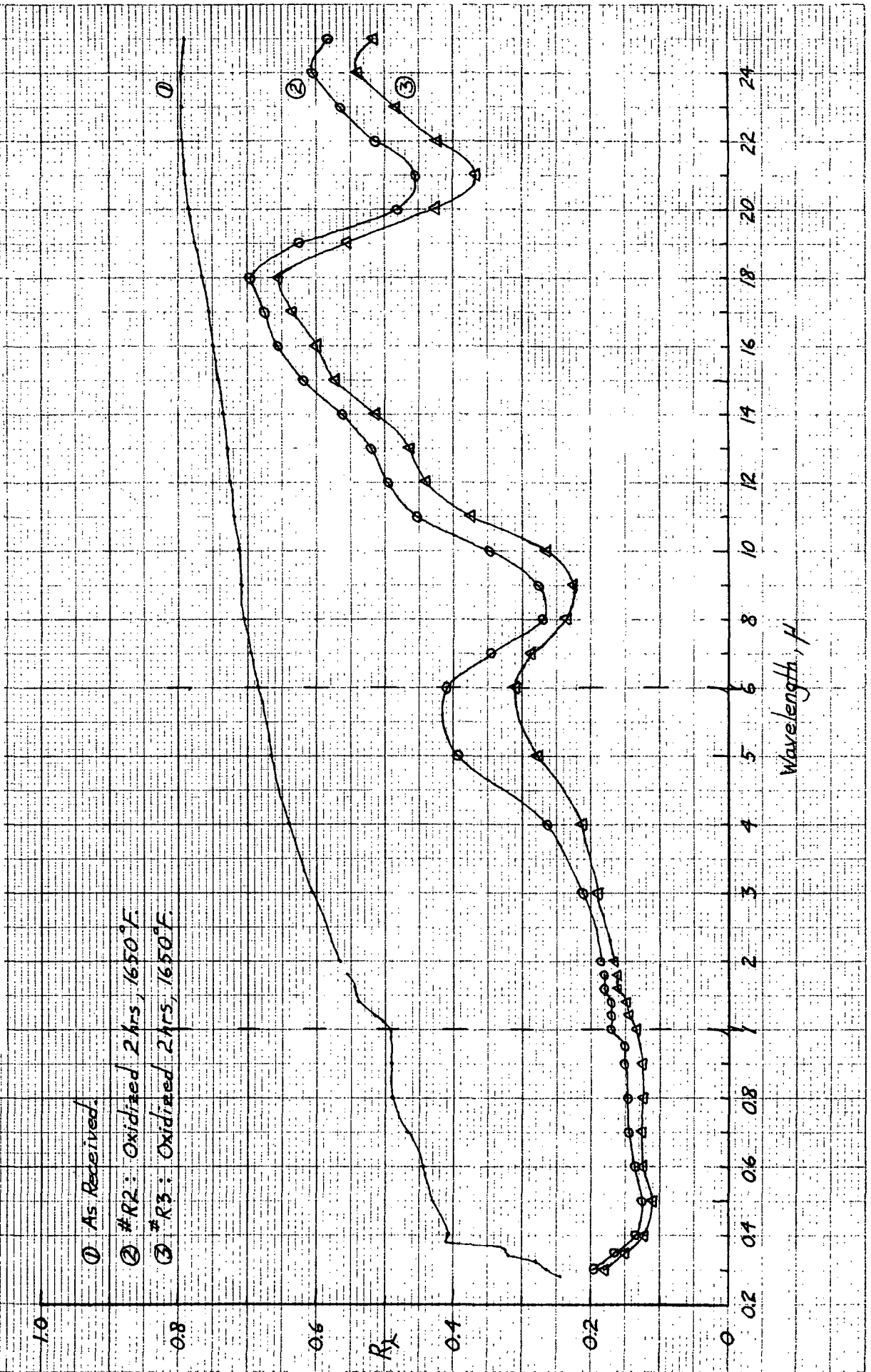
Figure 4-4. Room Temperature Spectral Reflectance of as Received and Oxidized HS-188 Samples

① As Received.
② #H1, Oxidized 1 Hr. at 1900°F.
③ #H2, Oxidized 2 Hrs. at 1900°F.
④ #H3, Oxidized 3 Hrs. at 1900°F.

R_λ

Wavelength, μ

28
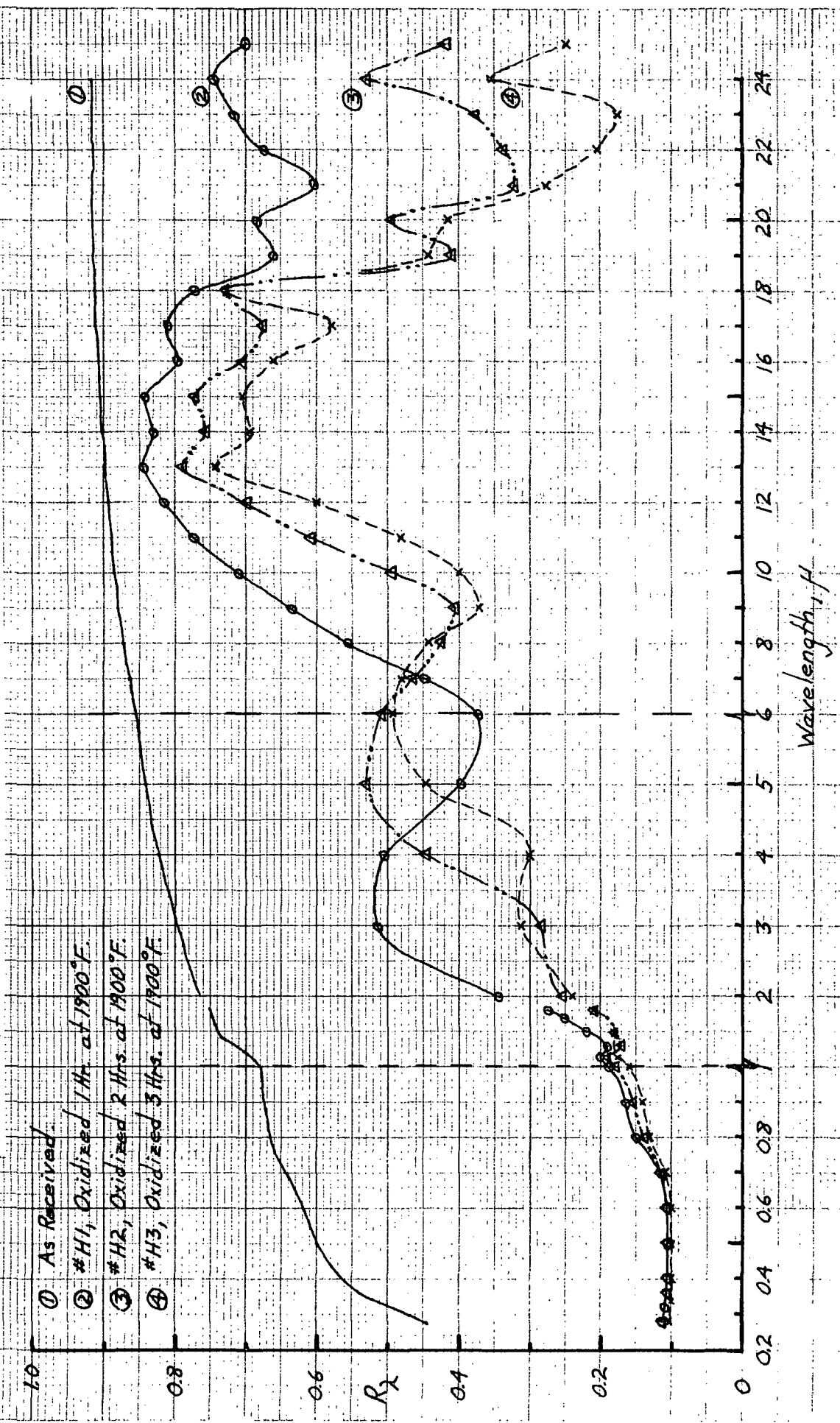
Figure 4-5. Variations in Spectral Reflectance for Four HS-188 Samples
Oxidized for 3 Hours at 1900°F

① Average for H4, H5, H6.
② H3

29
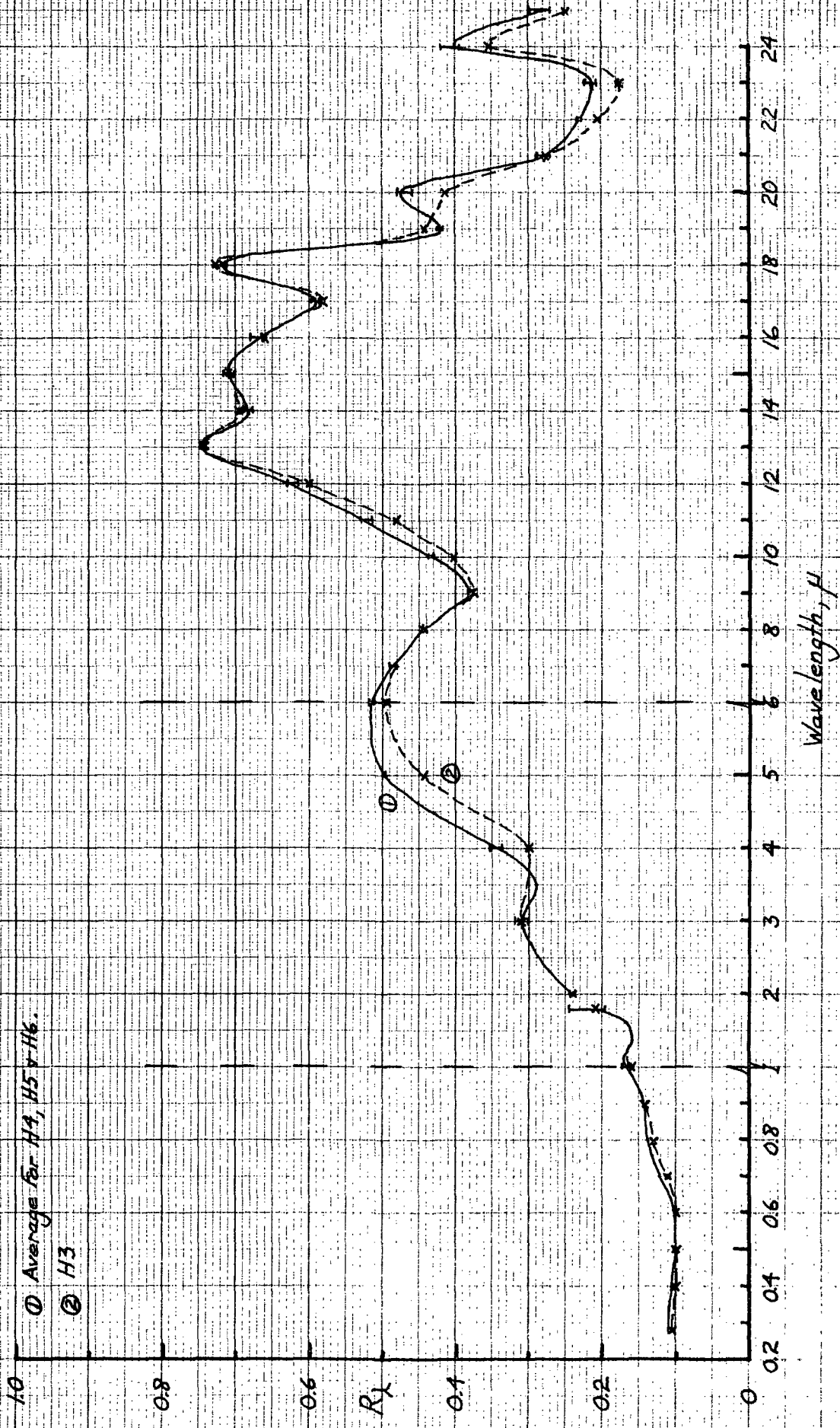
Figure 4-6. Room Temperature Spectral Reflectance of as Received
and Oxidized TD Ni-Cr Samples

① As Received
② #A1, Oxidized 1½ Hr. at 2200°F.
③ #A2, Oxidized 1 Hr. at 2200°F.
④ #A3, Oxidized 1½ Hrs. at 2200°F.

Wavelength, μ

R_λ
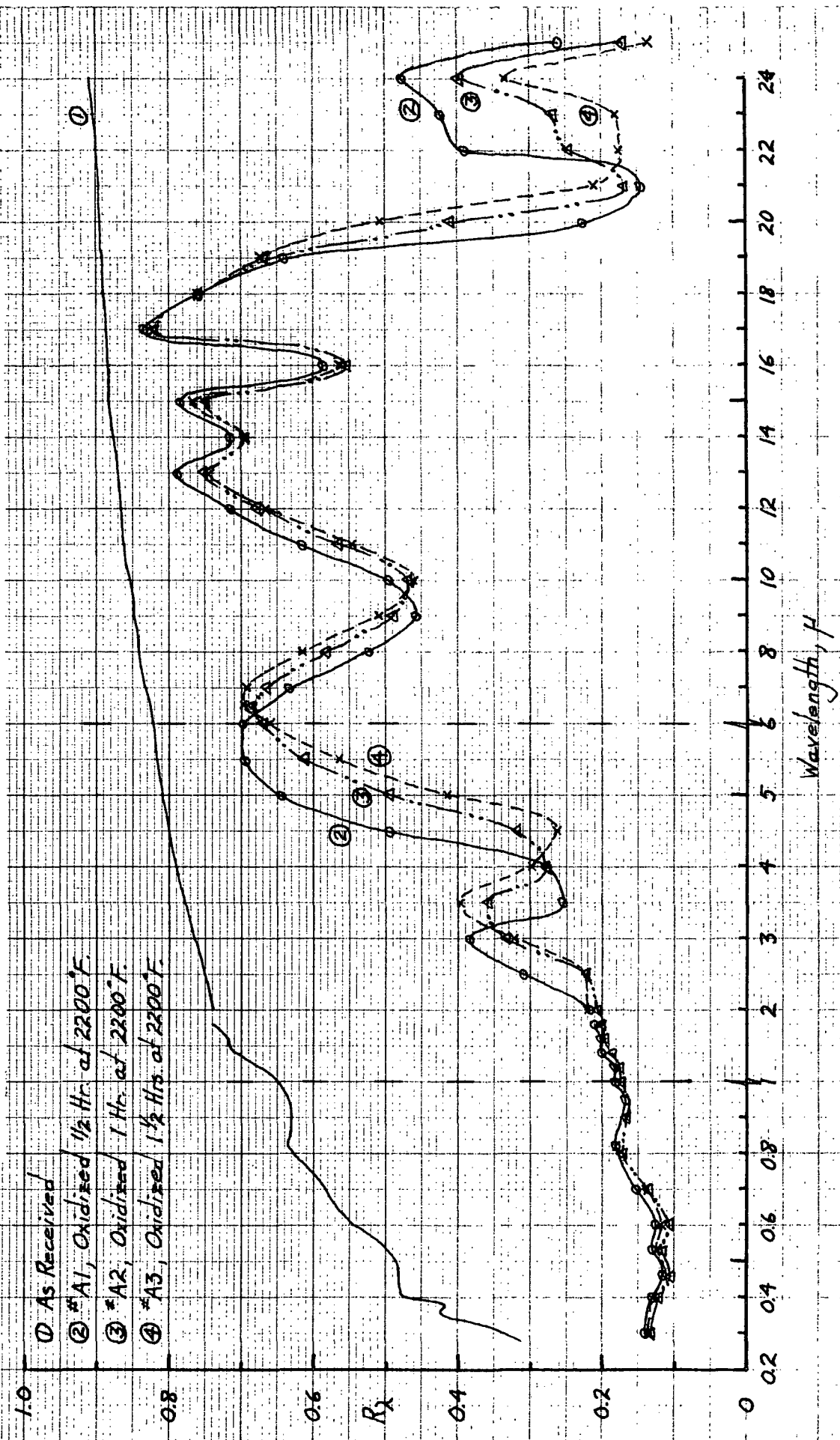
Figure 4-7. Variations in Spectral Reflectance for Four TD Ni-Cr Samples Oxidized for 1 Hour at 2200°F

① *Average for A5, A6 & A7*

② *A2*

Wavelength, μ

R_λ
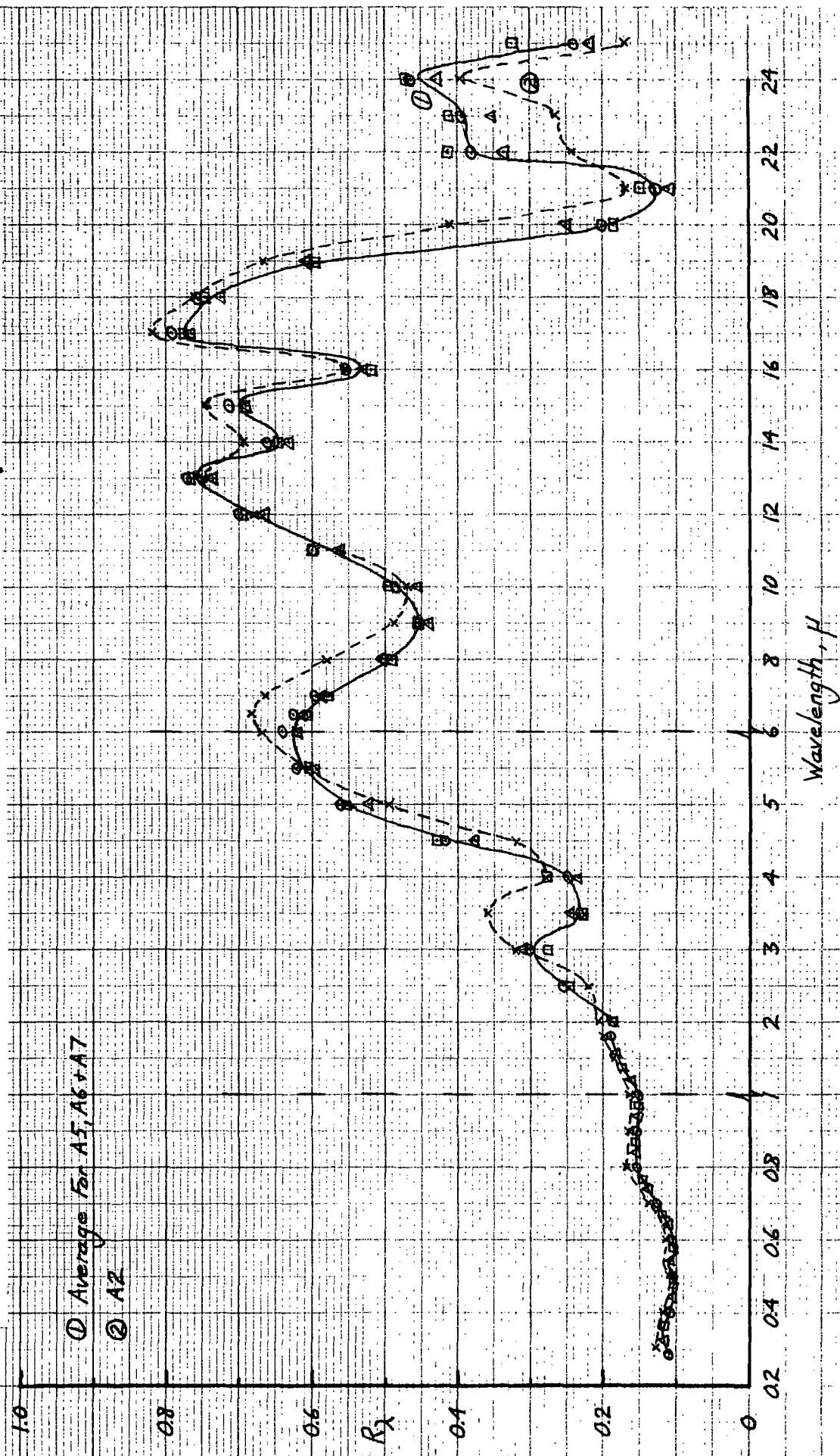
TABLE 4-3

Predicted* Total Normal Emittance Values at
Five Temperatures for Oxidized René 41,
HS-188 and TD Ni-Cr Samples

Alloy	Oxidation Exposure	$\epsilon_{TN}(T)$				
		T = 70°F	T = 500°F	T=1000°F	T=1500°F	T=2000°F
René 41	As Received	0.26	0.30	0.34	0.36	0.39
"	1650°F/2 Hrs.	0.54	0.64	0.70	0.74	0.77
HS-188	As Received	0.10	0.13	0.16	0.18	0.20
"	1900°F/ 1 Hr.	0.30	0.42	0.49	0.53	0.57
"	1900°F/2 Hrs.	0.46	0.49	0.55	0.60	0.65
"	1900°F/3 Hrs.	0.54[1]	0.55	0.60	0.64	0.68
TD Ni-Cr	As Received	0.13	0.16	0.19	0.21	0.23
"	2200°F/1/2 Hr.	0.46[1]	0.45	0.51	0.58	0.63
"	2200°F/1 Hr.	0.48[1]	0.47	0.54	0.61	0.67
"	2200°F/1-1/2 Hrs.	0.49[1]	0.48	0.55	0.62	0.67

*Calculated from room temperature spectral reflectance data.

[1]These values may be too high due to lower ϵ_λ values at $\lambda \geq 25\mu$ than were assumed. See discussion at the end of Section 4.

Figure 4-4 shows the spectral reflectance characteristics of the HS-188 disk samples that were oxidized for 1, 2 and 3 hours, and of the as received material. The progression of the significant reflectance peak that occurs between 3.5μ and 6μ towards longer wavelength with increasing exposure time indicates that the oxide coatings are probably not thick enough to be optically opaque at these wavelengths, and the occurrence of the peak is probably due to a film-thickness/substrate-reflectance interference condition. On the basis of these measurements, the oxidation exposure time of 3 hrs. was selected for this alloy in order to obtain maximum initial emittance characteristics along with a more stable, optically thick oxide layer.

The curves in Figure 4-5 are for four individual HS-188 disk samples oxidized for 3 hrs. at 1900°F and are shown to indicate the good repeatability of emittance characteristics that was obtained for these samples. The solid curve is a plot of the average ϵ_λ values for three samples oxidized on the same day, and the vertical bars through this curve indicate the maximum and minimum values obtained. The dashed curve is for the first disk sample oxidized for 3 hrs. which was prepared several weeks earlier. The differences are minor and can be attributed to slightly different furnace conditions. With these spectral characteristics, the total emittance of the oxidized, HS-188 strip samples should increase with temperature from about 0.48 at room temperature to about 0.68 at 2000°F.

Figure 4-6 shows the spectral reflectance characteristics of the TD Ni-Cr disk samples that were oxidized for 1/2, 1 and 1-1/2 hrs., and of the as received material. The change in location of the major reflectance peak occurring around 6μ again suggests that these oxide film thicknesses are not optically opaque at this wavelength; but the effect of increased exposure from 1 to 1-1/2 hrs. on the total emittance characteristics of the samples (see Table 4-3) is small, consequently the oxidation exposure time of 1 hr. was selected for this alloy.

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The curves in Figure 4-7 are for four separate TD Ni-Cr disk sample oxidized for 1 hr. at 2200^OF. and are shown in indicate the good repeatability of emittance characteristics that was obtained for these samples. The solid curves are for three samples that were oxidized on the same day and the dashed curve is for the first sample oxidized for 1 hr. which was prepared several days earlier. As with the HS-188 samples, the differences are small and can be attributed to slightly different furnace conditions for the oxidation exposures. With these spectral characteristics, the total emittance of the oxidized TD Ni-Cr samples should increase with temperature from about 0.44 at room temperature to about 0.70 at 2200^OF.

A comparison of the calculated ϵ_{TN} (T = 70^OF) values for the oxidized HS-188 and TD Ni-Cr samples listed in Table 4-3 with the DB-100 measured values for the oxidized strips listed in Table 4-2 shows the calculated values to be considerably higher than the measured values. The reason for this discrepancy is believed due to higher reflectance (lower emittance) of these oxides in the spectral region beyond 25μ than was assumed for the calculated ϵ_{TN} values. Approximately 18% of the room temperature black body energy distribution is contained in this far-IR region. If an average ϵ_λ value of 0.4 is assumed for this region for HS-188 sample H3 and for TD Ni-Cr samples A1, A2 and A3 instead of the ϵ_λ values at $\lambda = 25\mu$ which were used in the ϵ_{TN} calculations, then the calculated values are lower and in good agreement with the DB-100 readings. The calculated values of ϵ_{TN} at the higher temperatures are not significantly affected by the ϵ_λ values beyond $\lambda = 25\mu$.

Section 5
COLD AIR TESTS OF OXIDIZED RENE 41

Two series of cold air flow tests were conducted on the oxidized Rene 41 material. Specimen oxidization conditions and preliminary characterization results are described in Section 4. Maximum test temperatures were on the order of 1150 $^{\circ}$K (1610°F), and total pressure at the specimen measuring area ranged from 0.1 to 80 Torr. Flow conditions covered static air (no flow but chamber pressure followed typical total pressure-time cycle), and free stream Mach Number of 1.1 and 2.1. Radiometric measurements were made of E_{TN} and E_{λ} under these conditions as well as at low pressure (0.1 Torr) with calorimetric total hemispherical (ϵ_{TH}) measurements being performed concurrently. The results of the two test series are described in the following subsections.

5.1 Specimen C-2

The specimen configuration was a 12-inch long by 1-inch wide strip 40 mils thick which had a narrowed test section at its midpoint to account for convective heat transfer during flow tests. The width of this test section was 0.85-inch for a 1-inch length. Instrumentation consisted of six 3 mil diameter chromel-alumel thermocouples spotwelded to the edge and rear surface of the strip. Five of these thermocouples were located in the test area; two along the vertical centerline 0.1 inch above and below the midpoint; and three at the edge, one at the midpoint and one each 1/4-inch above and below this point. The sixth thermocouple was on the edge 1/4-inch above the narrow section.

The specimen was installed in the test apparatus in a similar fashion as for the NBS standard specimens, and the same control arrangement was utilized for this test series. Both the M = 1.1 nozzle (throat diameter of 1.19 inch) and the M = 2.1 nozzle (0.462 inch throat diameter with a 5°half angle supersonic section) were used for flow testing. The nozzle exit plane was located 5-3/8 inches from the specimen test area in both cases, and this corresponds

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to the location used with the pressure model to calibrate for test area local total pressure as a function of chamber and nozzle upstream pressures for both flow conditions.

On the basis of the flow calibration testing, the M = 1.1 nozzle was operated at an upstream total pressure to chamber pressure ratio (P_t/P_c) of 2.0. With this slight supersonic expansion the ratio of test area total pressure to nozzle upstream total pressure (P_t/P_s) remained constant at a value of 1.095 over the entire range of test conditions. During flow testing the nozzle chamber inlet valve and pump duct bleed valve are operated simultaneously to maintain the value of P_t/P_c constant at 2.0 while P_t is increased to give the desired values of P_s. For the M = 2.1 nozzle, as the pressure ratio P_t/P_c is varied . compression and expansion waves in the free jet pass across the specimen the pressure ratio P_t/P_s varies. However, this ratio was found to be slowly varying in the region of P_t/P_c = 12.7. At this optimum operating point the ratio of P_t/P_s was determined experimentally as a function of P_t, and a curve constructed for calculation of P_s. During the emittance testing both nozzle and bleed valves are again operated together to obtain the desired values of P_s while P_t/P_c is maintained at 12.7.

The objectives of the tests of specimen C-2 were to measure emittance as a function of a time variant total pressure for four temperature levels under M = 1.1 and M = 2.1 conditions and to evaluate temperature uniformity across the test area under flow. \Prior to each flow situation (sonic versus supersonic), radiometric measurements were made as a function of temperature in a static environment at 0.1 Torr, and these results are shown in Figure 5-1. The values of E_{TN} are essentially the same before and after the M = 1.1 test series indicating that the sonic condition did not effect the total emittance of this material.

For both M = 1.1 and M = 2.1 conditions E_{TN} was measured as a function of specimen total pressure at four temperatures. From the data shown in Figures 5-2 and 5-3 it is seen that these emittance values are the same as those measured under static conditions to within 0.02. The small variations in E_{TN} with changing pressure for each temperature level are attributed to small

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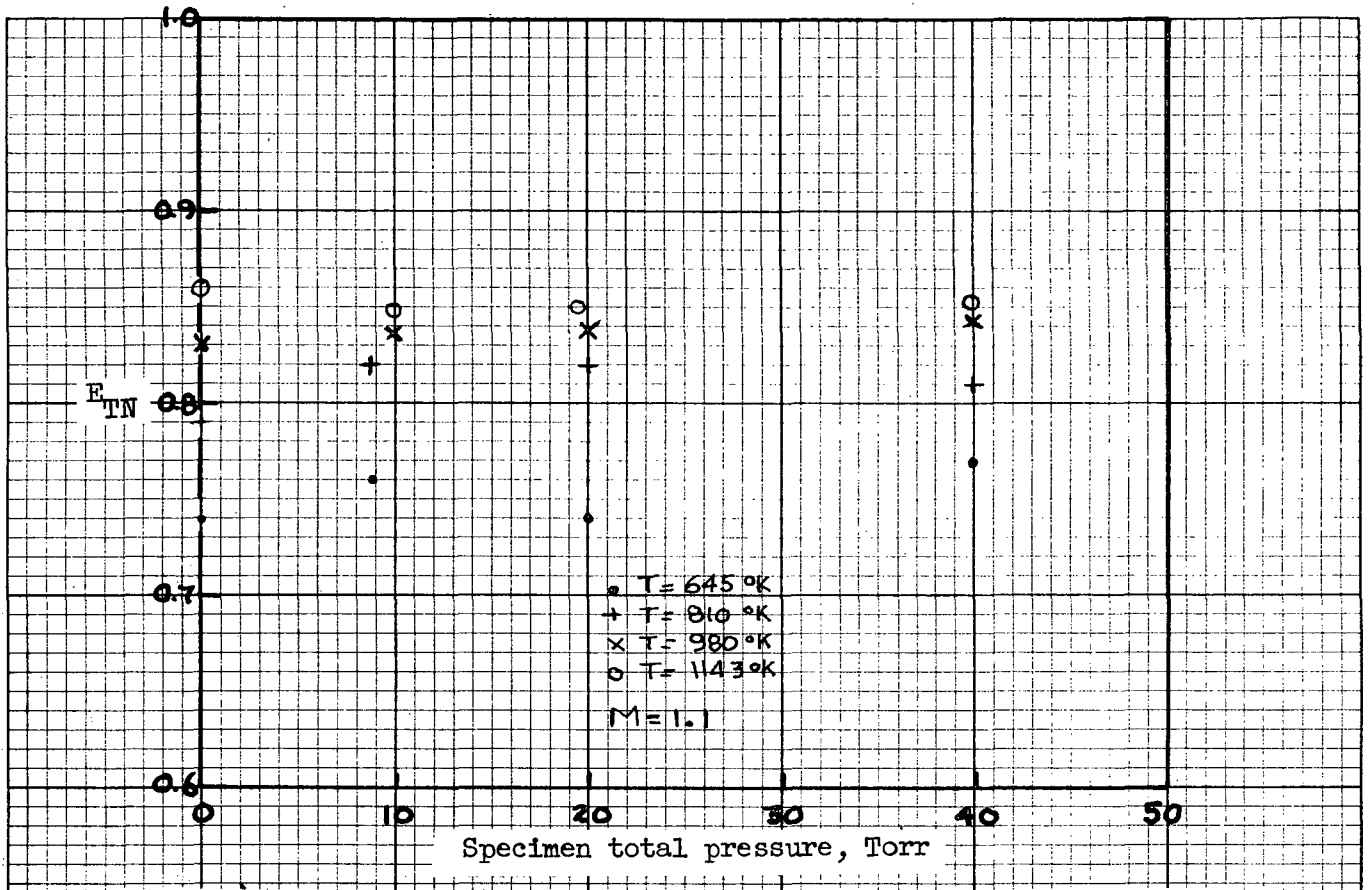
Figure 5-2. E_{TN} as a Function of Total Pressure for Oxidized Rene 41 for M = 1.1 Flow at Four Temperatures

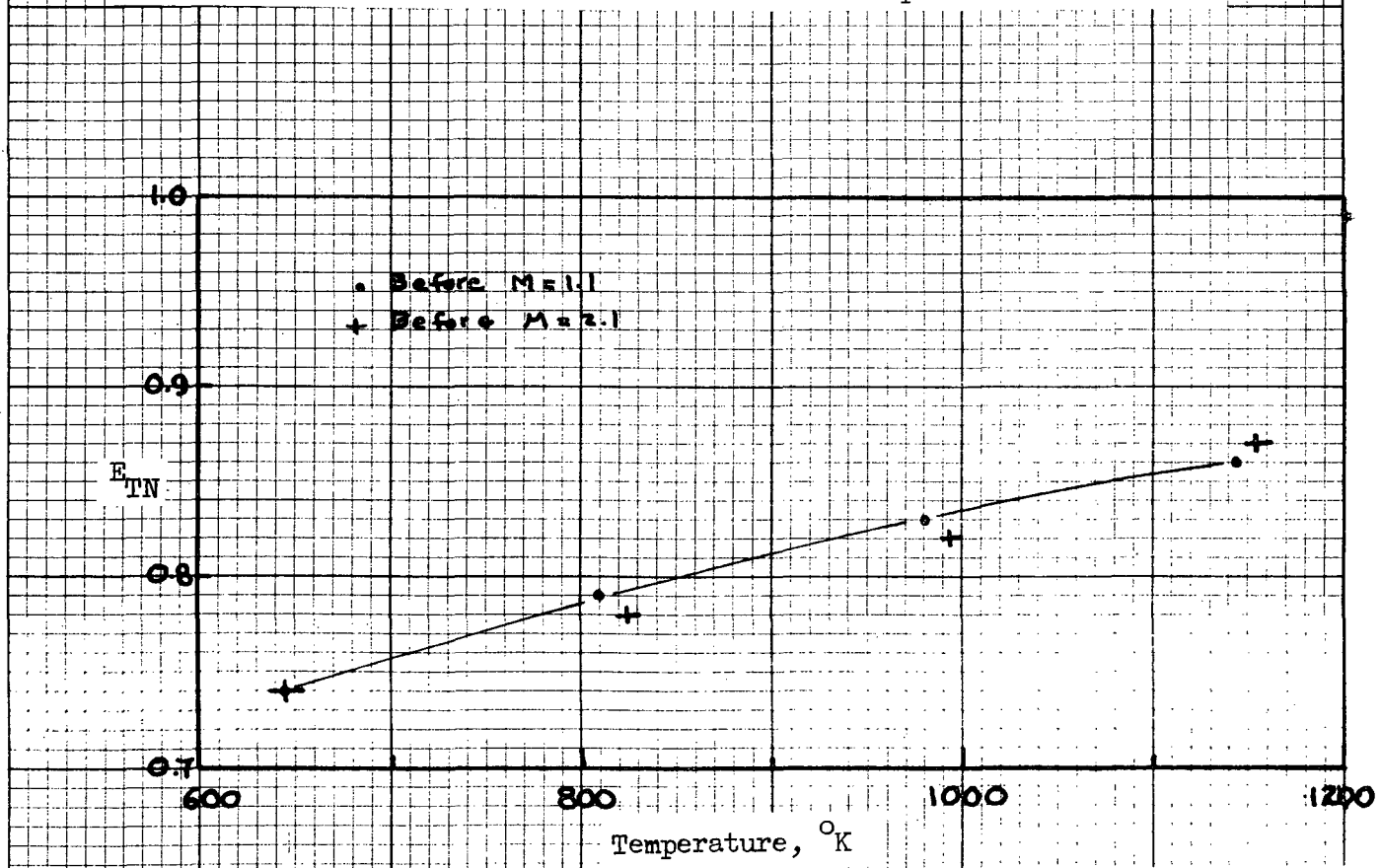Figure 5-1. E_{TN} as a Function of Temperature for Oxidized Rene 41 Specimen C-2 Before M = 1.1 and M = 2.1 Test Exposures
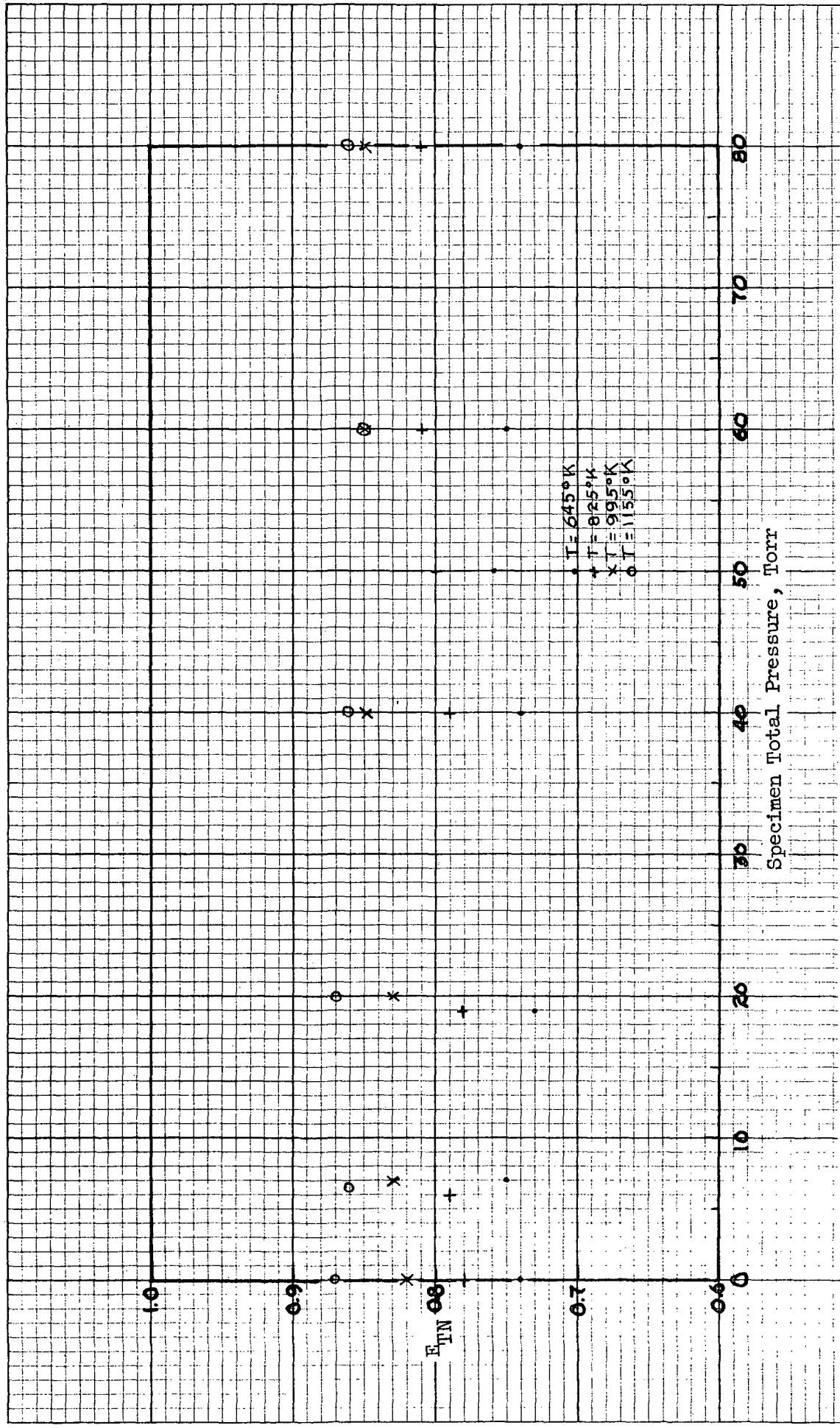
Figure 5-3. E_{TN} as a Function of Total Pressure for Oxidized Rene 41 for M = 2.1 Flow at Four Temperatures.

Specimen Total Pressure, Torr

E_{TN}

• T = 645°K
+ T = 825°K
x T = 995°K
○ T = 1155°K

temperature gradients in the measuring area. The radiometer viewed an area approximately 0.10 inch diameter, and the instrument was sighted nominally on an area opposite the center thermocouple used for determining surface temperature. However, small temperature gradients between center and edge were observed to vary with changing pressure level, and consequently, the viewed area probably contained gradients which caused slight differences in integrated radiance which were not reflected by the single center temperature measurement. As the variations in emittance calculated on the single temperature measurement were small, the data was not re-evaluated using an averaged temperature.

Spectral emittance data as a function of temperature for no flow and at one temperature for M = 1.1 conditions are shown in Figure 5-4. In the region of 1.5 to 5 μm no trend was observed as to the temperature dependence of spectral emittance. At the longer wavelengths spectral emittance increased with increasing temperatures. This was also evident from the broad band filter data (8 to 16.6 μm) which showed emittance in this band width to increase from 0.70 to 645°K to 0.73 at 811°K to 7.7 at 1143°K. Also, very little difference is noted in the values of E_λ for the no-flow and M = 1.1 conditions.

From both the total and spectral normal emittance data under no flow and flow conditions, the emittance properties of this material are stable and are not a function of the free stream Mach Number. Within the limits of experimental error emittance did not change; no effect of flow condition on emittance was observed. The values of ϵ_{TN} reported herein are in good agreement with those of Seban and Rolling (Ref. 2) for oxidized René 41 as shown in Table 5-1. Their data were for material oxidized at 1200°K (1700°F) for 3 hours, and the measurements were made in a static air environment.

TABLE 5-1

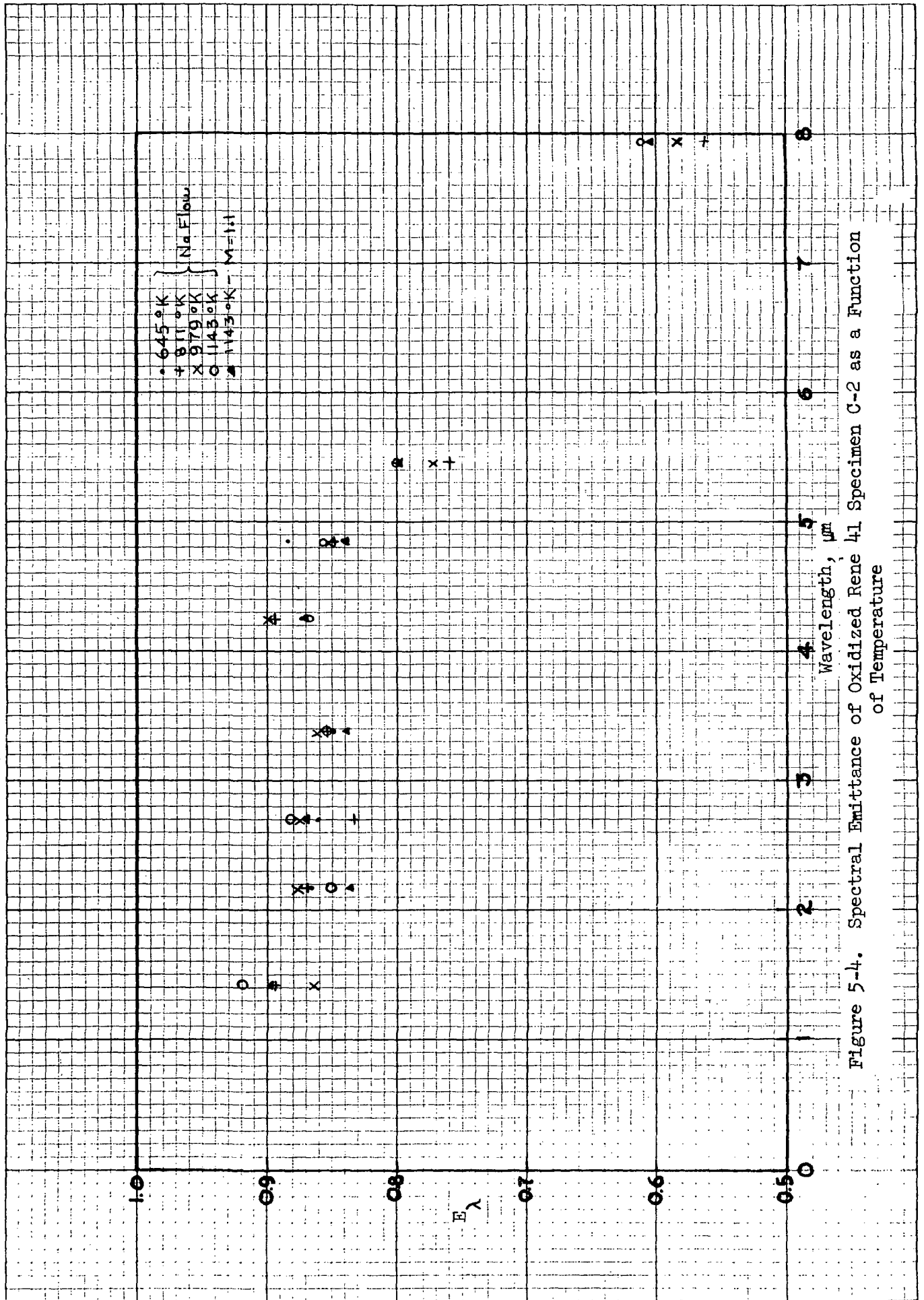| Temp. (°K)	E_{TN}	
	This Work	Ref. 2
700	0.76	0.79
900	0.81	0.82
1150	0.86	0.85

Figure 5-4. Spectral Emittance of Oxidized Rene 41 Specimen C-2 as a Function of Temperature

5.2 Specimen C-4

The configuration of this specimen was a 1-inch wide by 40 mils thick strip 12 inches long. The test area was not reduced in width for this specimen. Six platinum-Rhodium thermocouples, 3 mil diameter, were spotwelded to the edges and rear surface in the nominal 1-inch by 1-inch test area, four were in the central area along the vertical centerline and two on the edge. This specimen was installed in the test apparatus in the same manner as No. C-2 and operated using the same controls with the addition of a program unit to drive the controller at the appropriate temperature levels for the cyclic temperature-pressure conditions. A typical cycle trace is shown by figure 5-5. The two intermediate temperature plateaus on the ascending portion of the curve permitted emittance measurements to be made under steady temperature conditions during the cycle.

Testing was done under three flow conditions; static, M = 1.1 and M = 2.1 following the total pressure (P_s) of Figure 5-5. For static testing the chamber pressure was raised in a linear manner from 2 to 20 Torr over the 45 min. cycle. In both flow cases chamber and nozzle upstream pressures were adjusted to achieve the same type of specimen total pressure increase with time. The same program was used for all cycles, and temperatures were repeatable to $10^\circ K$ and pressures to 1 Torr from cycle to cycle.

The test measurement and exposure sequence for this specimen No. C-4 was as follows:

(1) Measure E_{TH}, E_{TN} and E_λ as a function of temperature in static environment of 0.1 Torr

(2) Conduct five static cycles of temperature and pressure varying with time as shown by Figure 5-5; measure E_{TN} and E_λ during cycles

(3) Measure E_{TH} and E_{TN} as per (1)

(4) Conduct five flow cycles, M = 1.1, following Figure 5-5; measure E_{TN}

(5) Repeat (1)

(6) Conduct five flow cycles, M = 2.1, following Figure 5-5; measure E_{TN}
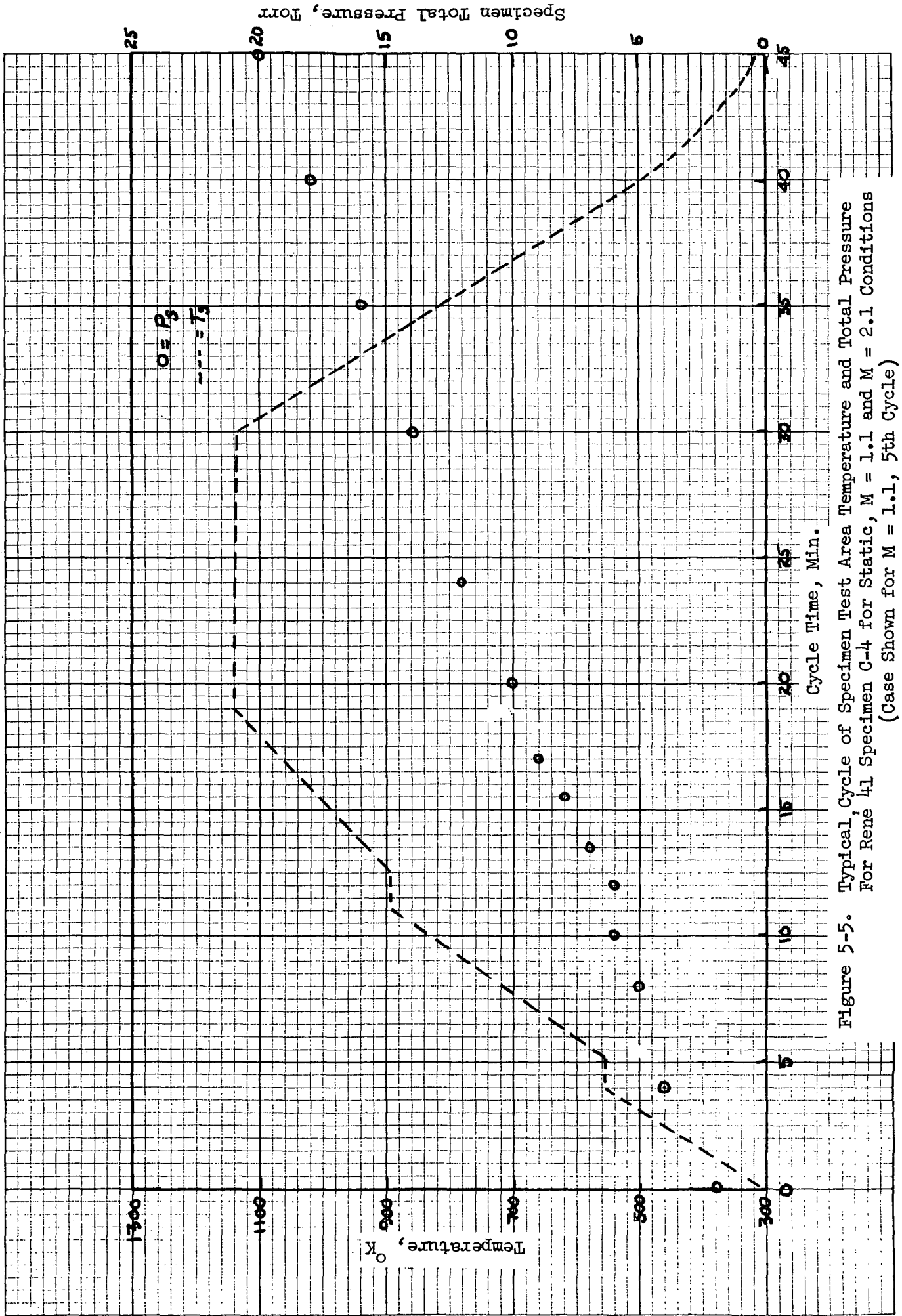
(7) Repeat (1)

Figure 5-5. Typical, Cycle of Specimen Test Area Temperature and Total Pressure For Rene 41 Specimen C-4 for Static, M = 1.1 and M = 2.1 Conditions (Case Shown for M = 1.1, 5th Cycle)

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A summary of the radiometric total normal emittance data for representative cycles of the static, $M = 1.1$ and $M = 2.1$ conditions is presented in Table 5-2. During the static cycling E_{TN} values between cycles were repeatable to within 0.01. Temperature measurement was not a problem in this natural convective environment. However, during the $M = 1.1$ and $M = 2.1$ cycles, somewhat larger cycle-to-cycle variations were observed (0.02 to 0.03), and the measured values of E_{TN} increased with exposure by 0.04 to 0.05 from the static to $M = 2.1$ cycles. To determine if these increases were attributable to real changes in radiative properties or are due to temperature measurement problems, a comparison is made between E_{TN} and E_{TH} values measured at 0.1 Torr at the start of testing and after completion of each flow environment testing. These data are tabulated in Table 5-3, and the results show that the total emittance remained essentially stable throughout the exposure testing. The measured changes in E_{TN} were 0.02 which are within the experimental uncertainty. Similarly, the changes in ε_{TH} were 0.01. From the conclusion that emittance was unchanged, the increases observed during flow testing are probably due to nonuniform temperatures across the specimen test area due principally to variations in local convective heat transfer to the cold gas. These center-to-edge gradients were verified by the thermocouple and pyrometer data. The narrower test section reduced this problem somewhat for the $M = 2.1$ nozzle which has a smaller flow field. Future testing will be done with the narrower specimen test area.

Spectral emittance data before exposure and after the $M = 1.1$ and $M = 2.1$ testing are shown in Figure 5-6. No significant changes in spectral emittance resulted from the exposure cycles. At the shorter wavelengths emittance appears to decrease slightly (0.04 to 0.05) with exposure, whereas at the longer wavelengths it is unchanged.

Comparing the data from specimens C-2 and C-4, the former showed a slightly greater total normal emittance, 0.02 in the range of $900°$ to $1160°K$. This trend is also borne out by the somewhat higher spectral emittance of C-2 at shorter wavelengths.

TABLE 5-2

Summary of Typical Measurements of E_{TN},
During Static and Flow Tests of Oxidized Rene 41

Sample C-4

Flow Condition	Time (Min)	Cycle No.	$T_{Surface}$ (°K)	ϵ_{TN}
Static, Chamber pressure varied from 2 to 20 Torr	11 1/2		888	0.78
	20		1129	0.83
	25	1	1131	0.83
	29		1135	0.83
	4 1/2		640	0.71
	11 1/2		891	0.78
	20	3	1136	0.83
	29		1142	0.83
	4 1/2		642	0.72
	11 1/2		893	0.78
	20	5	1138	0.83
	29		1144	0.84
Flow M = 1.1, P_s = 2 to 20 Torr	4 1/2		636	0.75
	11	3	887	0.81
	20		1135	0.86
	29		1138	0.86
	4-1/2		643	0.76
	11		894	0.83
	20	5	1137	0.87
	29		1138	0.88
Flow M = 2.1, P_s = 2 to 20 Torr	4-1/2		647	0.76
	11-1/2		892	0.81
	20	3	1127	0.89
	29		1132	0.89
	4-1/2		645	0.78
	11-1/2	5	887	0.82
	20		1132	0.87
	29		1137	0.88

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TABLE 5-3

Summary of ϵ_{TN} and ϵ_{TH} Data Before and
After Flow Testing of Oxidized Rene' 41

Specimen C-4

Conditions	T_s ($^\circ K$)	ϵ_{TN}	ϵ_{TH}
In vacuum before static tests	708 936 1161	0.76 0.79 0.85	0.748 0.767 0.801
In vacuum after static tests	711 940 1167	0.76 0.78 0.85	-- 0.769 0.797
In Vacuum after M = 1.1 tests	710 940 1160	0.77 0.79 0.85	0.781 0.803
In vacuum after M = 2.1 tests	710 939 1158	0.77 0.79 0.83	0.748 0.766 0.790

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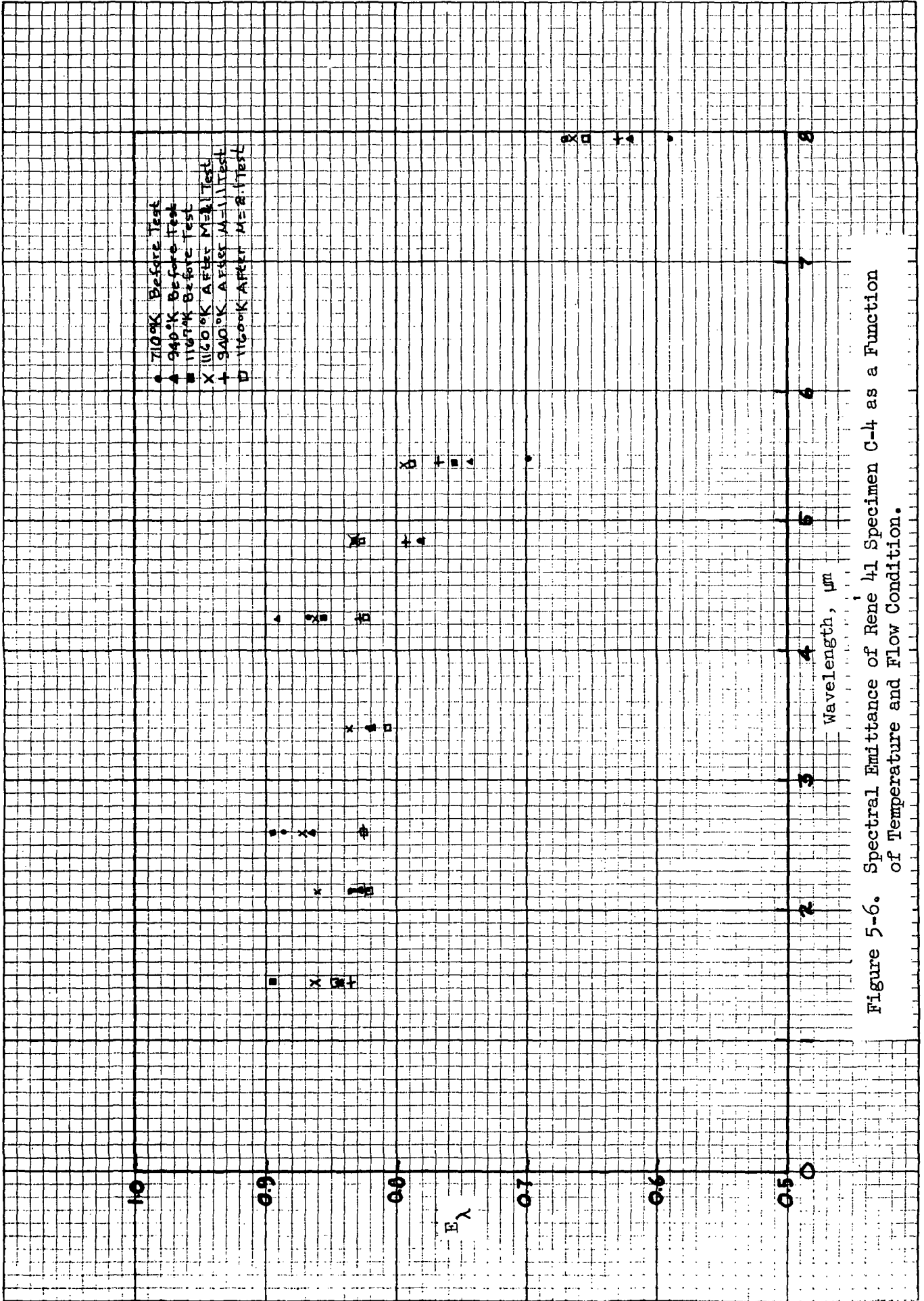
Figure 5-6. Spectral Emittance of Rene 41 Specimen C-4 as a Function of Temperature and Flow Condition.

Legend:
- ● 710°K Before Test
- ▲ 940°K Before Test
- ■ 1167°K Before Test
- ✕ 1160°K After M=4.11 Test
- + 940°K After M=11 Test
- □ 1160°K After M=2.1 Test

Wavelength, μm

E_λ

Section 6

REFERENCES

1. "Certificate of Normal Spectral Emittance," - "Standard Reference Materials 1402 to 1409" and "Standard Reference Materials 1420 to 1428", National Bureau of Standards, U. S. Department of Commerce, April 1966 and Dec. 1965.

2. R. A. Seban and R. E. Rolling, "Thermal Radiation Properties of Materials," WADD Technical Report 60-370, June 1961.