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EVALUATION OF A STANDARD TEST METHOD FOR TOTAL HEMISPHERICAL EMITTANCE OF SURFACES FROM 293 K TO 1673 K

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SUMMARY

Emittance tests were made on samples of Rene' 41, Haynes 188, and Inconel 625 superalloy metals in an evaluation of a standard test method for determining total hemispherical emittances of surfaces from 293 K to 1673 K. The intent of this evaluation was to address any problems encountered, check repeatability of measured emittances, and gain experience in use of the test procedure. Five test specimens were fabricated to prescribed test dimensions and surfaces cleaned of oil and residue. Three of these specimens were without oxidized surfaces and two with oxidized surfaces. The oxidized specimens were Rene' 41 and Haynes 188. The tests were conducted in a vacuum where the samples were resistance-heated to various temperature levels ranging from 503 K to 1293 K. The calculated results for emittance, in the worst case, were repeatable to a maximum spread of ±4% from the mean of five sets of plotted data for each specimen. While data were generated for oxidized and unoxidized specimens, an independent source of data was available for comparison only for the oxidized specimens. The entire temperature range of the test method was not covered in the evaluation of the test procedure; however, the emittance values calculated for oxidized Rene' 41 for the available temperature range were closely matched to those extrapolated from published data derived from other emittance measurements and test methods. The emittance values for oxidized Haynes 188 data, however, did not compare favorably. The poor comparison for the Haynes 188 data is attributed to differences in the surface oxide composition of the specimens.

INTRODUCTION

A calorimeteric method for determining total hemispherical emittance of surfaces described in ANSI/ASTM C835-76, (ref. 1) has been set forth by the American Society for Testing Materials as a standard test procedure. Total hemispherical emittance is a needed parameter to properly characterize heat loss/gain of materials by radiation. There is a growing need for this type of data to perform thermal analyses of high temperature systems such as the heat shield of Space Shuttle vehicles.

It is recognized that in the application of metallic materials, the surface properties of a material are highly dependent upon the service conditions. For example, high temperature exposure of metals in an oxidizing environment may result in growth of a surface oxide or scale which greatly alters its emittance characteristics. Also, some oxidized metals suffer a loss of the surface oxide on exposure to high temperatures under a hard vacuum. The present test method can be used to determine the effects of service conditions on the emittances of such materials.

The tests were conducted in a vacuum on preconditioned surfaces of the test specimen to simulate service conditions and will provide emittance values that are applicable to materials subjected to other environmental conditions. However, it must be considered that these emittance values may vary from those measured in air due to effects of a vacuum environment. With these considerations duly noted, such emittance measurements can be used in the calculation of radiant heat transfer from surfaces which are within the temperature range of a duplicate test specimen (ref. 1).

SYMBOLS

W_B total energy emitted from a blackbody surface, watts

W_s total energy emitted from the specimens test surface area, watts

Q time rate of heat flow, electrical energy applied to the specimen test area, watts

ϵ emissivity factor, (blackbody = 1)

ϵ_{TH} total hemispherical emittance of the specimens test surface area

A radiating surface area, m^2

A_s the total radiating test surface area of the specimen including edges that "see" the absorbing surface (ref. 1). Thus, $A_s = 2L(w + t)$ where

L = length
w = width
t = thickness

σ Stefan-Boltzmann constant, = 5.669×10^{-8} $W/m^2 \cdot K^4$

T absolute temperature, K

T_s specimen test surface temperature, K

T_w internal test vessel wall temperature, K

$\Delta\epsilon_{TH}$ the standard deviation in the total hemispherical emittance value

ΔQ deviation in the electrical energy applied to the specimen test area due to error in the metering instruments

ΔA_s deviation in the dimensions of the specimen test area due to fabrication and the installation of thermocouples

ΔT_s deviation in the surface temperature of the specimen test area due to error in the thermocouple wire

ΔT_w deviation in the surface temperature of the test vessel wall due to error in the thermocouple wire

TEST THEORY

If the transfer of energy (heat) could be limited to radiation only, then the electrical power used for resistance heating of a test specimen could be equated to a radiative heat transfer; hence power in will equal power out when in equilibrium. Therefore, using a vacuum environment to eliminate convection while minimizing conduction by restricting the test area to a small central region of a sufficiently long test specimen and applying the energy balance to the Stefan-Boltzmann law,

$$W_B = \varepsilon A \sigma T^4$$

or

$$Q = W_s$$

Making the appropriate substitutions while considering two interacting surfaces, the test specimen and the test vessel wall, the radiation interchange can be described as

$$Q = \varepsilon_{TH} A_S \sigma (T_S^4 - T_w^4) \quad \text{(ref. 1)}$$

or

$$\varepsilon_{TH} = \frac{Q}{A_s \sigma (T_S^4 - T_w^4)}$$

Consequently, the electrical power, Q dissipated across the test area and the temperature differential $(T_S^4 - T_w^4)$ are measured.

There are several assumptions made, however, in calculating emittance from the equation above. They are:

1. That the vacuum enclosure is a blackbody absorber at uniform temperature.

2. That the total hemispherical absorptance of the sample for completely diffuse blackbody radiation at the temperature of the enclosure is equal to total hemispherical emittance of the sample at its temperature.

3. That there is no heat loss from the sample test section due to convection or conduction.

Using this test method, the effects of these assumptions on the emittance calculation are small for most materials. Further, if the principle dictates of this method are followed, one can expect the measured values to be accurate to within ±5% (ref. 1).

The intent of this evaluation is to address any problems encountered, check repeatability of measured emittances, and gain experience in use of the test procedure.

APPARATUS

The test facility is shown in figure 1 with a sketch taken from reference 1 and photos of the system assembled for this evaluation. It is comprised of:

1. A vacuum system (bell jar)

2. Support hardware for the vertical suspension of test specimens.

3. A test specimen

4. Thermocouples to monitor temperatures of the specimen and the environment of the bell jar.

5. Power supply

6. Metering equipment to determine the power dissipated across the specimen test area and temperature readout of the thermocouples.

Vacuum System

The vacuum enclosure is a pyrex bell jar with an ID of 43 cm and a height of 76 cm equipped with 2 diffusion pumps and backed by one mechanical pump. The operating vacuum atmosphere obtained with this system is a nominal pressure of 0.49 m Pa (3.7×10^{-6} torr) which exceeds the vacuum requirement of 1.3 m Pa (9.75×10^{-6} torr) for this method (ref. 1). This enclosure meets the criterion for a uniform blackbody emitter since pyrex has the required emittance of 0.8 for this test method. However, the bell jar is transparent to the outside environment which could present a problem if the specimen is exposed to any external high energy radiation sources. This did not occur in this evaluation. In the presence of external high energy radiation sources, the bell jar should be coated to make it opaque to the outside. The coating should be selected to ensure that the inner surface has an emittance greater than 0.8. Types of coatings for this application are discussed in reference 1.

The test method specifies a minimum differential of 373 K between T_s and T_w to achieve the accuracy stated for the method. The enclosure in the present evaluation was at room temperature, hence, the lowest temperature consistent with this minimum temperature differential is 402 K. Measurements at lower temperatures would require cooling the enclosure walls (ref. 1).

Support Hardware

The support hardware was designed to accommodate specimens 25 cm to 50 cm long which are mounted vertically and held firmly by two terminal connectors clamped over a 1.3 cm protrusion of each end for good electrical contact. All portions of the support mechanism that serves as part of the electrical circuit is of low resistance to minimize extraneous heating. The upper connector is held fast to a support arm while the lower connector is pigtailed electrically to allow the specimen to hang freely for expansion during the test (fig. 2).

Test Specimen

Specimens 25 cm long and 1.3 cm wide were fabricated from 0.254 mm thick sheet material (figure 3a) to conform to the recommended dimensions of reference 1 and to minimize heat loss over the test surface. All edges are squared to minimize error in the calculated area. The surfaces are cleaned as follows to assure that they are free from oils:

1. Spar clean for 1 hour, i.e., detergent wash.

2. Ultrasonic rinse in distilled water.

3. Freon clean, 5 minutes over Freon vapor, 10 minutes immersed in liquid Freon, and again 5 minutes over vapor.

4. Distilled water rinse.

5. Verify with clean water sheet test. If water beads, the surface is not clean.

6. Forced air dry.

After cleaning, the specimens were treated as follows to form a heavy surface oxide.

1. Rene'41 - Heat treat 12 hours at 1500°F at 1 atm. in air.

2. Haynes 188 - 12 hours at 1800°F at 1 atm. in air.

3. Furnace cool. Specimen should remain in the furnace and cool with it.

Thermocouples

To accurately measure the temperature of the specimen it was necessary to give special consideration to the type of thermocouple, size, positions, and orientation of the thermocouple. Because of the high temperatures involved, platinum vs platinum 13% rhodium (type R) is required. A .08-mm-diameter thermocouple was used to minimize conduction losses through the wire. Three thermocouples are intrinsically attached to the specimen in the positions shown in figure 2. The test section length of 7.5 cm was selected as compatible with the overall length of the specimen in minimizing conduction errors in the surface temperature. An ac power supply was selected for the test system because the thermocouple alignment is less critical than for a dc power supply. A slight misalignment of the thermocouple attachments for dc application creates a voltage potential between them, thus inducing error in the temperature readings, figure 3b. Only one thermocouple is used to monitor the wall temperature of the vacuum vessel. This is an iron/constantan (type J) thermocouple located on the base plate of the support hardware (fig. 1).

Power Supply

Requirements for the power supply output were 10-16 volts and 0-100 amps. An ac power supply (variable transformer) with a rated output of 7 KVA at 50 amps. and a voltage range of 0-140 volts was used. Overload characteristics of this

supply permit a sustained 150 percent overload for a period of 15 minutes which meets the power and time necessary to obtain the upper range temperature of 1673 K.

Metering Equipment

The current through, and the voltage drop across, the test area of the test sample are measured with two digital multimeters, thus, permitting the calculation of power input to the test area. One meter was calibrated for a current range of 0-100 amps and the other for a voltage range of 0-10 volts.

All thermocouple voltages were measured, converted to temperature and recorded using a data logger.

TEST PROCEDURE

The test specimen is positioned in the vacuum vessel with the thermocouple leads extending 18 cm to a terminal strip attached to the upper support arm post. Connections are made at the strip to 0.4-mm-diameter platinum wire running to the outside of the vessel through a pressure fitting. Coupled to the platinum wire in the fitting is extension wire that connects to the data logger. One lead of each of the two end thermocouples is used as a voltage tap for a digital multimeter connection to measure the voltage drop across the test portion of the specimen. A second digital multimeter is connected electrically in series with the specimen to measure current through it. Subsequently, the circuit is completed to the voltage supply. The bell jar is evacuated to a nominal working pressure of 0.49 m Pa. Once this is achieved, electrical power is applied to the circuit and the specimen is resistance-heated to the desired temperature.

The test conditions (specimen temperature and power input, Q_s) are allowed to stabilize before recording these data for computation of emittance. To assure a steady state condition, the temperature and power should be observed at intervals of not less than 5 minutes until three successive sets of observations give emittance values differing by not more than 1 percent (ref. 1). In this evaluation the conditions of temperature and power were allowed to stabilize for 15 minutes for each test point (temperature level). The test temperature is computed by taking the average of the values indicated by the three thermocouples attached to the specimen. Since one of the primary goals of this evaluation was to determine repeatability of emittance values, data were taken at increments of 8 amps during the heating period up to approximately 60 amps and over a temperature range that varied for each specimen. This provided 7 to 8 test points for each of 5 tests performed on each specimen. Data recorded for each data point includes the temperature of, the voltage across and the current through the specimen test area.

TEST RESULTS

Table I presents a summary of test results from this evaluation. All emittance measurements for each specimen are shown in figures 4-8. Figures 4 and 5 show a comparison of emittance data versus temperature for oxidized Rene' 41 and Haynes 188 from this evaluation with data from the literature (refs. 2, 3, and 4). Two sets of the published data were recorded direct as total hemispherical emittance and the other two were extrapolated by analysis from total normal emittance data of reference 5 using the method of reference 1.

Figure 4 shows very good agreement of results for total hemispherical emittance of oxidized Rene' 41 from the present evaluation and published data.

The data for Haynes 188 from this study are not in good agreement with results from the literature (see figure 5). The differences in these data are attributed to differences in the surface oxides present on the specimens. The amount and types of oxides present depend on the high temperature exposure history of a specimen. Specimens for the present study were subjected to 12 hours oxidation at 1800°F compared to 3 hours at 1900°F for specimens whose results are cited from the literature. The data from references 2, 4, and 5 show the extent of variation in emittance of Haynes 188 alloy specimens resulting from different oxidation conditions.

Figures 6-8 show data for hemispherical emittance versus temperature for Rene' 41, Haynes 188, and Inconel 625 alloy specimens with no prior oxidation treatment. The lower values of emittance compared to the data in figures 4 and 5 are typical of results for metals with thin surface oxide layers. Each figure shows very little scatter in the results which attests to the repeatability of the test method.

ERROR ANALYSIS

Errors to be considered are those found in power dissipation, temperatures, and in the surface test area of the specimen. To determine the manifestation of these errors in the emittance value, ε_{TH}, the standard deviation of the afore mentioned parameters are applied to the following equation.

$$\frac{\Delta\varepsilon_{TH}}{\varepsilon_{TH_{RMS}}} = \sqrt{\left(\frac{\Delta Q}{Q}\right)^2 + \left(\frac{\Delta A_s}{A_s}\right)^2 + \left(\frac{\Delta T_s}{T_s}\right)^2 + \left(\frac{\Delta T_w}{T_w}\right)^2}$$

Error values for these are shown in Table II. The $\Delta\varepsilon_{TH}$ derived from these values is within the error margin for this procedure. Errors in conduction are beyond the scope of the paper. It was noted that there was no appreciable temperature gradient over the entire length of the specimen.

TEST DISCUSSION

Because of limitations in the power supply, the highest temperature recorded was 1293 K. The lowest temperature used for an emittance computation was 503 K. Both of these temperature extremes were for unoxidized Rene' 41. The highest temperature level for the two oxidized specimens was 1063 K.

The low temperature of 503 K represents a general level below which ε_{TH} values for Rene' 41 were inconsistent with values derived at higher temperatures, in that the low temperature results are much too high. This minimum temperature varied with each specimen and often occurred at temperatures, T_s, between the first and second heating steps of 8-16 amps. input. Since the heating cycle is done in increments of 8 amps., the first ε_{TH} value for documentation was calculated at the second heating level. Even though the temperature differential between T_s and T_w exceeds 373K, the power Q, is larger in relation to the quantity $(T_s^4 - T_w^4)$ for the lower values of T_s. Consequently, the ratio $\frac{Q}{(T_s^4 - T_w^4)}$ results in larger values than for those at the higher temperatures.

Perhaps this is due to the heating characteristics of some materials. One approach to resolving this problem for valid ε_{TH} values at the lower temperature is to cool the vacuum vessel wall with chilled water or LN_2, thus enabling one to make emittance measurements at surface temperatures as low as 293 K.

Another limitation to the highest temperature for some materials is the loss of vacuum due to outgassing. For some surface conditions such as those for the oxidized samples, this outgassing with loss of vacuum is excessive. Although there is a gradual decrease in vacuum during the heating period, the vacuum is maintained below the pressure dictated by the test method for the unoxidized samples, but the vacuum is not adequate for the two oxidized specimens at temperatures above about 1100 K. In most instances the loss of vacuum is slowed and limited with each test made as the outgassing diminishes. This better vacuum improves the stability in conditions of temperature and power dissipation for each test point. For instance, Inconel appears to be more stable during heating than Rene' 41. Therefore, the physical properties of materials will manifest themselves in many ways that affect test results derived from the same test method.

In addressing problems encountered, the test facility requires some modification and update of data acquisition instrumentation (ref. 1). Changes to be considered are:

1. Blackening of the bell jar to shield the specimen from external radiation (ref. 1).

2. Provide cooling of the vessel wall to increase the temperature test range from 293 K to 1673 K (ref. 1).

3. Additional thermocouples for the vessel wall for a more realistic sampling of T_w. Also increase the number of specimen thermocouples to determine the temperature gradient over the entire length of the sample (ref. 1).

4. A better technique in attaching thermocouples to the specimen to minimize alteration of the surface area. Currently the surface is being altered in three places where it is lightly sanded to produce good thermocouple welds.

5. Change terminal strip design for a quick connection and release of thermocouple leads to expedite the installation and removal of a test specimen.

6. Provide some means for keeping the specimen as straight as possible during a test, perhaps by weighting the unrestrained end.

7. A new power source capable of providing the heating capacity necessary for the specimen to reach an upper temperature of 1673 K (ref. 1).

8. Automate all data acquisition instrumentation. The data logger will provide this capability with the addition of an accessory option for recording the power, Q, directly. A watt-transducer designed for this purpose is also being considered.

CONCLUDING REMARKS

Even though the quantity of comparable emittance data is limited and has been derived under conditions less than the dictates of this test method, the goals of this evaluation were satisfied. The fact that only the emittance values for oxidized Rene' 41 compared favorably with published data, does not discredit the validity of the emittance values for Haynes 188. The high temperature exposure history and thus the surface composition of the sample tested in this evaluation and those of samples for published data were decidely different. This, of course, effects the differences in the two sets of emittance measurements for Haynes 188 and is a concern for future tests. The total quantity of data, including those of the unoxidized specimens, substantially supports repeatability in the emittance data. The experience gained and problems encountered in the evaluation served to define a modification of the test facility for a more expedient and accurate acquisition of emittance measurements. In effecting these changes, the test facility can be qualified and a standard test method for total hemispherical emittance measurements fully implemented at LaRC.

REFERENCES

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6. Temperature Measurement Handbook and Encyclopedia, Omega Engineering, Inc., 1984.

Table I.- Summary of Test Results for Emittance Measurements

Specimen Material	Data Sets I		II		III		IV		V	
	Temp	ε_{TH}	Temp	ε_{TH}	Temp	ε_{TH}	Temp	ε_{TH}	Temp	ε_{TH}
Rene'41 Oxidized	413	.78	428	.67	431	.70	419	.82	417	.70
	562	.76	556	.72	554	.72	561	.72	552	.71
	694	.77	678	.75	679	.75	679	.75	679	.75
	780	.77	783	.77	776	.77	781	.77	788	.77
	877	.78	883	.79	867	.79	861	.78	864	.78
	943	.78	950	.80	942	.79	941	.79	940	.78
	1017	.80	1007	.81	1008	.80	1009	.80	1008	.79
	1061	.81	1049	.81	1054	.81	1047	.81	1049	.81
Haynes 188 Oxidized	512	.80	508	.77	523	.77	404	.75	388	.83
	603	.80	649	.80	597	.80	527	.78	528	.79
	707	.81	701	.81	700	.82	580	.78	602	.81
	792	.82	781	.82	809	.84	746	.82	703	.83
	888	.82	863	.84	871	.85	822	.83	787	.85
	954	.82	946	.85	939	.86	871	.85	873	.86
	1005	.84	1000	.85	994	.86	938	.85	927	.89
					1048	.87	1023	.87	971	.88
Rene' 41	690	.36	700	.32	708	.32	518	.35	502	.37
	954	.36	852	.34	859	.35	723	.33	714	.33
	1130	.38	967	.36	970	.36	857	.35	857	.35
	1214	.38	1086	.37	1077	.38	972	.37	967	.37

Table I (Concluded)

Specimen Material	Data Sets I		II		III		IV		V	
	Temp	ε_{TH}	Temp	ε_{TH}	Temp	ε_{TH}	Temp	ε_{TH}	Temp	ε_{TH}
Rene' 41,			1058	.37	1160	.39	1060	.38	1057	.38
(Continued)			1162	.38	1166	.39	1148	.39	1157	.39
			1262	.39	1251	.39	1241	.39	1247	.40
			1286	.39	1283	.40	1278	.40	1294	.40
Haynes 188	651	.25	674	.24	679	.24	513	.23	685	.23
	791	.27	820	.26	824	.25	673	.23	823	.25
	971	.30	941	.27	953	.27	810	.25	954	.27
	1034	.31	1052	.28	1076	.28	941	.27	1049	.28
	1106	.31	1136	.29	1140	.29	1074	.28	1159	.29
	1215	.31	1221	.30	1210	.30	1159	.29	1237	.30
	1288	.31	1288	.31	1219	.30	1235	.30	1284	.30
Inconel 625	638	.31	640	.32	625	.32	628	.32	672	.32
	765	.35	779	.34	761	.34	772	.34	768	.34
	892	.36	902	.36	894	.35	892	.35	898	.35
	992	.37	985	.36	989	.36	991	.36	998	.36
	1074	.38	1063	.37	1076	.37	1078	.37	1079	.37
	1156	.38	1142	.38	1160	.38	1155	.38	1160	.38
	1195	.38	1199	.38	1196	.38	1199	.38	1201	.38

Table II.- Parametric Errors

Specimen	Test No.	Run No.	$\frac{\Delta Q}{Q}$	$\frac{\Delta A_s}{A_s}$	$\frac{\Delta T_s}{T_s}$	$\frac{\Delta T_w}{T_w}$	$\frac{\Delta \epsilon_{TH}}{\epsilon_{TH}}$
Haynes 188 Oxidized	5	4	.28	.064	1.2	2.9	3.15
Rene' 41 Oxidized	3	4	.28	3.5	1.3	2.9	4.74
Haynes 188	4	4	.28	1.3	1.42	2.9	3.5
Rene' 41	4	4	.28	.02	1.44	2.9	3.25
Inconel 625	4	4	.28	2.02	1.39	2.9	3.8

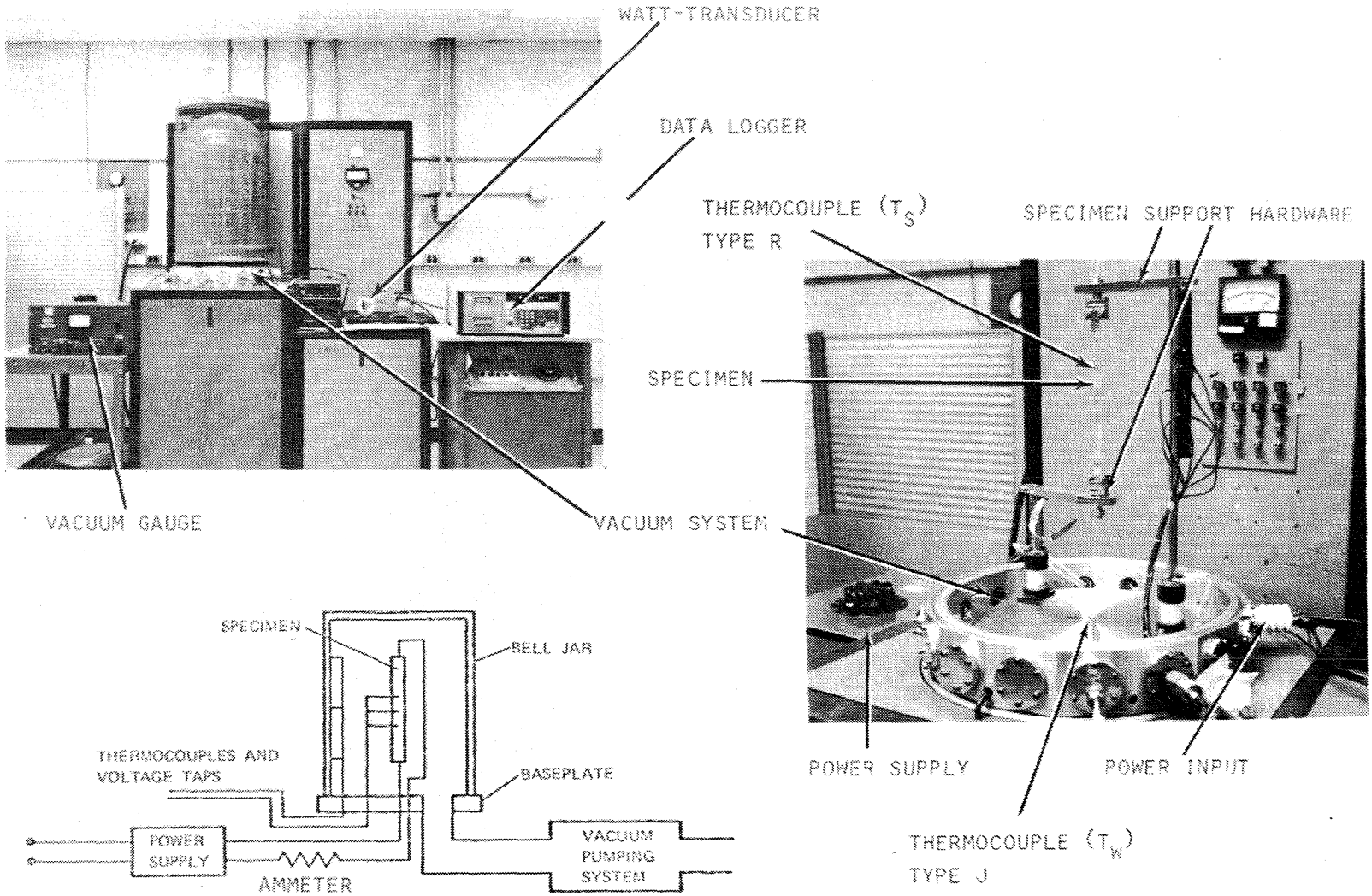


FIGURE 1. - TOTAL HEMISPHERICAL EMITTANCE TEST FACILITY

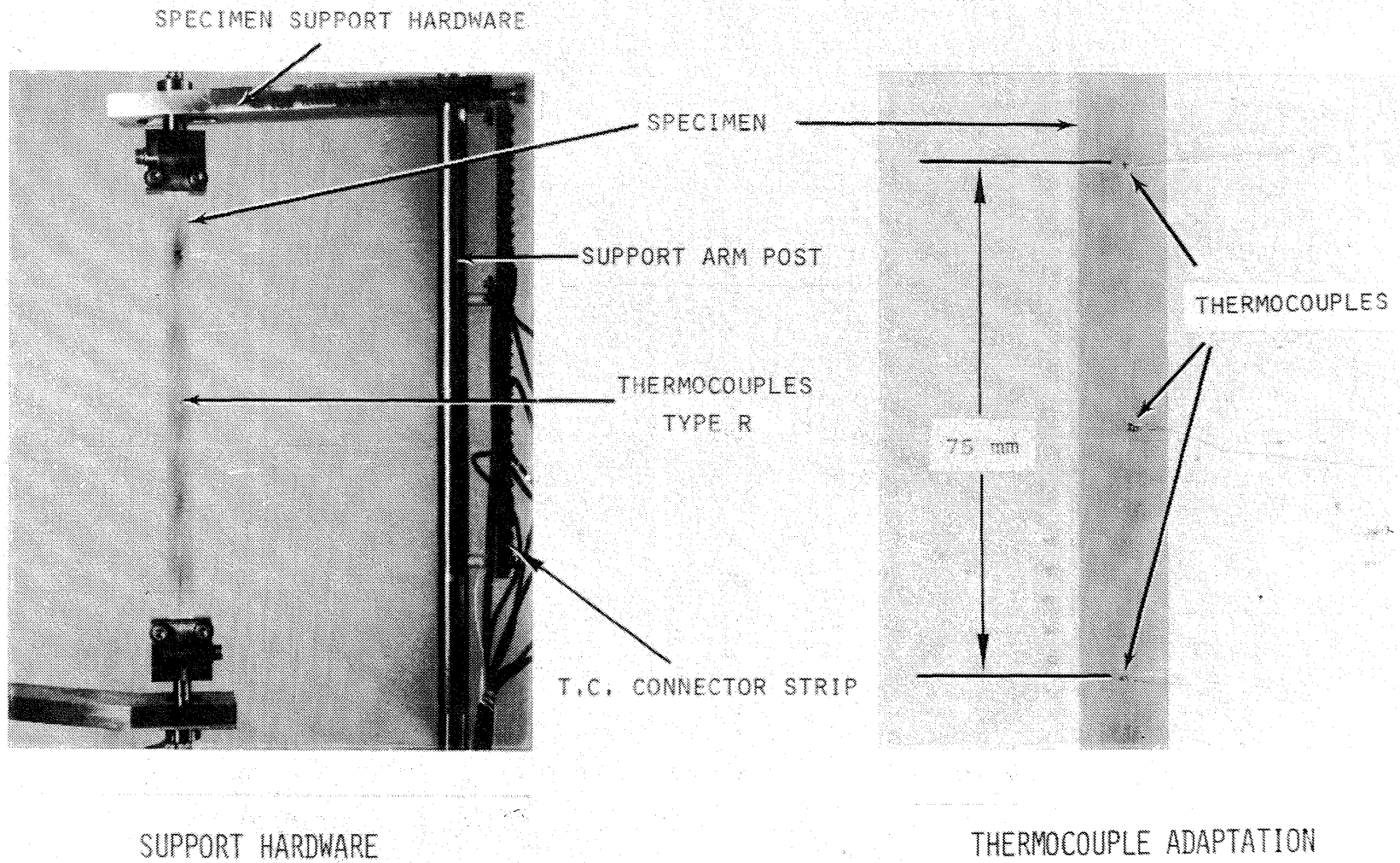
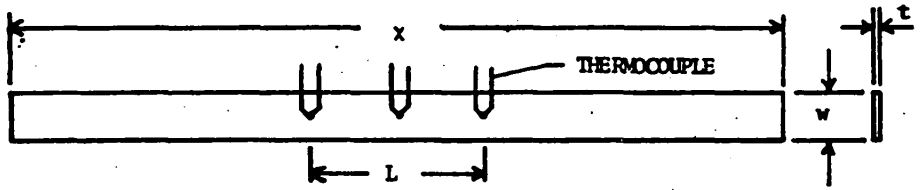


FIGURE 2. SPECIMEN INSTRUMENTATION AND ASSEMBLY DETAIL



MATERIAL	t	w	L	X
RENE' 41	0.25	13	75	250
HAYNES 188	0.25	13	75	250
INCONEL 625	0.25	13	75	250

NOTE: ALL DIMENSIONS IN MM

FIGURE 3A. - TYPICAL TEST SPECIMEN DIMENSIONS (REF. 1)

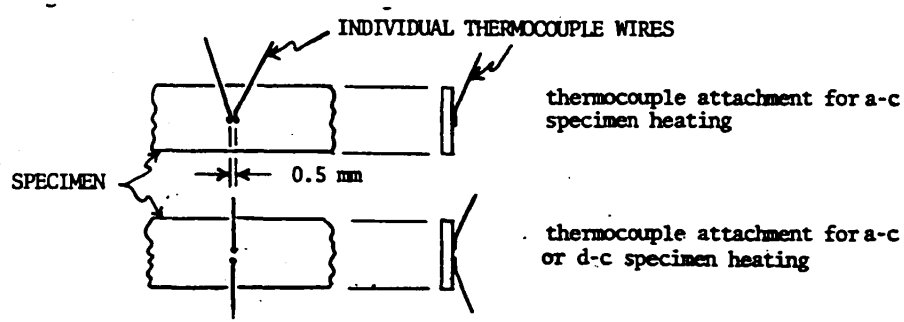


FIGURE 3B. - THERMOCOUPLE ATTACHMENT (REF. 1)

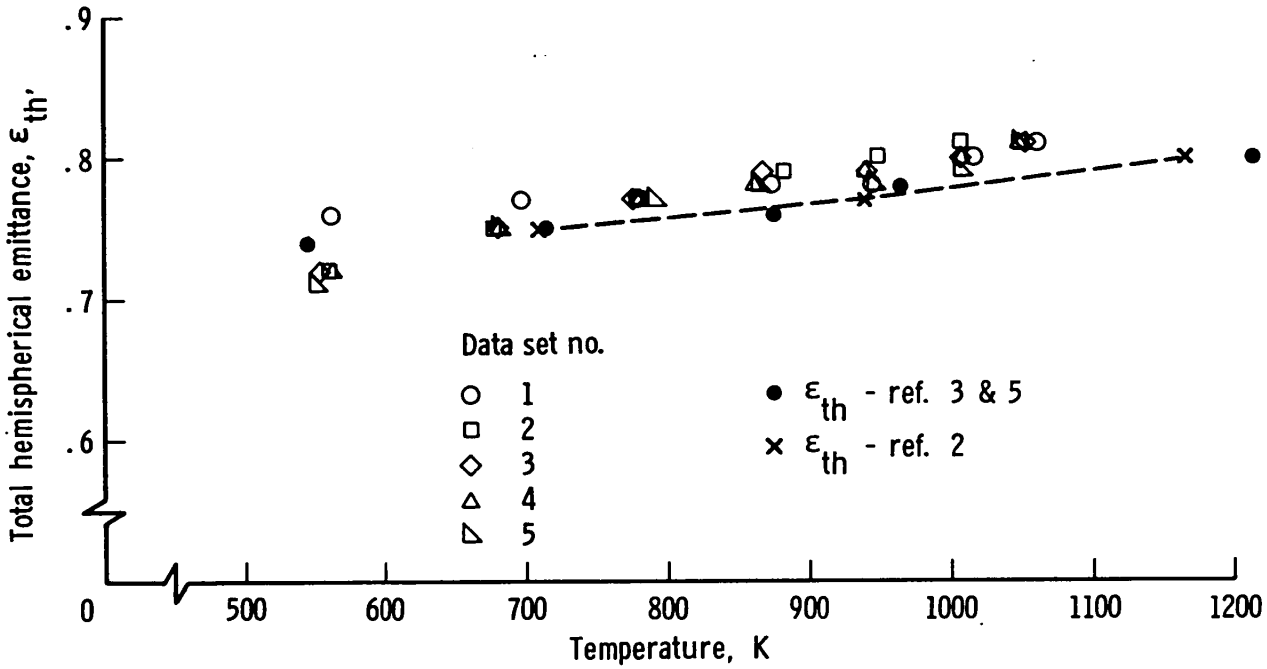


Figure 4. - Total hemispherical emittance data versus temperature for oxidized Rene' 41

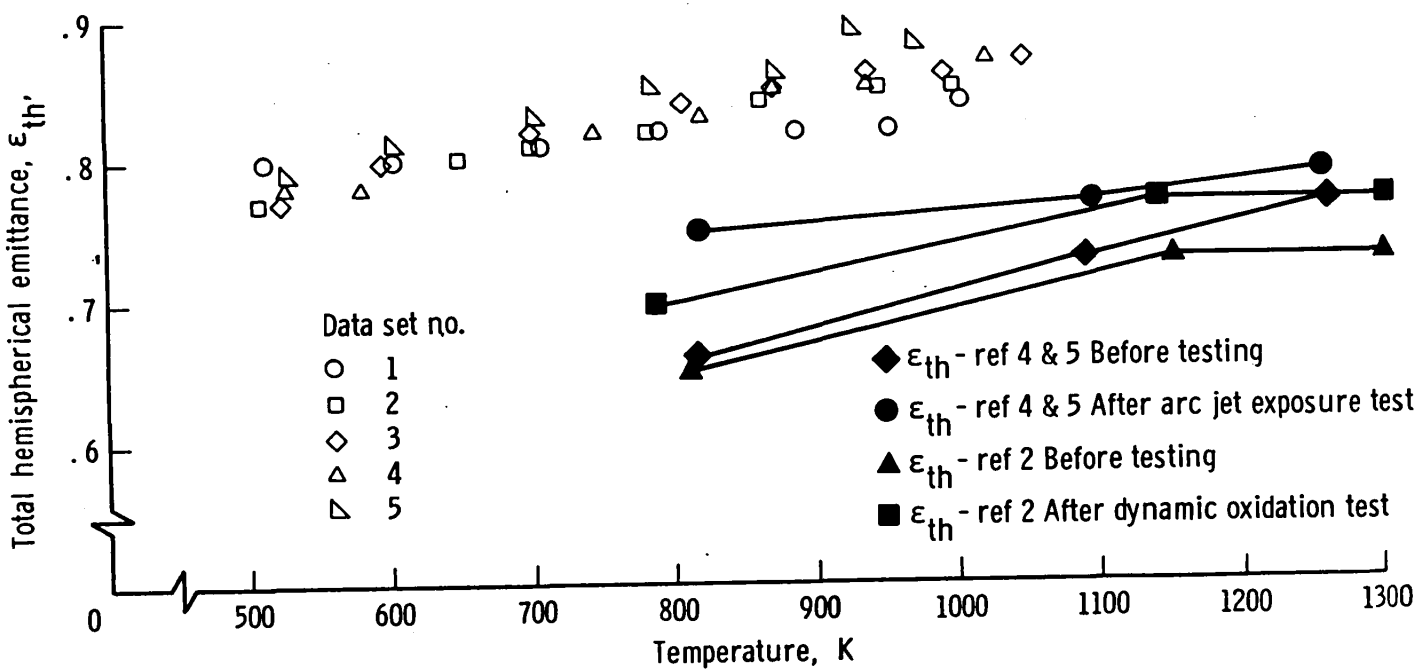


Figure 5. - Total hemispherical emittance data versus temperature for oxidized Haynes 188

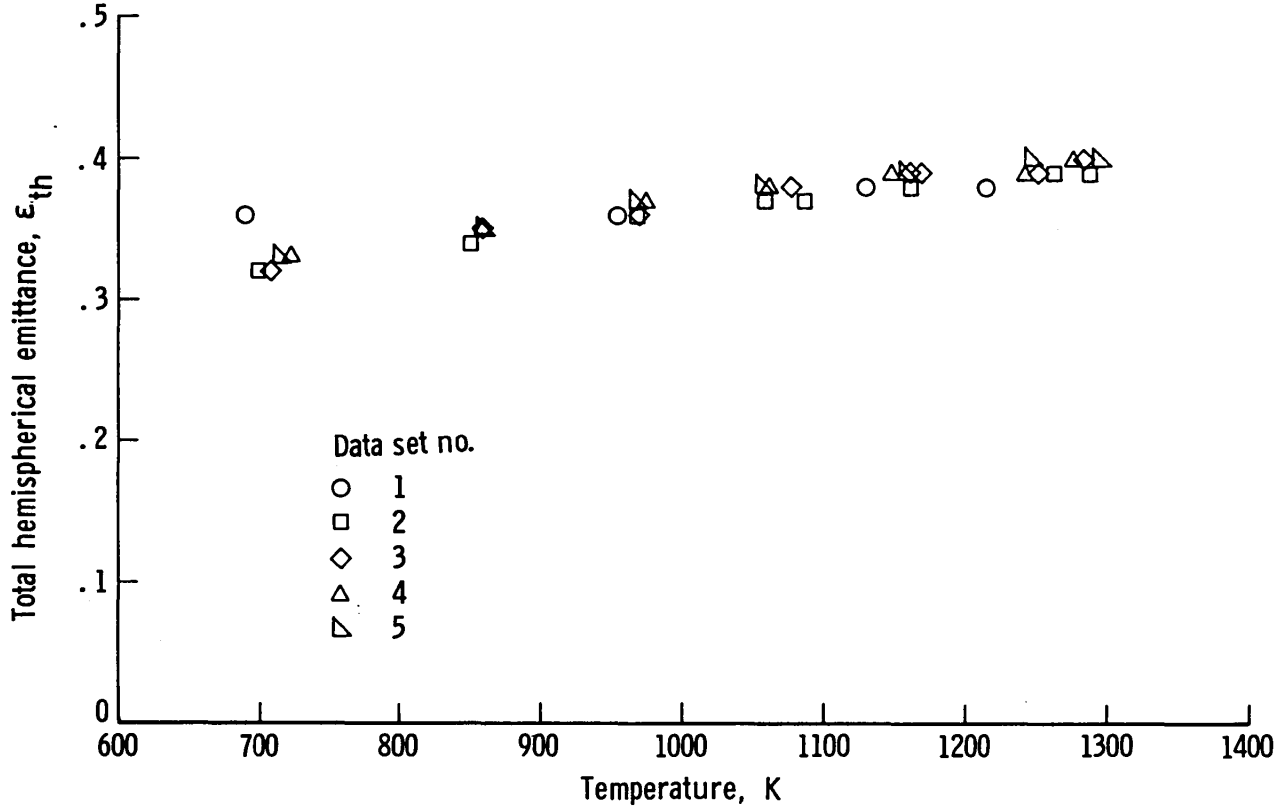


Figure 6. - Total hemispherical emittance data versus temperature for Rene' 41

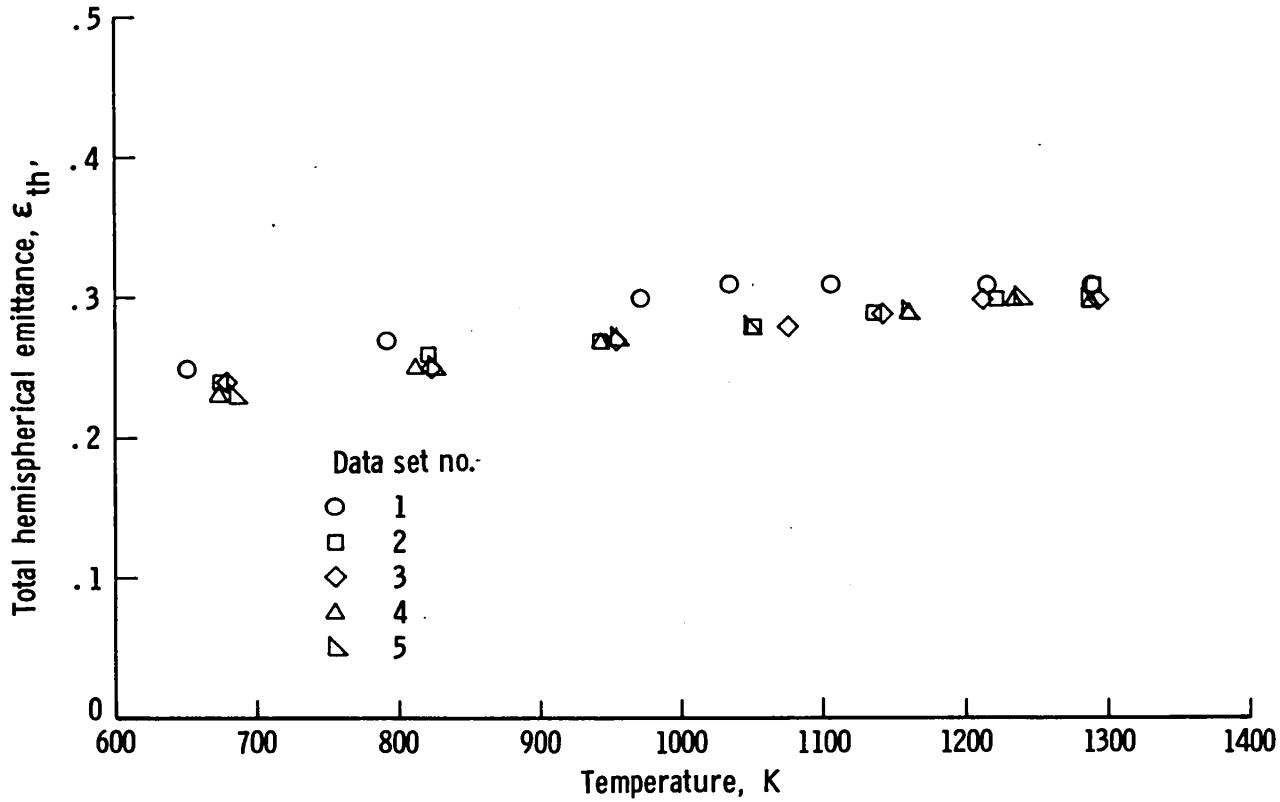


Figure 7. - Total hemispherical emittance data versus temperature for Haynes 188

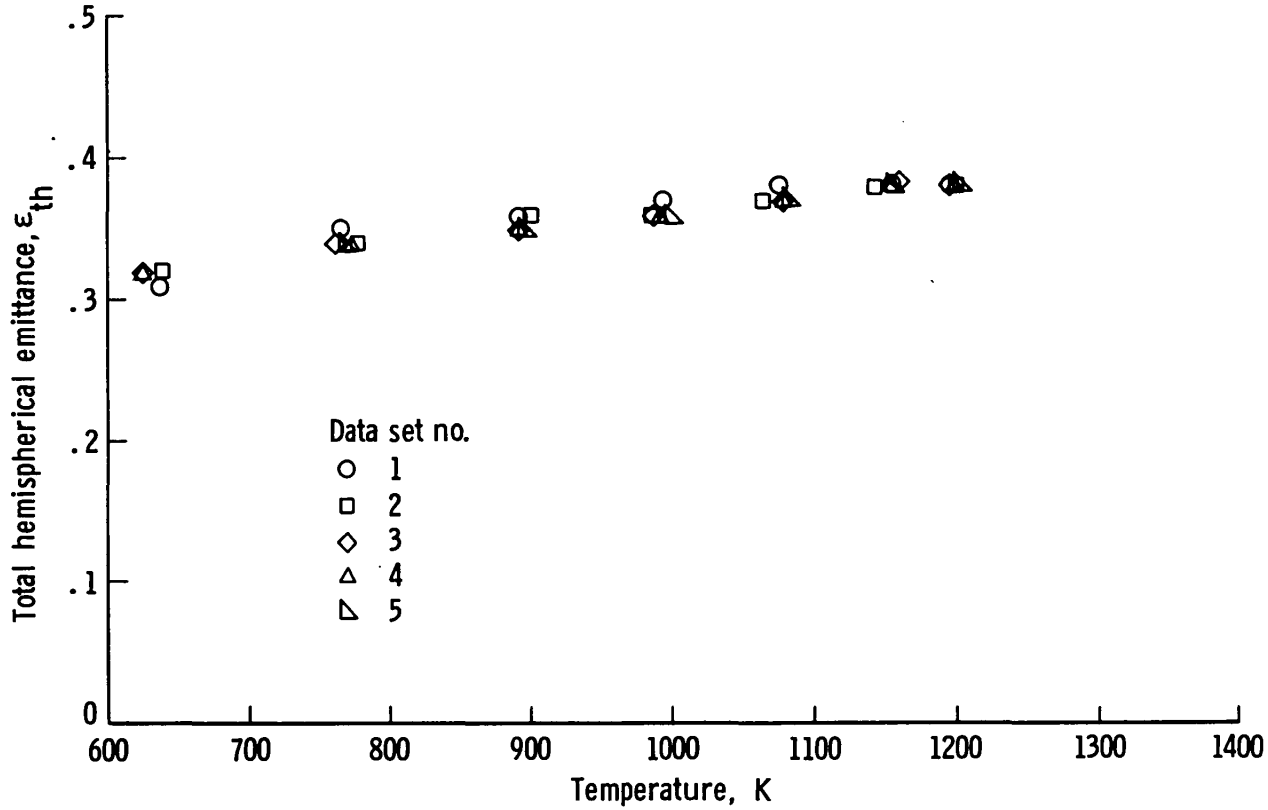


Figure 8. - Total hemispherical emittance data versus temperature for Inconel 625

Standard Bibliographic Page

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16. Abstract

Emittance tests were made on samples of Rene' 41, Haynes 188, and Inconel 625 superalloy metals in an evaluation of a standard test method for determining total hemispherical emittances of surfaces from 293 K to 1673 K. The intent of this evaluation was to address any problems encountered, check repeatability of measured emittances, and gain experience in use of the test procedure. Five test specimens were fabricated to prescribed test dimensions and surfaces cleaned of oil and residue. Three of these specimens were without oxidized surfaces and two with oxidized surfaces. The oxidized specimens were Rene' 41 and Haynes 188. The tests were conducted in a vacuum where the samples were resistance-heated to various temperature levels ranging from 503 K to 1293 K. The calculated results for emittance, in the worst case, were repeatable to a maximum spread to ±4% from the mean of five sets of plotted data for each specimen.

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