NATIONAL BUREAU OF STANDARDS REPORT

8175

EQUIPMENT FOR THERMAL EMITTANCE MEASUREMENTS ABOVE 1400°K

ANNUAL SUMMARY REPORT

January, 1964

Contract No. H-41986
04-84-3-75-0905-09-000-8400-2561-1
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80X0108 (63) R&D, MSFC, OBJECT 25

GEORGE C. MARSHALL SPACE FLIGHT CENTER
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
Huntsville, Alabama

NBS

U.S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS

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NATIONAL BUREAU OF STANDARDS REPORT

NBS PROJECT

1009-11-10493

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Prepared for

George C. Marshall Space Flight Center
National Aeronautics and Space Administration
Huntsville, Alabama

U.S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS

FOREWORD

A research program aimed at developing equipment and procedures suitable for reliable measurements of the total and spectral emittance of both metals and nonmetals in the temperature range 1400 - 2500°K was initiated on January 1, 1961 at the National Bureau of Standards under the sponsorship of the George C. Marshall Space Flight Center of the National Aeronautics and Space Administration. The contract was under the technical supervision of Dr. Klaus Schocken, M-RP-T, Bldg. 4488.

The present report summarizes the progress that was made during the calendar year 1963. The report was prepared by D. G. Moore of the National Bureau of Standards staff (Div. 10, Sec. 9) who served as the group leader during the course of the investigation. Staff personnel who were active in the investigation included H. E. Clark, F. J. Kelly, J. L. Alderman, J. C. Richmond and W. N. Harrison.

ABSTRACT

The furnaces used with the rotating cylinder equipment were redesigned to give improved performance at the higher operating temperatures. At the same time an automatic temperature control system was incorporated that permitted the specimen and reference blackbody thermocouples to be maintained at the same temperature to within ± 0.5°K.

Normal spectral emittances from 1 to 15μ were measured for platinum at 1400°K and for 11 ceramic oxide specimens at 1200°K. Room-temperature reflectance for six of these oxide materials was measured from 0.26 to 2.10μ under conditions of near normal illumination and hemispherical viewing. The normal spectral emittances computed from these reflectances were then compared in the range 1.0 to 2.1μ with emittances measured with the rotating-specimen equipment.

The Gouffé expression as used in the shallow-hole method was tested experimentally through use of a reflectance approach. The theoretical values were in excellent agreement with the experimental values, thus indicating that no significant error is introduced when the Gouffé equation is used for converting flux ratios to emittances; the same study showed, however, that an error will occur if the specimen is formed from a material that is a poor diffuser of radiant energy.

A few total normal emittance measurements on specimens of beryllium were made in the temperature range 1000 - 1550°K.

Two manuscripts were prepared describing different phases of the program. Both papers were accepted for presentation at the March 1964 Symposium on the Thermal Radiation of Solids.

I. OBJECTIVES

The primary objective of this project is to develop reliable and accurate techniques for the measurement of the thermal radiation properties of both metals and nonmetals at temperatures up to 2500°K. A secondary objective is to provide emittance data of known accuracy for materials of interest to the space program.

Two measurement approaches are being used in the investigation. The first is for normal spectral emittance (1 to 15μ) in the range of about 1000° to 1800°K (rotating-cylinder method) and the second for total normal emittance at temperatures as high as 2500°K (induction-heating method). In both approaches emphasis has been placed on devising measurement procedures that will reduce temperature gradients in nonmetals and that will permit the specimen and the reference blackbody to operate at very nearly the same temperature.

Progress made in perfecting both types of measurement during the period Jan. 1, 1963 to Jan. 1, 1964 is described in the present report.

II. ROTATING SPECIMEN METHOD FOR MEASUREMENTS TO 1800°K (2780°F)

A. Description of Method

In the rotating specimen method a hollow cylindrical specimen, 1" outside diameter with a 1/8" wall, is rotated in a platinum-wound furnace equipped with a water-cooled viewing port. Because the specimen is rotating, a freshly heated surface is arriving continuously at the port. The method is based on the premise that at a sufficiently high speed of rotation, the temperature change of any given area on the specimen surface while passing the port will be too low to significantly affect the measurements and, also, that temperature gradients from the surface inward will be largely eliminated. A theoretical analysis of the periodic heat flow in such a specimen, which was performed as one phase of the investigation, has been published (Ref. 1). Although this analysis was valuable in predicting the general pattern of temperature fluctuations in the specimen with increased speed, the expressions were not useful for quantitative calculations because reliable thermal property data were not available on the specimen materials. Instead a method was devised for determining the minimum permissible speeds experimentally. This was done by increasing the speed in increments until the apparent emittance as measured at 1.4μ reached a constant value. Details of this experimental method are given in Ref. 2.

The emittance measurements are made by comparing the radiant flux density from the rotating specimen with the flux density from a blackbody at the same temperature. This is done by using suitable transfer optics to focus the two beams onto the entrance slits of a double-beam monochromator which continuously records the ratio of the flux densities in the two beams. Possible error from uncompensated differences in optical conditions affecting the beams is largely eliminated by using a second blackbody furnace to obtain a calibration or "100% curve" prior to obtaining the specimen data. Figure 1 is a schematic showing the arrangement of the equipment and the optical paths.

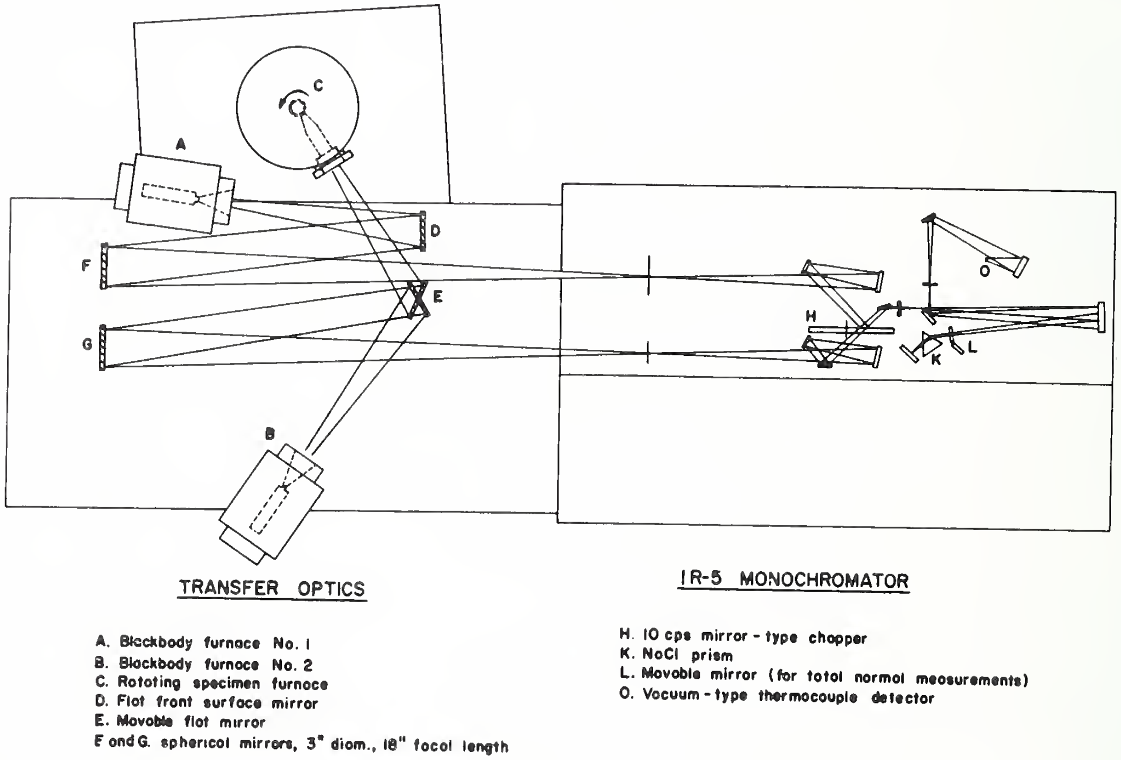

Figure 1. Optical paths in rotating-cylinder equipment.

The spectrometer being used for the measurements is a modified Beckman IR-5A. Before modification this instrument provided a linear response to within 1%. However, because the modifications that were made to convert it for emittance measurements could have disturbed this linearity, an independent test of linearity was made after the equipment was installed and ready for operation.

To conduct this test, the two blackbodies were first adjusted to 1200 ± 1°K. A sector-disc attenuator was then inserted into the beam from blackbody no. 2 (B in Fig. 1) and rotated at high speed (> 500 RPM). Six different discs were used which had nominal transmittances of 75.1, 50.0, 25.3, 12.7 and 5.1%. The resulting spectral curves, which were found to be reproducible to within 2%, showed that instrument response was lower than the actual beam attenuation. This lowering was the same at all wavelengths. Since no simple method could be devised to restore linearity to the instrument, a correction curve (Fig. 2) was prepared and used to correct all spectral emittance values that were obtained from the recorder charts.

Further details relative to the procedures used for making measurements are given in ref. 2.

B. Modification of Blackbody Furnaces

The blackbody furnaces described in the preceding annual summary report were found to have a short operating life at temperatures above 1400°K. Hence, it was necessary to incorporate several new design features and rebuild the furnaces so as to achieve the required trouble-free operation at the higher test temperatures.

Fig. 3 is a schematic of the new design. The inner cavity is formed of fused alumina bonded with 20% by weight of a calcium aluminate cement. This mixture, which sets hydraulically, was mixed with water and vibrated into a greased plaster mold. The mold was made by using a Norton alumina core[1] as a pattern; a brass mandrel was positioned at the center of the mold to form the outline of the cavity. After allowing 20 hrs. for curing, the core was removed from the mold, dried in an oven for 24 hours, and then heated to 1925°K for 1 hr. prior to the winding with 0.032 in. diam. platinum-40% rhodium resistance wire. As shown in Fig. 3, the spacing of the winding was varied to minimize temperature gradients. No power taps were used on the windings.[2]

The alumina core material has a low emittance in the wavelength region 1 to 5μ. Inasmuch as the cavity emittance depends, in part, on the emittance of the walls, it was necessary to line the cavity with a coating that would have high emittance at the short as well as the long wavelengths. The coating developed for this purpose consisted of black nickel oxide (Ni_2O_3) bonded with 7.5% by weight of clay. This coating was prepared as a water suspension (slip) and applied at a thickness of about 0.005 in. to the inner wall surfaces of the cavity by first pouring the slip into the cavity, allowing it to remain for a few seconds, and then pouring it out again. After drying, the core was heated to 1850°K for 1 hr. to bond the coating to the alumina. The spectral emittance of a cylindrical specimen of the core material coated in this way was found to be above 0.875 at all wavelengths from 1 to 15μ; hence, the lowest possible cavity emittance, at any wavelength from 1 to 15μ, when computed by the expression given by Gouffé (Ref. 3) was 0.990.

Temperature gradients in the two blackbody furnaces were measured at each of the four test temperatures by means of a calibrated Pt vs Pt-10% Rh thermocouple inserted into the furnace through the entrance slit. The arrangement was such that the bead of the thermocouple could be positioned within 1/8 in. of the core wall at any point along the length of the cavity.

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[1] Norton RA139 core no. 10445 (Norton Co., Worcester, Mass.)

[2] Power taps were used in a preliminary design. Temperature gradients could be minimized by increasing power to the ends; however, this procedure caused overloading of the end coils with a subsequent short operating life of the furnace at 1800°K.

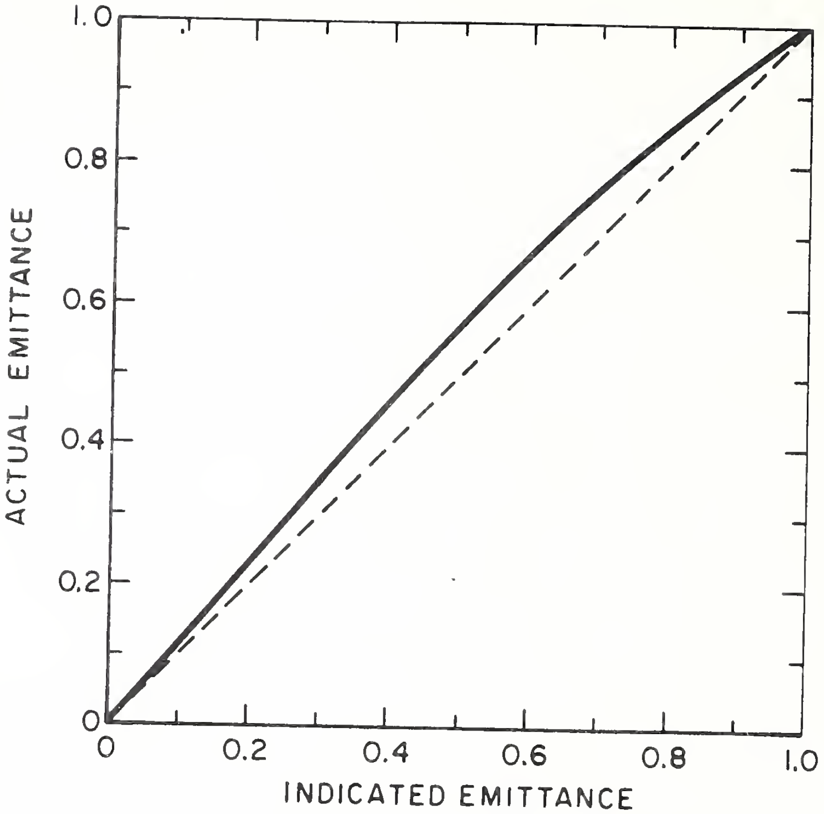

Fig. 2. Calibration curve (solid line) for correcting for lack of instrument linearity. Dashed line shows type of behavior desired of an instrument (linear response).

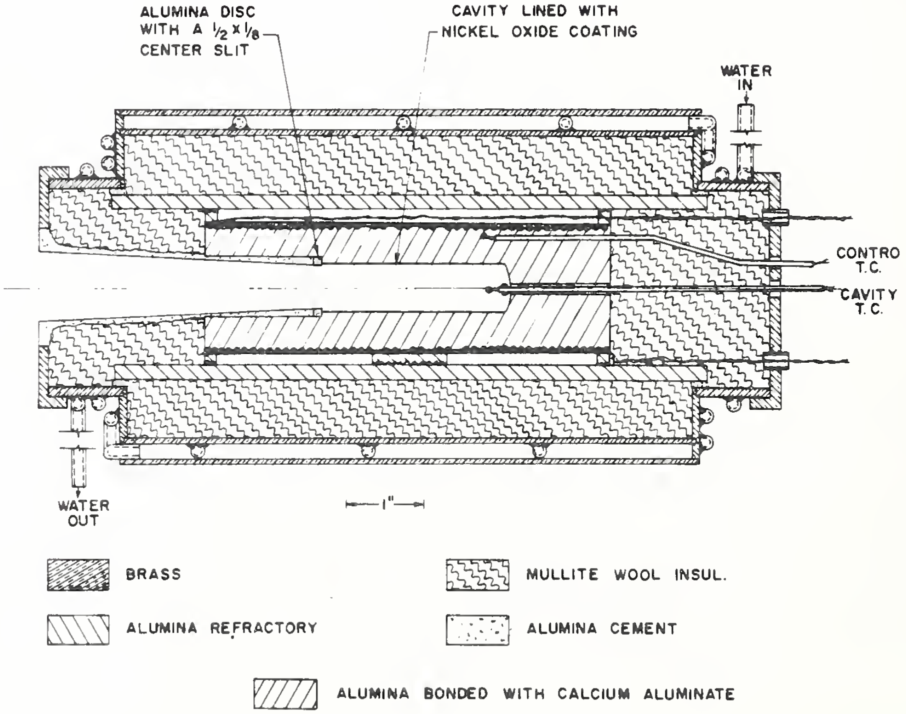

Fig. 3. Schematic of blackbody furnace.

These measurements showed that at 1800°K the back wall of the cavity was 2°K below the 1½ in. long isothermal region near the center and the front of the cavity (rear wall of the entrance slit) was 3°K below the isothermal region. The cavity thermocouple (see Fig. 3) gave an emf corresponding to the temperature of the isothermal region of the cavity.

C. Modification of Specimen Furnace

Preliminary trials indicated that because of large heat losses, the temperature of the specimen furnace could not be raised above about 1600°K. The furnace was therefore rebuilt with the following changes: (a) a more durable wire (60% Pt;40% Rh) was used for the windings, (b) a new top insert with improved thermal insulation properties was added, (c) platinum radiation shields were placed at both the top and bottom of the furnace tube, and (d) gold wire rather than copper was used for bringing the power leads through the glass-metal seals.

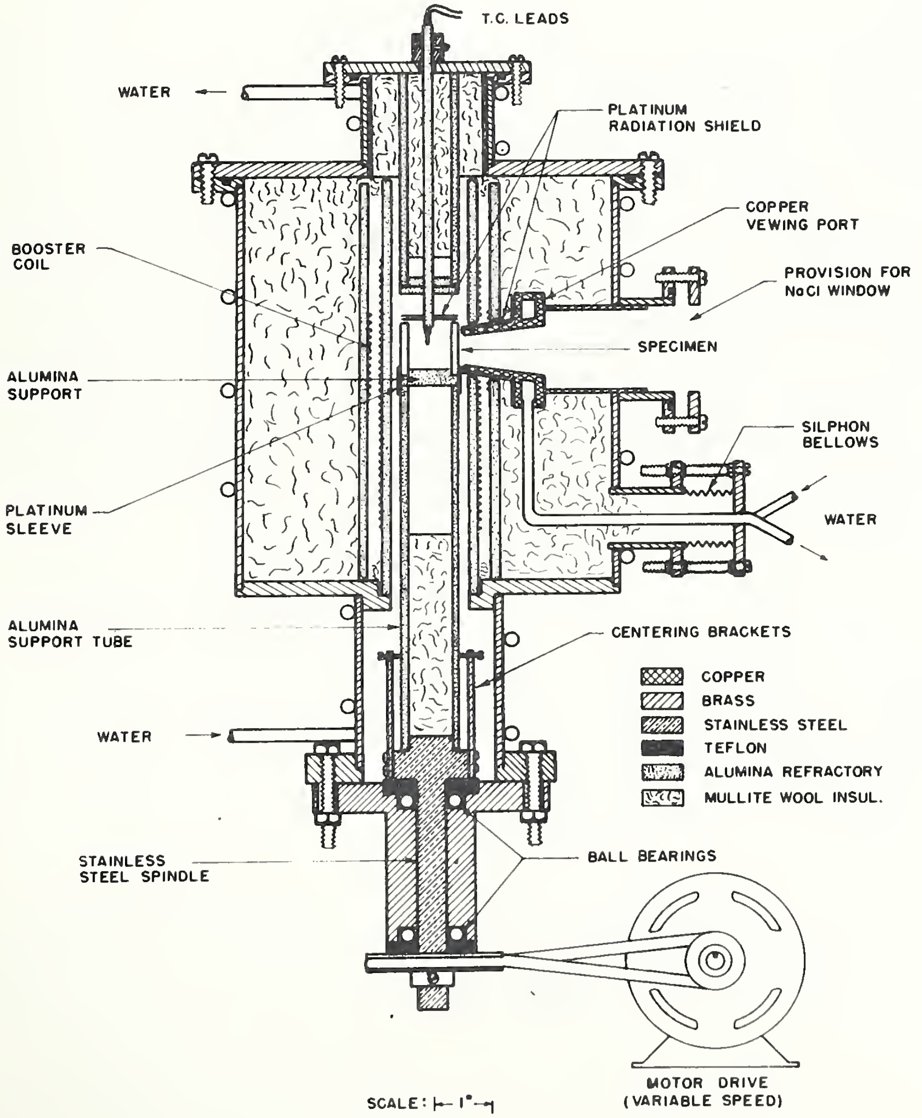

Fig.4. Schematic of rotating-specimen furnace.

Fig. 4 is a schematic of the modified design. Although this particular version was completed late in the contract year, it has nevertheless been operated at 1800°K on numerous occasions for periods of several hours without difficulties developing. Power requirements at 1800°K are moderate and there is no overheating of the shell. Also, the refractory pedestal has shown no evidence of distortion.

Adjustment of power to the booster coil is used to control vertical gradients in the specimen furnace which would otherwise occur because of the large heat losses through the viewing port. When the power to the booster coil is adjusted at the proper level, no thermal gradient can be detected from top to bottom on a rotating alumina specimen when temperatures are measured with a micro-optical pyrometer sighted on the specimen through the viewing port. The pyrometer is capable of detecting differences of as small as 2°K.

D. Temperature Control System

The measurements given in the preceding annual summary report were obtained after adjustment of the temperature of the specimen furnace to that of blackbody no. 1 by manual control of power input. This manual control was not only time consuming but in those cases where the temperature of the specimen furnace changed slightly during measurement, the data had to be discarded and the test repeated. To eliminate these difficulties, a differential thermocouple control system was installed during the contract year. This system is similar to the one described by Harrison, et al (Ref. 4). The thermocouples in both furnaces were made from calibrated Pt;Pt-10% Rh wires taken from the same spools. The new controller meters the power into the specimen furnace as required to attain a zero temperature difference between the two thermocouples. Temperature equality between the two furnaces is reached in as short a time as 5 min.; the temperatures of the two furnaces can then be automatically maintained to better than 1°K until such time as the control point is changed. The new system is expected to improve not only the precision of the spectral measurements, but also it will appreciably reduce the time required to complete a series of determinations.

E. Emittance Measurements

Because of the large amount of time spent in refining the equipment during the contract year it was not possible to obtain large quantities of emittance data. However, some preliminary measurements were made in air on specimens of ceramic oxides at 1200°K and also on platinum at 1400°K. The measurements are included in this summary report to indicate, in a general way, the capabilities of the equipment; they should not be interpreted as representing highly accurate data on well characterized specimens.

Table 1 identifies the specimens that were measured. All were in the form of cylinders approximately 1 in. outside diameter with smooth surface finishes. The wall thickness of the platinum specimen was approximately 1/16 in.; that of the sintered ceramic specimens approximately 1/8 in.

Table 1. Identification of platinum and sintered ceramic specimens.

Letter Symbol	Material	Description[a]	No. of Spec. Tested	Porosity in % Pores by Vol.
P	Platinum	Commercially pure	1	0
AN	Alumina	Coarse grained furnace tube refractory; > 95 Al_2O_3	1	45
AD	Alumina	Fine grained, dense; 96 alumina	5	11
AL	Alumina	Fine grained; 99 + Al_2O_3	3	36
CZ	Calcium Zirconate	Fine grained body CaO 31; ZrO_2 68	1	30
M	Magnesium Oxide	Fine grained magnesia 99.2 MgO	2	35
AS	Mullite	Fine grained body Al_2O_3 67; SiO_2 30	1	32
S	Silica	Fine grained body[b] SiO_2 95; Al_2O_3 4.0	1	8
MA	Spinel	Fine grained body Al_2O_3 71; MgO 28	1	25
T	Thoria	Fine grained; 99+ ThO_2	1	36
ZS	Zircon	Fine grained body ZrO_2 66; SiO_2 32	2	26
Z	Zirconia	Fine grained, lime stabilized ZrO_2 + HfO_2 94; CaO 4.5	1	18

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[a] Compositions are nominal for major constituents in percent by weight.

[b] Major crystalline phases - crystobalite and quartz.

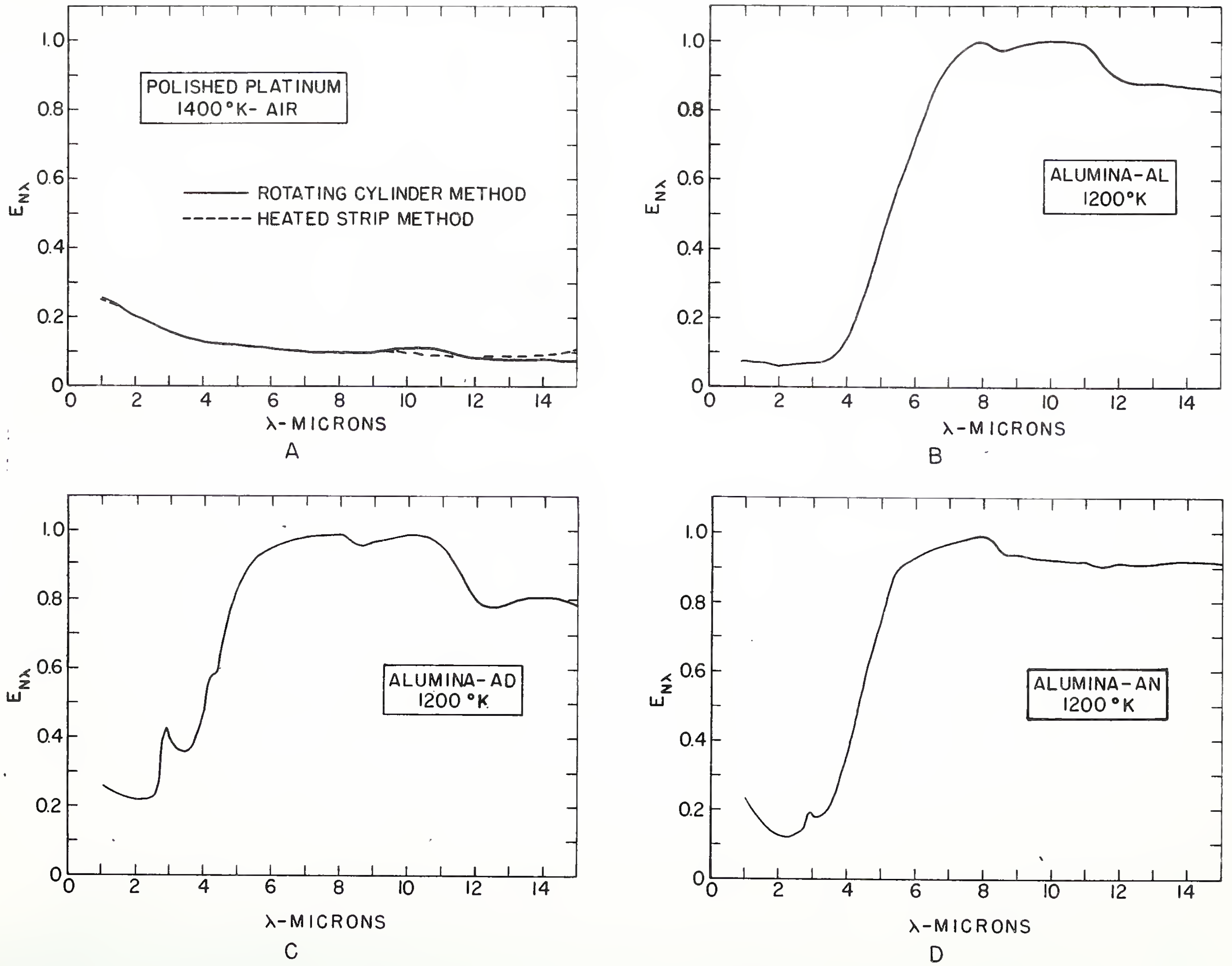

Figure 5. Spectral emittance curves of polished platinum and three types of sintered alumina.

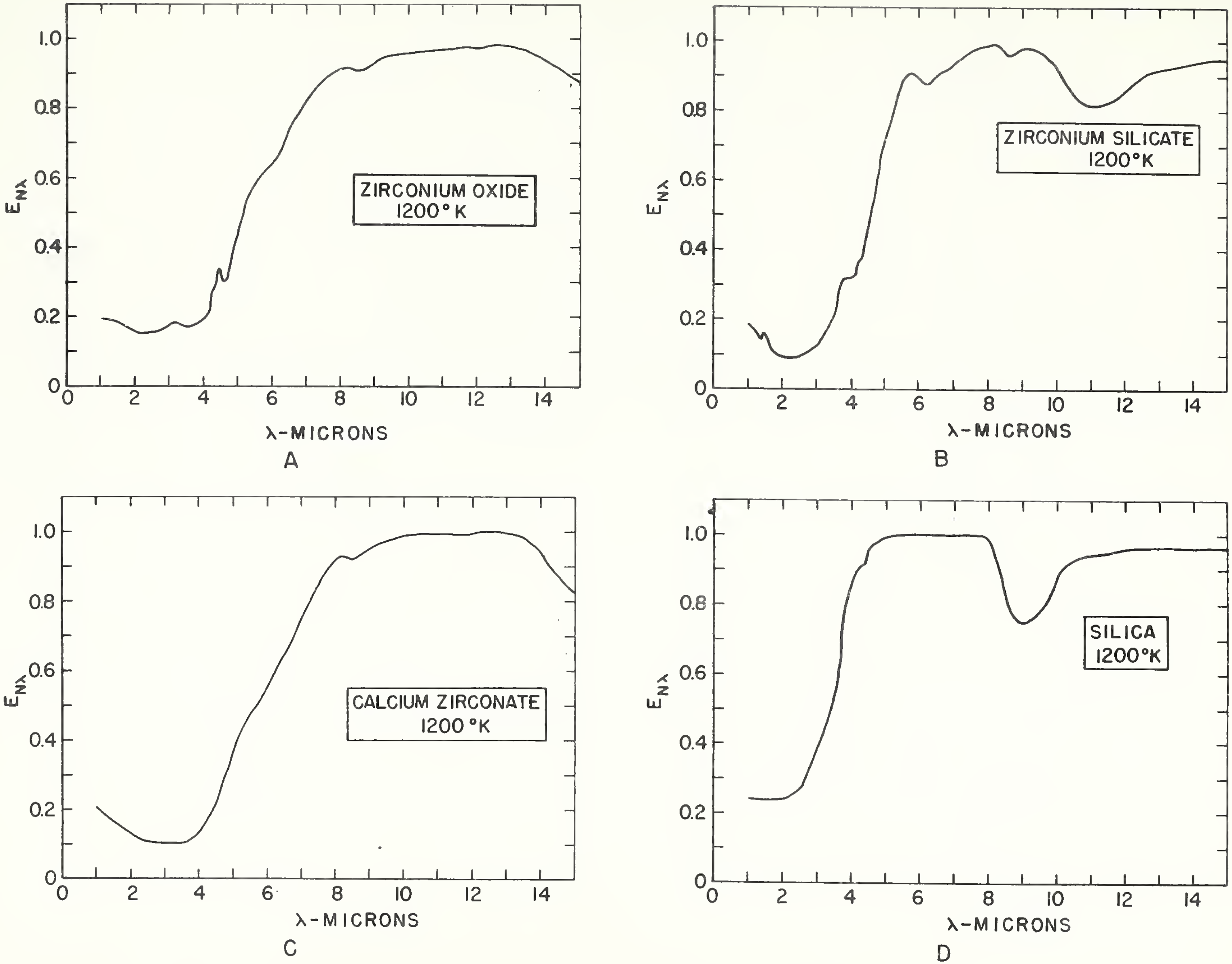

Figure 6. Spectral emittance curves for sintered ceramic oxide specimens.

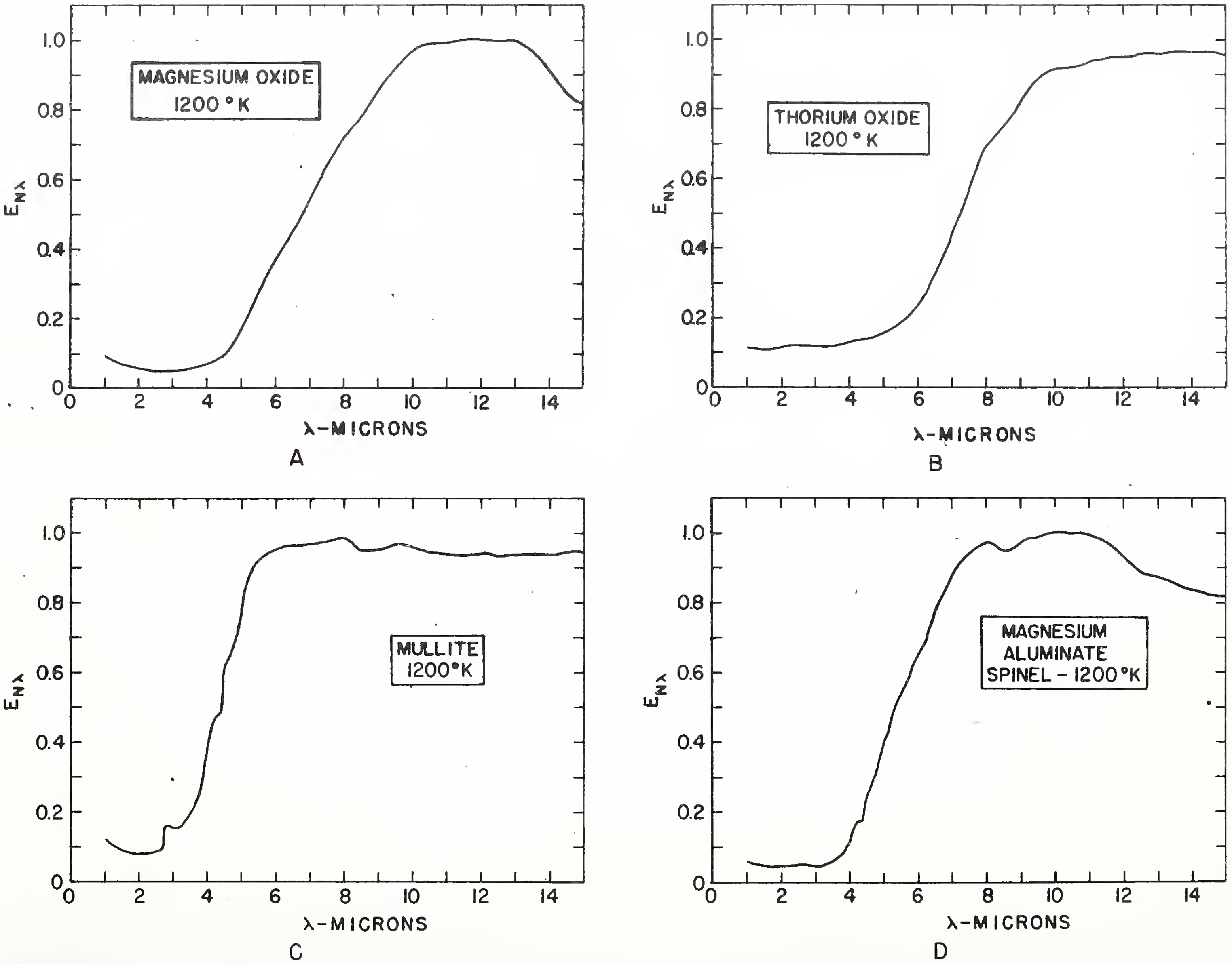

Figure 7. Spectral emittance curves for sintered ceramic oxide specimens.

The spectral curves are shown in Figs. 5, 6 and 7. Fig. 5A is for a polished platinum specimen that had been annealed for one hour in air at 1525°K prior to testing. The agreement of the curve determined with the rotating specimen method with that reported by Harrison, et al (Ref. 5) for polished platinum as measured by a carefully calibrated heat-strip method is good and these data, by themselves, indicate that the transfer optics and monochromator are capable of satisfactory operation.

Fig. 5B, 5C and 5D show the curves obtained for three types of sintered alumina. All three of these curves are in fair agreement with data for alumina specimens reported by Blau (Ref. 6) and by Slemp and Wade (Ref. 7) at wavelengths beyond about 5μ, but below 5μ the present measurements give values which are more in keeping with those reported by Folweiler (Ref. 8). It should be pointed out, however, that cross comparison of emittance measurements are of only limited value unless the specimens are prepared from the same material by the same processing and with the same surface finish. Such factors as grain size, porosity, and the presence of impurities are known to have an appreciable effect on measured emittance.

The emittance peak at 2.9μ for AD and AN alumina (Fig. 5C and 5D) was obtained for all specimens of this material that were tested. A similar peak has been noted for flame-sprayed alumina (Ref. 9 and 10). It is probably caused by chemically-held water in the structure. An observation made in the present measurements that the intensity of the peak decreased with time of heating at 1200°K tends to support this explanation. A similar maximum was observed for mullite (Fig. 7C).

With the exception of the emittance peak at about 4.6μ for zirconia (Fig. 6A) and the low emittance band for silica at about 9.0μ (Fig. 6D), the other deviations are relatively minor and need confirmation by additional measurements especially at higher test temperatures before they can be fully accepted. The low emittance band for silica at 9.0μ has been observed previously (Ref. 11).

Of the 11 ceramic materials measured, thorium oxide and magnesium oxide have the longest wavelength region of low emittance. The emittance of thorium oxide does not reach 0.9 until the wavelength is approximately 10μ; the wavelength at which this emittance is attained by magnesium oxide is about 9μ. This behavior should be reflected in the computed total normal emittances and Table 2 shows that such is the case. These two oxides gave the lowest total normal emittances at 1200°K of any of the oxides measured; the silica specimen had the highest.

The selected ordinate method (Ref. 5) with 25 ordinates was used for computing the total normal emittances from the spectral data.

Table 2. Total normal emittances at 1200°K as computed from spectral data.

Material	Ident. Symbol	ϵ_{TN}	Material	Ident. Symbol	ϵ_{TN}
Aluminum Oxide	AL	0.29	Silica	S	0.60
Aluminum Oxide	AD	0.52	Calcium Zirconate	CZ	0.29
Aluminum Oxide	AN	0.42	Zirconium Silicate (Zircon)	ZS	0.39
Zirconium Oxide	Z	0.34	Aluminum Silicate (Mullite)	AS	0.40
Thorium Oxide	T	0.23			
Magnesium Oxide	M	0.20	Magnesium Aluminate (Spinel)	MA	0.26

F. Room Temperature Reflectance Measurements

Because of the many possible sources of error in measuring spectral emittance of ceramic materials, especially in the low wavelength region, it seemed desirable to test the reliability of the preliminary emittance data by means of spectral reflectance measurements. Spectral reflectance of an opaque material determined under conditions of normal illumination and hemispherical viewing is the complement of normal spectral emittance; hence, reflectances determined in this way can be converted to emittances by subtracting them from unity. Since the spectral emittance varies only slightly with temperature, such measurements should provide a reasonably good check of the emittance data even though the spectral reflectances were obtained at room temperature.

A Cary Model 14M recording spectrophotometer equipped with an integrating sphere was used for these reflectance measurements. Data were obtained over three different ranges (0.26 to 0.4μ, 0.36 to 0.7μ, and 0.6 to 2.1μ) with different source-receiver combinations for each range. The specimens were flat discs, 1½ in. diam. by ¼ in. thick, prepared of materials AL, M. Z, ZS, CZ, and T (Table 1). The discs were prepared of the same materials and with the same processing used for the 1 in. diam. cylinders.

The measurements were made relative to smoked magnesium oxide and corrected to "absolute" values using the data of Middleton and Sanders (Ref. 12 and 13). The values used for conversion are listed in Table 3, while the reflectance curves, corrected through use of these data, are shown in Figs. 9, 10 and 11. The curves in each case were plotted from the average values obtained from two specimens. These values were in good agreement over the entire range except for the two magnesium oxide specimens in the region 0.26 to 0.60μ where deviations in spectral reflectance of as great as 0.2 were observed. Because of this poor agreement, the average curve for magnesium oxide is not shown below 0.6μ (Fig. 8B).

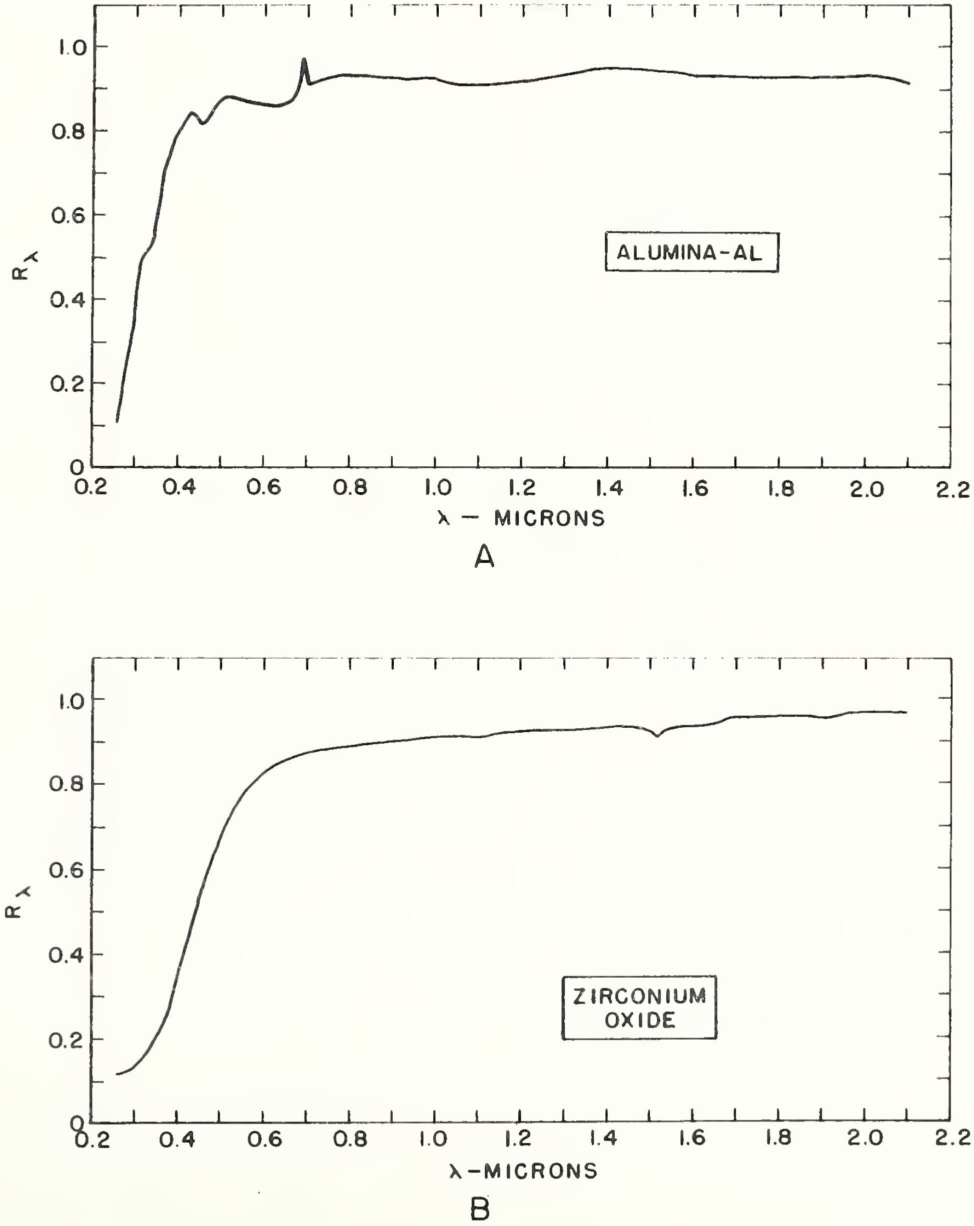

Fig. 8. Room-temperature spectral reflectance curves for alumina and zirconia specimens.

Table 3. Spectral diffuse reflectances of smoked magnesium oxide.

λ	R_λ [a]	λ	R_λ [b]	λ	R_λ [b]
0.20μ	0.955	0.80μ	0.965	1.50	.945
.30	.970	.90	.960	1.60	.945
.40	.980	1.00	.960	1.70	.945
.50	.975	1.10	.955	1.80	.940
.60	.970	1.20	.955	1.90	.925
.70	.965	1.30	.950	2.00	.925
.80	.965	1.40	.950	2.10	.930

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[a] Selected from data given by Middleton and Sanders (J. Am. Opt.Soc., 41, 419, 1951).

[b] Taken from Fig. 2 of paper by Sanders and Middleton (J. Am. Opt.Soc., 43, 58, 1953).

G. Comparison of Reflectance and Emittance Measurements

Between 1.0 and 2.0μ it is possible to compare the spectral emittances computed from the reflectance measurements with those obtained with the rotating cylinder equipment. Table 4 shows this comparison at 1.0, 1.5 and 2.0μ.

Considering both the difference in temperature of the two measurements (900°K) and the uncertainty in converting reflectances from relative to "absolute" values, the agreement is surprisingly good for AL, CZ and M; reasonably good for T and ZS; but poor for Z.

In general, spectral emittance increases only very slowly with increasing temperature; hence this comparison at the two different test temperatures is of interest. However, until such time as measurements are available at 1200, 1400, 1600 and 1800°K and temperature coefficients are established, it is not possible to obtain a more valid comparison of the two sets of data. The data listed in Table 4 are nevertheless of value in obtaining an "order of magnitude" comparison.

Table 4. Spectral emittances of oxide specimens at 1.0, 1.5, and 2.0 microns.

Material	Ident. Symbol	Wave-length μ	Normal Spectral Emittance: Rot. Cyl. Method 1200 °K	Normal Spectral Emittance: Comp. from R. T. Refl.
Aluminum Oxide	AL	1.0	0.08	0.08
		1.5	0.07	0.06
		2.0	0.06	0.06
Calcium Zirconate	CZ	1.0	0.21	0.11
		1.5	0.16	0.13
		2.0	0.13	0.11
Magnesium Oxide	M	1.0	0.09	0.10
		1.5	0.07	0.07
		2.0	0.06	0.04
Thorium Oxide	T	1.0	0.11	0.07
		1.5	0.11	0.09
		2.0	0.11	0.06
Zirconium Oxide	Z	1.0	0.19	0.09
		1.5	0.18	0.08
		2.0	0.16	0.03
Zirconium Silicate	ZS	1.0	0.18	0.11
		1.5	0.16	0.21
		2.0	0.09	0.02

H. Discussion of Errors in Spectral Emittance Measurements.

The most serious sources of error in emittance measurement of this type are (1) lack of optical equivalence in the two beams, (2) reflected flux entering the specimen beam, (3) temperature gradients in the specimen, (4) lack of temperature equality between specimen and blackbody, and (5) radiant flux from the specimen cavity being transmitted through the wall of the specimen and into the specimen beam. Any sizeable error from the first of these sources is avoided through use of the two blackbodies to establish the "100% line"; the error from the second source was minimized by maintaining the clearance between the viewing port and the specimen as small as possible; and error from the third source was reduced to a low level by rotating the specimen at high speed.

Lack of temperature equality between specimen and blackbody, although potentially a large source of error, was minimized by reducing the temperature difference, as indicated by the blackbody and specimen thermocouples, to less than 1°K. Even if the temperature of the specimen surface had been as much as 3°K lower than that indicated by the thermocouple at the specimen center, the error in emittance at 1μ for T = 1200°K would be only ±3%. Since the precision with which the emittance could be obtained from the recorder chart was 0.005 units, this error would be detectable only when the emittance was above 0.17. Errors from this source would, of course, be smaller both at higher temperatures and at longer wavelengths.

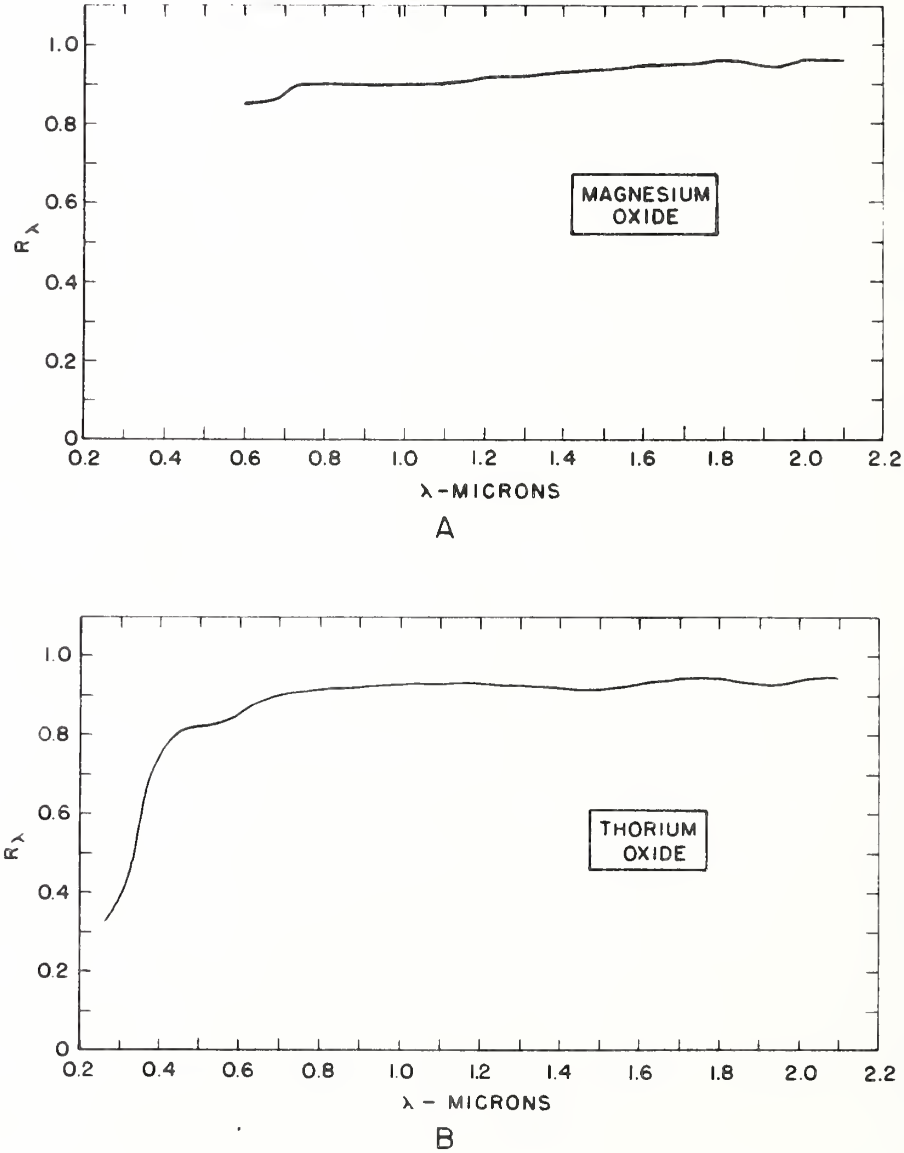

Fig. 9. Room temperature spectral reflectance curves for magnesia and thoria specimens.

The greatest uncertainty that remains in the measurement is the possibility of transmission of blackbody radiation from the specimen cavity through the wall of the specimen. This radiation, if it were present, would reinforce the emitted flux from the specimen and cause the measured emittance to be high. For example, a specimen with a true emittance of 0.15 in the near infrared would show a measured emittance of 0.20 if as little as 5% of the radiation from the specimen cavity was being transmitted through the wall. This amounts to an error of 33%.

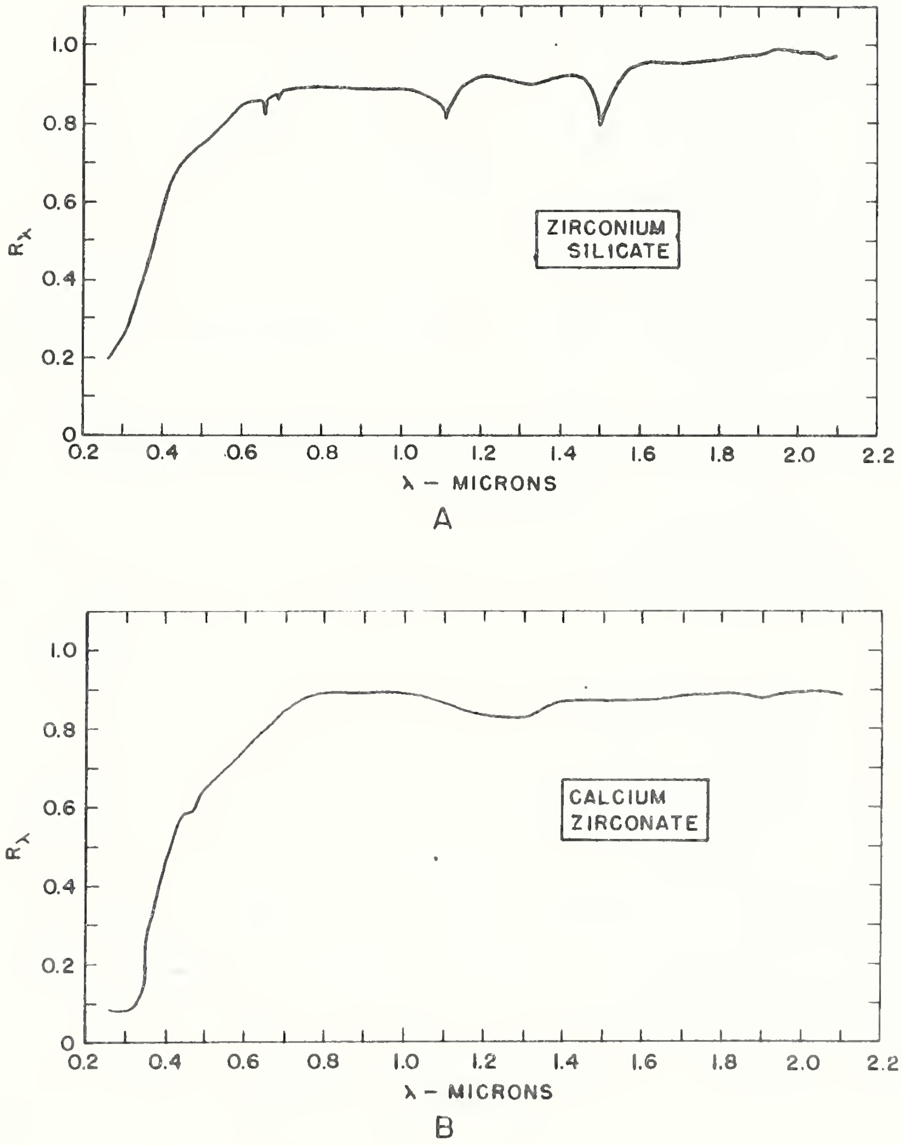

Fig. 10. Room-temperature spectral reflectance curves for zirconium silicate and calcium zirconate specimens.

Since the transmittance of the specimen walls could not be measured readily, an effort was made to obtain a qualitative indication of wall transmission at room temperature. This was accomplished by placing each specimen in the beam of a He-Ne laser ($\lambda = 0.6328$) with the assumption that if the 1/8 in. thick wall was opaque at 0.63μ it would also be opaque in the range of 1.0 to 2.0μ. The relative flatness of the reflectance curves (Figs. 9, 10, 11) in the region 0.6 to 2.1μ suggests that this assumption may be reasonably valid. The results of this qualitative type of test indicated that the ZS, T and Z specimen walls were opaque; the AL walls were nearly opaque; the AD, AN, M and CZ walls were slightly transmitting; and the S and MA walls were appreciably transmitting.

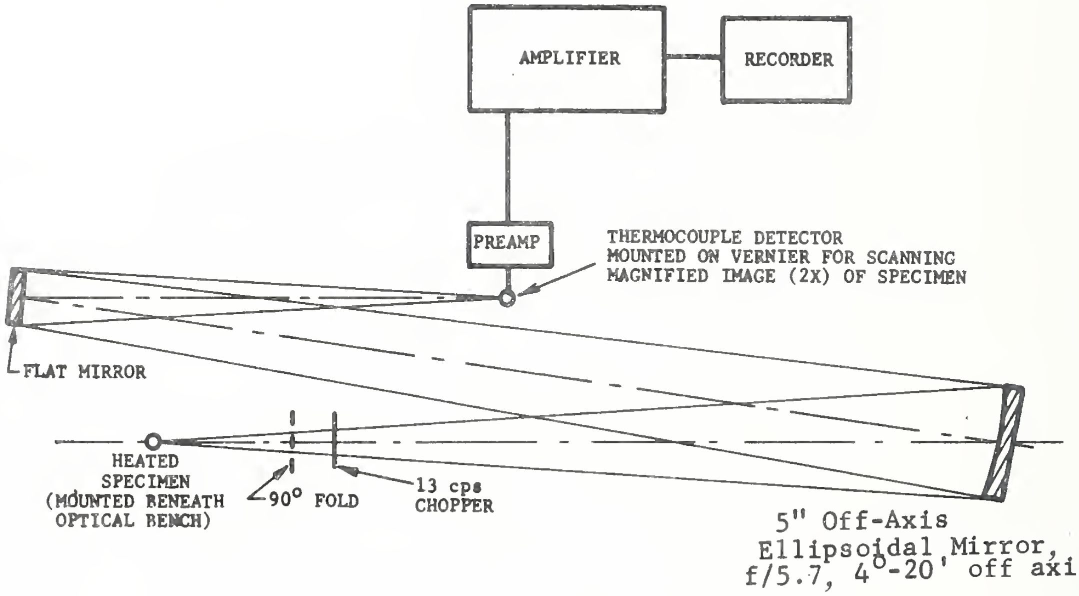

Fig. 11. Schematic of optical paths and electronic components for total normal emittance equipment.

I. Future Plans

The work for calendar year 1964 will include the following:

(1) Factors affecting the accuracy of the rotating-cylinder method will be analyzed and computations will be made to arrive at a realistic value for the accuracy of the measurements.

(2) Spectral emittance data will be obtained for specimens of representative nonmetals at 1200, 1400, 1600 and 1800°K.

(3) The variation with temperature of the spectral emittance of the tested materials will be evaluated and an attempt will be made to arrive at the most probable room-temperature spectral emittance curve from a knowledge of the temperature dependency in the 1200-1800°K range. If this extrapolation proves to be feasible, comparisons will be made of these extrapolated room-temperature emittances with those computed from room-temperature spectral reflectance curves measured on the same materials.

III. INDUCTION HEATING EQUIPMENT FOR TOTAL NORMAL EMITTANCE MEASUREMENTS TO 2500°K (4040°F)

A. Equipment

The design of the induction heating equipment and the reasons for its selection have been given in an earlier report (Ref. 14). Basically, the measurement consists of determining the ratio of the radiant flux density from the surface of a small specimen heated to a high temperature to the flux density from a cylindrical hole drilled into the specimen surface.

The holes are of different dimensions for metals than for nonmetals as indicated in Fig. 11 and the method of arriving at the emittance from the flux ratio is also different.

A schematic of the equipment is shown in Fig. 12. The only major change that was incorporated during the contract year was the substitution of a 5 in. diameter off-axis ellipsoidal mirror for the ordinary spherical mirror used in the early design. This substitution resulted in an improved quality of the image at the detector, which in turn resulted in less error from optical aberrations.

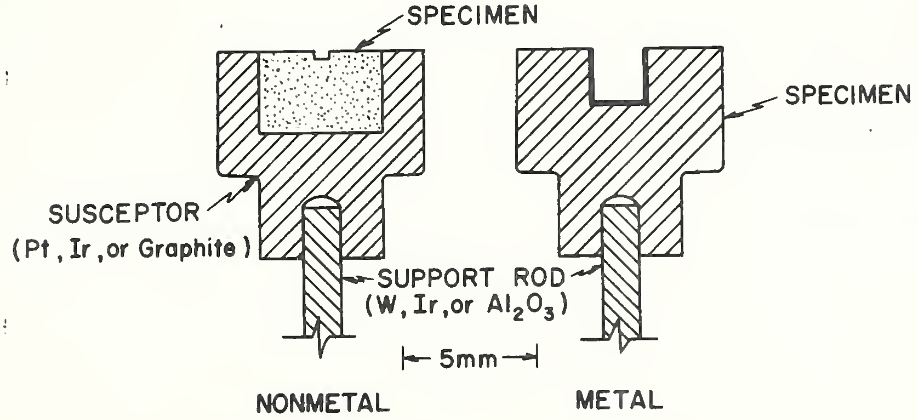

Fig. 12. Two types of specimen used in induction heating equipment. Heavy line on hole walls of metal specimen represents high emittance coating.

B. Measurements on Metals

A number of total normal emittance measurements were made on beryllium specimens submitted by NASA. The specimens were of the type shown in Fig. 12B. The beryllium had a reported purity of 98.5% beryllium metal with BeO as the principal impurity.

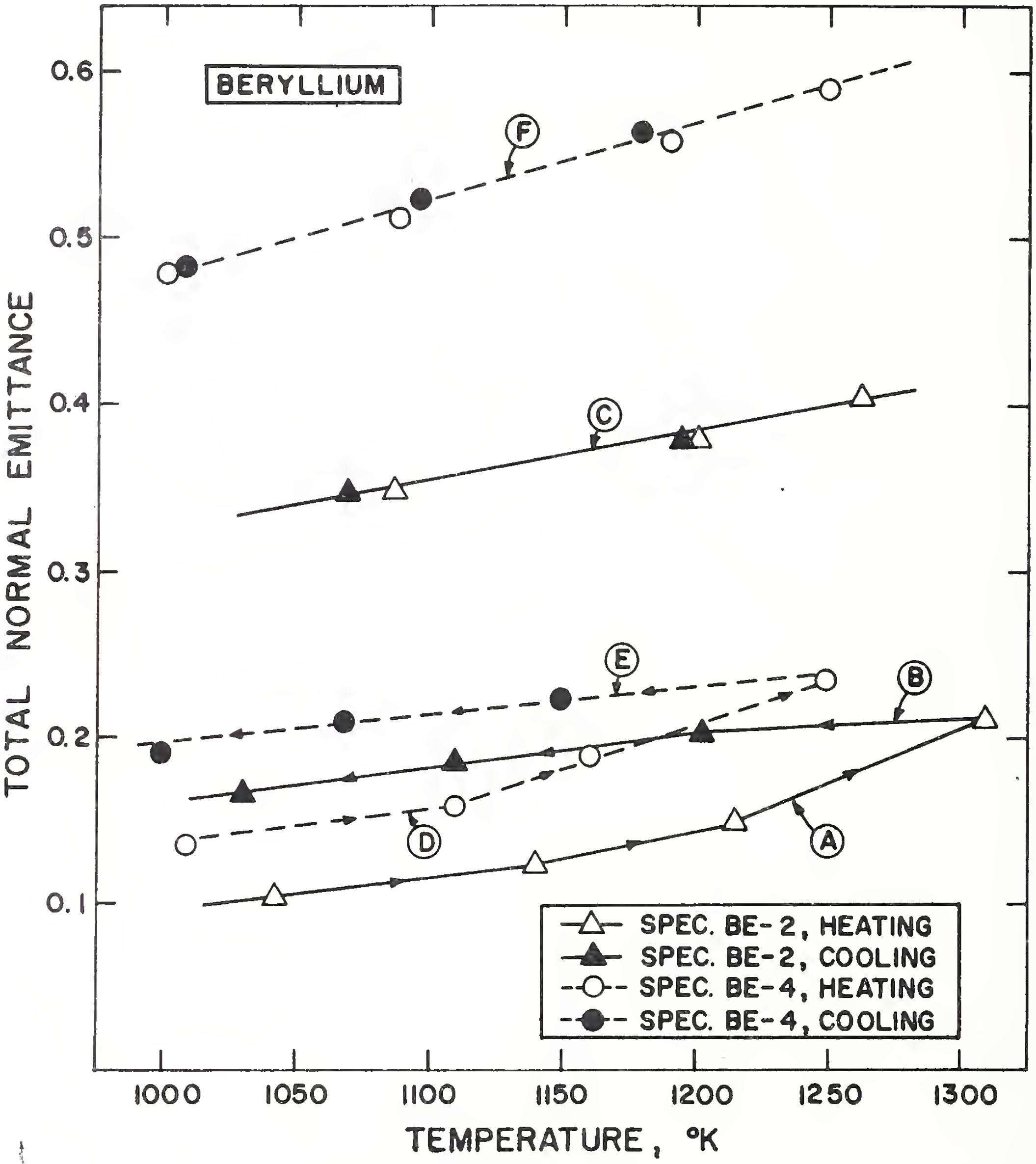

Fig. 13. Total normal emittance of beryllium specimens before treatment (Curves AB and DE) and after heat treatment of 15 sec. in air at 1 atm. at 1500°K (Curves C and F). Specimen B-2 was polished and Be-4 was abraded with 400C paper. All measurements were made in a vacuum of 2 x 15^{-5} of Hg.

Fig. 13 shows the results of measurements on two of the four specimens that were submitted. Specimen Be-2 was polished (RMS 8) while Be-4 was surface ground to RMS 20[3].

The difference in ϵ_{TN} between the two specimens is in keeping with what might be expected from the variation in surface finish. The clean untarnished beryllium appears to have both a total normal emittance and a temperature coefficient comparable to those of tunbsten and several other metals. However, the tests showed that beryllium tarnishes rapidly at temperatures above about 1150°K even in a vacuum as low as 2×10^{-5} torr. This tarnish, which has a dark gray appearance, increases the emittance. Other tests showed that treatment in air at 1275°K for as short a time as 1 sec. could increase the emittance by as much as 20%.

C. Measurements on Nonmetals

In the case of nonmetals with their relatively low thermal conductivity and relatively high thermal emittance, a small, shallow reference hole is needed so as to minimize the temperature difference between the surface and the bottom of the hole (Ref. 14). A shallow hole of this type does not, of course, radiate as a blackbody. Nevertheless, if the geometry of the hole is known, and if the emittance of the hole walls is the same as that of the test surface, then it is possible through use of an expression derived by Gouffé (Ref. 3) to compute the emittance from the measured ratio of the flux from the surface to that from the hole.

The Gouffé expression for the apparent emittance of a cavity may be written as follows:

$$\epsilon_c = \frac{\epsilon_w \left[1 + (1-\epsilon_w)\left(\frac{a}{A} - f\right)\right]}{\epsilon_w \left(1- \frac{a}{A}\right) + \frac{a}{A}} \qquad (1)$$

where: ϵ_c = cavity emittance

ϵ_w = emittance of cavity walls

$f = \dfrac{1}{1 + (L/R)^2}$ where L = cavity depth and R = cavity radius

a/A = ratio of area of cavity opening to total area of cavity, opening included, =

$$\frac{1}{2\left[1 + (L/R)\right]}$$ for a cylindrical cavity

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[3] RMS finish refers to root-mean-square average of the peak-to-valley depth of surface irregularities as expressed in microinches.

Now, if E is the measured ratio of normal radiant flux from surface and hole, I_s is the normal radiant flux per unit area from a plane (non-cavity) surface of a specimen, I_c is the normal radiant flux per unit area from a cavity formed in the specimen material, I_{BB} is the radiant flux from a blackbody radiator under the same conditions at the same temperature, ϵ_s is the emittance of the specimen, and ϵ_c is the emittance of the cavity, then it is possible to write the following:

$$E = \frac{I_s}{I_c} = \frac{I_s/I_{BB}}{I_c/I_{BB}} = \frac{\epsilon_s}{\epsilon_c} \qquad (2)$$

Assuming that the emittance of the cavity wall, ϵ_w, is the same as that of the specimen surface, ϵ_s, Gouffé then combines equation (2) with equation (1) to obtain:

$$\epsilon_s = \frac{E(1 + \frac{a}{A} - f) - \frac{a}{A}}{1 - \frac{a}{A} + E(\frac{a}{A} - f)} \qquad (3)$$

Equation (3) is the expression that is used in the shallow-hole method. Obviously, it can be valid only if equation (1) is valid. Therefore, before an accuracy can be ascribed to measurements made by the shallow-hole method, it was first necessary to test equation (1) experimentally. This was done by determining the spectral reflectance from 0.4 to 0.75μ of an adjustable-depth, paper-lined cylindrical brass cavity, and then converting the spectral reflectances to emittances by subtracting from unity. Because the incident beam of the spectrophotometer was near normal and the viewing was hemispherical, the reflectance measurement was, in effect, the complement of the normal emittance, which is the property of interest in the shallow-hole method.

The results for a cavity lined with a coating that was a good diffuser are shown in Fig. 14. The measured values fall on the curve predicted by the Gouffé expression (equation 3) to within the precision of measurement. The more important conclusions from this study of cavity emittances were:

(1) When the wall material was a good diffuse reflector, the Gouffé expression predicted the emittance of shallow cylindrical holes to within 0.01.

(2) When the wall material was somewhat specular in nature (a poor diffuser) the experimental and theoretical emittances still agreed well for a hole with a depth-to-radius ratio (L/R) of 0.5.

(3) The Gouffé expression (equation 3) can be used for converting flux ratios obtained by the shallow-cavity method to emittance without significant error for all those materials that are good diffuse reflectors. Fortunately, this includes most of the nonmetals.

A paper entitled "A Test of Analytical Expressions for the Thermal Emittance of Shallow Cylindrical Cavities" was prepared. This will be presented at the March 1964 Symposium on Thermal Radiation of Solids.

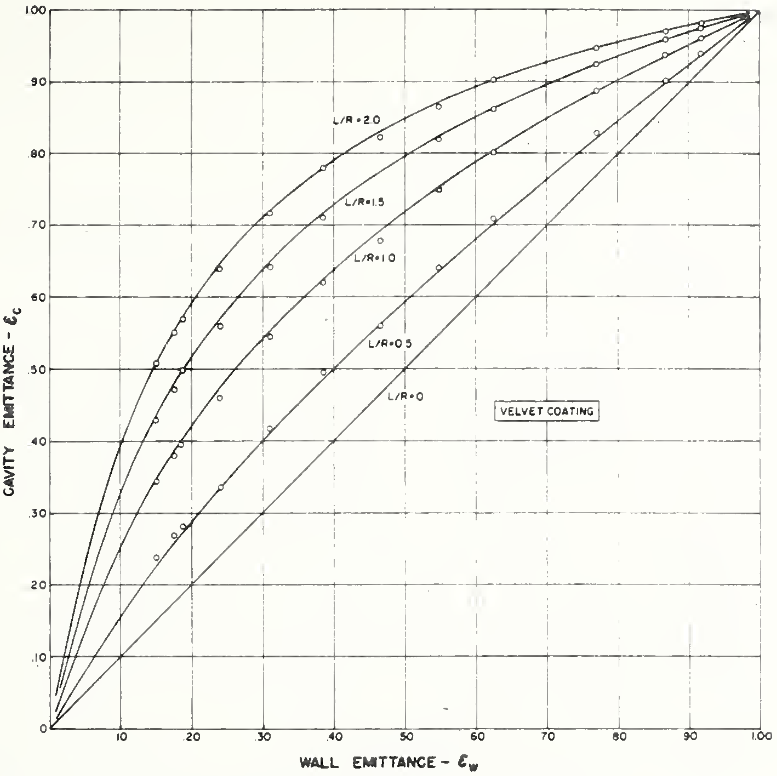

Fig. 14. Room-temperature emittances (circled points) determined by a reflectance technique for cavities lined with a diffusely reflecting coating. Solid lines were computed from Gouffé expression.

D. Future Plans

The following work is planned for calendar year 1964:

(1) An analysis will be made of all potential sources of error in the shallow-hole method and an attempt will be made to arrive at a figure for its overall accuracy.

(2) Emittances will be measured on specimens at temperatures in the range 1200° - 1800°K and compared to total normal emittance values for the same materials obtained with the rotating-specimen equipment.

(3) Methods of heating oxide specimens to high temperatures without the aid of a metal crucible will be explored. This is needed to avoid contamination of the specimen by the crucible material, which has occurred in a number of measurements at temperatures above 2000°K.

(4) Emittance-temperature curves will be determined for several representative metallic and nonmetallic specimens at temperatures up to 2500°K.

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