

45110

NATIONAL BUREAU OF STANDARDS REPORT

7857

EQUIPMENT FOR THERMAL EMITTANCE MEASUREMENTS
ABOVE 1400°K

ANNUAL SUMMARY REPORT

January, 1963

Contract No. H-22727
3-84-2-41-1306-01-000-8400-2561-4
80X0108 (62) R&D, MSFC, OBJECT 25

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



U. S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS

THE NATIONAL BUREAU OF STANDARDS

Functions and Activities

The functions of the National Bureau of Standards are set forth in the Act of Congress, March 3, 1901, as amended by Congress in Public Law 619, 1950. These include the development and maintenance of the national standards of measurement and the provision of means and methods for making measurements consistent with these standards; the determination of physical constants and properties of materials; the development of methods and instruments for testing materials, devices, and structures; advisory services to government agencies on scientific and technical problems; invention and development of devices to serve special needs of the Government; and the development of standard practices, codes, and specifications. The work includes basic and applied research, development, engineering, instrumentation, testing, evaluation, calibration services, and various consultation and information services. Research projects are also performed for other government agencies when the work relates to and supplements the basic program of the Bureau or when the Bureau's unique competence is required. The scope of activities is suggested by the listing of divisions and sections on the inside of the back cover.

Publications

The results of the Bureau's research are published either in the Bureau's own series of publications or in the journals of professional and scientific societies. The Bureau publishes three periodicals available from the Government Printing Office: The Journal of Research, published in four separate sections, presents complete scientific and technical papers; the Technical News Bulletin presents summary and preliminary reports on work in progress; and the Central Radio Propagation Laboratory Ionospheric Predictions provides data for determining the best frequencies to use for radio communications throughout the world. There are also five series of nonperiodical publications: Monographs, Applied Mathematics Series, Handbooks, Miscellaneous Publications, and Technical Notes.

A complete listing of the Bureau's publications can be found in National Bureau of Standards Circular 460, Publications of the National Bureau of Standards, 1901 to June 1947 (\$1.25), and the Supplement to National Bureau of Standards Circular 460, July 1947 to June 1957 (\$1.50), and Miscellaneous Publication 240, July 1957 to June 1960 (includes Titles of Papers Published in Outside Journals 1950 to 1959) (\$2.25); available from the Superintendent of Documents, Government Printing Office, Washington 25, D.C.

NATIONAL BUREAU OF STANDARDS REPORT

NBS PROJECT

1009-11-10493

NBS REPORT

7857

EQUIPMENT FOR THERMAL EMITTANCE MEASUREMENTS
ABOVE 1400 K

ANNUAL SUMMARY REPORT

January, 1963

Contract No. H-22727

3-84-2-41-1306-01-000-8400-2561-4

80X0108 (62) R&D, MSFC, OBJECT 25

Prepared for

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

IMPORTANT NOTICE

NATIONAL BUREAU OF STANDARDS
for use within the Government. Before
and review. For this reason, the report
whole or in part, is not authorized for
Bureau of Standards, Washington
the Report has been specifically prepared

Approved for public release by the
Director of the National Institute of
Standards and Technology (NIST)
on October 9, 2015.

Accounting documents intended
subjected to additional evaluation
listing of this Report, either in
Office of the Director, National
the Government agency for which
copies for its own use.



U. S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS

FOREWORD

A research program aimed at developing equipment and procedures suitable for accurate measurements of the total and spectral emittance of both metals and nonmetals in the temperature range 1400 - 2500°K was initiated on Jan. 1, 1961 at the National Bureau of Standards under the sponsorship of the George C. Marshall Space Flight Center at 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 1962. 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 A. G. Eubanks, B. A. Peavy, H. E. Clark, A. W. Crigler, J. C. Richmond, and W. N. Harrison.

ABSTRACT

A spectrometer was procured for use with the rotating cylinder equipment. The source optics were removed and replaced with transfer optics that permitted focusing of beams from a blackbody furnace and from the rotating specimen respectively on the two slits of the monochromator. Preliminary results with platinum and sintered alumina at 1 to 15 μ indicated that the equipment was capable of satisfactory operation; however more work is indicated to eliminate several potential sources of error.

A large number of measurements of total normal emittance were made with the induction heating equipment. Tests with specimens of steatite (magnesium aluminum silicate) indicated that the measured emittances were too low when the depth-to-radius ratios of the reference holes were greater than 2.0. On the other hand, total normal emittance measurements on platinum and tungsten specimens were as much as 30% higher than prevailing literature values when the blackbody reference holes had a depth-to-radius of 10 (3.5 mm depth by 0.35 mm radius). Reexamination of the optics used with the induction heating equipment showed that aberrations were present to an extent that would cause high emittance readings if the reference hole was less than 0.35 mm in radius. This source of error can be eliminated either through use of larger holes or by substitution of an ellipsoidal mirror for the present 5" spherical mirror.

Typical results obtained with both the rotating cylinder and induction heating equipment are included.

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 measurements (1 to 15μ) in the range of about 1000° to 1800°K (rotating cylinder method) and the second for total normal emittances at temperatures that may go 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, 1962 to Jan. 1, 1963 is described in the present report.

II. ROTATING SPECIMEN METHOD FOR MEASUREMENTS TO 1800°K

A. A Brief Description of Method

The rotating specimen method has been described in detail in the first summary report (ref. 1) which also includes a description of the furnace.

Briefly, the procedure is as follows: 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. The method by which these gradients were analyzed is given in the next section.

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.

B. Analysis of Periodic Heat Flow in a Rotating Specimen

A theoretical analysis of periodic heat flow in a specimen rotating before a viewing port was performed, and a report of this analysis has been prepared for publication (ref. 2). This analysis is more rigorous than that given in the first annual summary report. The equations that were developed permitted computation of cyclic temperatures of a specimen as a function of system geometry, rotational speed, thermal properties of the specimen material, and a derived average heat flux for the portion of the cycle during which the specimen is being heated by the furnace.

A comparison was made between temperatures of a porous alumina specimen computed theoretically and those obtained experimentally at low speeds of rotation. The agreement in the two temperatures was within 6°K . This agreement seemed well within both the possible experimental error and the uncertainty as to the thermal properties of the specimen.

The theoretical analysis showed that at low rotational speeds the surface temperature of a specimen, at the center of the viewing port opening, is substantially lower than the temperature of the thermocouple in the specimen cavity. However, this difference decreases with increasing speed reaching in the case of the porous alumina specimen a computed value of 4°K at about 450 RPM. In the actual measurements, if the temperature near the specimen surface at the viewing port is lower than cavity temperature, the measured emittance will be low. However, as the speed increases the measured emittance will rise to some maximum value, after which a further increase in speed will have no measurable effect. This predicted type of behavior is used to determine experimentally the optimum speed of rotation for each specimen at each test temperature (See Sect. II-E).

C. Blackbody Furnaces and Temperature Control

The blackbody furnaces used with the rotating-specimen equipment were constructed as indicated in fig. 1. The alumina core was wound with Pt-20% Rh resistance wire. Power taps (not shown in fig. 1) were positioned $1\frac{1}{4}$ " from each end of the winding. These taps permitted adjustment of power inputs to equalize temperatures from front to back. A thermocouple placed near the center winding of one of the furnaces was connected to a controller which maintained the temperature in the furnace cavity constant to within $\pm 0.5^{\circ}\text{K}$. The second blackbody furnace and the rotating specimen furnace were adjusted to this same temperature to within about $\pm 1^{\circ}\text{K}$ by manual control of power input.^{1/}

^{1/} Final adjustment of the blackbody furnaces was based on the appearance of the 100% spectral curve when blackbody no. 1 was inserted in the reference beam and blackbody no. 2 in the specimen beam of the spectrometer. If blackbody no. 2 was at a slightly higher temperature than no. 1, the curve from 1 to 2μ rose above 100% while if the temperature was slightly lower, the curve fell below the 100% line. When the two temperatures coincided the curve was substantially flat over the entire range of 1 to 15μ . This method of determining temperature equivalence has high sensitivity and it is more reliable for precise work than depending entirely on thermocouple indications.

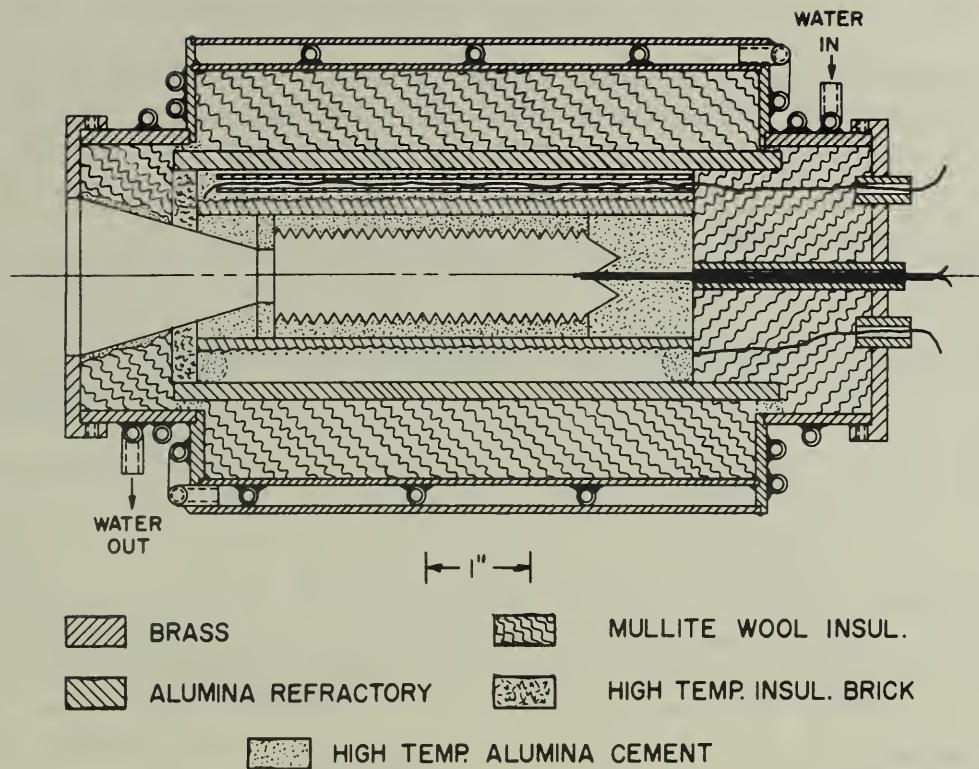


Figure 1. Schematic of blackbody furnace

D. Monochromator and Transfer Optics

A Beckman IR-5A spectrometer with NaCl prism is being used for the spectral measurements. A flip mirror was inserted into this instrument so that the prism could be bypassed when total normal emittance measurements were desired (see fig. 2).

The source optics of the IR-5 instrument were removed and replaced by the transfer optics as shown schematically in figure 2. The off-axis angle of the spherical mirrors is approximately 5° .

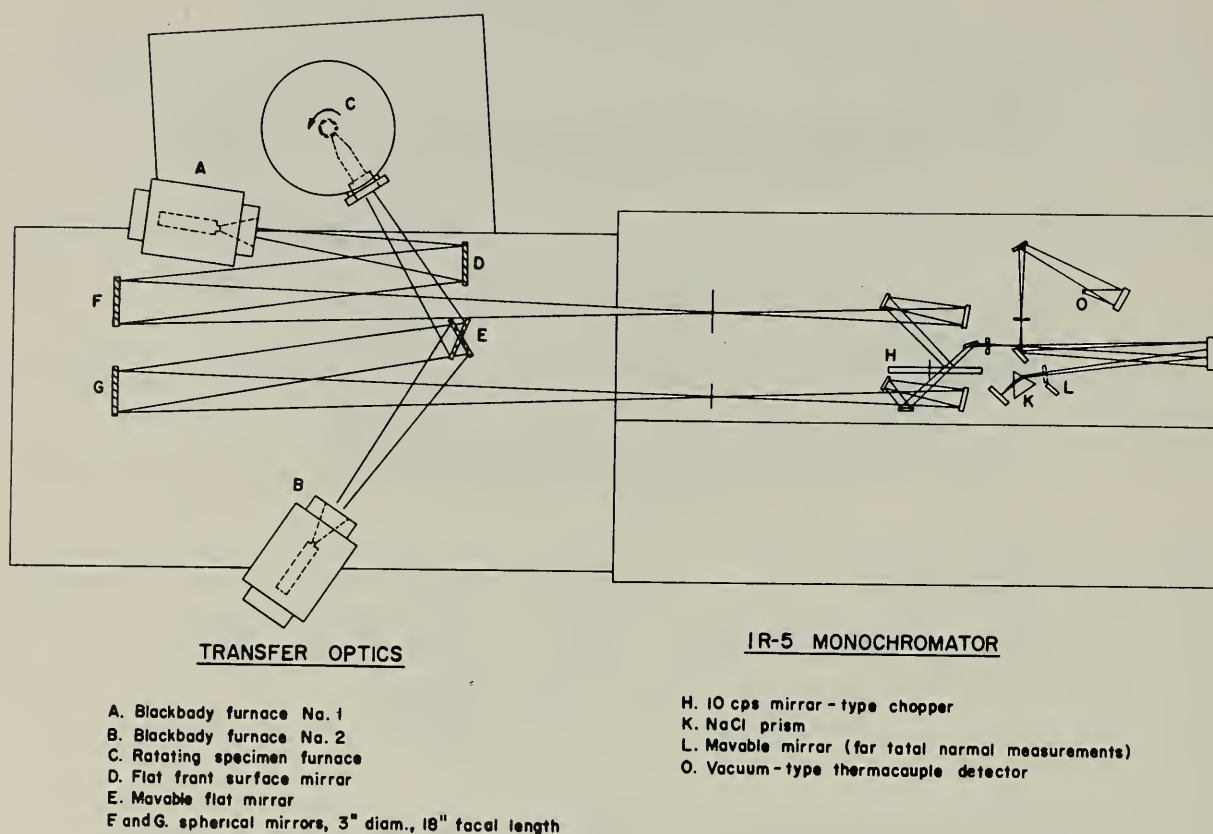


Figure 2. Sketch of optical paths in rotating-specimen equipment

The monochromator is operated in double-beam mode. Energy from each beam is chopped at 10 cps. Reference and specimen beams are alternately directed through the prism to a Littrow mirror and the dispersed radiation is then brought to a focus on a vacuum-type thermocouple detector. The thermocouple converts the radiant energy to an electrical signal proportional to the difference in energy in the two beams. This signal passes through a preamplifier and a signal amplifier to a pen-comb servo amplifier. The amplified signal then drives a servomotor to return a linear reference comb to null position, equalizing the intensity of the specimen and reference beams. A mechanical connection to the comb drives the pen which in turn records the ratio of the energies in the two beams. A separate motor drives the wavelength and slit cams as well as the lateral movement of the pen carriage.

E. Experimental Procedure for Determining Optimum Rotational Speed

The following procedure, which is based largely on the theoretical analysis described in Section II-B, has been developed to determine experimentally the optimum speed of rotation for any given specimen at any given test temperature:

- (1) The specimen is heated to the test temperature while rotating at approximately 20 RPM.
- (2) The monochromator is adjusted to a wavelength setting of 1.4μ and the measured emittance at this wavelength is recorded^{2/}.
- (3) The specimen speed is next increased in increments until the emittance at 1.4μ shows no measurable increase with increasing RPM.
- (4) At this rotational speed the temperature in the specimen cavity and the temperature of the specimen surface are very nearly equivalent and hence this speed can be used as the optimum speed of rotation for obtaining the spectral curve. The speed may be increased somewhat beyond this optimum, but in no case are the measurements made at slower speed.

F. Measurement Procedure

Once the rotational speed has been fixed by the procedure outlined in the preceding section, the specimen beam is blocked and a "zero line" obtained. Next mirror E in the transfer optics (fig. 2) is moved into position to focus radiation from blackbody No. 2 on the specimen slit of the monochromator. The wavelength drive is then started and a "100% line" is obtained, after which Mirror E is shifted to the rotating specimen position and the specimen curve is determined. All three curves are recorded on the same graph. The true emittance of the specimen is obtained for each wavelength by measuring the height of the recorded specimen curve above the "zero curve", and dividing this distance by the height of the 100% line above the zero line. Due mostly to a lack of perfect optical equivalence in the two beams neither the "100% line" nor the "zero line" is completely flat; hence errors would result if a spectral correction of this type were not made.

Total normal emittances are obtained in the same way as spectral emittances except that Mirror L is moved into position to bypass the prism in the monochromator and the wavelength drive is not used. The slit openings, while not critical, must be sufficiently narrow to prevent overloading the detector.

Fig. 3 is a photograph of the equipment.

^{2/} Difference in temperature between specimen and blackbody can be shown to have the greatest effect on emittance at the shorter wavelengths. The selected setting of 1.4μ is well below the energy peak of blackbody radiation for all four of the preselected test temperatures of 1200, 1400, 1600, and 1800°K.

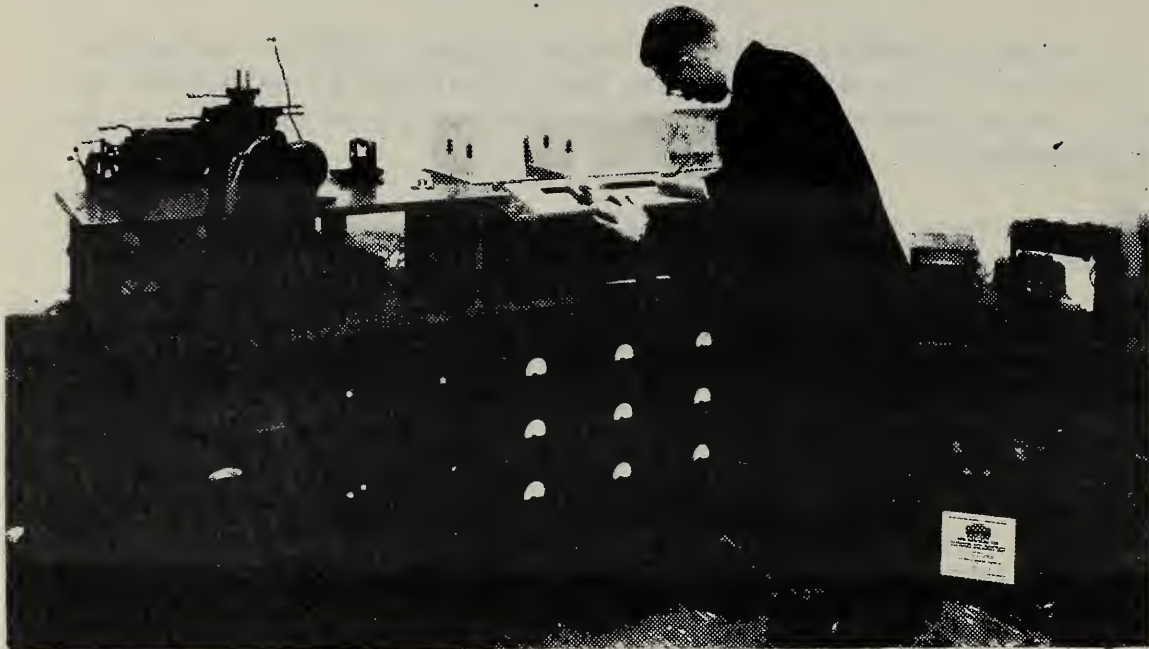


Figure 3. Photograph of rotating-specimen equipment

G. Preliminary Measurements

Figure 4 shows a curve obtained for a polished platinum specimen at 1400°K that had been annealed for one hour in air at 1525°K prior to test;

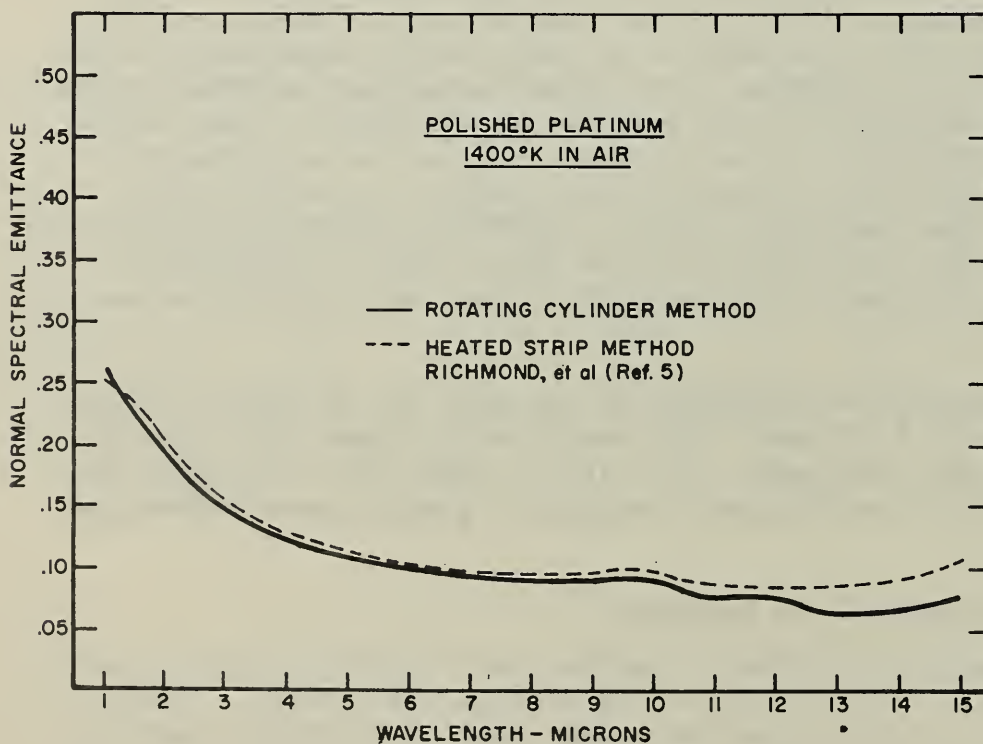


Fig.4. Preliminary measurements of normal spectral emittance of a platinum specimen.

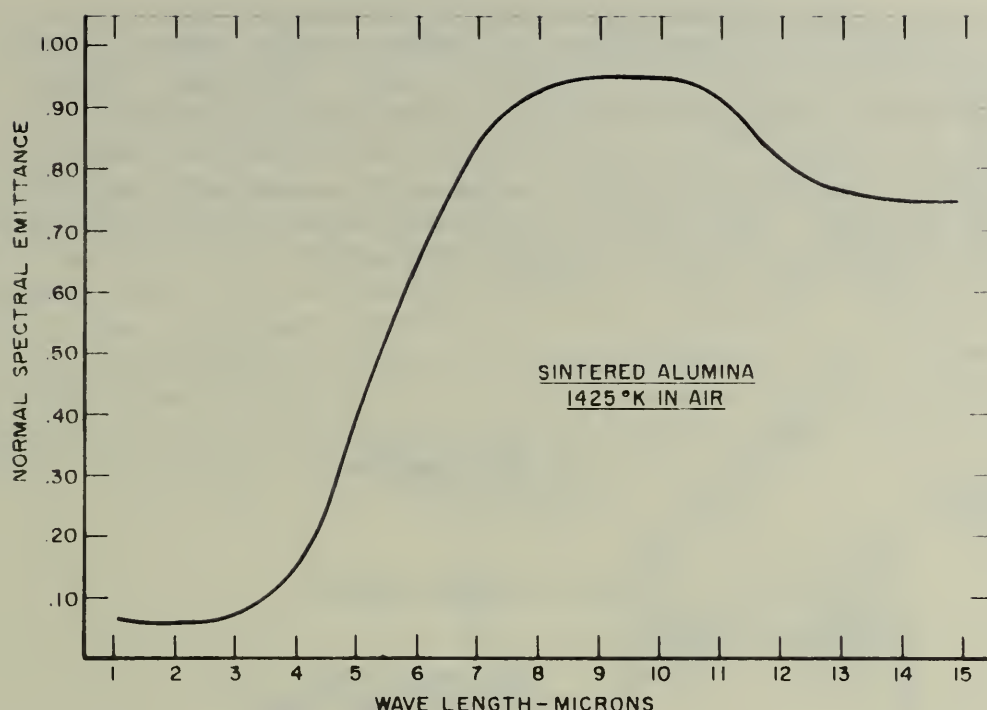


Figure 5. Preliminary measurements of normal spectral emittance of a sintered alumina specimen.

fig. 5 is a spectral curve for a sintered alumina specimen at 1425°K. Both of these curves must be considered preliminary because certain possible sources of error had not been eliminated at the time of measurement. The values of emittance in the wavelengths range 1 to 5 μ for the sintered alumina are suspect because of their poor agreement with recently reported data for a similar material (ref. 3 and 4). On the other hand, the agreement of the curve obtained for platinum with that reported by Richmond, et al (ref. 5) is fairly good and these data, of themselves, indicate that the equipment is capable of satisfactory operation. The total normal emittances measured with the equipment at 1400°K were 0.13 for platinum and 0.21 for alumina.

H. Present Status of Measurements and Future Plans

Current work with the equipment is concerned with adjustment of the blackbody furnaces and the specimen furnace to minimize temperature gradients. When this task has been completed, the transfer optics will be realigned, and response linearity will be investigated with sector disc attenuators at each of the four selected test temperatures (1200, 1400, 1600, and 1800°K). If the response is found to be sufficiently linear, an error analysis will be performed to determine the overall accuracy of measurement. In this analysis special attention will be given to the effect of temperature uncertainties and also to the effect of possible translucency of the

specimen walls over certain wavelength regions. Finally, the spectral and total normal emittance will be determined at 1200, 1400, 1600 and 1800°K on three specimens each of the following materials: platinum, sintered alumina, magnesia, zirconia, thoria, silica, calcium zirconate, mullite, magnesium aluminate spinel, zirconium silicate, and steatite (magnesium aluminum silicate). Specimens of all of these materials have been procured for the testing program. The nominal chemical analysis of each has been obtained. All 11 materials are of commercial purity.

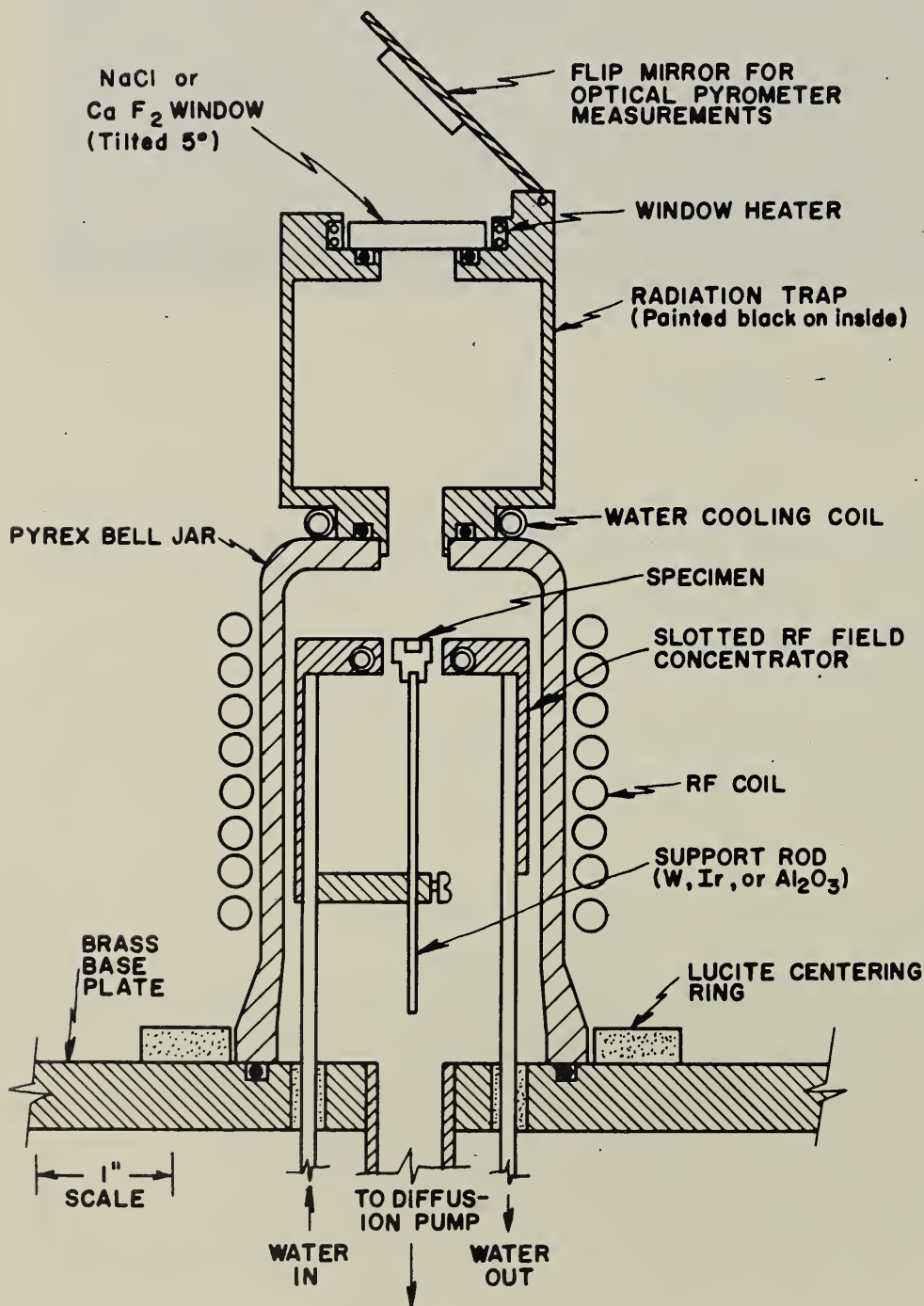


Fig.6 Schematic of induction furnace.

III. INDUCTION HEATING EQUIPMENT FOR TOTAL NORMAL EMITTANCE MEASUREMENTS TO 2500°K

A. Brief Description of Equipment and Theory of Method

The design of the induction equipment and the reasons for its selection were given in the first annual summary report (ref. 1), and also in a paper to be published as a part of the Proceedings of the Symposium on the Measurement of Thermal Radiation Properties of Solids (ref. 6). Fig. 6 is a schematic of the furnace equipment while fig. 7 is a schematic of the optics. Basically, the measurement consists of determining the ratio of the density of radiant flux from the surface of a small specimen to the density of flux from a cylindrical hole drilled into the specimen surface. Fig. 8 shows the two types of specimen in current use.

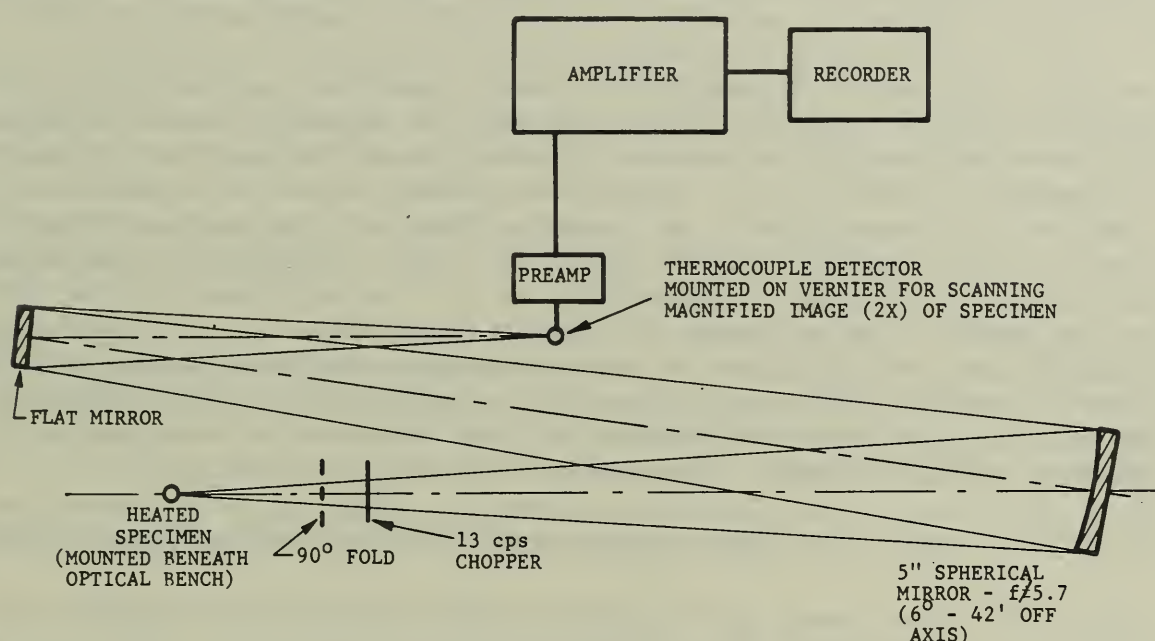


Figure 7. Sketch showing both optical paths and electronic components.

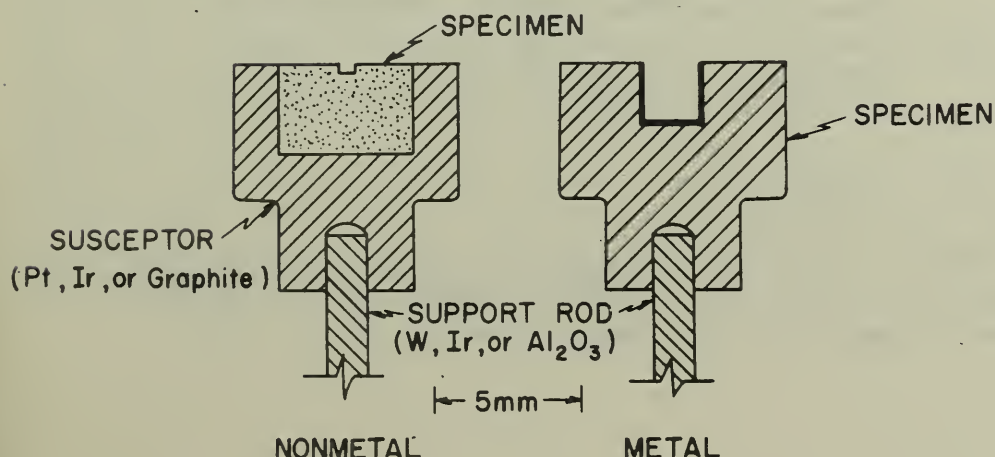


Fig.8 Two types of specimen. Metal specimen shown has type of reference hole on which best results were obtained. Heavy line on hole walls represents high emittance coating.

The principle modification introduced into the equipment during the contract year was the introduction of a radiation trap at the top of the bell jar to prevent back-reflected energy from the top of the bell jar and from the NaCl or CaF₂ window from entering the specimen beam. This trap is water cooled so as to eliminate overheating of the window "O" ring which was one of the more serious problems encountered with the earlier design.

The equipment is used for both metals and nonmetals. For metals the reference cavity is designed so as to radiate very nearly as a blackbody. This is accomplished through the use of cavities in which the wall surfaces have been modified by roughening and/or application of a high emittance coating. Fortunately, temperature differences between surface and reference cavity are not serious with most metals because of their high heat transfer coefficients and their low emittances which means that reasonably good temperature equality can be achieved even when sizeable reference cavities are used. However, this is not the case with nonmetals and for this class of materials a small, shallow reference cavity is imperative (fig. 8) so as to reduce as much as possible the differences in temperature between the surface and the bottom of the cavity. An expression derived by Gouffe' (ref. 7) permits the emittance of a specimen with a shallow reference hole to be computed from (a) the ratio of the flux densities from surface and reference hole and (b) the hole dimensions. This expression was derived by assuming that (1) the emittance of the hole walls is the same as the surface, (2) the specimen material is a perfectly diffuse reflector, and (3) there is no temperature difference between hole and surface. None of these conditions can be achieved in a real specimen but they can be very closely approached by most nonmetallic specimens if the reference hole is of small diameter and of shallow depth.

The Gouffe' expression that is employed with the shallow-hole method is:

$$\epsilon = \frac{E(1 + R - R_0) - R}{1 + R + E(R - R_0)} \quad (1)$$

where:

$$\begin{aligned} \epsilon &= \text{emittance of specimen} \\ E &= \text{Ratio of radiant flux density from the surface to the flux density from the hole} \\ R &= \frac{1}{2(1+h/r)}, \text{ (for a cylindrical cavity of depth, } h, \text{ and radius, } r.) \\ R_0 &= \frac{1}{1+(h/r)^2} \end{aligned}$$

Note: The R_0 given by Gouffe' is an approximation. It can be shown that a more rigorous expression for R_0 is $2(1-h/\sqrt{h^2+r^2})$. However, no significant error is introduced through use of the approximation at h/r values above about 2.5.

B. Measurements on Nonmetals

Data already presented in ref. 6 will not be repeated in this report but instead attention will be focused on results obtained since preparation of the paper for the Symposium.

1. Steatite. Steatite was selected for an investigation of the effect of hole depth on the apparent emittance. Earlier tests with alumina had indicated that specimens with reference holes of 0.25 mm radius and having h/r ratios of 2.5 and greater gave lower emittance values than did specimens with holes having h/r ratios of less than 2.0. This type of result suggested that the temperature of the bottom of the deeper holes was higher than that in the shallow holes, possibly because of less heat loss by radiation when the hole is deep. Steatite, because of its easy machineability, appeared to be an excellent material with which to investigate this possible effect.

Six specimens were machined from a rod of unfired steatite. The reference hole in each specimen was precision drilled with a flat bottom so that after firing it would have a radius of about 0.25 mm. The hole in each specimen was drilled to a different depth so as to give a range of h/r values from about 1 to 4. After firing at 1475°K for 1 hr., measurements were made at 1360°K .

The results are plotted in fig. 9. The upper curve, A, shows the apparent total normal emittance when the hole is considered as a blackbody (E in Equation 1); the middle curve, B, results when the relative flux density ratios from surface and hole are reduced to emittance by Equation 1, while the lower curve, C, shows the corrected emittance values when the exact solution for R_0 is used. It should be noted that curves B and C are practically superimposed for h/r values greater than 2.

Two tentative conclusions can be implied from these data. The first concerns the use of the exact solution for R_0 . If the Gouffe' expression is sound, and if no significant temperature difference exists between hole and surface, then the emittance as computed from the flux ratio, E , should be independent of the h/r ratio. Fig. 9 shows that this independence is achieved at h/r values between 1.0 and 2.0 only when the exact solution for R_0 is used in the Gouffe' equation (Curve C). At h/r ratios greater than 2.0, the computed emittances decrease with increasing hole depth. This is the type of behavior that would occur if the bottom of the hole was at a higher temperature than the specimen surface which is precisely the condition that would be expected for relatively deep reference holes. Hence a second implication from the data is that the h/r ratio of the reference hole is that the h/r ratio of the reference hole in an oxide specimen such as steatite should be less than 2.0 if significant errors from temperature differences are to be avoided.

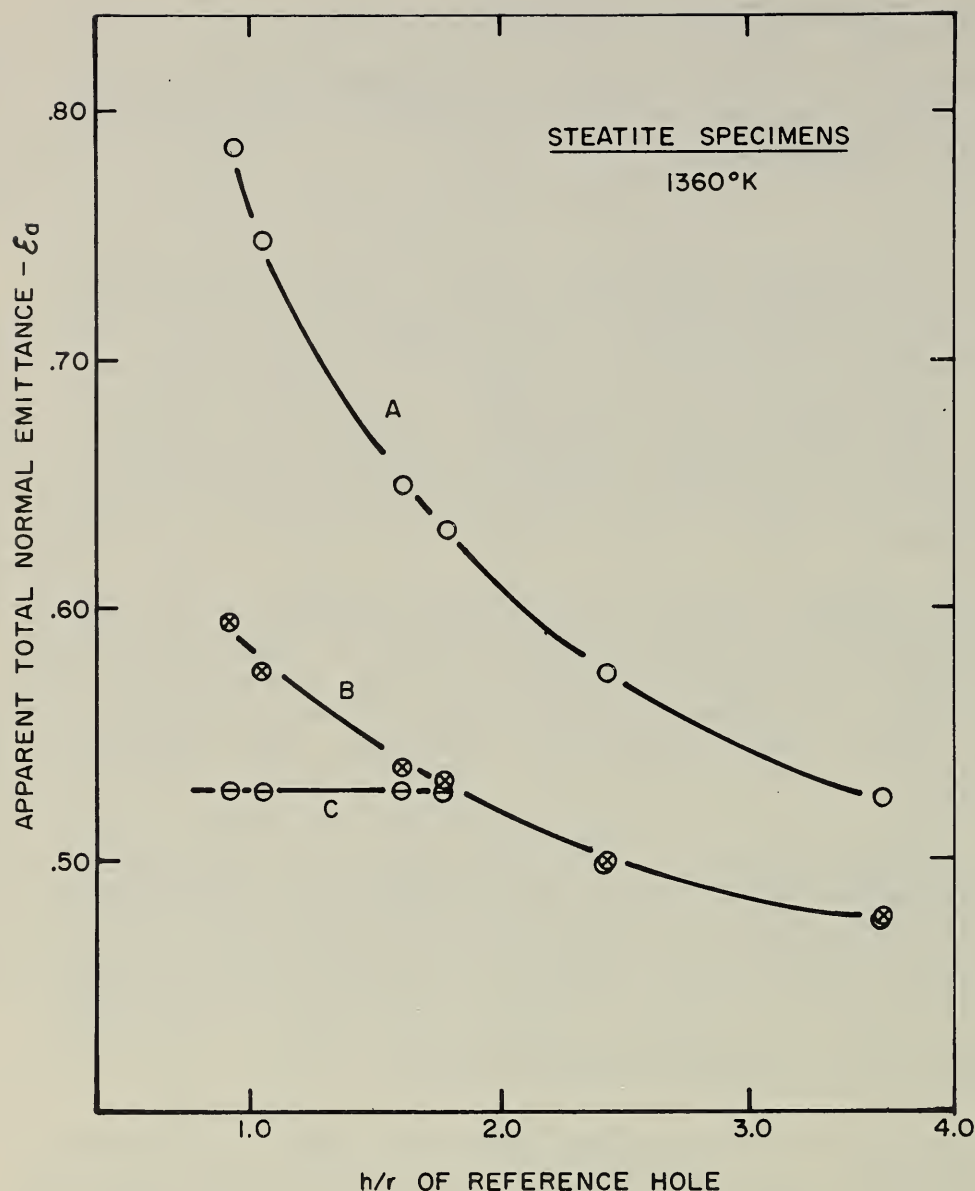


Fig.9 Effect of depth-to-radius (h/r) ratio of the reference hole on the total normal emittance of steatite specimens: (A) ϵ_a considering reference hole as a blackbody; (B) ϵ_a computed from Equation 1; (C) ϵ_a computed from Equation 1 with exact solution for R_0 .

2. Other nonmetals: A considerable number of measurements were made on such nonmetals as alumina, zirconia, graphite, and silicon carbide. Some of these data are given in ref. 6 and some have been presented in the monthly letter reports. In most cases, the measurements were made on specimens with reference holes, 0.25 mm radius, and depths of 0.25 to 0.50 mm (h/r ratios of 1.0 to 2.0). Fig. 10 shows a recently obtained curve for a commercial grade of sintered alumina while fig. 11 shows results for a dense recrystallized grade of silicon carbide (code 45 in ref. 8).

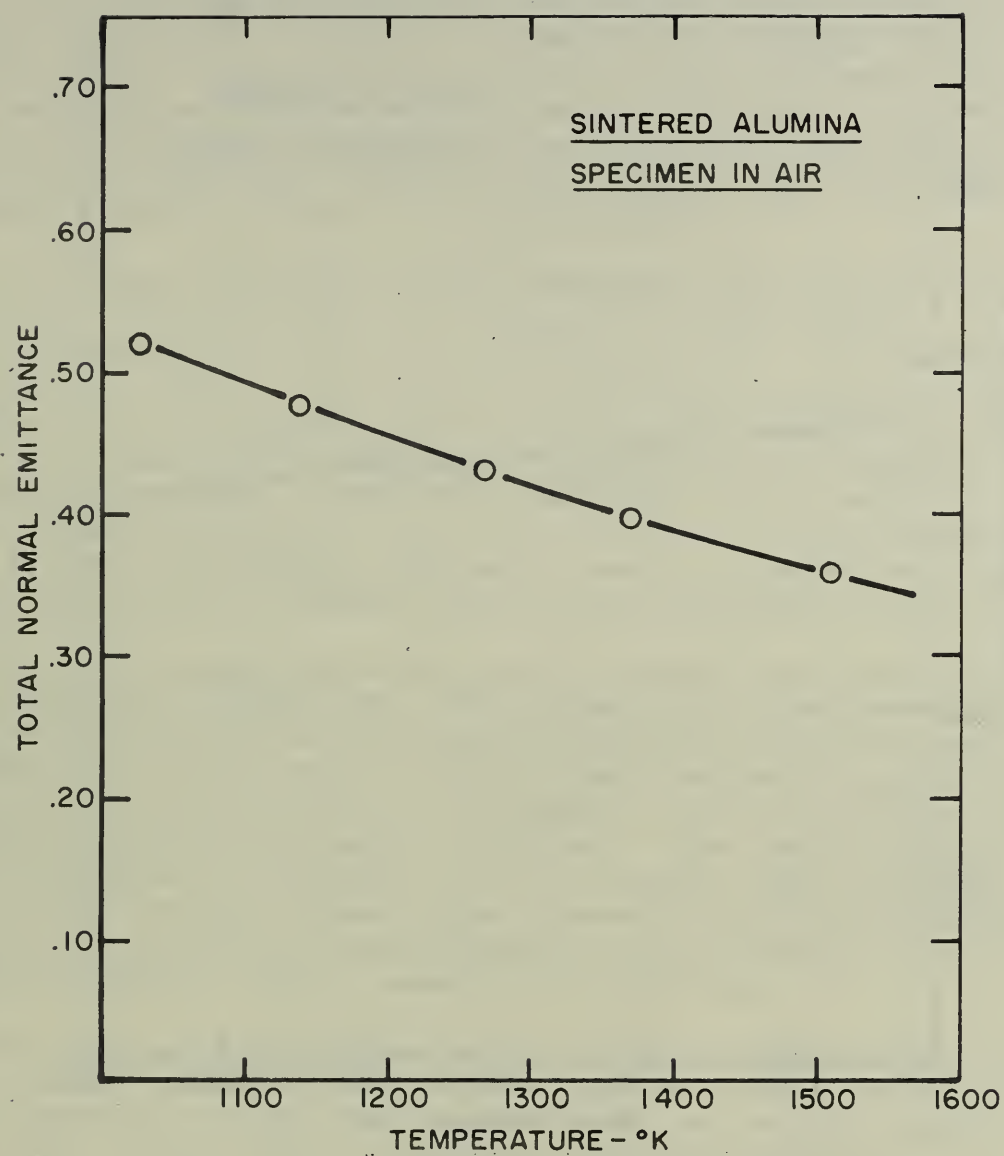


Figure 10. Change in normal emittance with temperature for a specimen of sintered alumina.

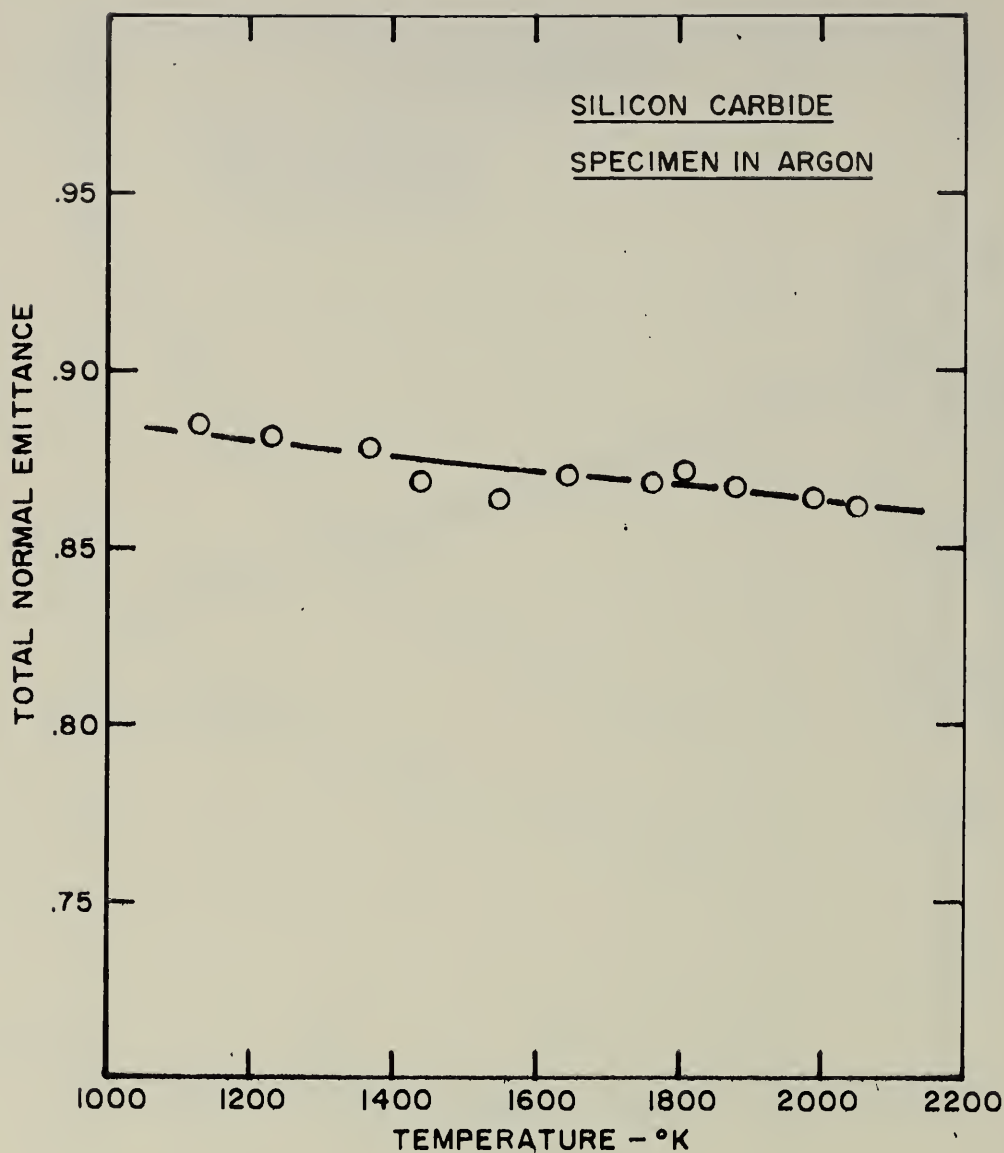


Figure 11. Change in normal emittance with temperature for a specimen of recrystallized silicon carbide.

C. Measurement on Metals

The Gouffe' equation does not apply to metals because materials of this type, when the surfaces are smooth, are specular rather than diffuse reflectors. Metals, however, have relatively high thermal conductivity and low emittance; hence fairly deep reference holes can be used without encountering a serious temperature difference between the bottom of the hole and the surface. If such a hole can be made to approximate a black-body closely, then the emittance can be determined directly from the ratio of the relative flux density from the surface to that from the hole.

The first attempts at such measurements were made with platinum and tungsten specimens into which flat-bottomed holes 0.25 mm radius by 5 mm deep ($h/r = 10$) had been drilled. The walls of these holes were purposely made rough; also, a special tool was used to roughen the bottoms. The purpose of the roughening was to produce a diffusely reflecting surface that would raise the effective emittance of the hole to an emittance of 0.99 or better (ref. 9 and 10). Measurements made with these specimens consistently gave total normal emittance values that were higher than accepted literature values by as much as 30%.

An attempt was made to devise conditions that would bring the emittance values for polished metals, as determined by this method, into line with the accepted measurements reported in the literature. Different polishing methods were tried. Also, methods of specimen heating and support were changed so as to reduce as much as possible any temperature gradients that might be present in the specimens. In addition hole geometry was modified by making the bottom of the hole cone shaped rather than flat. None of these modifications were successful in reducing the measured emittance. In fact the only modification that gave results in keeping with best literature values was to use a reference hole of the type shown in fig. 8.

Up to the present time this cavity has been used only in a single platinum specimen. The hole was 1.90 mm diam. by 2.05 mm deep ($h/r = 2.16$). The hole was drilled and the test surface mechanically polished after which the side walls and bottom of the hole were roughened by grit blasting with a small dental unit. Finally, successive layers of a silicon carbide coating were applied to the walls of the hole. The coating consisted of 5% by weight 600 mesh SiC suspended in a 1% by weight sugar solution. A small drop of this coating suspension was placed in the hole and worked around the sides with a wire of small diameter. After drying, the specimen was tested at 1100°K. Table 1 summarizes the results.

The specimen surface remained virtually the same in all five tests and only the hole treatment was changed. With no coating in the hole, the apparent emittance of the platinum was 0.183; yet with an applied coating layer only 0.02 mm thick the measured emittance dropped to 0.117. The coating thickness does not appear to be critical for layers thinner than about 0.2 mm. Coatings prepared in this way contain numerous pores that tend to cause low heat conduction. At thicknesses of the order of 0.2 mm and greater the temperature drop through the coating is apparently sufficiently high to cause the surface of coating to operate at a significantly lower temperature than the platinum, thus causing the measured emittance values to be high.

Fig. 12 shows the results obtained when the emittance of this same specimen (coating thickness in hole, 0.02 mm) was measured up to 1500°K. Agreement is good with recent values reported for polished platinum by Abbott, Alvares and Parker and also with the emittance computed from electrical resistivity measurements (ref. 11).

Table 1

Effect of thickness of a silicon carbide coating in the reference hole on the measured total normal emittance of a platinum specimen

Coating Thickness ^{a/} in mm	Apparent ϵ_{TN} of Platinum Surface
0	.183
0.02	.117
0.06	.116
0.21	.120
0.30	.134

a/ Measured with a microscope at center of hole bottom.

The emittance of silicon carbide is about 0.90 (fig. 11). Assuming the applied coating to have this value, the emittance of the cavity as computed by the Gouffe' expression is 0.98. The results plotted in fig. 12 and listed in Table 1 suggest that the coated-wall cavity closely approximates a black-body when the coating is applied at a thickness as low as 0.02 mm.

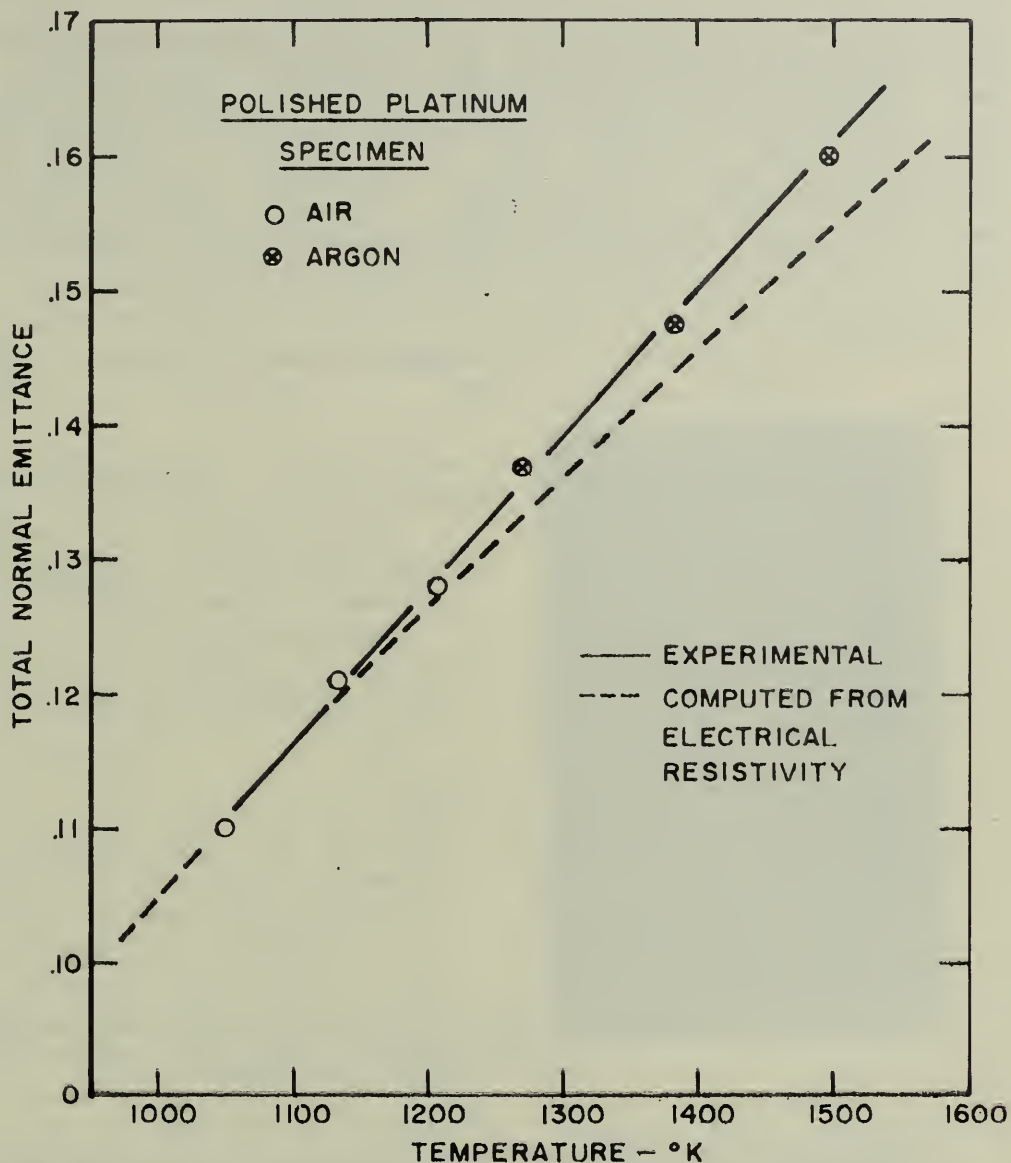


Fig.12. Change in total normal emittance with temperature for a specimen of polished platinum.

D. Investigation of Possible Sources of Error

A large difference in the total normal emittance of sintered alumina from the same source was observed when measurements were made by the rotating cylinder and the shallow hole methods (0.21 and 0.39 at 1400°K). This type of result required a careful review of both methods for possible sources of error. The approaches being used with the rotating cylinder equipment are discussed in Section II-G; the present section is concerned with possible sources of error that might still be present in the induction heating method.

(1) Lack of flat response from detector: The thermocouple detector used in the induction heating equipment is of a type commonly used in infrared monochromators. The sensing area of this detector is coated with a special black paint that supposedly provides non-selective (flat) spectral response. A rapid method of determining whether or not the detection system does in fact react in this manner, is to determine whether the detector response follows the Stefan-Boltzmann relation, i.e., increases linearly with the fourth power of the temperature of a blackbody source. As the temperature increases the peak energy in the blackbody radiation shifts to lower wavelengths in accordance with Wien's law. It follows that if the detector gives a flat response, a straight line will result when the detector response is plotted against the fourth power of the temperature. Likewise if the detector is selective some deviation from the straight-line relation will occur. This test method involves the premise that the detector responds linearly to radiant flux density at a given temperature, which fact was established earlier (ref. 1).

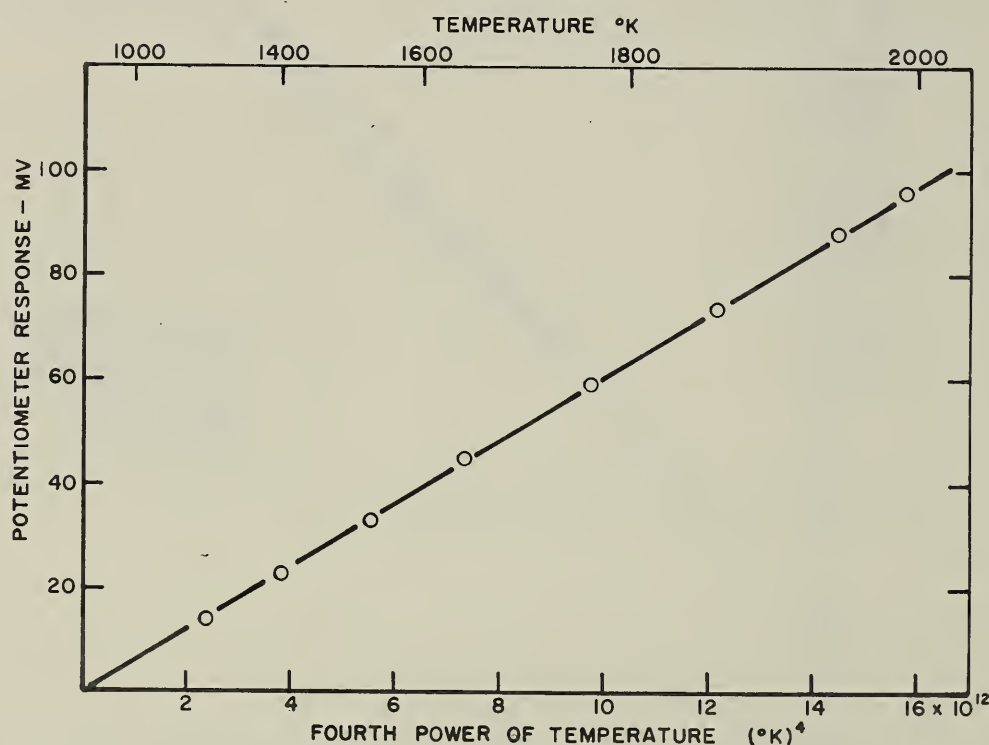


Figure 13. Potentiometer response with temperature for a deep hole drilled in a specimen of graphite. Temperatures were measured with a calibrated optical pyrometer.

The data plotted in fig. 13 were obtained with the detector focused on a hole drilled in a graphite specimen. The emittance of this hole when computed by the Gouffe equation was 0.99. The measurements were made in vacuum with a sodium chloride window at the top of the radiation trap. The plotted points all fall on a straight line which intercepts the temperature axis at very close to room temperature ($T^4 = 0.007 \times 10^{12}$). These results show that the detector has a substantially flat response to the wavelengths of interest, hence there is no measurable error from this source.

(2) Error from detector not being at exact focal point: Alignment of the optics was accomplished initially by determining visually the point of sharpest focus of the specimen image. To determine this point more accurately, the detector was replaced with a camera with a focal plane shutter. The lens was removed from the camera and a series of photographs taken of a platinum specimen at distance increments of 1 mm along a 2 cm length near the focal point.

An enlargement of these pictures showed that the image of the reference hole tended to be elliptical because of the slight astigmatism introduced by the off-axis optics except at one distance where the picture of the hole was sharp and circular (see fig. 14). This position, which was almost 1 cm from the focal point originally located by less precise means, was about 7 mm farther when either a NaCl or CaF₂ window was inserted at the top of the radiation trap; hence, the detector position was made adjustable so that it could be properly located for different test conditions. Numerous emittance determinations showed that earlier measurements were approximately 10% too high because of the slightly off-focus positioning of the detector.

(3) Error from aberrations: A densitometer curve was made from the negative used for preparing fig. 14. The effect of this aberration would be to cause a blurring of the hole image which, in the case of holes of small diameter, would mean that the measured flux densities would be too low. However, for holes that are sufficiently large, no significant error from this source would be expected.

The effect of this aberration on the relative flux densities from the hole and the surface was investigated with specially prepared nickel specimens. The reference holes were drilled completely through the specimens. A narrow shoulder was machined near the top surface of each specimen so that the specimen could be supported from the top of the field concentrator by means of a thin mica sheet into which a hole was drilled so as to support the specimen at the shoulder. A water-cooled blackened cone was placed at the bottom of the concentrator beneath the hole in the specimen. With this arrangement, the detector when placed on the image of the hole in a heated specimen would "see" the cold blackened cone and, hence, the detector, at this position, should indicate a zero response with respect to the hot specimen surface. However, if optical aberrations were present some of the flux from the specimen surface would be "smeared out" onto the hole image. This, in turn, would be evidenced by hole readings that were greater than zero, as the edges of hole were approached.



Fig.14. Photograph taken at image plane of a heated platinum specimen with a deep reference hole.

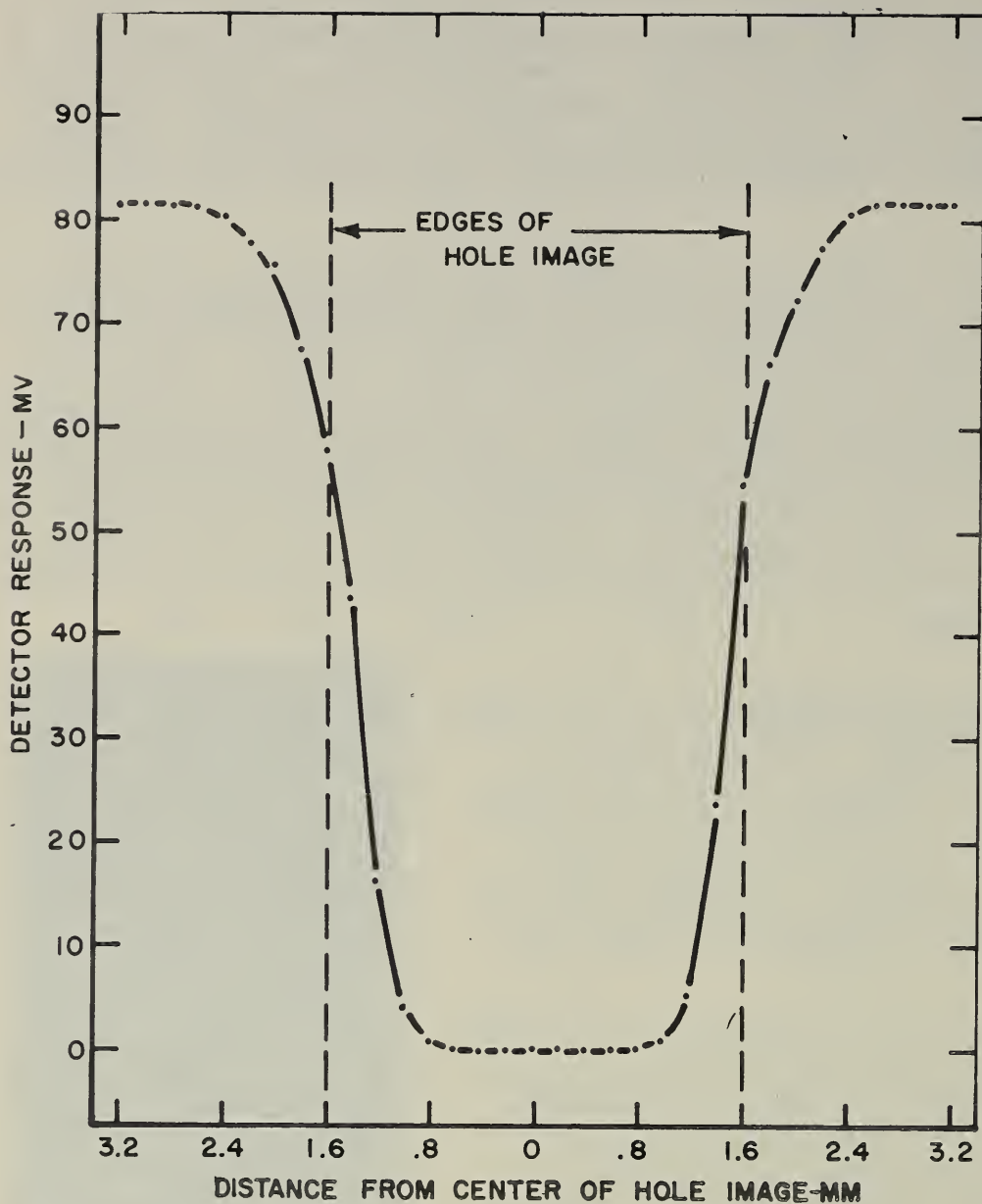


Figure 15. Change in detector response on scanning across the image of a 1.6 mm diam. hole in a heated oxidized nickel specimen. Hole was drilled through specimen so as to permit detector, when centered on hole image, to "see" water-cooled blackened surface at bottom of field concentrator. Resolution is determined by 0.5 mm diameter aperture on detector.

Fig. 15 shows the results obtained for an oxidized nickel specimen with a hole 1.6 mm diam. (image size 3.2 mm diam.). It is obvious from this graph that there are aberrations in the optics, and furthermore that an error from optical aberrations can be expected unless hole diameters of greater than about 0.7 mm are used. In similar test made with a platinum specimen having a 0.5 mm diam. hole, the flux from the center of the

hole was about 10% of that from the surface. It follows that all of the emittance measurements previously made on specimens with 0.5 mm diam. (0.25 mm radius) holes are too high, because of aberration in the optics. There should, however, be no measurable error from this source when the reference holes have diameters appreciably larger than 0.5 mm, such as was the case for the platinum specimen used in obtaining the data plotted in fig. 12.

Obviously the most desirable solution to the aberration error is to employ optics that will give an image free of aberrations. Replacement of the 5" spherical mirror with an off-axis ellipsoidal mirror should result in a significant improvement. Use of larger holes is not a desirable solution because of the larger temperature differences that the increased depth of such holes would create in the nonmetallic specimens.

(4) Window error: The window error mentioned in the Symposium paper (Ref. 6) has been completely eliminated through use of the radiation trap at the top of the bell jar coupled with the repositioning of the detector to the point of sharpest focus.

D. Summary of Present Status and Future Plans

Considerable effort during the latter part of the contract year was devoted to investigating possible sources of error. The measurements with steatite specimens indicated that a modified R_0 should be used for those specimens with reference holes having h/r values below 2. Although the measured emittances of these specimens are undoubtedly too high because of focus and aberration errors, they should all be high in the same proportion; hence, the conclusion on h/r ratio should be valid. Errors from deviation of the detector position from that of sharpest focus were detected and these will be eliminated from future measurements, as will be the error from the presence of a viewing window.

The most serious error that still remains is caused by the aberration which is present with the current optical system when hole diameters are below 0.7 mm. Since the use of larger, and hence deeper holes would introduce troublesome temperature differences between the surface and the bottom of the reference hole, the elimination of existing aberrations will be sought through substitution of an off-axis ellipsoidal mirror for the present spherical mirror.

Even when all measurement errors have been reduced to negligible amounts there will still be uncertainties caused by the failure of real specimens to fulfill the assumptions made in the derivation of the Gouffe expression (Section III-A). The magnitude of the error caused by these uncertainties will be evaluated during the coming year by comparing emittances obtained by the shallow-hole method with those obtained with the rotating cylinder equipment.

E. Restrictions on Measurements Above 2000°K

Many of the measurements that have been attempted at temperatures above 2000 K have encountered difficulties from two sources; (1) filming of the viewing window, and (2) change in specimen emittance due to such factors as thermal etching, specimen contamination from the crucible, volatilization of impurities, and specimen shrinkage. For many but not all materials these difficulties will present serious obstacles to the achievement of reliable measurements up to the target temperature of 2500°K.

REFERENCES

1. Equipment for thermal emittance measurements above 1400°K. First annual summary report to NASA on Contract No. H-3646. NBS report No. 7514, Jan. 1962.
2. Peavy, B. A. and Eubanks, A. G. Periodic heat flow in a hollow cylinder rotating in a furnace with a viewing port. Prepared for publication in the Proceedings of Symposium on Measurement of Thermal Radiation Properties of Solids, Dayton, Ohio, Sept. 1962.
3. Slemp, W. S. and Wade, W. R. A method for measuring the spectral normal emittance in air of a variety of materials having stable emittance characteristics. Vol. 11, p. 75 of preprints of papers for Symposium on Measurement of Thermal Radiation Properties of Solids, Dayton, Ohio, Sept. 1962.
4. Blau, H. H., Marsh, J. B., Martin, W. S. and Chaffee, E. Infrared spectral emittance properties of solid materials. Rept. No. AFCRL-TR-60-416, Geophysical Res. Dir., AF Res. Div., Oct. 1960.
5. Richmond, J. C., Harrison, W. N., and Shorten, F. J. An approach to thermal emittance standards. Vol. 11, p. 102 of Preprints of Papers for Symposium on Measurement of Thermal Radiation Properties of Solids, Dayton, Ohio, Sept. 1962.
6. Moore, D. G. Investigation of shallow reference cavities for high-temperature emittance measurements. Ibid. Vol. iii, p. 159, Sept. 1962.
7. Gouffe', A. Revue d'Opt. 24, 1 (1945).
8. Lang, S. M. Properties of high-temperature ceramics and cermets; elasticity and density at room temperature. NBS Monograph No. 6, 1960.
9. Vos, J. C. de. The emissivity of tungsten ribbon. Thesis Amsterdam 1953. Also, Physica 20, 690 (1954).
10. Buckley, H. Phil.Mag. 4, 753 (1927); 6, 447 (1928); 17, 576 (1934).
11. Abbott, G. L., Alvares, N. J. and Parker, W. J. Total normal and total hemispherical emittance of polished metals. WADD TR 61-94, Nov. 1961.



THE NATIONAL BUREAU OF STANDARDS

The scope of activities of the National Bureau of Standards at its major laboratories in Washington, D.C., and Boulder, Colorado, is suggested in the following listing of the divisions and sections engaged in technical work. In general, each section carries out specialized research, development, and engineering in the field indicated by its title. A brief description of the activities, and of the resultant publications, appears on the inside of the front cover.

WASHINGTON, D. C.

Electricity. Resistance and Reactance. Electrochemistry. Electrical Instruments. Magnetic Measurements. Dielectrics. High Voltage.

Metrology. Photometry and Colorimetry. Refractometry. Photographic Research. Length. Engineering Metrology. Mass and Scale. Volumetry and Densimetry.

Heat. Temperature Physics. Heat Measurements. Cryogenic Physics. Equation of State. Statistical Physics. **Radiation Physics.** X-ray. Radioactivity. Radiation Theory. High Energy Radiation. Radiological Equipment. Nucleonic Instrumentation. Neutron Physics.

Analytical and Inorganic Chemistry. Pure Substances. Spectrochemistry. Solution Chemistry. Standard Reference Materials. Applied Analytical Research. Crystal Chemistry.

Mechanics. Sound. Pressure and Vacuum. Fluid Mechanics. Engineering Mechanics. Rheology. Combustion Controls.

Polymers. Macromolecules: Synthesis and Structure. Polymer Chemistry. Polymer Physics. Polymer Characterization. Polymer Evaluation and Testing. Applied Polymer Standards and Research. Dental Research.

Metallurgy. Engineering Metallurgy. Microscopy and Diffraction. Metal Reactions. Metal Physics. Electrolysis and Metal Deposition.

Inorganic Solids. Engineering Ceramics. Glass. Solid State Chemistry. Crystal Growth. Physical Properties. Crystallography.

Building Research. Structural Engineering. Fire Research. Mechanical Systems. Organic Building Materials. Codes and Safety Standards. Heat Transfer. Inorganic Building Materials. Metallic Building Materials.

Applied Mathematics. Numerical Analysis. Computation. Statistical Engineering. Mathematical Physics. Operations Research.

Data Processing Systems. Components and Techniques. Computer Technology. Measurements Automation. Engineering Applications. Systems Analysis.

Atomic Physics. Spectroscopy. Infrared Spectroscopy. Far Ultraviolet Physics. Solid State Physics. Electron Physics. Atomic Physics. Plasma Spectroscopy.

Instrumentation. Engineering Electronics. Electron Devices. Electronic Instrumentation. Mechanical Instruments. Basic Instrumentation.

Physical Chemistry. Thermochemistry. Surface Chemistry. Organic Chemistry. Molecular Spectroscopy. Elementary Processes. Mass Spectrometry. Photochemistry and Radiation Chemistry.

Office of Weights and Measures.

BOULDER, COLO.

Cryogenic Engineering Laboratory. Cryogenic Equipment. Cryogenic Processes. Properties of Materials. Cryogenic Technical Services.

CENTRAL RADIO PROPAGATION LABORATORY

Ionosphere Research and Propagation. Low Frequency and Very Low Frequency Research. Ionosphere Research. Prediction Services. Sun-Earth Relationships. Field Engineering. Radio Warning Services. Vertical Soundings Research.

Radio Propagation Engineering. Data Reduction Instrumentation. Radio Noise. Tropospheric Measurements. Tropospheric Analysis. Propagation-Terrain Effects. Radio-Meteorology. Lower Atmosphere Physics.

Radio Systems. Applied Electromagnetic Theory. High Frequency and Very High Frequency Research. Frequency Utilization. Modulation Research. Antenna Research. Radiodetermination.

Upper Atmosphere and Space Physics. Upper Atmosphere and Plasma Physics. High Latitude Ionosphere Physics. Ionosphere and Exosphere Scatter. Airglow and Aurora. Ionospheric Radio Astronomy.

RADIO STANDARDS LABORATORY

Radio Physics. Radio Broadcast Service. Radio and Microwave Materials. Atomic Frequency and Time-Interval Standards. Radio Plasma. Millimeter-Wave Research.

Circuit Standards. High Frequency Electrical Standards. High Frequency Calibration Services. High Frequency Impedance Standards. Microwave Calibration Services. Microwave Circuit Standards. Low Frequency Calibration Services.

