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SPECTRAL EMISSIVITY AT HIGH TEMPERATURES

Six Months Report to

Air Force Office of Scientific Research

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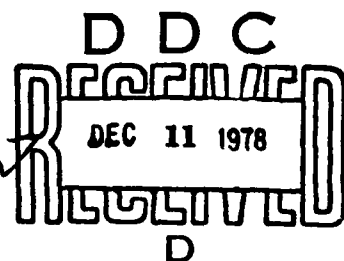
January - July 1978

by

P. E. Johnson, D. P. DeWitt, and R. E. Taylor

September 1978

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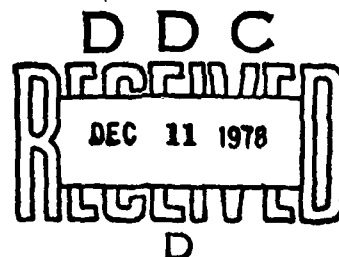
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ABSTRACT

The status of the high-temperature emissometer at the Properties Research Laboratory is summarized. The emissometer is an adjunct to the Multiproperty Apparatus which provides simultaneous measurement of ten thermophysical properties on the same sample of an electrically-conducting solid under various environmental conditions. The apparatus features rapid time-to-temperature and data acquisition under minicomputer control yielding state-of-the-art accuracy.

The calibration of the emissometer, including the incorporation of some new devices and the modification of the calibration data, has been more thoroughly studied.

Spectral emissivity measurements in the spectral range 2 to 12 μm have been made on tantalum (reference material), silicon nitride (Si_3N_4), silicon carbide (SiC), and a graphite composite from 1400 K to their degradation temperatures. The new data on the ceramics provides better understanding of their high temperature behavior including the effect of fabrication process and impurities.

The initial phase of the study to determine the feasibility of the emissometer to handle semitransparent materials has been completed. A model has been developed for relating the observed spectral radiance from a small cylindrical sample mounted within a heating tube to the optical properties (indices of refraction and absorption) and emissivity of the sample. Preliminary results of a parametric sensitivity analysis are discussed.

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Six Months Report
SPECTRAL EMISSIVITY AT HIGH TEMPERATURES

INTRODUCTION

The goal of this research program is to expand the capabilities of the Multiproperty Apparatus to include measurement of the thermal radiative or optical properties. The Multiproperty Apparatus represents a unique approach for simultaneously obtaining several transport properties. The utilization of the minicomputer to collect observations, control apparatus parameters, and calculate experimental results has been innovative. The method has become widely recognized as the most reliable method for detailed studies at high temperatures.

The thrust of the program for the past two and one-half years was the development and evaluation of the "integral blackbody" method for obtaining normal spectral emissivity on electric conductors and on nonmetallic materials. During the first year attention was focused on the method to study metallic systems.

When the sample is an electrical conductor and can be direct electrically heated, the method is readily implemented. In this instance, it is straightforward to assess the systematic errors associated with the quality of the blackbody cavity, temperature gradients in the vicinity of the sample-target areas, and radiometric parameter measurements. The basic features of this method are not new since this approach has been used by other investigators. However, we have improved the method in two significant ways. First, we

are able to simultaneously measure the normal spectral emissivity and other non-radiometric properties such as the hemispherical total emissivity and electrical resistivity. One of the advantages of this feature is the opportunity to characterize the sample surface by measurement of other transport properties. Second, the functions of the minicomputer provide both flexibility in the experimental procedure and improvement in precision over previous investigators.

During the second year of the research program, attention was given to developing a method for measurement of nonmetallic materials.

In this new method, the sample, in short-cylindrical form, is mounted within a heating tube whose functions are to create an isothermal zone around the sample and to serve as a blackbody cavity. The systematic errors due to temperature gradients in the tube and near the surface (target area) of the sample have been carefully evaluated. This new method has three important features which make it an improvement over other methods which have been previously described in the literature. First, the sample is small and in a form convenient for fabrication; this is especially attractive since new, nonmetallic materials are difficult and expensive to fabricate. Second, the measurement procedure including heat-up time can be accomplished in a short time; this is important in order to reduce effects of the environment on the sample. Third, the heating tube-sample interaction influences are minimized by avoiding solid contact; that is, the sample is thermally coupled by radiation exchange with the heating tube. While some interaction is likely, this can

be determined by monitoring electrical resistivity changes of the heating tube in the vicinity of the sample.

The capability to measure the normal spectral emissivity of non-conductors represents a major contribution to high temperature technology. The existing data in the literature is sparse and poorly characterized (Ref. 1 and 2). For certain classes of materials useful in high-energy, laser-related applications there is insufficient data to understand temperature effects above normal ambient. Most of the materials are alloys or mixtures and it is expected that higher temperatures (and temperature cycling), environmental conditions (air vs. vacuum), and fabrication methods will have an appreciable, if not dominating, effect on the thermal radiative properties. Without the benefit of some careful experimental results on well-characterized materials we are unable to predict such material behavior. As will be seen from the accomplishments of our research to-date, we have the tools to generate high quality data and new information is being generated which provide some basis for predictive schemes.

The report is organized to provide a summary of the total program and the measurement capability that exists today. The objectives for the third year are outlined and discussed. A description of the emissometer is concisely presented so that details of the measurement method, hardware and associated minicomputer data acquisition/control system can be appreciated.

During the reporting period attention was given to the wavelength calibration procedures. Apparatus and software (programming) modifications were made to permit greater reliability and flexibility

in establishing the wavelength scale for the emissometer. A continuing activity of the program has been the studies on new ceramic materials. New data on silicon nitride and silicon carbide are presented; for the latter material, results for the α - and β -phases give an indication of the effect of structure and impurity on the spectral emissivity.

Additional effort was given to evaluation of a proprietary (U. S. Naval Surface Weapons Center) graphite composite sample. Initial studies in 1977 permitted operation of the emissometer to 2000 K with a special configured sample. After several failures using various sample shapes and heat shield combinations, operation to 3100 K was achieved at which point the material failed.

An important objective for this third year is the study to determine the feasibility of measuring the emissivity/optical properties of semitransparent materials in the Multiproperty Apparatus. A heat transfer model has been developed and a parametric study performed, the implications of which are discussed in the final section of this report.

OBJECTIVES OF THE RESEARCH

In our proposal dated 15 November 1977 we outlined four objectives for the third year of this research program. Briefly these objectives will be reviewed and the progress made on them will be presented in subsequent sections of this report.

The first objective related to measurement method studies which would result in improved emissometer performance. One aspect of this work was the installation of the new trivalent (HgCdTe) detector

which substantially extends the wavelength capability of the emissometer. This work was started in late 1977 and subsequent experiences during the first six months of the present contract year have helped to establish further confidence in our measurements. As a consequence of this activity, we became aware of the need to improve our wavelength calibration procedures. This was a rather substantial effort and is summarized in the subsequent section entitled "CALIBRATION OF THE EMISSOMETER".

The second objective for the current year is to study new ceramic materials at high temperatures. The work on silicon nitride and silicon carbide reported in the section, "CERAMIC SAMPLE EMISSIVITY MEASUREMENTS", shows that the spectral emissivity is very dependent upon the impurities and fabrication method of the material. There are numerous other ceramics which need to be carefully studied in order to gain a more fundamental understanding of high temperature behavior.

The third objective is related to the use of radiation shielding in order to obtain higher temperature operation. The need to consider the approach arose from the studies summarized in the section "GRAPHITE COMPOSITE EMISSIVITY MEASUREMENTS". Operation to 3100 K was achieved but attempts to use radiation shields were not very successful. At this time it appears to be more practical to modify the sample configuration rather than to employ radiation shields. Hence, we have not given any serious attention to heat transfer analysis that would help in identifying optimal radiation shielding.

The fourth and final objective of this year's program is to study the extension of the Multiproperty Apparatus capability to measure

the optical properties of semitransparent materials. The section entitled "FEASIBILITY STUDY ON METHOD FOR SEMITRANSSPARENT MATERIALS" summarizes our activities to date suggesting that the method might be a contribution to high temperature technology.

DESCRIPTION OF THE EMISSOMETER

The Multiproperty Apparatus

The experimental apparatus is shown schematically in Fig. 1. The apparatus has been described in detail previously [3] and only a brief description will be presented here.

The metallic sample in the form a thin-walled, conducting tube is supported vertically between two water cooled movable electrodes as shown in Fig. 2. The nonmetallic sample, as shown in Fig. 3, is of a short-cylindrical form mounted with a heating tube. The electrode separation distance is adjustable between 0 and 35 cm. To accommodate length changes in the sample due to thermal expansion, a strain-relief system is provided with the lower electrode adjustable. The 76 cm diameter bell jar containing the sample holder is raised and lowered by power-driven hoist. Vacuum in the low 10^{-6} torr range can be achieved in a few minutes and vacuum in the mid 10^{-7} torr range can be attained with longer pump out times. The bell jar rests on a feed-through collar which contains instrumentation leads, electrical connections and water lines. Signals to be measured are patched into a terminal board connected to the mini-computer. Power is supplied to the sample through regulated DC power supplies. A 500 ampere-50 volt supply, transformer, calibrated

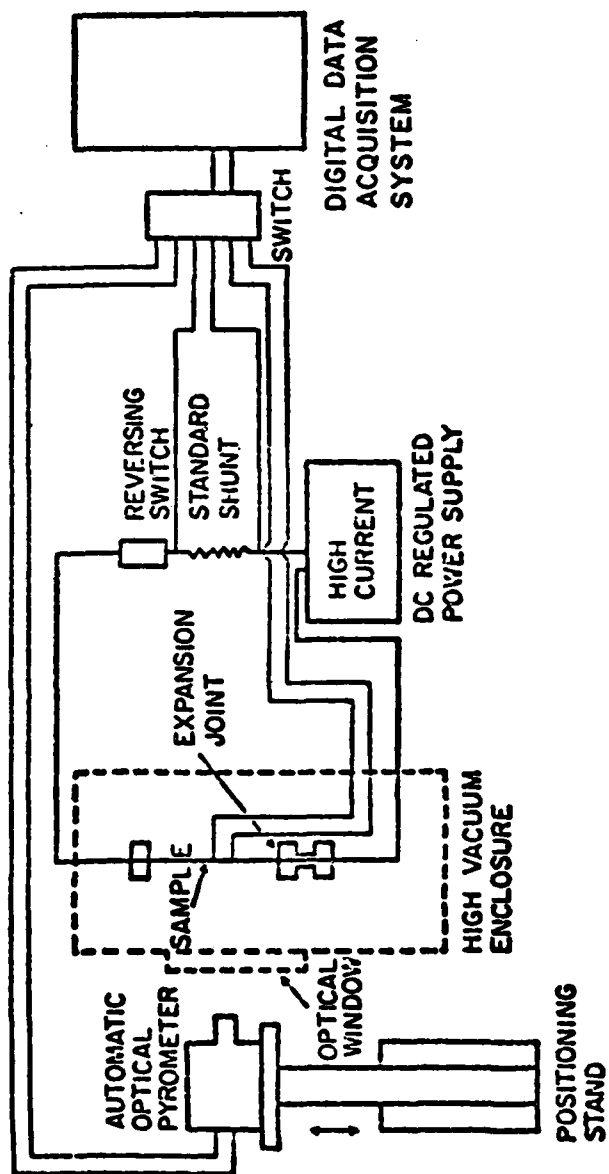


Figure 1. Schematic of the Multiproperty Apparatus

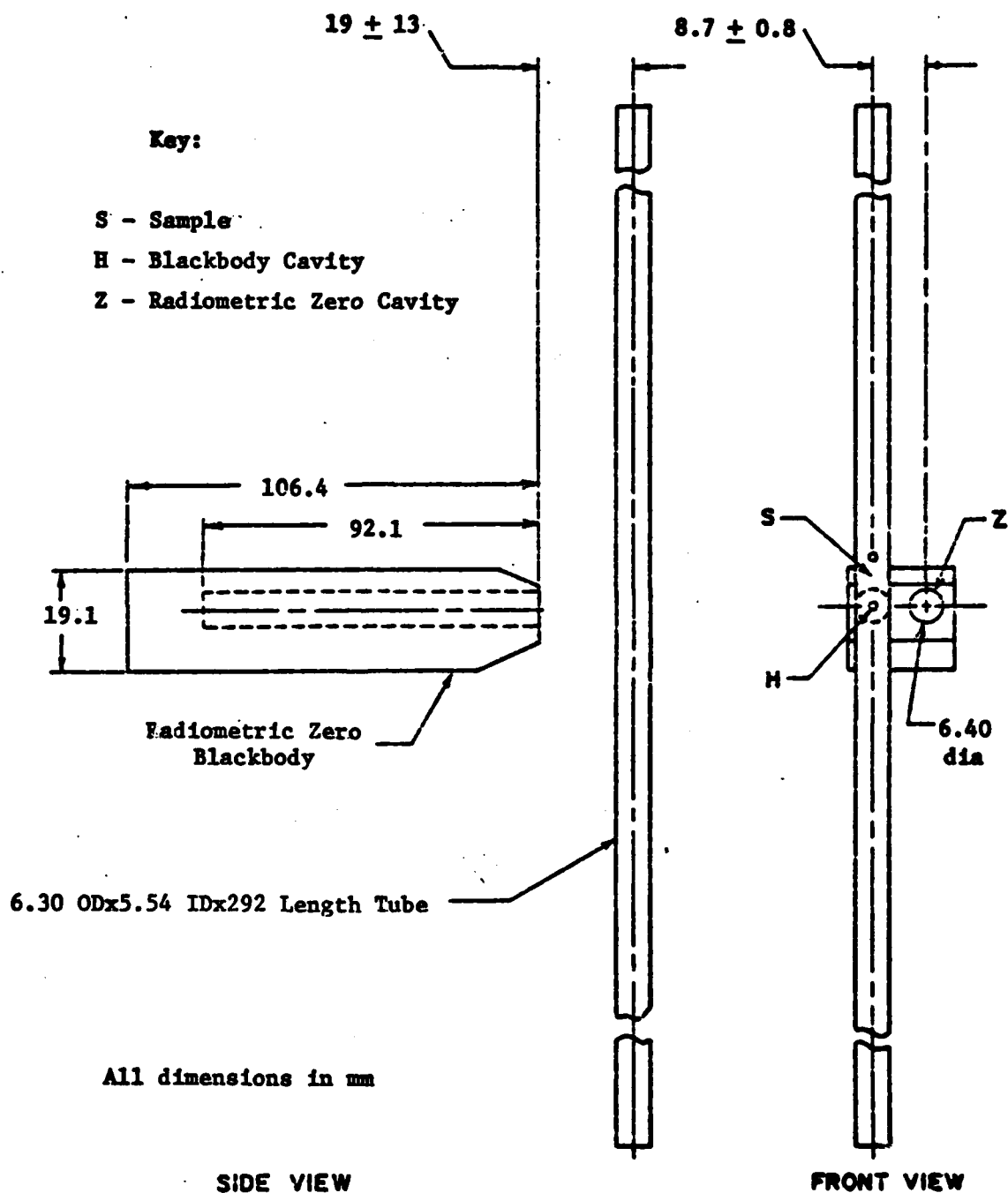


Figure 2. Sample heating tube showing the sample and blackbody cavity target positions within the test section and separate radiometric zero cavity.

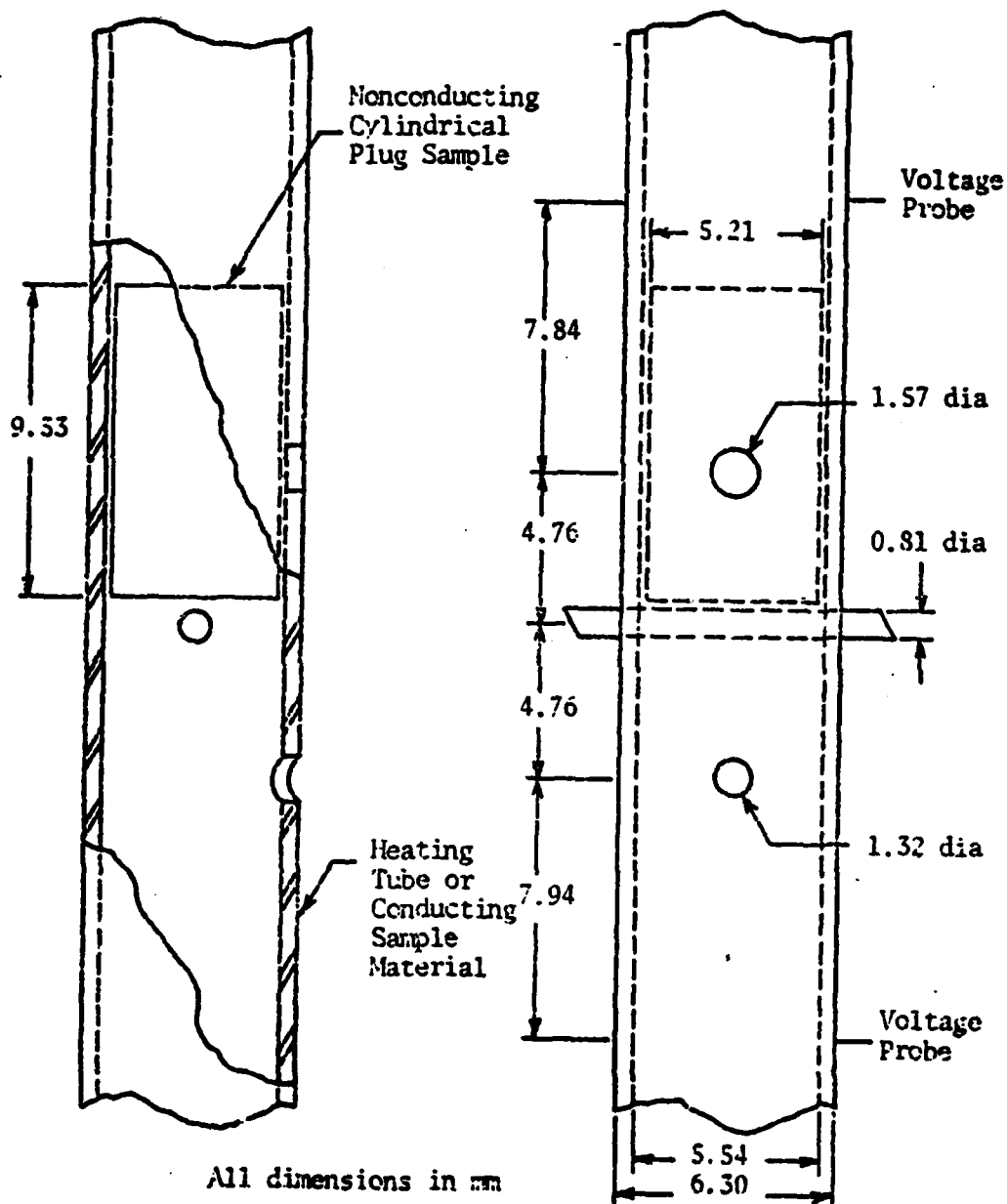


Figure 3. Test section configuration with nonconducting sample (upper cavity) and blackbody cavity.

shunt, reversing switches and remote control circuitry, are used for operations where sample temperatures exceed 1800 K while three, 100 amp-10 volt Kepco power supplies are used for lower sample temperatures.

The Optical and Detection System

The emissometer optical system shown schematically in Fig. 4 is mounted on a table located in front of the Multiproperty Apparatus. The position of mirror M1 is set by solenoids prior to the start of the spectral data collection such that signals from the three targets - sample surface (S), blackbody cavity (H), and the radiometric zero (Z) - can be measured. During the collection of data the minicomputer controls the solenoids (via programmable switches) such that the three targets are viewed in a time sequence set at the start of the experiment.

The spherical mirror M2 takes the image which is being viewed by M1 (sample surface, blackbody cavity or radiometric zero) and places the image at the aperture (A) plane. A 1000 hz chopper (C) is located just behind the aperture plane.

Mirror M3 can be positioned such that the target image can be placed by the ellipsoid mirror M4 onto a thermocouple detector. In this position the flux reaching the detector is undispersed and the total emissivity can be measured. This feature has not yet been studied. Mirror M3 can also place the target on the plane mirror M5 and then onto the spherical mirror M6. Mirror M6 then places an image of the aperture (A), with a magnification of two, onto the entrance slit of the prism monochrometer, and from here the target

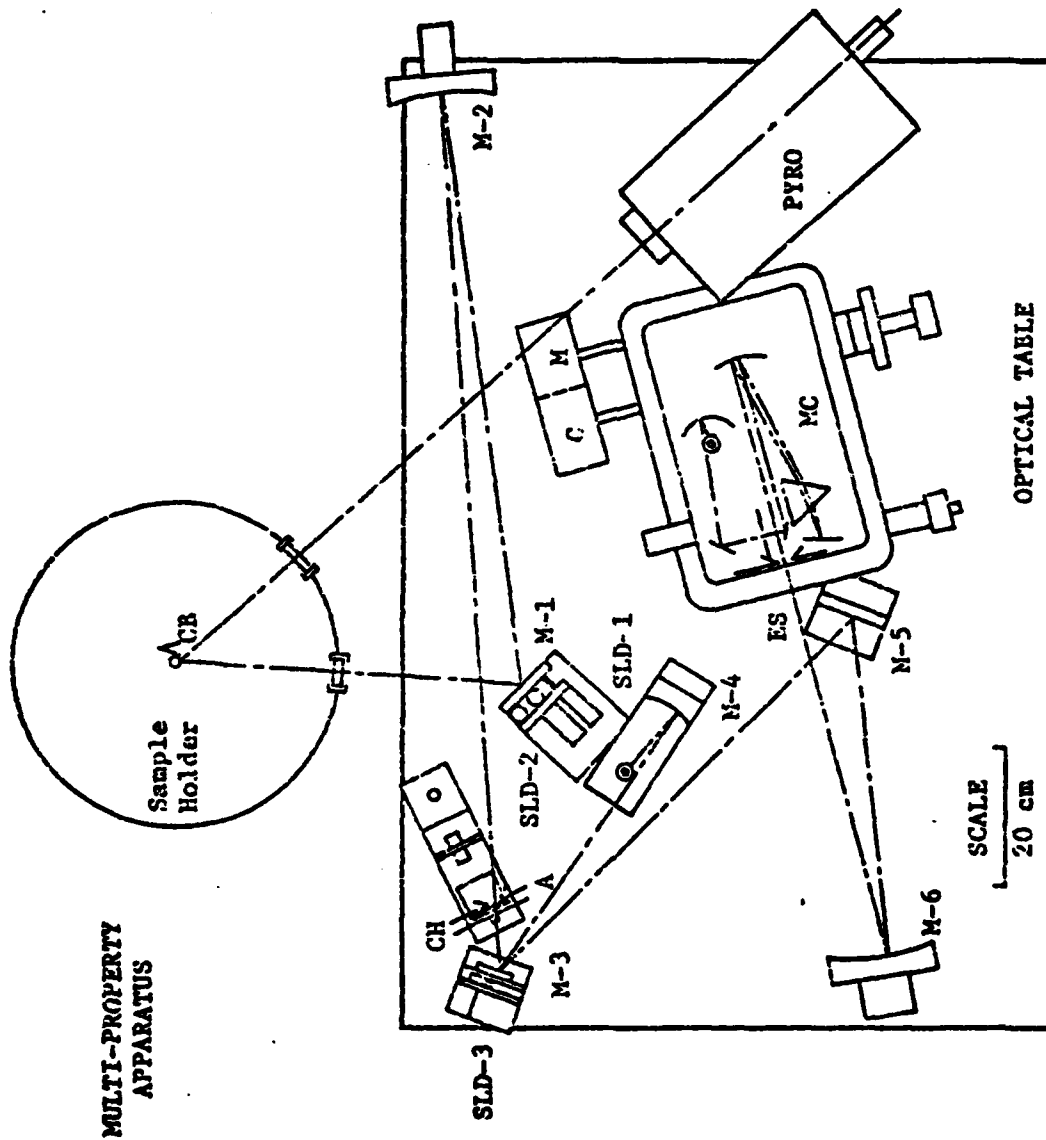


Figure 4. Schematic of the Emisometer illustrating positioning of sample, optical train, and detection system.

image is optically dispersed and focused onto the detector for measurement.

A photograph of the entire optical detection system with the Multiproperty Apparatus in the background is provided in Fig. 5.

The Minicomputer System

The system contains a PDP-8/E minicomputer (central processing unit) with 32,000 words of core connected to an omnibus. Connected to the omnibus through a "data break" is a controller and disk drive for a disk system which is subdivided into four parts, System (SYS), disk 1, disk 2, and disk 3. The SYS system with 1×10^6 word capacity and DSK1 with 6×10^5 word capacity reside on a removable cartridge with a floating head. The other two parts (DSK2 and DSK3 with 1×10^6 and 6×10^5 word capacity, respectively) reside on a fixed head cartridge. Information can be swapped readily among the various subdivisions. The capacity of the DDAS is sufficiently large (3.3×10^6 words) that programs and experimental data may be stored and used without input-output to any other device such as magnetic tape, punched tape, punched cards, etc., although a high speed paper tape reader/punch and magnetic tape units are included. Other disk cartridges may be readily substituted for the removable cartridge, providing additional storage and back-up capabilities. Instructions and programs may also be entered through the Tektronix Display Terminal (Model 4010-1), which is the master input/output device or through the Decwriter (high-speed teletype) or standard teletype units. The Decwriter is essentially used as a line printer to give printed output. In addition, hard copies of whatever is displayed



Figure 5. Photograph of Emissometer showing DC power supplies on the right, amplifiers in front and the Multiproperty Apparatus in the background

on the Display Terminal can be made by the Tektronix Hard Copy Unit (Model 4610).

Experimental data are directly entered into the system through an analog-to-digital converter (ADC) or through a VIDAR Model 521-01 integrating digital voltmeter (IDVM). The IDVM is controlled via a master Scanner (Vidar 610) and is equipped for 100 input channels. The Vidar DVM has full scale ranges from 10 MV to 1000 V in steps of 10 and has three integration periods (166.7, 16.7 and 1.67 milliseconds) so chosen as to essentially provide infinite rejection of 60 hz signals. The resolution of the IDVM depends upon the integration period and for the 10 MV full scale range is ± 0.1 microvolt for the longest integration period. Operating in conjunction with the IDVM is a special crystal-controlled timer which accurately records the time the data are taken, without resort to software timing.

In addition, the DDAS is equipped with a real time clock and with programmable switches and digital-to-analog converters (DAC) for controlling experiments.

Details of the measurement procedure and the nature of the software programs have been reported in Reference 4. This will all be omitted for the sake of brevity; however, it should be stated that extensive effort was required to develop flexible programs which are being expanded as the emissometer is upgraded.

CALIBRATION OF THE EMISSOMETER

A properly executed calibration procedure that can reliably and conveniently be accomplished is essential if high confidence is to be placed on the spectral emissivity measurements. Extensive effort

has been given to installation of accessories which will permit wavelength calibrations to be performed on a routine basis. Changes to the minicomputer (software) program for performing the calibration and for storing the calibration results were made.

Incorporation of New Devices

To avoid tying up the Multiproperty Apparatus for calibration purposes and to make the procedure as straightforward as possible, several new devices were incorporated into the emissometer.

A globar is used as the source of continuous infrared radiation. It is a silicon carbide rod 4.8 mm diameter and 5.0 cm long. The rod, surrounded by a water-cooled jacket, is electrically heated up to 1100°C with an AC power supply. Throughout the region covered by the spectrometer, the infrared radiation from the globar has approximately the same distribution as that of a blackbody at the same temperature. By mounting the globar casing onto a stand, which screws directly into the optical table, the need for the Multiproperty Apparatus is eliminated and the calibration can be performed in a separate room altogether. Furthermore, this arrangement will permit spectral transmittance measurements on infrared window materials at room temperature to be routinely performed.

Another added feature was heaters on the spectrometer base. These heaters serve to thermally stabilize the spectrometer optics and prism at a $37^{\circ}\text{C} \pm 0.7^{\circ}\text{C}$. By heating the prism, the wavelength accuracy of the instrument is insured and it also prevents prism fogging. With the heating pads operational, the option existed for performing the calibration "hot" or "cold". After performing both,

it was determined that having the prism heated resulted in a more stable scan of the spectrum, and therefore, a more reproducible calibration. As a result of this experiment, the calibration data was modified to account for the effect of the heated prism and consequently, the prism is heated during all spectral emissivity runs.

A variable speed control was also adopted which enables the operator to speed up or slow down the wavedrive mechanism, thereby scanning the spectrum at a faster or slower rate. This is particularly useful in those regions of the spectrum where sharp absorption bands occur. By scanning at a slow rate the band can be spread out and more readily recognized. Likewise, a fast scan rate is desired where the energy level is low, where there is no spectral character to the data, or where the sensitivity is low.

Wavelength Calibration

The function of the wavelength calibration is simply to relate a potentiometer signal to a particular wavelength. This is the calibration which is used in program SEM2 to determine the wavelength from measured potentiometer signals. The potentiometer signal is a function of the angular position of the monochromator wavelength drum and the angular position of this wavelength drum determines the wavelength of the target signal reaching the detector.

The calibration of the wavelength was based on measuring the transmittance as a function of potentiometer signal for a standard polystyrene sample. The standard polystyrene sheet was supplied along with its transmission versus wavelength characteristics by Perkin - Elmer, the manufacturer of the Model 98 monochromator. The

absolute maximum and minimum transmission peaks of the supplied transmittance versus wavelength curve were compared with the peaks of the measured transmittance versus potentiometer signal curve to arrive at a correspondence between measured potentiometer signals and wavelength. This correspondence, actually being the wavelength calibration sought, was then compared to the existing wavelength calibration to check its accuracy.

The first step in generating the transmittance versus potentiometer signal curve for the polystyrene material was to place the emissometer in front of the globar source such that a strong detector signal was obtained. For the present study the globar stand was mounted on the optical table such that the globar was the same distance from the target mirror as a sample being heated in the bell jar would be. The next step was to engage the wavelength drum drive motor such that the drum would rotate at a constant speed. This in turn produced a linearly varying potentiometer signal. As the drum rotated, the amplified detector signal was being recorded by a strip chart, feeding at a constant rate. Also recorded on the strip chart were the positions at which the wavelength drive motor was engaged and disengaged and the potentiometer signals corresponding to these positions. Since the wavelength drum drive motor and the strip chart were both moving at constant speeds, the potentiometer signal should vary linearly between the two recorded points. Therefore, the detector signal produced by the globar has been recorded as a function of the linearly varying potentiometer signal. It should be noted that the amplifier time constant (30 ms), chart speed (2 inches/min), and rate of wavelength drum rotation (1, 2 or 4 rpm),

were chosen such that the time delay was negligible between the detector sensing the signal and the signal being recorded.

Since the radiation from the globar has a considerably different distribution than the radiation from the previously used tantalum source, and in an effort to obtain the best resolution and clarity, four parameters were varied throughout the scanning of the spectrum. These parameters were the (1) monochromatic entrance slit width (.10 mm to .40 mm); (2) amplifier sensitivity setting or gain (10 μ V (x2) to 1 μ V (x1)); (3) wavelength drum drive motor speed (1 rpm to 4 rpm); and (4) volt box setting ($\frac{\text{MULTI BY } 200}{\text{MAX VOLTS } 30}$ to $\frac{20}{3}$). Because this was solely a trial and error process, a great deal of time was required in determining the initial value of these parameters and in learning exactly when to change them.

The next step in determining the transmittance as a function of potentiometer signal for the polystyrene standard was to place the standard over the entrance slit of the monochrometer such that the incoming beam would have to pass through the polystyrene. The globar was maintained at the same temperature level and all parameters were re-set to their initial values as in the previous run. The wavelength drum drive motor and the strip chart were then engaged, as in the previous run, and again the detector signal produced by the globar was recorded as a function of the linearly varying potentiometer signal. In short, this run was identical to the previous run except for the polystyrene placed over the entrance slit of the monochrometer.

Taking the ratio of the detector signal with the polystyrene present to the detector signal without the polystyrene present for

the same potentiometer signal level gives the transmittance at that particular potentiometer signal. Following this procedure for a number of potentiometer signal levels gives the spectral transmittance versus potentiometer signal curve for the standard. Then, as previously explained, the absolute maximum and minimum transmittance peaks of this curve are compared to the known spectral characteristics of the polystyrene standard and the potentiometer signal-wavelength relationship is established.

Modification of Calibration Data

Once the wavelength versus potentiometer signal-calibration curve is obtained, it is then necessary to compare this curve with the result stored in the minicomputer memory and used in previous experiments. The difficulty in the recent past has been the inability to change the calibration file so that a given potentiometer signal corresponds to the proper wavelength. A periodic check of the calibration is essential for high confidence in measurements and hence a routine method for modifying the minicomputer calibration file is necessary.

For each value of wavelength drum number, the corresponding potentiometer signal can be determined manually by rotating the wave drum and observing the signal on a digital voltmeter. This allows both potentiometer signal and wavelength drum number to be plotted against wavelength.

Subroutine EVM of program SEM3 determines the wavelength from the measured potentiometer signal by means of linear interpolation. The data from which this subroutine operates is the calibration

file, SALT CL.DA. This file lists potentiometer ratio times 40 (convenient factor) and the corresponding electron volts which are given by dividing 1.2398 by wavelength (in micrometers). The potentiometer ratio is, therefore, easily obtained from this file. The next step is to take the value of potentiometer ratio obtained from the calibration file and go to the wavelength versus potentiometer signal curve and read off the corresponding value of wavelength (in microns). Then divide 1.2398 by this value of wavelength to obtain the new value of electron volts. Once this procedure has been carried out for all points, the new data can be put back into SALT CL.DA and the modification of the calibration data will be complete.

SPECTRAL EMISSIVITY OF TANTALUM

To evaluate emissometer performance and to verify that the spectrum could be scanned from 1.5 to 15 μm , measurements were made on a used tantalum tube sample and compared to previous experimental results. Tantalum has been used in our in-house reference material and measurements made when any modifications are made to the emissometer. Tantalum was selected as the reference material for several reasons, key among these being our experiences with the material were extensive, the material is relatively inexpensive and its spectral emissivity is stable with repeated thermal cycling in vacuum.

Figure 6 represents the results of an observation of the spectral emissivity of tantalum at 1900 K for the spectral region 1.5 to 15 μm ; the results are also given in Table 1. The spectral emissivity values of this sample were slightly higher than those of virgin tantalum. This result was to be expected, however, because

of the change in surface conditions which resulted from the tube being used previously to contain a sample material which reacted slightly with the tube, thereby contaminating it somewhat. It should be noted that the graph of Fig. 6 is obtained from the computer display unit screen; at the start of the observations, only the coordinate system appears and then as the readings are taken for each wavelength, dots appear representing the blackbody (H) and sample (S) signal levels. As the spectral emissivity for each wavelength is calculated, a solid line connecting the points is drawn, eventually giving the full emissivity spectra when the experiment is completed. This manner of displaying the emissivity spectra as the data is collected allows the operator to view the progress of the experiment and make any corrections that might be necessary.

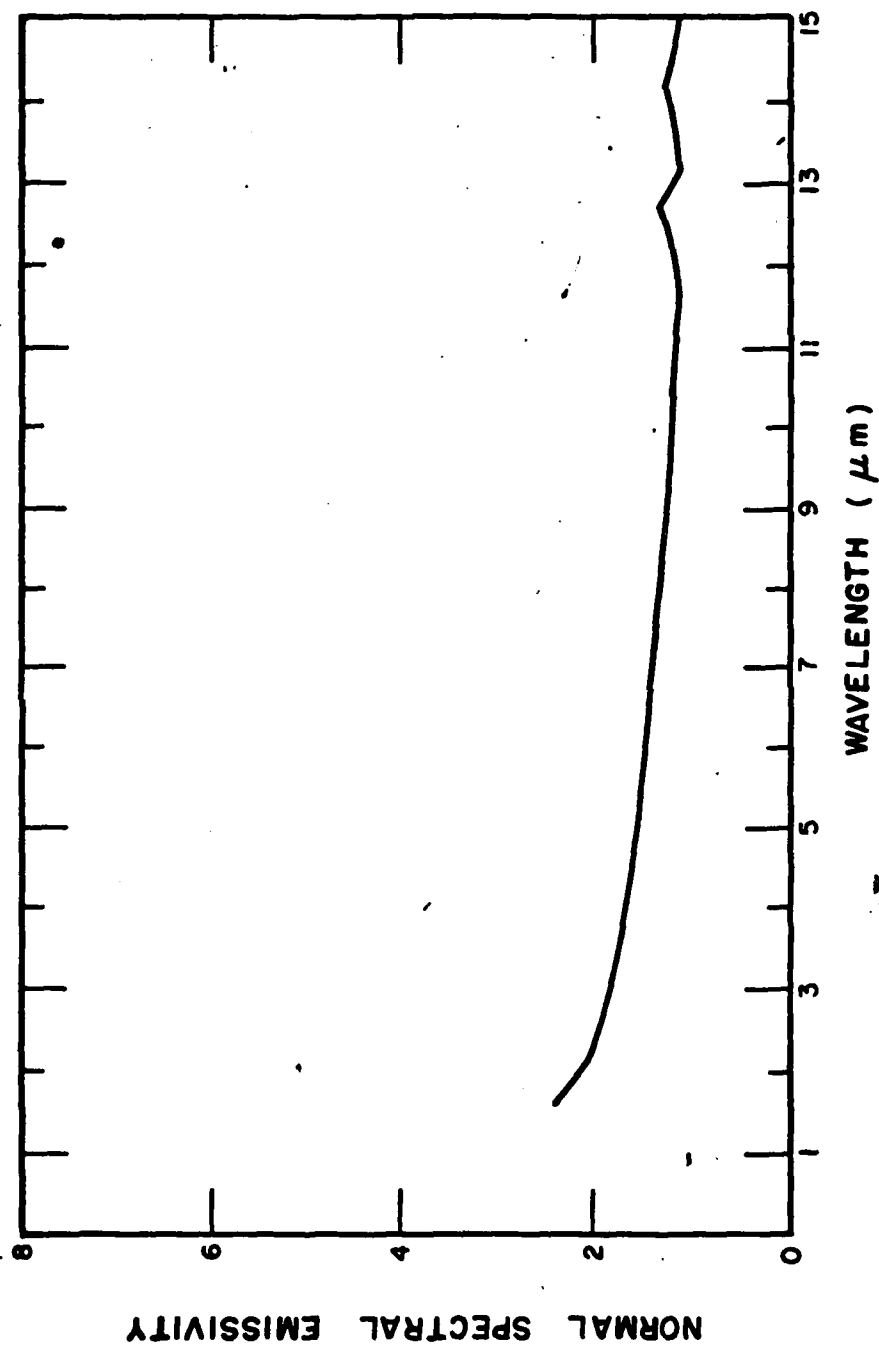


Figure 6. Spectral emissivity of tantalum

Table 1 Normal Spectral Emissivity of Tantalum at 1900 K

<u>Wavelength (μm)</u>	<u>ϵ_{λ}</u>
1.53	0.241
2.09	0.204
2.66	0.189
3.20	0.183
3.62	0.175
4.14	0.166
4.61	0.161
5.10	0.156
5.62	0.148
6.10	0.146
6.60	---
7.10	0.137
7.60	0.138
8.09	0.131
8.59	0.128
9.09	0.126
9.60	0.124
10.09	0.122
10.59	0.117
11.09	0.116
11.59	0.110
12.10	0.114
12.60	0.130
13.09	0.108
13.59	0.111
14.10	---
14.59	0.111
15.11	0.104

CERAMIC SAMPLE EMISSIVITY MEASUREMENTS

During the second year, the thrust of our activities was toward development of the method to measure the spectral emissivity of non-conducting materials. In our annual report we present the result for several (4) materials which demonstrated the precision and acceptability of the method. Additionally, a heat transfer analysis was performed to indicate that there were no significant temperature nonuniformities which could cause appreciable systematic errors. In the first six months during the present contract period, we have studied two new ceramic materials, the results of which have helped us to better understand the measurement method and the nature of material behavior at high temperatures.

Norton Silicon Nitride

The Norton silicon nitride sample had been heat treated for 30 hours, however, no further conditioning information was made available.

Because of the instability of silicon nitride at high temperatures under vacuum, emissivity data were first taken at 1400 K, the temperature level was increased in 100 K steps to 1800 K and at each level it was carefully observed if instabilities occurred. The sample was quite unstable, as indicated by the spectral emissivity versus wavelength results of Fig. 7 and Table 2. The data from 1400 to 1500 K show an increase in emissivity from 3 to 8 μm , a decrease from 8 to 11 μm and an increase from 11 to 14 μm . The spectral emissivity continued to drop in magnitude and to flatten out in shape

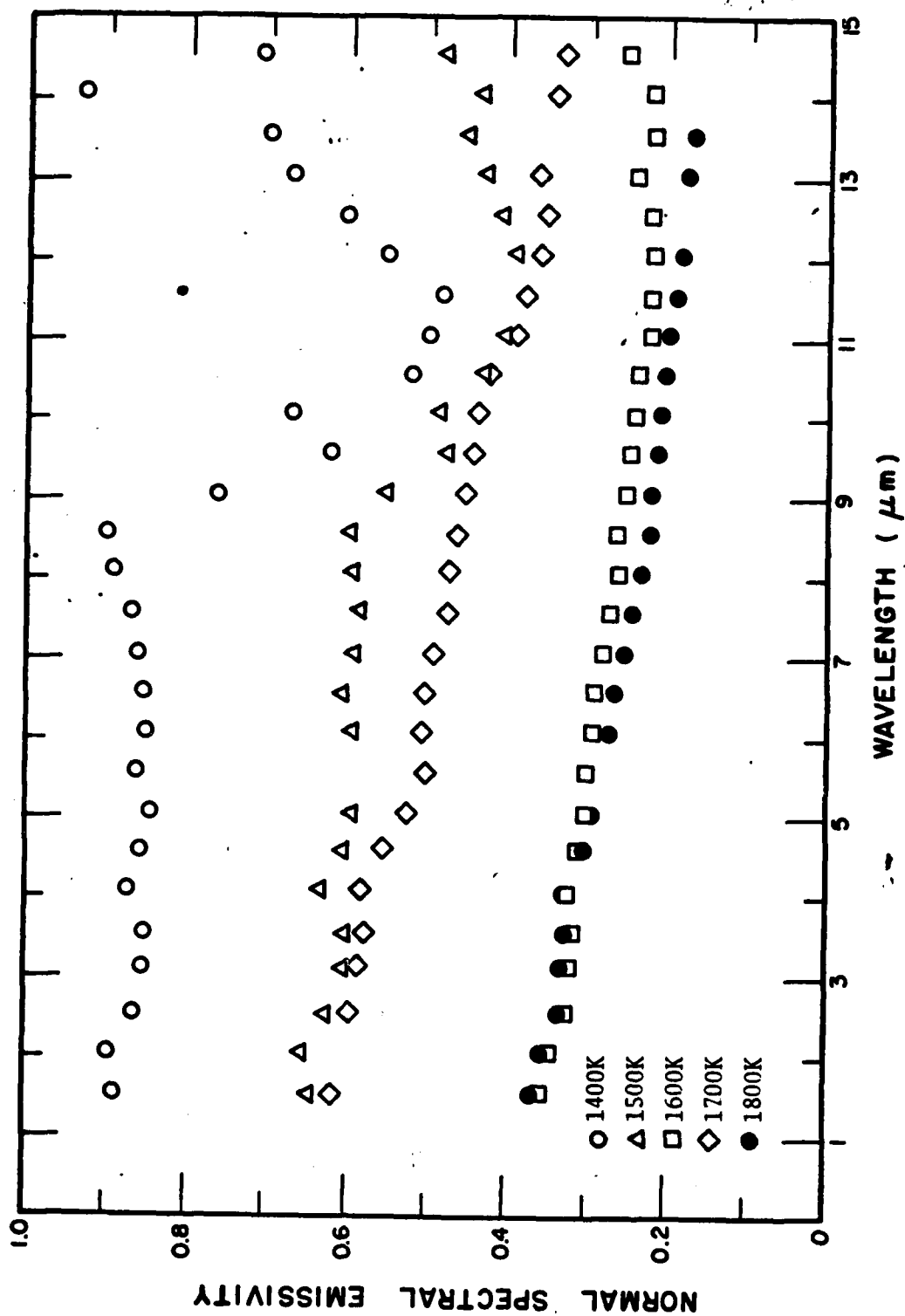


Figure 7. Spectral emissivity of Norton silicon nitride

Table 2 Normal Spectral Emissivity of Norton Silicon Nitride

λ (μm)	Temperature (K)				
	1400	1500	1600	1700	1800
1.52	0.889	0.644	0.353	0.618	0.370
2.06	0.898	0.659	0.344	0.653	0.358
2.57	0.868	0.622	0.326	0.599	0.337
3.13	0.854	0.603	0.321	0.585	0.331
3.57	0.851	0.605	0.318	0.579	0.322
4.10	0.873	0.632	0.327	0.582	0.327
4.60	0.860	0.607	0.312	0.554	0.308
5.09	0.848	0.593	0.302	0.527	0.294
5.58	0.862	---	0.302	0.502	---
6.09	0.852	0.593	0.295	0.508	0.276
6.58	0.857	0.608	0.292	0.504	0.269
7.08	0.863	0.592	0.281	0.493	0.257
7.59	0.871	0.589	0.274	0.479	0.247
8.09	0.896	0.598	0.264	0.475	0.233
8.58	0.901	0.599	0.265	0.466	0.225
9.07	0.767	0.552	0.253	0.454	0.222
9.59	0.624	0.481	0.250	0.445	0.217
10.08	0.672	0.490	0.246	0.440	0.211
10.58	0.522	0.433	0.240	0.424	0.208
11.08	0.501	0.408	0.225	0.396	0.204
11.58	0.488	0.140	0.226	0.382	0.191
12.08	0.555	0.394	0.222	0.366	0.186
12.57	0.607	0.413	0.224	0.357	---
13.08	0.671	0.434	0.243	0.368	0.182
13.58	0.701	0.453	0.221	---	0.174
14.08	0.933	0.438	0.226	0.345	0.213
14.58	0.711	0.482	0.258	0.334	---

from 1500 to 1600 K, but at 1700 K there was an increase in emissivity, followed by a decrease at 1800 K. The sample definitely deteriorated, as evidenced by the difference in the before and after weighings as well as dimensional changes. Unfortunately, the exact temperature at which this occurred is unknown. This might explain the sudden increase in emissivity at 1700 K.

G. E. Silicon Carbide, β -Phase

This G. E. sample, a virgin sample of SiC, was heated to 1800 K where two spectral emissivity runs were made. Following this, a single run at 1900 K was made. The maximum number of data points (49) were taken of each target (H, S, Z), and therefore, during each run the sample was at temperature for about 30 minutes. The emissivity was higher than that obtained by Riddle in his measurements [5] on a similar G. E. sample of this material. This could be due to the fact that the percentages of boron, plus any impurities, were not the same, even though both samples were G. E. silicon carbide. However, the same trends were observed. The plateau occurring at 11 μm is thought to be caused by boron carbide. At 1800 K this rise is quite distinct, while at 1900 K this effect is not quite so noticeable. The values of emissivity were very consistent over the temperature range considered as revealed in Fig. 8 and Table 3.

G. E. Silicon Carbide, α -Phase

This G. E. sample was the boron-doped, sintered silicon carbide which was measured and reported by Riddle in report PRL 152 [5]. The sample was heated at 600°C/hr. to 2200°C in helium at 1 atm. and

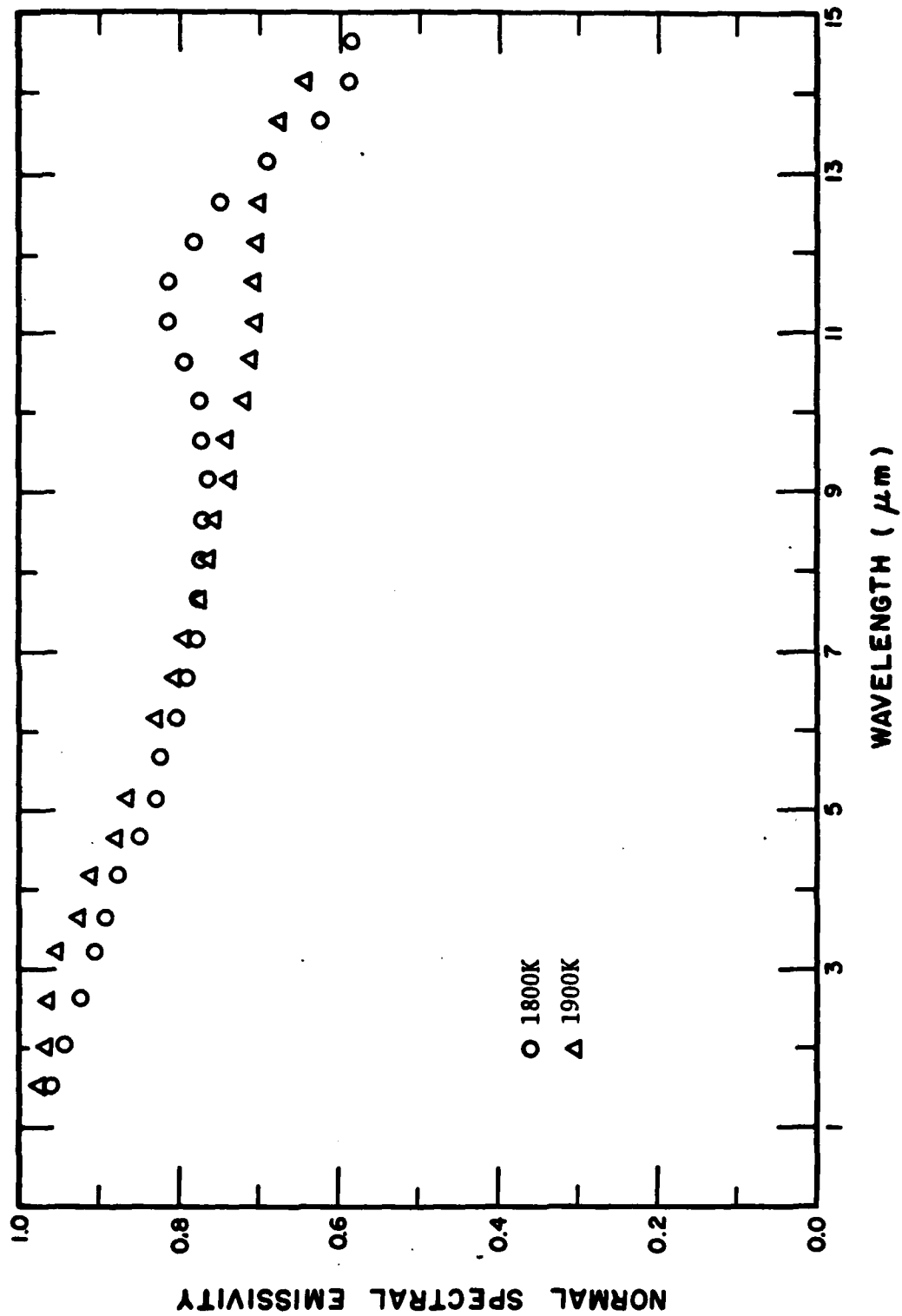


Figure 8. Spectral emissivity of G. E. silicon carbide, β -phase

Table 3 Normal Spectral Emissivity of G. E. Silicon Carbide, β -Phase

λ (μm)	Temperature (K)	
	1800	1900
1.52	0.961	0.977
2.06	0.945	0.969
2.62	0.922	0.966
3.22	0.909	0.953
3.66	0.892	0.927
4.19	0.876	0.912
4.68	0.850	0.880
5.17	0.830	0.864
5.69	0.825	---
6.18	0.805	0.830
6.67	0.797	0.807
7.18	0.780	0.794
7.67	0.778	0.778
8.17	0.775	0.768
8.66	0.771	0.756
9.16	0.768	0.740
9.67	0.772	0.742
10.17	0.778	0.720
10.67	0.796	0.711
11.17	0.816	0.708
11.66	0.816	0.708
12.17	0.786	0.706
12.67	0.750	0.702
13.17	0.693	---
13.66	0.627	0.679
14.17	0.591	0.649
14.66	0.587	---

held at temperature for 30 minutes by the Physical Chemistry Lab at General Electric. This treatment was believed to have transformed the cubic β -SiC to hexagonal α -SiC. It was thought that this transformation would cause the spectral emissivity curve to follow the same pattern exhibited by the other α -phase SiC samples.

The sample holder was the same tantalum tube used to run the G. E. β -SiC sample. The sample was run twice at 1800 K and twice at 1900 K. Once again the maximum number of data points were taken at each target, and therefore, each run took about 30 minutes. The emissivity runs were very smooth, stable, and reproducible. Changing the temperature to 1900 K did not have a pronounced effect upon the value of the spectral emissivity. The four runs were very similar. However, the expectation that the curve would flatten out was not observed. The curve was found to steadily drop off until 8 μm and then it started to level out, as Fig. 9 indicates. At this time we have no explanation for this behavior. A tabulated listing of the data is given in Table 4.

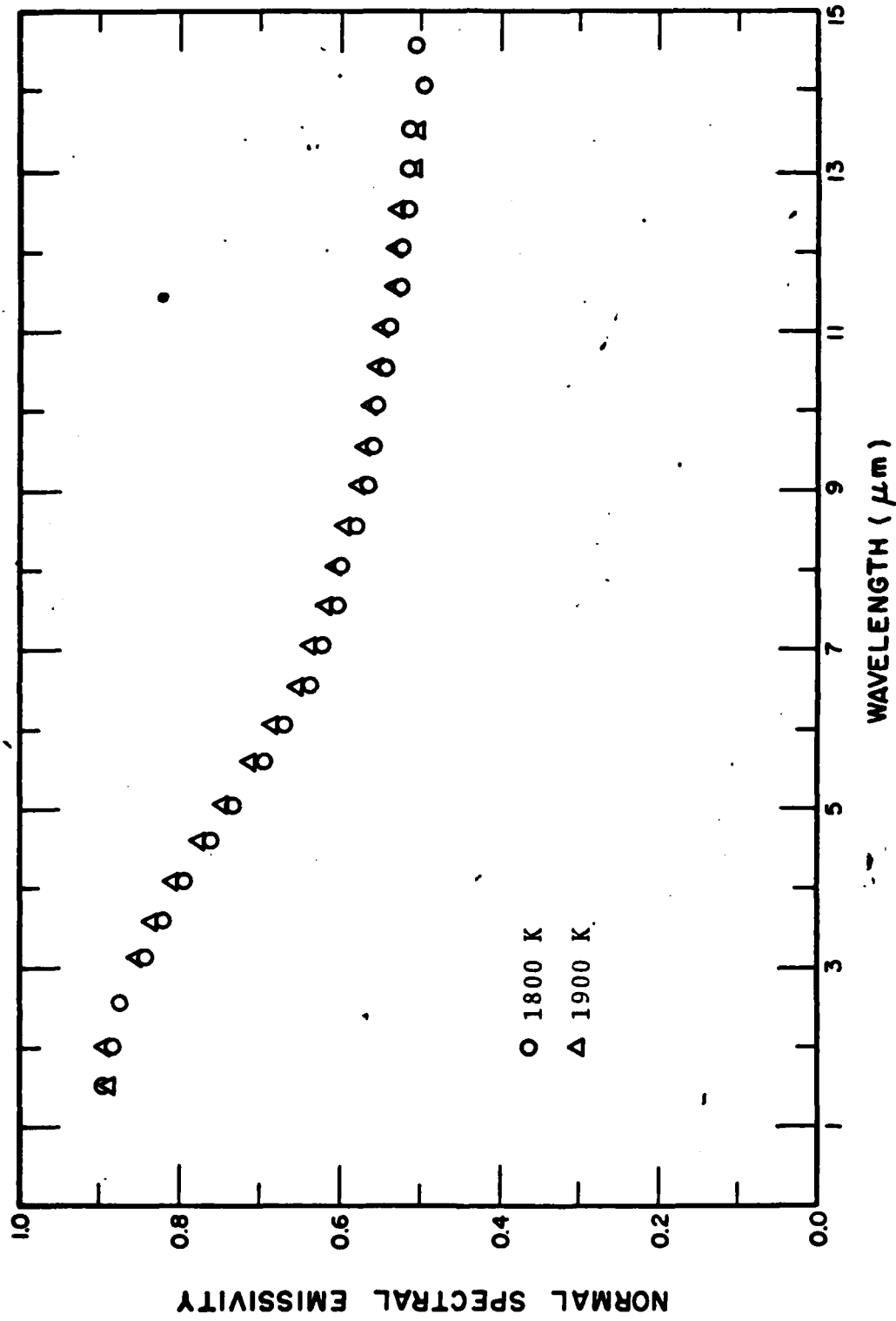


Figure 9. Spectral emissivity of G. E. silicon carbide, α-phase transformed from a β-phase sample

Table 4 Normal Spectral Emissivity of G. E. Silicon Carbide, α -Phase

<u>λ (μm)</u>	<u>Temperature (K)</u>	
	1800	1900
1.52	0.896	0.893
2.04	0.885	0.891
2.58	0.874	0.875
3.14	0.849	0.859
3.60	0.827	0.837
4.10	0.799	0.811
4.59	0.764	0.778
5.07	0.734	0.746
5.60	0.699	0.716
6.07	0.671	0.686
6.57	0.640	0.654
7.08	0.626	0.636
7.57	0.609	0.620
8.07	0.600	0.608
8.57	0.583	0.595
9.07	0.569	0.579
9.58	0.560	0.570
10.07	0.556	0.565
10.57	0.548	0.556
11.08	0.540	0.548
11.57	0.528	0.536
12.07	0.523	0.531
12.57	0.518	0.531
13.07	0.516	0.506
13.58	0.514	0.508
14.07	0.499	---
14.57	0.507	---

GRAPHITE COMPOSITE EMISSIVITY MEASUREMENTS

The feasibility of using the Multiproperty Apparatus for determining the spectral emissivity of a proprietary graphite composite material (U. S. Naval Surface Weapons Center) was demonstrated in June 1977 [6]. In a subsequent study completed in January 1978 [7] the objective was to extend the measurements to higher and lower temperatures and to extend the wavelength region well beyond 10.5 μm . One major modification to achieve these goals was the substitution of a trimetal detector for the thermocouple detector. Using the trimetal detector, excellent data was obtained from 1200 to 2000°C over the wavelength range from 2 to 14 μm .

The data showed that the spectral emissivity of this composite material to decrease slightly with increasing wavelength with typical values of 0.83 ± 0.02 at 4 micrometers and 0.80 ± 0.03 at 10 μm . The spectral emissivity was found to be essentially independent of temperature from 1750 to 2400°C. However, the sample failed during measurements at 2400°C and data at this temperature were obtained only from 2 to 4 micrometers. The sample configuration (1978-A) used in these tests is shown in Figure 10. The walls were kept thin in order to minimize the corrections for temperature drop across the walls. Ideally, the surface and blackbody should be at the same temperature, but since the surface radiates to the cooler surroundings while the blackbody does not, the surface temperature is less than the blackbody temperature. This temperature difference depends upon the thermal conductivity of the material, the thickness of the wall, the total hemispherical emittance, and the absolute temperature.

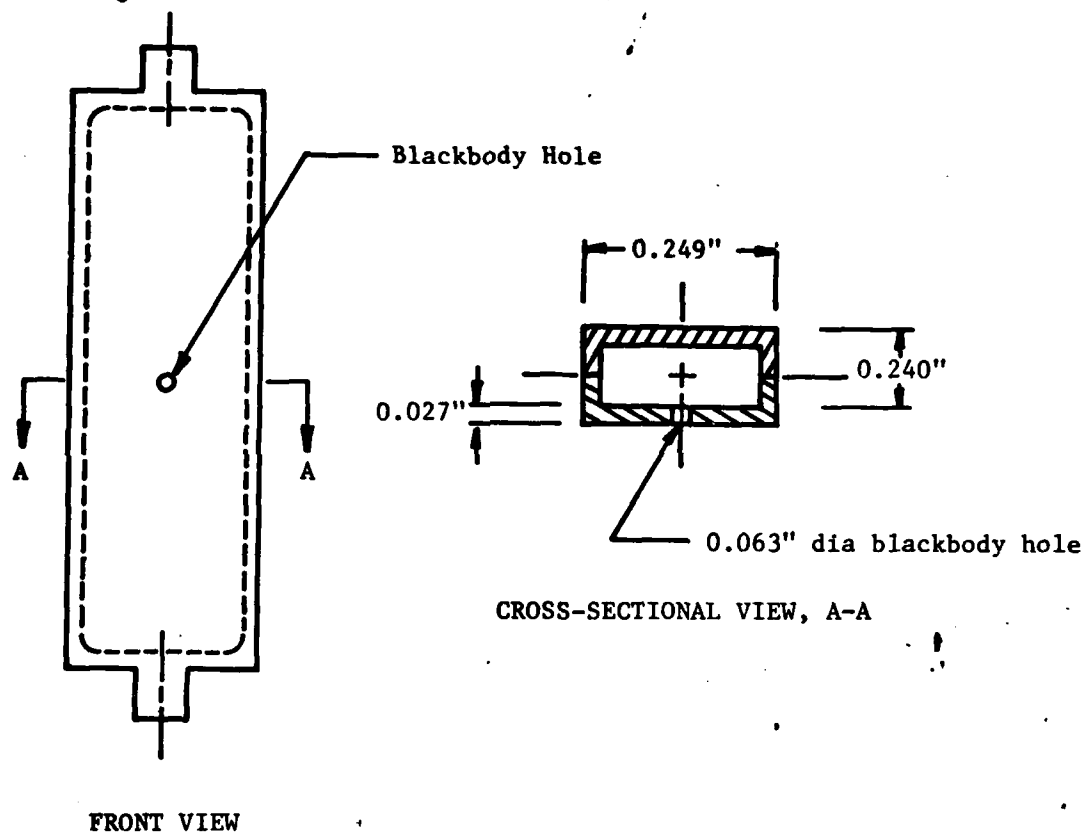


Figure 10. Configuration (1978-A) for the thin-walled graphite composite sample used in the preliminary study [7]

In addition, the correction to the spectral emissivity depends upon wavelength, being less at longer wavelengths. For the geometry of Figure 10, the correction term was 1.004 at 1473 K and 10 micrometers, increasing to 1.039 at 2673 K and 4 micrometers. However, the thin sides (0.027") of the sample split during heating at 2400°C.

The objective of the present work, a summary of which follows, was to determine whether the spectral emittance, particularly at longer wavelengths, changed rapidly with increasing temperature at higher temperatures and not necessarily to obtain high accuracy data at these temperatures. Full details of the work are given in Reference 8.

Apparatus and Procedures

The main features of the Multiproperty Apparatus are adequately described elsewhere. Hence, in this section, only those special modifications required for the sample configuration and mounting will be summarized.

The sample for the present work initially was a rectangular sample 0.375 in (9.53 mm), 0.290 in (7.34 mm) by 3.705 in (9.41 cm) long. Extender end pieces were rectangular shaped at the ends joined to the sample and round at the ends connected to our normal sample holders. The sample configuration (1978-B) is shown in Figure 11. An axial hole of 0.094 in diameter was drilled through the sample. A hole of 0.052 in diameter was drilled from the surface to join the axial hole to form a blackbody cavity of about 0.95 quality. Because of the large radiating surface, heat shields were used to decrease

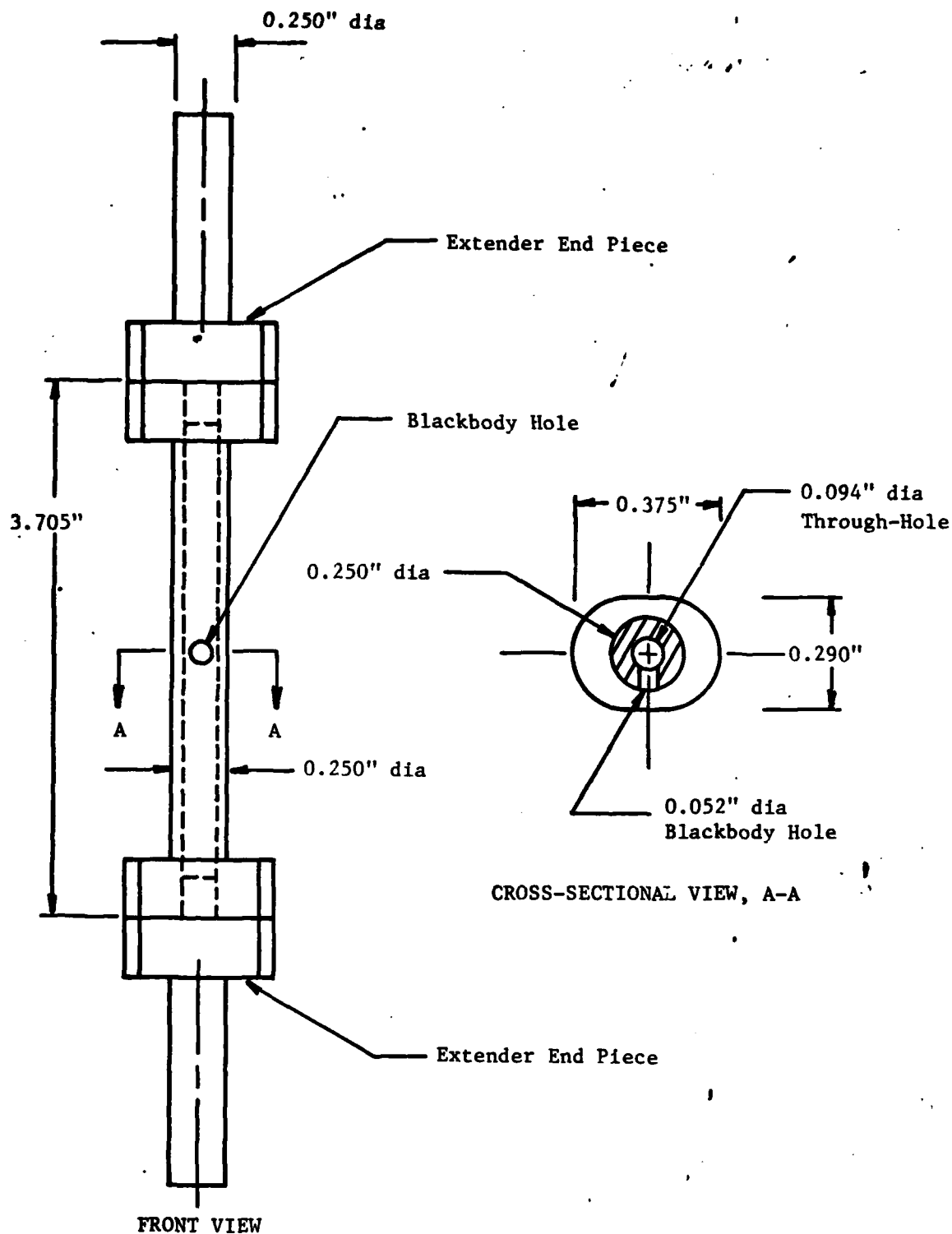


Figure 11. Initial graphite composite sample configuration (1978-B) used in the present study

the power consumption to levels compatible with our power supplies and feed-through capabilities. These shields were cut out in the vicinity of the blackbody hole to eliminate scattered radiation from entering the detector.

Four attempts were made using this sample geometry. In each case, the sample failed at the slip-joints between the sample and extenders. These joints could not be made as tightly as desired, resulting in a high resistance at the interface. Finally, the sample geometry (1978-C) shown in Figure 12 was used. The sample was circular 0.250 in diameter with a 0.094 in diameter center hole. Near the sample ends, the sample shape was left rectangular to provide a low resistance, higher strength contact. With this geometry, heat shields were not required to reach high temperatures. Measurements were made in vacuum up to 2800 K at which point vaporization became noticeable. Measurements were then made in 2/3 atmosphere of argon from 2800 to 3100 K where sample failure occurred at the blackbody hole.

Results

During the first three trials the sample-to-extender joint failed at about 2000 K. Nevertheless, some data at 1700 and 1800 K were obtained during these attempts. These data are given in Table 5 and plotted in Figure 13. In addition, data were obtained on the sample from 2000 to 2400 K where the sample-to-extender joints failed during the fourth trial. These data are given in Table 6 and are also plotted in Figure 13. Since the same sample geometry, Figure 11, was used during the first four trials, the results should be

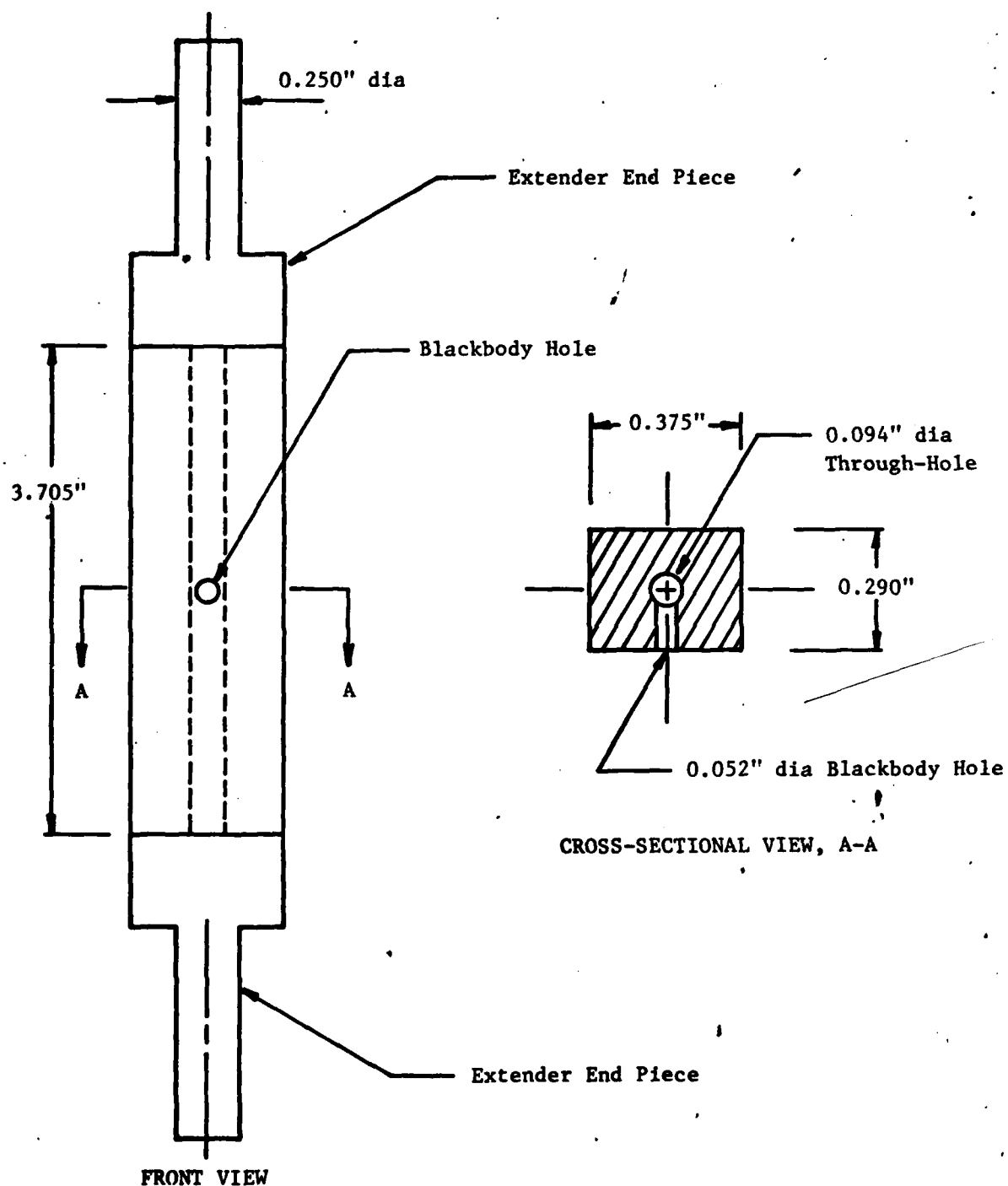


Figure 12. Final graphite composite sample configuration (1978-C) used in the present study

Table 5 Spectral Emissivity Results for the Graphite Composite (Trial 1)

Run Temp. No. (°K)	Wavelength (Micrometers)											
	2.6	3.6	4.6	5.6	6.6	7.6	8.6	9.5	10.4	11.3	12.2	13.0
1-1 1700	0.878	0.862	0.838	0.821	0.815	0.787	0.770	0.760	0.743	0.739	0.740	0.738
1-2 1800	0.880	0.898	0.860	-	0.846	0.821	0.803	0.813	0.799	-	-	-

Table 6 Spectral Emissivity Results for the Graphite Composite (Trial 4)

Run Temp. No. (°K)	Wavelength (Micrometers)											
	2.0	3.1	4.0	5.0	6.0	7.0	8.0	9.0	9.9	10.8	11.7	12.5
4-1 2000	0.887	-	0.853	0.824	-	0.811	0.769	0.752	0.751	0.738	-	0.752
4-2*2200	0.847	-	0.794	0.778	-	0.759	0.723	0.706	0.703	0.691	0.694	0.704
4-3 2400	0.854	0.839	0.807	-	0.783	0.736	0.708	-	-	-	-	-

Table 7 Spectral Emissivity Results for the Graphite Composite (Trial 5, Round Sample)

Run Temp. No. (°K)	Wavelength (Micrometers)											
	2.0	3.1	4.1	5.1	6.1	7.1	8.1	9.0	10.0	10.8	11.7	12.6
5-1 2000	0.794	0.788	0.780	0.771	0.767	0.742	0.743	0.725	0.729	0.721	0.712	-
5-2 2200	0.761	0.770	0.762	0.753	0.747	0.736	0.727	0.740	0.727	0.731	0.730	0.714
5-3 2400	0.743	0.752	0.748	0.742	0.741	0.742	0.734	0.727	0.726	0.715	0.727	0.715
5-4 2600	0.737	0.741	0.733	0.731	0.723	0.721	0.721	0.703	-	0.714	0.709	0.692
5-5*2700	0.739	0.750	0.749	0.743	0.731	0.727	0.727	-	0.701	0.715	0.720	-
6-1 2800	0.770	0.800	0.808	0.812	-	0.824	-	0.802	0.819	-	-	-
6-2 2900	0.775	0.810	0.810	0.808	0.814	0.812	0.814	0.812	0.814	0.813	0.815	0.813
6-3 3000	0.796	0.826	0.824	0.815	0.817	0.819	0.816	0.813	0.813	0.812	0.811	0.814
6-4*3100	0.798	0.825	0.812	0.802	0.802	0.796	0.793	0.790	0.791	0.787	-	-

*Not shown in figure.

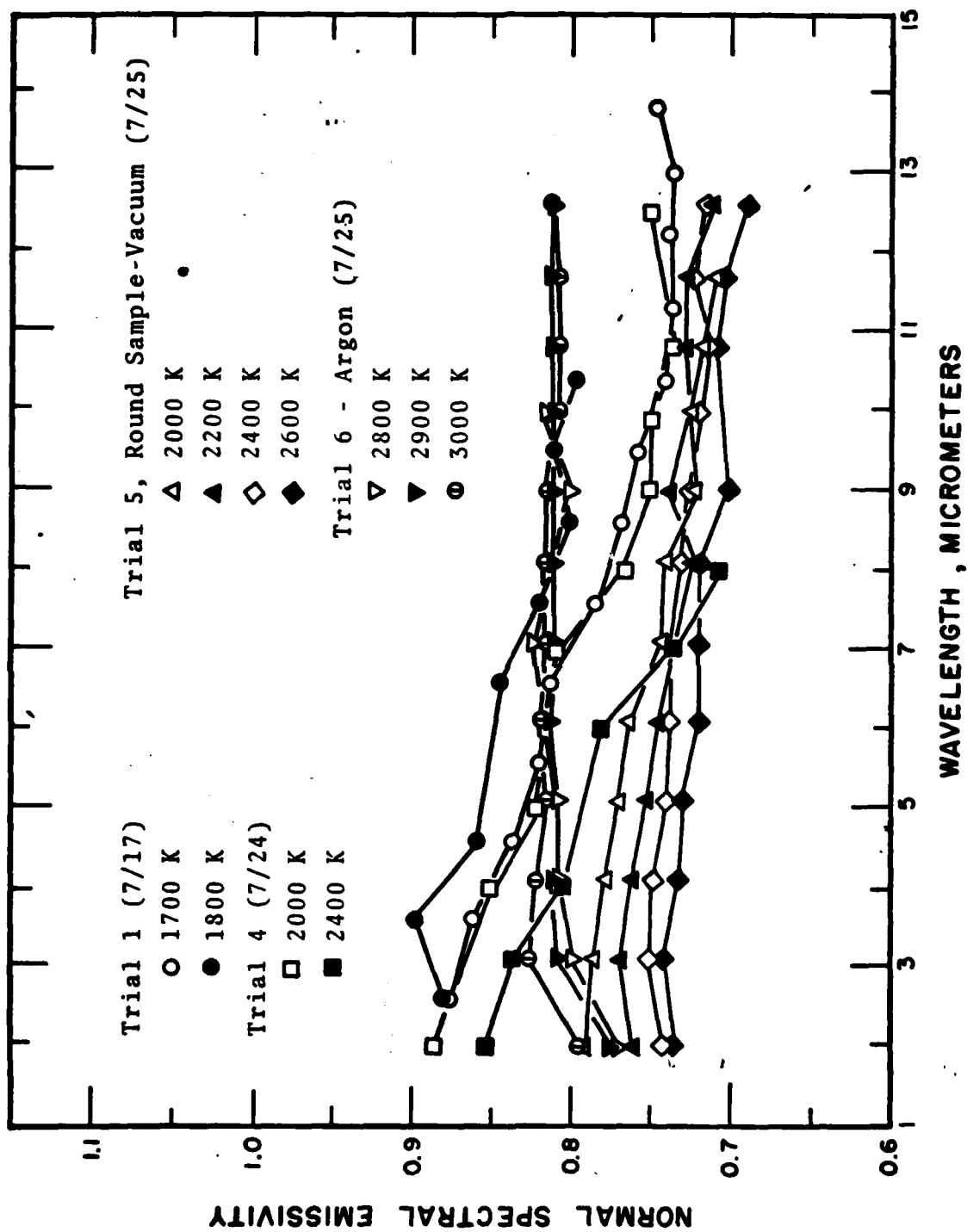


Figure 13. Spectral emissivity of the proprietary (U. S. Naval Surface Weapons Center) graphite composite sample (1978-C)

considered together. The spectral emissivity from 1700 to 2400 K is $0.84 \pm .02$ at $2 \mu\text{m}$, decreasing to $0.77 \pm .04$ at $13 \mu\text{m}$, with the emissivity values decreasing as the temperature is raised. These values were measured under vacuum. The temperature across the specimen walls will cause the surface radiance to be lower than it should be. This causes the measured emittance to be low. This error increases with increasing temperature.

Runs 5-1 through 5-5 were made on the round sample in vacuum. These values are generally lower than the present data on the square sample, but exhibit less wavelength dependency. The emittance values decrease from $0.75 \pm .02$ at $2 \mu\text{m}$ to $0.72 \pm .02$ at $12.5 \mu\text{m}$. These data cover the temperature range 2000 through 2600 K.

Above 2600 K the sample began to evolve carbon vapor in vacuum, so it was necessary to cool the sample to room temperature and then pressurize with argon before heating to higher temperatures. Data were obtained from 2800 to 3100 K under argon. The sample failed at the blackbody hole during the 3100 K measurements, but the data had already been recorded from 2 to $12 \mu\text{m}$ before sample failure. This failure was caused by the weakening of the structure caused by drilling the radial hole from the surface to meet the sample ID. The emissivity results were $0.81 \pm .01$ at $3 \mu\text{m}$ and were independent of wavelength and temperature from 3 to $13 \mu\text{m}$ and 2800 to 3100 K.

Discussion

In order to correct the emissivity data for the temperature drop across the sample wall, it would be necessary to know the thermal conductivity and electrical resistivity as a function of temperature.

Because two different sample geometries were used and data were taken at various temperatures and in argon and vacuum, the corrections are different for each run. The corrections are also a function of wavelength, being less as the wavelength increases. The corrections are probably less than 2% at 1700 K at 12 μm increasing to perhaps 15% at 3100 K at 2 μm . Thus, the present data does not have the high accuracy of that reported earlier. However, the objective of determining whether there is any anomalous behavior of the spectral emittance from 2 to 13 μm between 1700 and 3100 K was met. It is obvious that no anomalies occur over this region.

FEASIBILITY STUDY ON METHOD FOR SEMITRANSSPARENT MATERIALS

The most commonly used methods for measuring the high temperature optical properties of semitransparent materials are variations of the McMahon furnace [9, 10] or the high-speed method such as recently reported by Russian workers [11]. The McMahon method is cumbersome, requiring a large sample and long time, to obtain measurements; however, the error analysis for estimating accuracy can be readily performed. The high-speed method permits rapid measurement cycles, however, the apparatus and data acquisition features are rather complicated. Hence there is the need for a new method which can overcome the disadvantages of the existing technology yet become a useful tool for high temperature materials research. The objective of the present study is to determine the feasibility of performing optical property measurements on a sample configuration compatible with the Multiproperty Apparatus.

The first sample configuration considered in this feasibility

study is shown schematically in Fig. 14. The optical system views the semitransparent sample through a view hole in the heating tube; this is the same arrangement used with the non-conducting samples as illustrated in Fig. 3. However, a second hole on the backside of the tube is provided and hence the optical system can view the separate blackbody position a convenient distance from the sample. The temperature of this blackbody, T_3 , can be controlled and changed as required, but must be carefully measured. The lower lateral hole in the heating tube serves as a blackbody cavity at temperature, T . It is further assumed that the heating tube in the vicinity of the lateral holes and the semitransparent sample are isothermal at temperature T .

As the Fig. 14 suggests the ratio of the signal outputs from the optical system when the sample, S , and blackbody, H , targets are viewed will be

$$\frac{S}{H} = \frac{L_\lambda}{L_{\lambda,b}(\lambda, T)} = \frac{L_{\lambda,e} + L_{\lambda,t}}{L_{\lambda,b}(\lambda, T)}$$

where the sample radiance, L_λ , is the sum of the sample self-emission component, $L_{\lambda,e}$, and the transmission by the sample of the blackbody (T_3) spectral radiance, $L_{\lambda,t}$. The blackbody spectral radiance at temperature T is expressed as $L_{\lambda,b}(\lambda, T)$.

The simplest situation to consider is when the separate blackbody temperature is very low in which case $L_{\lambda,t} = 0$ in the above equation and the ratio becomes

$$\frac{S}{H} = \frac{L_{\lambda,e}}{L_{\lambda,b}(\lambda, T)} = \frac{\epsilon_\lambda L_{\lambda,b}(\lambda, T)}{L_{\lambda,b}(\lambda, T)} = \epsilon_\lambda$$

We need to recognize that the spectral emissivity measured in this

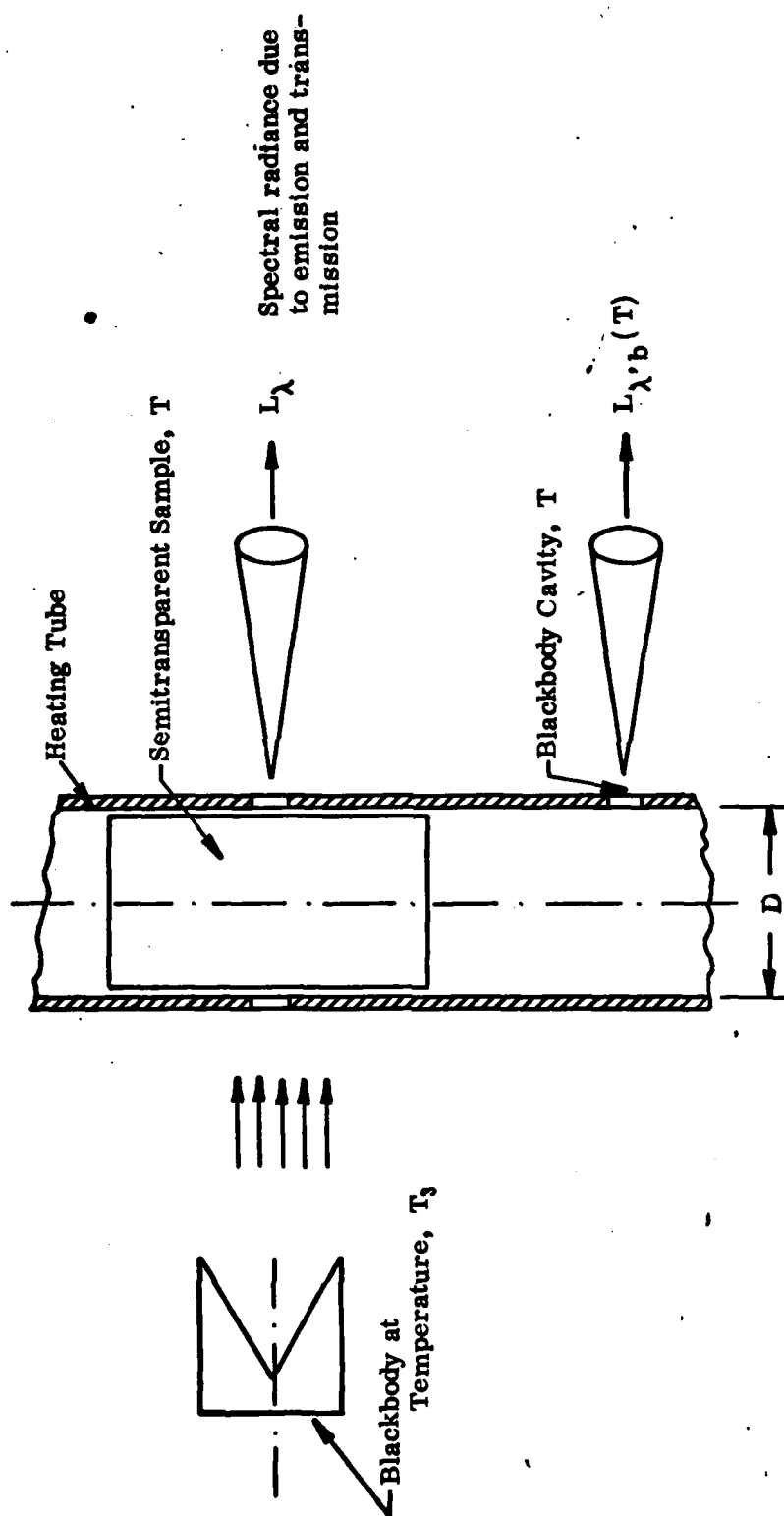


Figure 14. Proposed method for measuring the high temperature optical properties of semitransparent materials in the Multiproperty Apparatus.

instance will depend upon the thickness of the sample. In this sense, the measurement doesn't give fundamental information since there is no way to determine the emissivity for another sample thickness. However, even this information would be sufficient for many purposes particularly those relating to materials development where no data on high temperature emissivity presently exists.

The sample configuration shown in Fig. 14 has the potential for providing the optical properties (n , k) and the spectral transmittance, τ_λ , as well as the spectral emissivity. An analysis of the configuration based upon the radiative transfer model of Viskanta, et al. [12] has been performed yielding relations which could be written in this functional form;

$$n, k = f\{\text{geometry}, T_3, L_\lambda/L_{\lambda,b}\}$$

The relationships are complicated and for the sake of illustrating the method, they have been summarized in Table 8. It should be obvious that while the measurement procedure is relatively straightforward, determining the n , k values will be somewhat involved. Based upon this radiative transfer model, it now remains to be determined what the sensitivity of the various properties will be to the observations of the ratio, S/H . To perform this parametric sensitivity analysis, we are presently looking at the materials, fused quartz and sapphire, whose optical properties at high temperature are known with some precision. We have relied on the staff at CINDAS to provide this input and they are giving valuable assistance in this phase of the feasibility study.

Table 8 Relations for the Proposed Method to Obtain Optical Properties of the Semitransparent Sample

$$L_{\lambda} = [1 - \rho](1/n_{\lambda})^2 \frac{1}{[1 - \rho_{\lambda}^2 \cdot \exp(-\kappa_{\lambda} D)]} \{n_{\lambda}^2 L_{\lambda, b}(T_3) \exp(-\kappa_{\lambda} D)\}$$

$$+ \int_0^D n_{\lambda}^2 L_{\lambda, b}(\lambda, T) [\exp(-\kappa_{\lambda} Y) + \rho \exp(-\kappa_{\lambda} D) \cdot \exp(-\kappa_{\lambda} [D - Y])] \kappa_{\lambda} dy$$

where

n, κ optical properties

ρ reflectivity

$$\rho = \frac{(n-1)^2 + (n\kappa)^2}{(n+1)^2 + (n\kappa)^2}$$

It is our expectation to complete the analysis of the proposed method by the end of the present contract year. Our expectations are that there will be a positive outcome and further utility of the method could be demonstrated by developing the necessary hardware to make measurements.

PUBLICATIONS

The following reports and publications have been prepared on the emissometer studies during sponsorship by AFOSR.

- (1) PRL Report 116, "Spectral and Total Emissivity and Reflectivity at High Temperature," April 30, 1976, D. P. DeWitt, B. Hoenle, and R. E. Taylor.
- (2) PRL Report 126, "Description of the PRL High Temperature Emissometer," November 1976, D. P. DeWitt, R. E. Taylor, and B. D. Hoenle.
- (3) PRL Report 130, "Spectral and Total Emissivity and Reflectivity at High Temperatures," March 31, 1977, R. E. Taylor and D. P. DeWitt.
- (4) PRL Report 136, "Spectral and Total Emissivity and Reflectivity at High Temperatures," Progress Report of AFOSR, September 15, 1977, D. P. DeWitt, T. K. Riddle and R. E. Taylor.
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SUMMARY

The progress of our activities on the major objectives have been described. The study on the new ceramic materials promise to provide a better understanding of high temperature behavior. Such information will be very useful to the manufacturers and users of these materials; additionally, such data will be welcomed by those studying the theory of solids.

The Multiproperty Apparatus has proved to be a useful tool for studying exotic materials like the graphite composite. The flexibility of the apparatus permits changes in sample configuration and other features allowing operation to 3100 K. This capability helped to solve a very critical problem for the Naval Surface Weapons Center and is the result of sustained support by AFOSR for the past several years.

The extension of the Multiproperty Apparatus capability to the measurement of semitransparent materials should be a significant contribution to high temperature technology. Such capability will provide much needed support to several important problem areas such as ceramic coatings on refractory materials, insulation coatings, laser applications (windows, structural components), and electronic crystals. The thrust of our proposal for continuation of the research will be to use the completed feasibility study to evaluate the method after making necessary modifications to the apparatus.

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