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THE DETERMINATION OF THERMAL DIFFUSIVITIES OF THERMAL ENERGY STORAGE MATERIALS PART-1, SOLIDS UP TO MELTING POINT

By

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ABSTRACT

This paper describes a method for the determination of thermal diffusivities which has been developed specifically for substances which are poor conductors, and which have high melting points. Materials which are useful for thermal energy storage fall into this category.

The method has several unique features. The basic principle involved consists of raising the surface temperature of a solid specimen at a uniform rate. After the initial transients have died out, the diffusivity can be determined from temperature measurements alone.

The advantages of the method are:

1. Heat flux measurements are not needed.
2. Materials can be tested right up to the melting point, since the specimens can be encapsulated and softening can be tolerated.
3. Large temperature ranges can be tested quickly.
4. Precision and accuracy are good.

The method has been extended to the liquid range, and results will be published as PART-II.

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Results of measurements are reported for alumina and lithium fluoride. The results for alumina (Lucalox) check results reported previously. The results on LiF differ from published results.

Data on other substances are still being produced, and results will be published at a later date.

INTRODUCTION

Space vehicles may be equipped with power generators such as thermoelectric and thermionic direct energy conversion devices. For these devices solar energy may be harnessed and stored in materials having high heat of fusion. In designing these thermal energy storage systems, accurate thermal diffusivity data are needed. Because the materials suitable for energy storage are usually moderately poor thermal conductors and some have relatively high melting points[1], the present technique was developed to measure the thermal diffusivity of these materials at high temperatures. The method, although geared toward studying thermal energy storage materials, can also be used to study other moderately poor thermal conducting materials.

Thermal diffusivity is defined as the thermal conductivity divided by the product of density and specific heat; therefore by obtaining the thermal diffusivity experimentally, thermal conductivity can be calculated from the specific heat and density data available in the literature.

In measuring the thermal diffusivity, unlike measuring thermal conductivity, it is not necessary to measure the rate of heat flux which is extremely difficult to obtain accurately. The reason for this difficulty is due to the fact that the heat flux entering a test sample is the difference between the total heat supplied from the external source and the heat loss incurred in the apparatus. Despite the many ingenious methods which were invented to minimize this loss, there still exists a certain amount of heat loss in the apparatus which is very difficult to account for accurately. In the light of the above discussion, it was decided to measure the thermal diffusivity using a transient method in which only temperature differences have to be measured. An additional advantage of this method is that the measurements can be obtained in a short time; this reduces the effects of vaporization of test samples at high temperatures.

RELEVANT PREVIOUS WORK

Space limitations preclude a detailed description of the many methods which have been used in the past to obtain thermal diffusivities. It is appropriate, however, to list the important contributions, and to justify the choice of the method which was adopted for the present work.

Angström[2] developed a technique whereby one end of a long rod was subjected to periodic temperature changes. After the initial starting transient has died out, the temperature distribution along the entire bar becomes periodic, and the diffusivity can be computed from the mean temperature at different locations along the bar.

Angström's method was further developed by Billington[3], and Sidler and Danielson[4].

The basic drawback of this method is the difficulty of accounting for radiation losses at high temperatures, and for this reason it was not considered for the present work.

Fitzsimmons[5] developed a method of measuring diffusivities by quenching or heating long solid cylinders by sudden immersion in a well stirred fluid. The diffusivity may be computed from the temperature history of the center of the rod. The difficulty of insuring proper stirring and adjusting initial conditions at high temperatures made this method unattractive for our work.

Levine[6] heated specimens by radiation and convection from a constant temperature source. In this method, assumption is made that the heat flux entering the specimen is directly proportional to the temperature difference between source and specimen surfaces. This limits the method to low heating rates, and small temperature differences. Crandall[7] and Potter applied this technique to temperature ranges up to 2,000°C., but found great difficulty in meeting the specified boundary conditions.

A novel technique was developed by Parker, Jenkins, Butler and Abbott[8]. Here, a high intensity short-duration light pulse is absorbed by the front face of a thermally insulated specimen which is thin, and is coated with camphor black. The temperature history of the black face allows the computation of the thermal diffusivity. This method was also adopted by Rudkin, Jenkins and Parker[9] who found it very difficult to account for radiation losses at elevated temperature, and to insure uni-directional heat flow.

The light pulse technique was further improved by Deen and Wood[10] who used a laser as the light source. Denman[11] obtained data up to 1,100°C., but reported great difficulties due to "noise" at elevated temperatures which make it difficult to read the oscilloscope trace. His data scattered badly. Rudkin[12] extended the pulse technique to ceramics at high temperatures, and found several major drawbacks. The samples were partially translucent, and radiation losses were severe. For these reasons, pulse techniques were rejected for the present work.

A technique which appeared most promising to the authors, and which was finally adopted for this work was suggested by a brief note in Ref. 13. After the inception of the present work it was discovered that the idea underlying the present technique had already been applied by Flieger[14], but that the apparatus was limited to temperature ranges of below 1000°C. This technique will now be discussed.

DESCRIPTION OF THE METHOD

A long cylindrical specimen is heated by a concentric heat source in such a manner that the temperature of a region close to the surface increases at a uniform (constant) rate. The temperature response is measured at the center of the specimen, and also at some intermediate radius. These measurements are made roughly at the longitudinal center of the specimen to insure that only radial heat flow occurs. This radial heat flow is insured by providing a sufficiently long uniform temperature zone, and by taking advantage of the axial symmetry which exists.

The equation[15] (in cylindrical coordinates) governing the radial heat conduction in an infinite solid cylinder having radius r_1, and thermal diffusivity a, is

$$\frac{\partial \theta}{\partial t} = a\left(\frac{\partial^2 \theta}{\partial r^2} + \frac{1}{r}\frac{\partial \theta}{\partial r}\right) \quad \text{For the region} \quad 0 < r < r_1 \qquad (1)$$

where $\theta\ (r, t)$ = Temperature rise fron an initial uniform temperature (°C) i.e., $T - T_i$

$T\ (r, t)$ = Temperature (°C) at any radius r and time t.

T_i = Initial temperature (°C) at any radius r at $t = 0$.

r = Radius (cm.)

t = Time (sec.)

r_1 = Radius of isothermal surface where temperature increases at a constant rate.

a = Diffusivity (cm^2/sec)

The solution to equation 1 is:

$$\frac{\theta}{c} = \left(t - \frac{r_1^2 - r^2}{4\alpha}\right) + \frac{2}{r_1 \alpha} \sum_{n=1}^{\infty} e^{-\alpha Z_n^2 t} \frac{J_o(rZ_n)}{\cdot\, Z_n^3 J_1(r_1 Z_n)} \qquad (2)$$

where c = the constant rate of temperature increase ($^{\circ}$C/sec) at r_1

J_1 = Bessel function of order one of the first kind

J_o = Bessel function of order zero of the first kind

Z_n is given by $J_o(r Z_n) = 0$ for $n = 1, 2, 3 \ldots$

The boundary and the initial conditions for the above solution are

$$\begin{aligned} &\theta\,(r_1\ t) = ct \\ &\theta\,(0,\ t) \text{ is finite} \\ \text{and } &\theta\,(r,\ 0) = 0 \end{aligned} \qquad (3)$$

For a homogeneous and isotropic material the thermal diffusivity is uniform throughout the material and may also be assumed to be independent of temperature for small values of the temperature.

As t increases, the third term in equation 2 contributes less and less to the total solution, $\theta\,(r,\ t)$ in equation 2. The third term of equation 2 is the transient part of the solution. After a short interval of time the first two terms of equation 2 predominate.

Equation 2 with the third term deleted is:

$$\frac{\theta}{c} = t - \left(\frac{r_1^2 - r^2}{4\alpha}\right) \text{ for any } t \geq \text{ initial transient} \qquad (4)$$

Equation 4 indicates that, as the temperature of the cylindrical surface with radius r_1 rises linearly with increasing time, the temperature of any other cylindrical surface of radius r_2 also rises linearly with increasing time. The temperature versus time curve for the two cylindrical surfaces under consideration eventually become parallel to each other. (See Fig. 1)

Equation 4 provides two methods for calculating the diffusivity after the temperature rise from T_1 for both cylindrical surfaces, having radii r_1 and r_2 respectively has been measured as a function of time.

Method 1. The thermal diffusivity, α , may be obtained by equating the temperature rise on the cylindrical surface ot radius r_1 at time t_1, $\theta(r_1, t_1)$, to the temperature rise on the cylindrical surface with radius r_2, at the time t_2 , $\theta(r_2, t_2)$. t_2 is chosen arbitrarily, t_1 is the time elapsed from $t = 0$ when $\theta(r_1, t)$ reaches the value of $\theta(r_2, t_2)$ at time t_2. The thermal diffusivity is found to be inversely proportional to the time increment $(t_2 - t_1)$. (See Figure 1). Equation 4 yields:

$$\theta(r_1, t_1) = ct_1 ; \; \theta(r_2, t_2) = c \left[t_2 - \frac{(r_1^2 - r_2^2)}{} \right]$$

$$\theta(r_1, t_1) = \theta(r_2, t_2)$$

$$\alpha = \frac{r_1^2 - r_2^2}{4} \; \frac{1}{(t_2 - t_1)} \qquad (5)$$

If we choose $r_2 = 0$, the axis of the specimen, Equation 5 reduces to

$$\alpha = \frac{r_1^2}{4} \; \frac{1}{(t_2 - t_1)} \qquad (6)$$

Method 2. The thermal diffusivity, α, may also be obtained from the difference of the temperatures between the cylindrical surfaces of radii r_1 and r_2 respectively, since this difference remains constant for any arbitrary t_2. The thermal diffusivity is found to be inversely proportional to the difference in the temperature rise between the two cylindrical surfaces under consideration. (See Figure 1).

Equation 4 yields at t_2:

$$\theta(r_1, t_2) = ct_2 \ ; \ \theta(r_2, t_2) = c\left[t_2 - \frac{(r_1^2 - r_2^2}{4\alpha}\right]$$

$$\frac{\theta(r_1, t_2) - \theta(r_2, t_2)}{c} = \frac{r_1^2 - r_2^2}{4\alpha}$$

$$\alpha = \frac{(r_1^2 - r_2^2)}{4} \quad \frac{c}{\theta(r_1, t_2) - \theta(r_2, t_2)} \tag{7}$$

If we choose $r_2 - 0$, the axis of the specimen, equation 5 reduces to:

$$\alpha = \frac{(r_1^2)}{4} \quad \frac{c}{\theta(r_1, t_2) - \theta(0, t_2)} \tag{8}$$

It may be seen that method 1 is independent of c, the constant rate of temperature increase on the isothermal cylindrical surface of radius r_1, whereas Method 2 indicates that for α constant, c is directly proportional to $\theta(r_1, t_2) - \theta(r_2, t_2)$.

The thermal diffusivities calculated by both methods are intended to be taken as the diffusivities at the mean of the two temperatures of the two cylindrical surfaces under consideration attained at t_2. It may be pointed out that the diffusivities calculated by either method are identical.

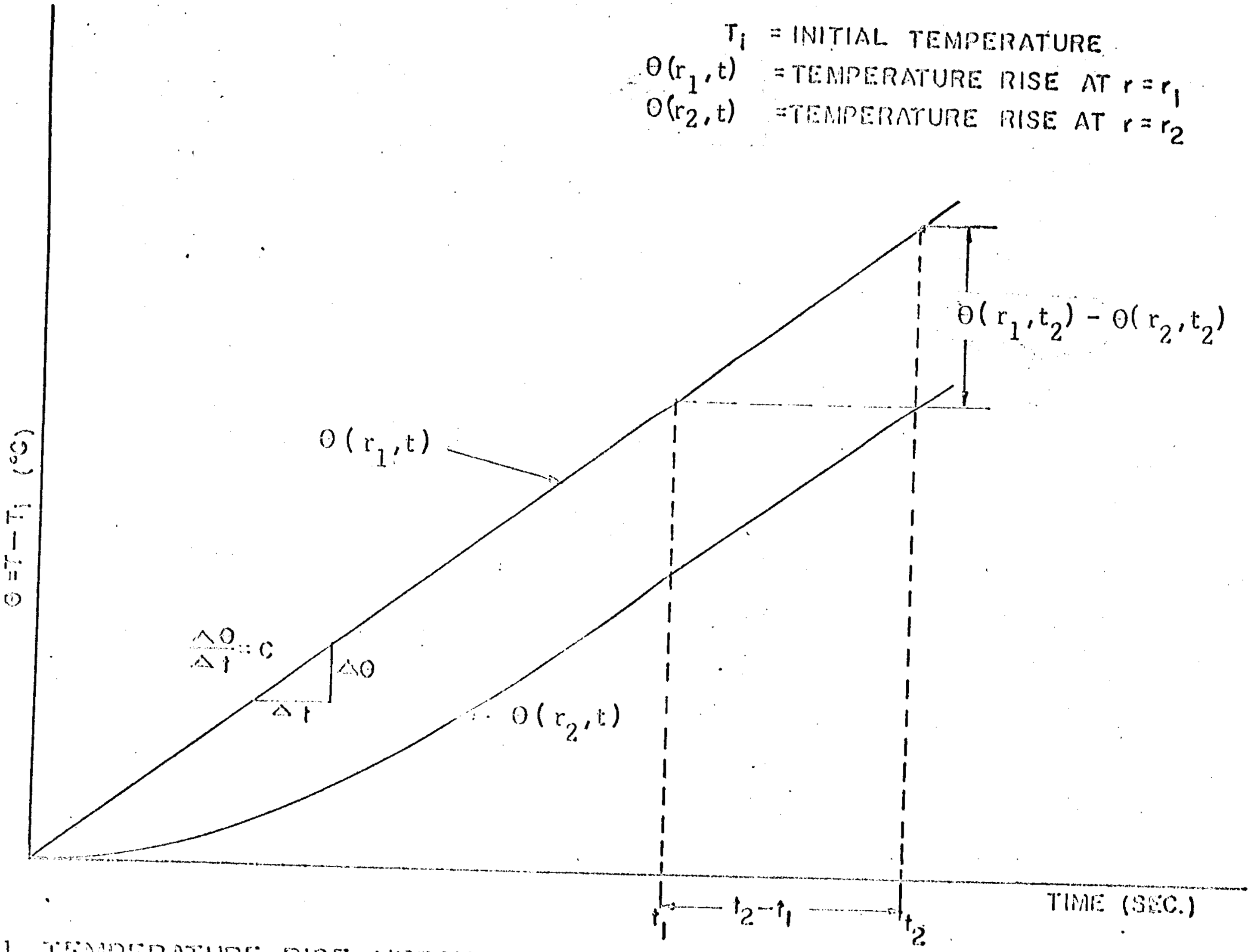


FIGURE 1. TEMPERATURE RISE VERSUS TIME IN CYLINDRICAL SAMPLE

APPARATUS

Two test furnaces were designed and fabricated. The first one was built primarily to gain experience, and to make it possible to have an independent check on the results of the final furnace. In order to save time and expense, the furnace was designed for a useful temperature range of below $1750^{\circ}C$.

The second furnace was much more elaborate, and is capable of operating at temperatures up to $2300^{\circ}C$.

The initial apparatus consists of a platinum - 40% rhodium wire-wound tubular furnace with an I.D. of 2 inches and 29 inches long.

In order to obtain a more uniform temperature in the central portion of the furnace, the windings were spaced more closely at the ends than in the middle portion of the tube. Another tube, 39 inches long, and with an I.D. of 1.5 inches was placed inside the furnace. The specimen was positioned within this inner tube by an adjustable supporting stand.

Figures 2 and 3 show schematically the details of the power supply, the heater controls, recording instruments and position of the sample inside the furnace.

Temperature measurements were made with Pt/Pt-10% Rh thermocouples, with a diameter of 0.001 in.

The details of the final apparatus is shown schematically in Figures 4 and 5. High temperature capability was obtained by the use of a tungsten mesh heater. This heater has an I.D. of 4 inches, and is 20 inches high. It is made of three separate elements which are connected on the lower end by a tungsten ring. This arrangement allows the heating element freedom for expansion due to temperature variation. The upper ends of the heating elements are connected individually to a tungsten connector. Electrodes made of Ta are attached rigidly at the center and extend radially outward.

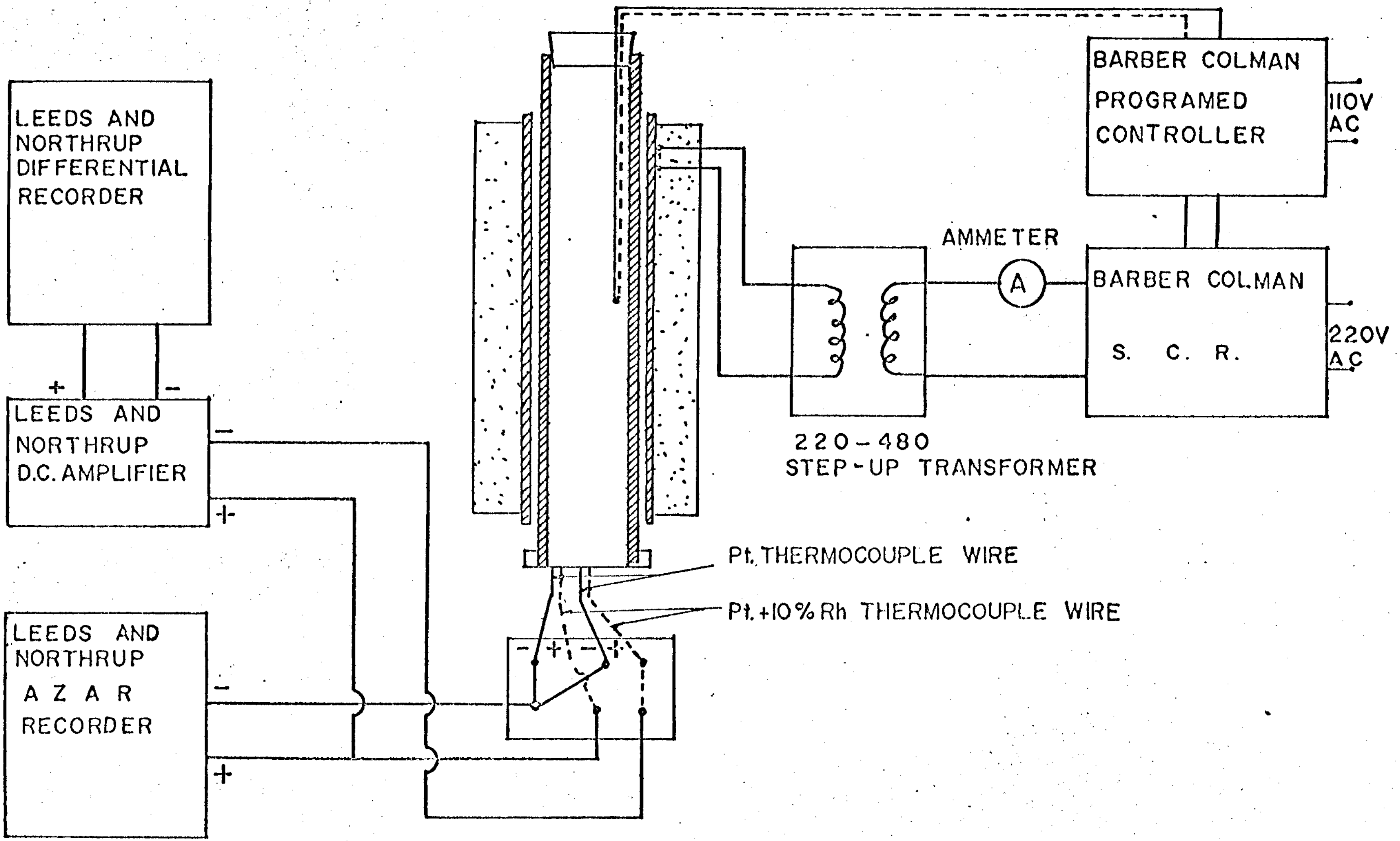


FIGURE 2: SCHEMATIC DIAGRAM OF THE APPARATUS FOR MEASURING THERMAL DIFFUSIVITY OF SOLIDS UP TO 1750°C

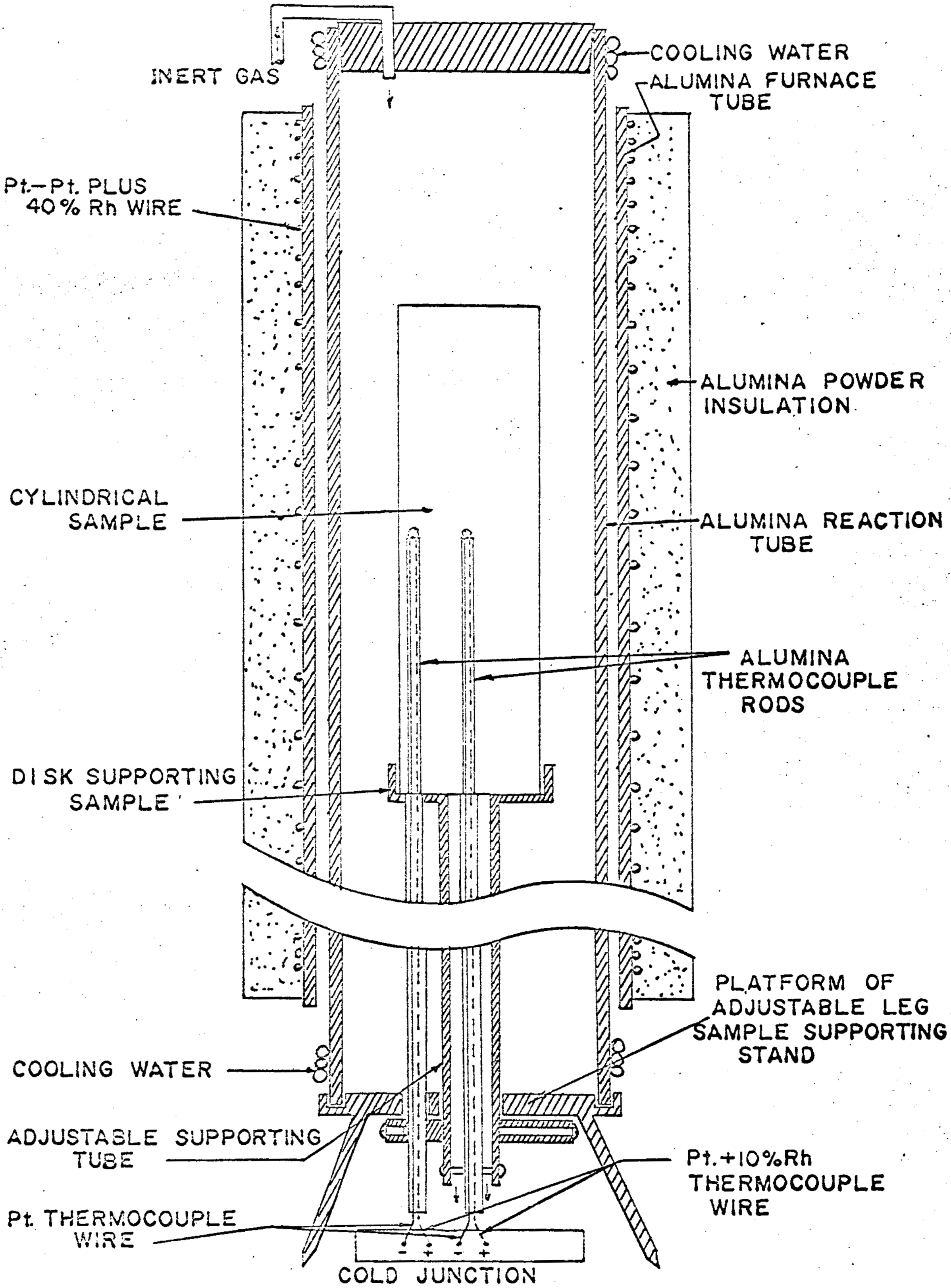


FIGURE 3 DETAILS OF APPARATUS FOR MEASURING THERMAL DIFFUSIVITY OF SOLIDS UP TO 1750°C

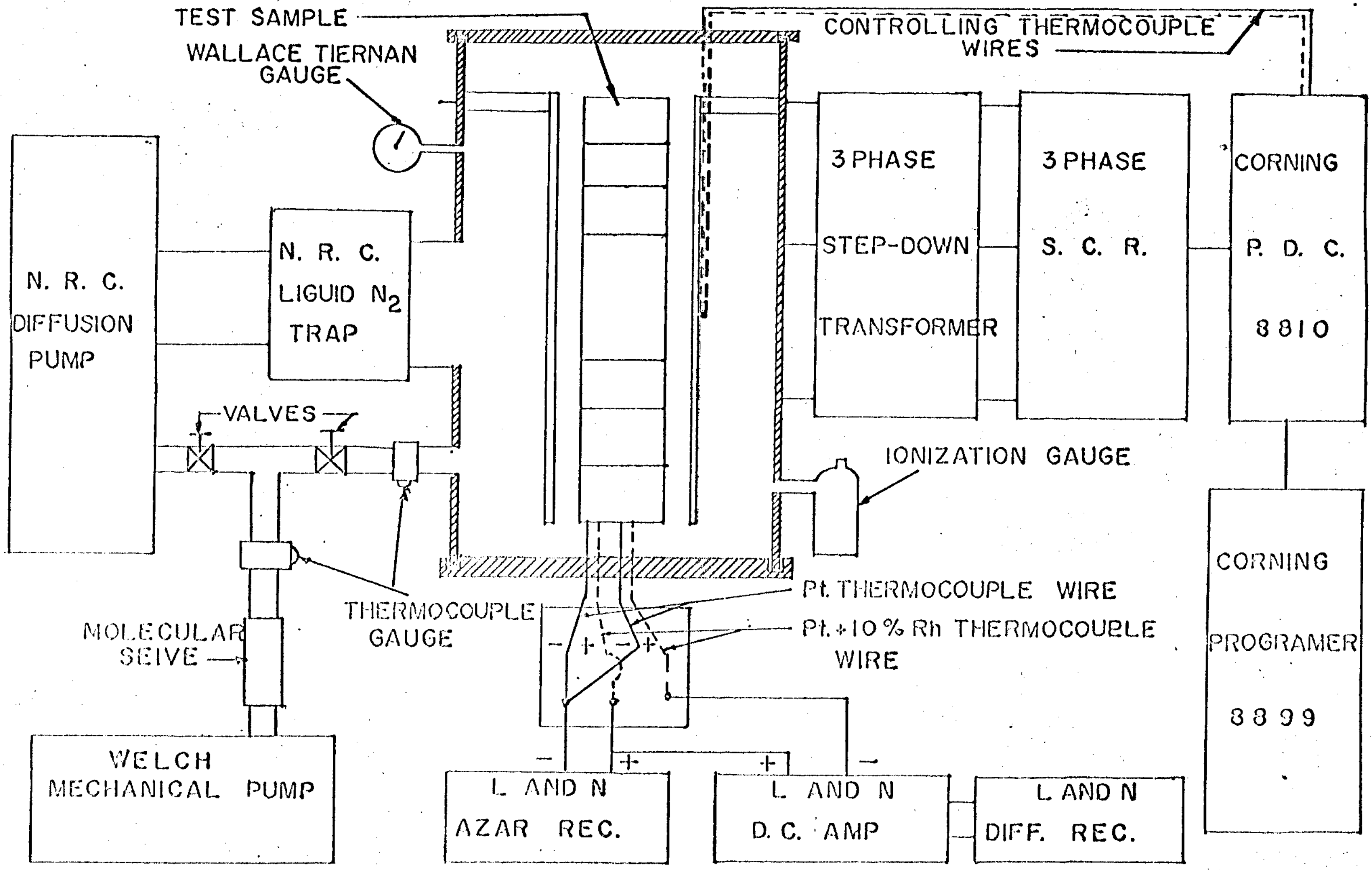


FIGURE 4 SCHEMATIC DIAGRAM OF THE APPARATUS MEASURING THERMAL DIFFUSIVITY OF SOLIDS UP TO 2300°C

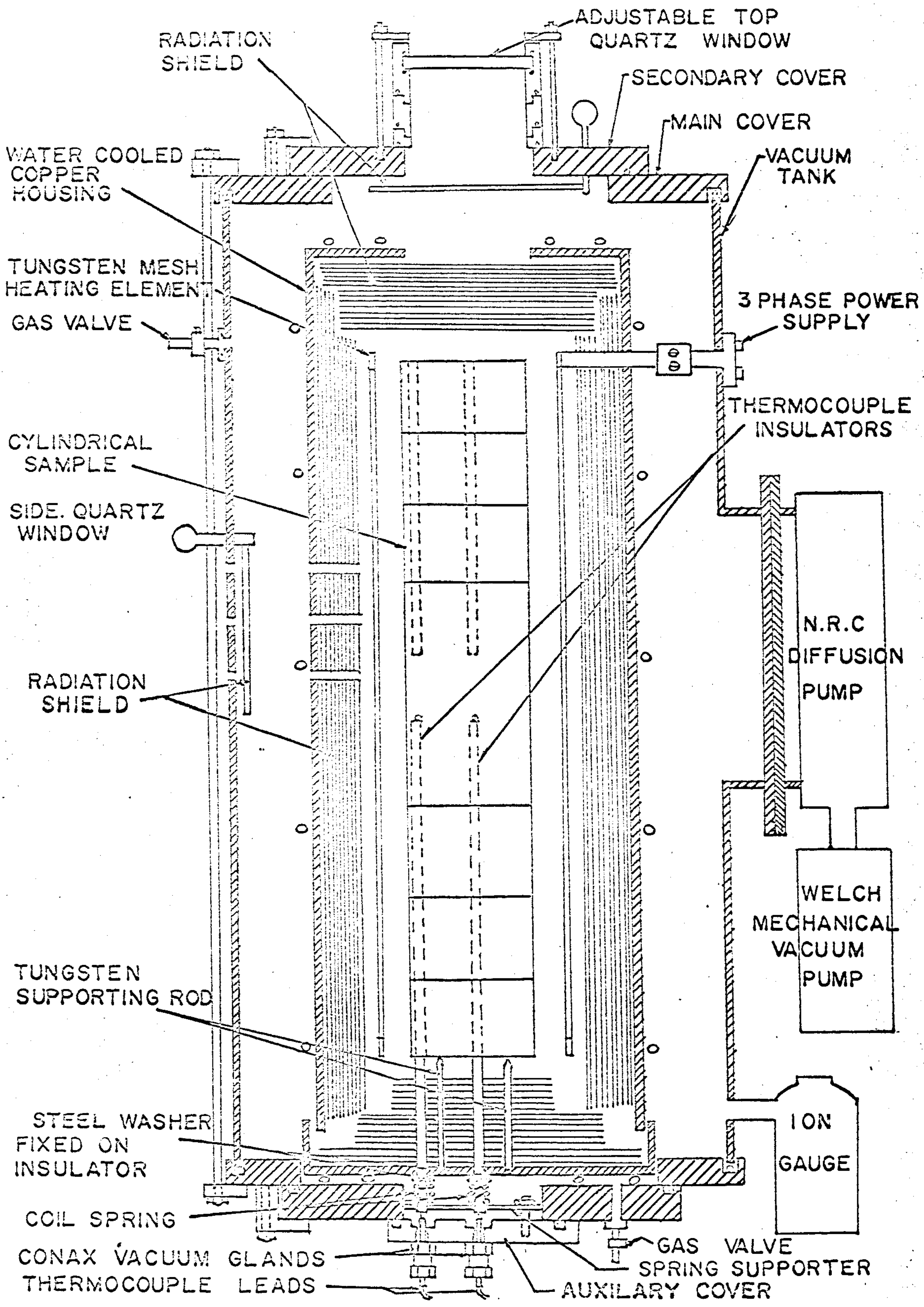


FIGURE 5 DETAILS OF APPARATUS FOR MEASURING THERMAL DIFFUSIVITY OF SOLIDS UP TO 2300°C

The heating element is enclosed by 10 side and 15 top and bottom radiation shields. The heating element with its radiation shields is housed in a copper water-cooled shell, through which the current-carrying leads protrude. The entire assembly is placed in an electro-polished double-wall stainless steel vacuum tank, 17" in dia. and 29" high. The ends of the current-carrying leads are clamped to the water-cooled copper electrodes. Cold or hot water may be circulated through the tank. The copper water-cooled electrodes are connected directly to the leads. Sight ports fitted with General Electric optical quartz disks are provided on the vacuum tank (see Figure 5) for use with optical pyrometers.

The temperature along the middle 7 in. of the heater could be observed through the optical parts by the use of a disappearing filament type pyrometer.

A Welch 1397 mechanical pump and a NRC - 1500 diffusion pump of 8" diameter are used to maintain a vacuum of 10^{-6} torr inside the vacuum chamber. A liquid nitrogen trap, NRC type 0314 - 6 and a molecular sieve foreline trap are used against back streaming.

A Wallace and Tiernan absolute pressure gauge, two thermocouple gauges and an E.I.T. Bayard-Alpert ionization gauge tube connected to a Fredericks Televac 3A-1-7C2 vacuum gauge unit are used to read pressures of 1-50 mm., 10-1000 microns and 10^{-4} torr - 10^{-9} torr respectively.

Provision has also been made to maintain an inert atmosphere inside the vacuum chamber at any desired pressure. The inert gas used (He) is purified by passing it through a U-tube molecular sieve. The U-tube made of copper, and containing Linde 13 x, ($0.8 \pm 0.05\ Na_2O$, $1.00\ Al_2O_3$, $2.48 \pm 0.03\ SiO_2 \cdot 1/2\ H_2O$), is kept in liquid notrogen.

The heating element is designed to operate at approximately 20 volts with a maximum current of 1440 amps in each phase. The power to the heating element is supplied through a three phase 60 KVA Hunterdon Step-down transformer, from a three phase Norbatrol silicon-controlled rectifier, operated on a 3 phase Y-connected 440 volts A.C. Mains. A current limiter is incorporated into the rectifier to protect the heating element from overheating.

The desired heating or cooling rates are obtained by regulating the power output of the Norbatrol through a feedback system consisting of a Corning precision deviation controller (Series 8810) and a Corning Graphic Programmer (8899).

PREPARATION OF TEST SPECIMEN

Specimens used for thermal diffusivity tests are in the form of long right cylindrical cylinders; for the wire-wound furnace they are 1" in diameter and 6" long, for the tungsten-mesh furnace, the samples are 2" in diameter and 18" long, which is formed by stacking pieces of shorter length (2" to 6") one on top of the other. Thermal diffusivity measurements were made for Lithium Fluoride (LiF) and Alumina (Al_2O_3).

1. The Al_2O_3 sample in the desired shape was made by the General Electric Lamp and Glass Department. The Al_2O_3 sample, commercially referred as Lucalox, is an α alumina polycrystallite with a density of 3.890 ± 0.09 gms/cc which is close to the density of single-crystal synthetic sapphire, i.e., 3.98 gm./cc. A typical analysis of Lucalox is Al_2O_3 - 99.8% minimum, MgO - 1000 ppm. SiO_2 - 300 ppm, Fe_2O_3 - 100 ppm, CaO - 100 ppm, plus traces of CuO, Cr_2O_3, and Ni.

The size and the location of the holes in the 1" diameter and 6" long Lucalox sample are shown in Figure 6A.

The 2" diameter and 18" long Lucalox sample is formed by stacking three 2" high cylinders on top of a 6" high cylinder on top of another three 2" high cylinders. The dimensions and the locations of the holes are shown in Figure 6B.

2. Lithium Fluoride samples were made by melting LiF powder (Fisher certified Reagent Powder) with the following analysis:
Acidity (as HF), 0.005%; Chloride (Cl), 0.005%; Sulfate (SO_4), 0.00%; Barium (Ba), 0.00%; Heavy metals (as Pb), 0.001% in carbon containers.

The powder is first heated up slowly in an inert atomosphere of argon to drive off the absorbed moisture, and it is then taken up to a temperature where it is completely molten, in small temperature increments. The liquid LiF is allowed to solidify slowly from the bottom of the container. This prevents the formation of voids in the solid sample. The sample generated is a polycrystalline aggregate.

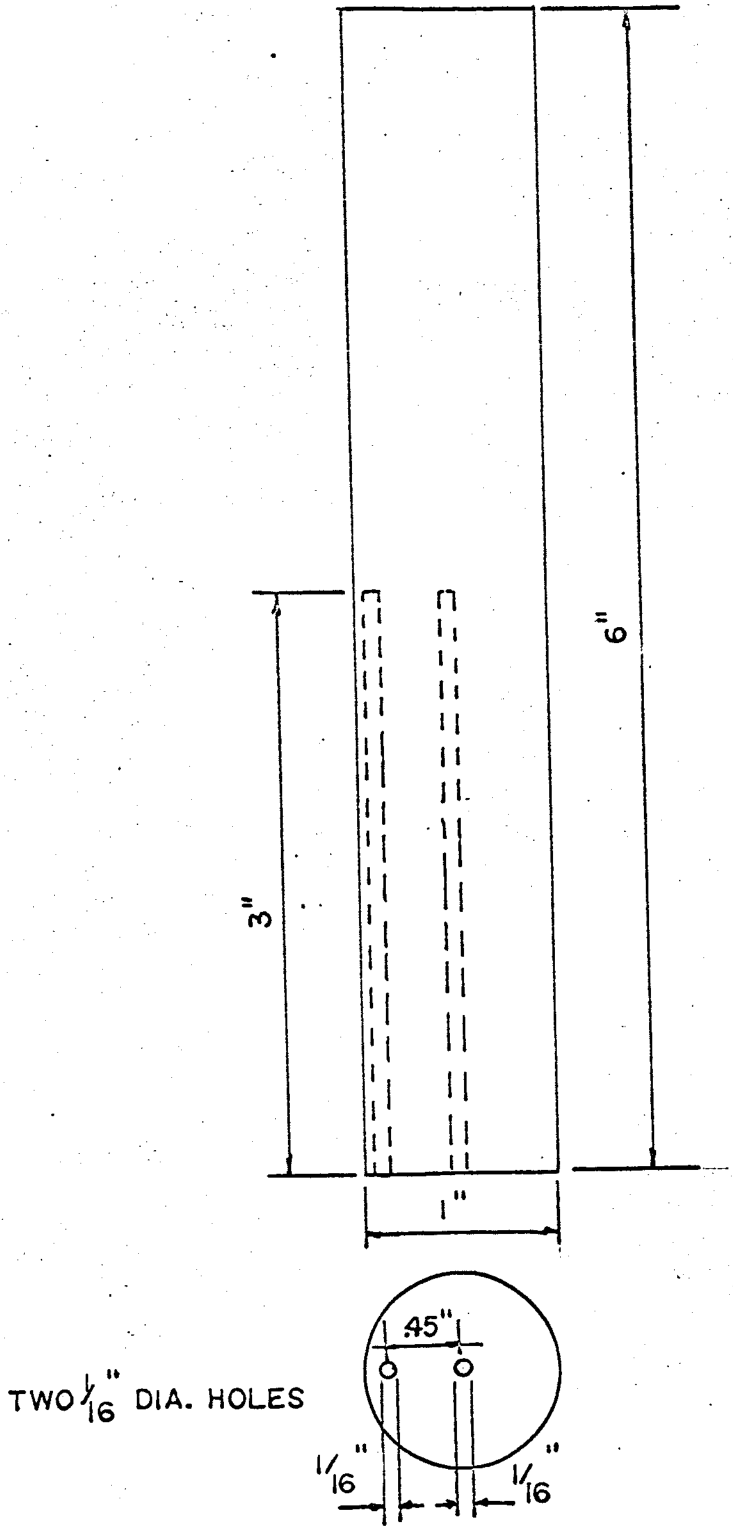


FIGURE 6A DIMENSIONS OF THE 1" DIAMETER TEST SAMPLE

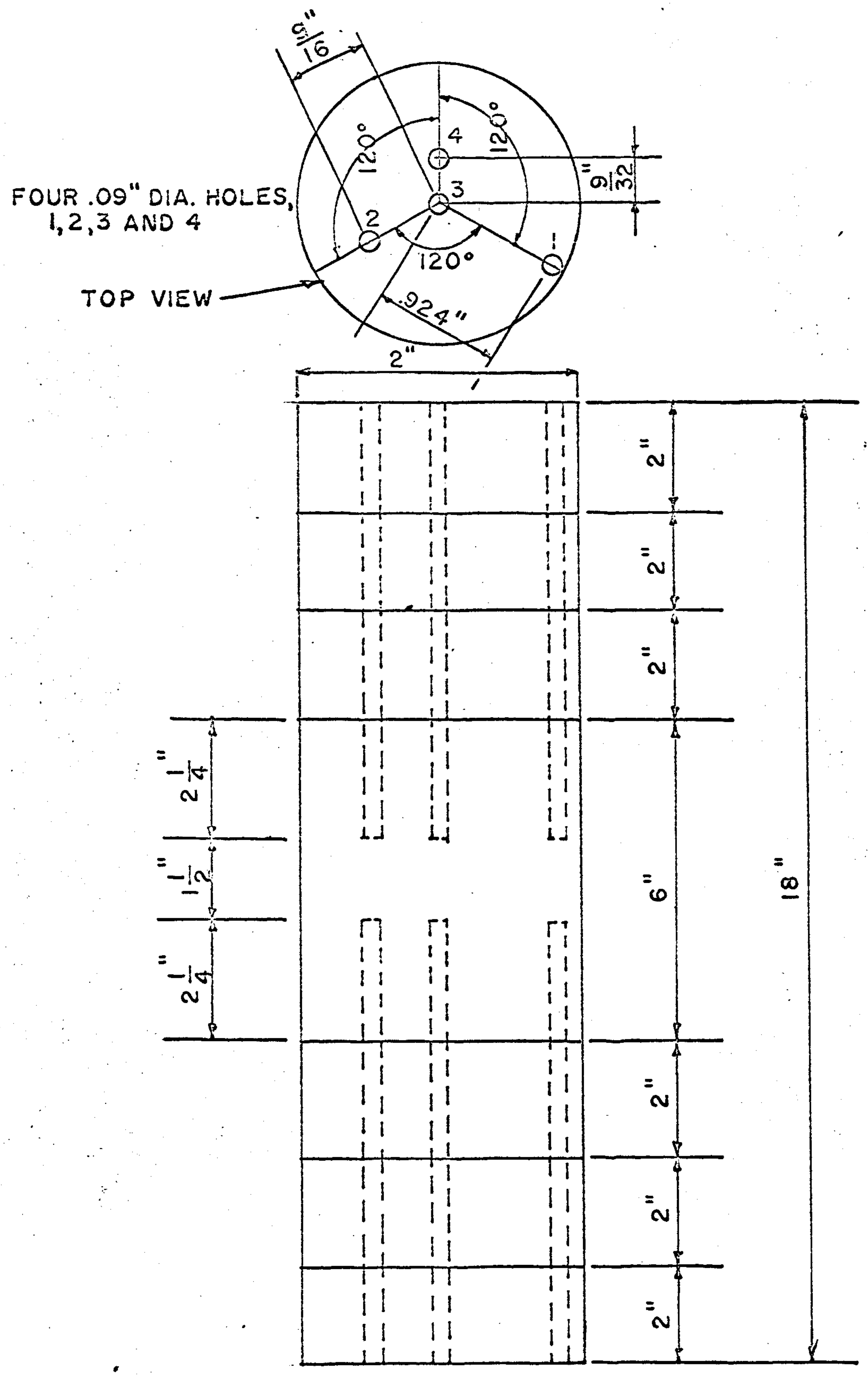


FIGURE 6B DIMENSIONS OF THE 2" DIAMETER TEST SAMPLE
(NOT TO SCALE)

Both the 1" diameter and the 2" diameter samples are fabricated in this fashion to 6" and 3" lengths respectively.
The argon used is purified by passing it over a glass flask containing phosphorous pentoxide to remove water vapor, and an alumina tube containing heated copper turnings to remove O_2. This process is accomplished by inserting the alumina tube in a Hoskin Electric Furnace operated on a 110 volt line at 5 amperes, maximum; this provides temperatures up to $800^{\circ}C$.

The density of the LiF sample, D, was found by the following method. It was determined from its weight, W; the density and the weight of mercury displaced by the sample volume, D_{Hg} and W_{Hg}, i.e., $D = \frac{W}{W_{Hg}} \times D_{Hg}$. The weight of the cylindrical LiF sample 2" long and 2" in diameter is measured to be 330.5 gm.; the weight of mercury is measured to be 1718.5 gm.; the density of mercury is taken as 13.456 gm./cc. and the density of LiF is calculated to be 2.61 ± 0.01 gm./cc.

For the purpose of inserting thermocouples to measure the temperatures, holes are drilled in the LiF sample parallel to the axis with high speed steel drills purchased from Butterfield Division UTD Corporation (Derby Line - Vermont). The dimensions and the locations of the 1" diameter sample are given in Figures 6A and 6B.

EXPERIMENTAL ERRORS

Space limitations make it impossible to present a detailed discussion. A careful analysis has been made, and it is estimated that the experimental data should be accurate to within ± 10 per cent.

RESULTS

The thermal diffusivity of Lucalox (Al_2O_3) measured in the Pt-Pt 40% Rh wire-wound furnace is tabulated in Table 1 and plotted in Figure 7. The thermal diffusivity of Lucalox measured in the Tungsten-mesh heater furnace is tabulated in Table II and also plotted in Figure 7. A curve giving the diffusivity of Lucalox as a function of temperature is drawn through both sets of data.

The thermal diffusivity of Lithium Fluoride (LiF) measured in the Pt-Pt 40% wire-wound furnace is tabulated in Table III and plotted in Figure 8. The thermal diffusivity of Lithium Fluoride measured in the Tungsten-mesh heater furnace is tabulated in Table IV and also plotted in Figure 8. Figure 9 shows a typical set of test data obtained during an experimental measurement of Lucalox in the Tungsten-mesh heater furnace, the difference between the center and the periphery thermocouple readings is recorded to be 12.5 microvolts, while the Pt-Pt 10% Rh periphery thermocouple reading records a linear rise of .19 mv./10 min. The thermal diffusivity, α, is calculated to be 0.0321 cm^2/sec. at 368°C.

TABLE I

THERMAL DIFFUSIVITY OF LUCALOX (Al_2O_3)

(Measured in Pt-Pt 40% Rh wire wound furnace)

Temperature (°C)	Thermal diffusivity (cm^2/sec.)
535	0.0228
561	0.0221
586	0.0213
622	0.0232
824	0.0135
916	0.0126
1002	0.0111
1206	0.0094

Density of the test sample, 3.98 gm./cc. at 25°C.

Purity of the test sample, not less than 99.8% Al_2O_3.

Constitution and structure of the test sample = σ - Alumina, polycrystalline.

Melting point of the test sample = 2040°C = 2313°K.

Geometry of the test sample, cylindrical cylinder 1" diameter 6" long.

TABLE II

THERMAL DIFFUSIVITY OF LUCALOX (Al_2O_3)

(Measured in Tungsten-mesh heater furnace)

Temperature (°C)	Thermal diffusivity (cm.^{2}/sec.)
95	0.0786
130	0.0645
144	0.059
178	0.0576
231	0.0456
273	0.0415
295	0.0376
440	0.0270
598	0.0216
627	0.0191
703	0.0171
788	0.0152
963	0.0124
1042	0.0123
1320	0.011
1419	0.012

Density of the test sample, 3.98 gm./cc. at 25°C.

Purity of the test sample, not less than 99.8% Al_2O_3

Constitution and structure of the test sample = α - Alumina, polycrystalline

Melting point of the test sample = 2040°C = 2313°K.

Geometry of the test sample, cylindrical cylinder 2" diameter 18" long

TABLE III

THERMAL DIFFUSIVITY OF LITHIUM FLUORIDE (LiF)

(Measured in Pt-Pt 10% Rh wire-wound furnace)

Temperature (°C)	Thermal diffusivity
185	0.0180
265	0.0157
360	0.0130
385	0.0129
390	0.0124
395	0.0119
405	0.0122
430	0.0119
437	0.0102
460	0.0110
471	0.0102
476	0.0111
490	0.0103
512	0.0993
520	0.0097
530	0.0107
565	0.0099
572	0.0102
575	0.0110
590	0.0097

Density of the test sample, 2.61 ± .01 gm./cc. at 25°C.
Purity of the test sample, 98.99% LiF
Structure of the test sample, polycrystalline
Melting point of the test sample = 848°C = 1121°K
Geometry of the test sample, cylindrical cylinder 1" diameter 6" long

TABLE IV

THERMAL DIFFUSIVITY OF LITHIUM FLUORIDE (LiF)

(Measured in Tungsten-mesh heater furnace)

Temperature (°C)	Thermal Diffusivity
40	0.038
80	0.0298
102	0.0296
115	0.028
134	0.0254
160	0.021
167	0.0217
212	0.0179
242	0.0164
270	0.0148
310	0.0138
350	0.0130
374	0.0125
395	0.0124
423	0.0118
505	0.0110
555	0.0117
602	0.0110
640	0.0105
665	0.0107
675	0.0112
684	0.0105
735	0.0115
790	0.0113
845	0.0112

Density of the test sample, 2.61 ± .01 gm./cc. at 25° C
Purity of the test sample, 98.99% LiF
Structure of the test sample, polycrystalline
Melting point of the test sample = 848° C = 1121° K
Geometry of the test sample, cylindrical cylinder 2" diameter 18" long.

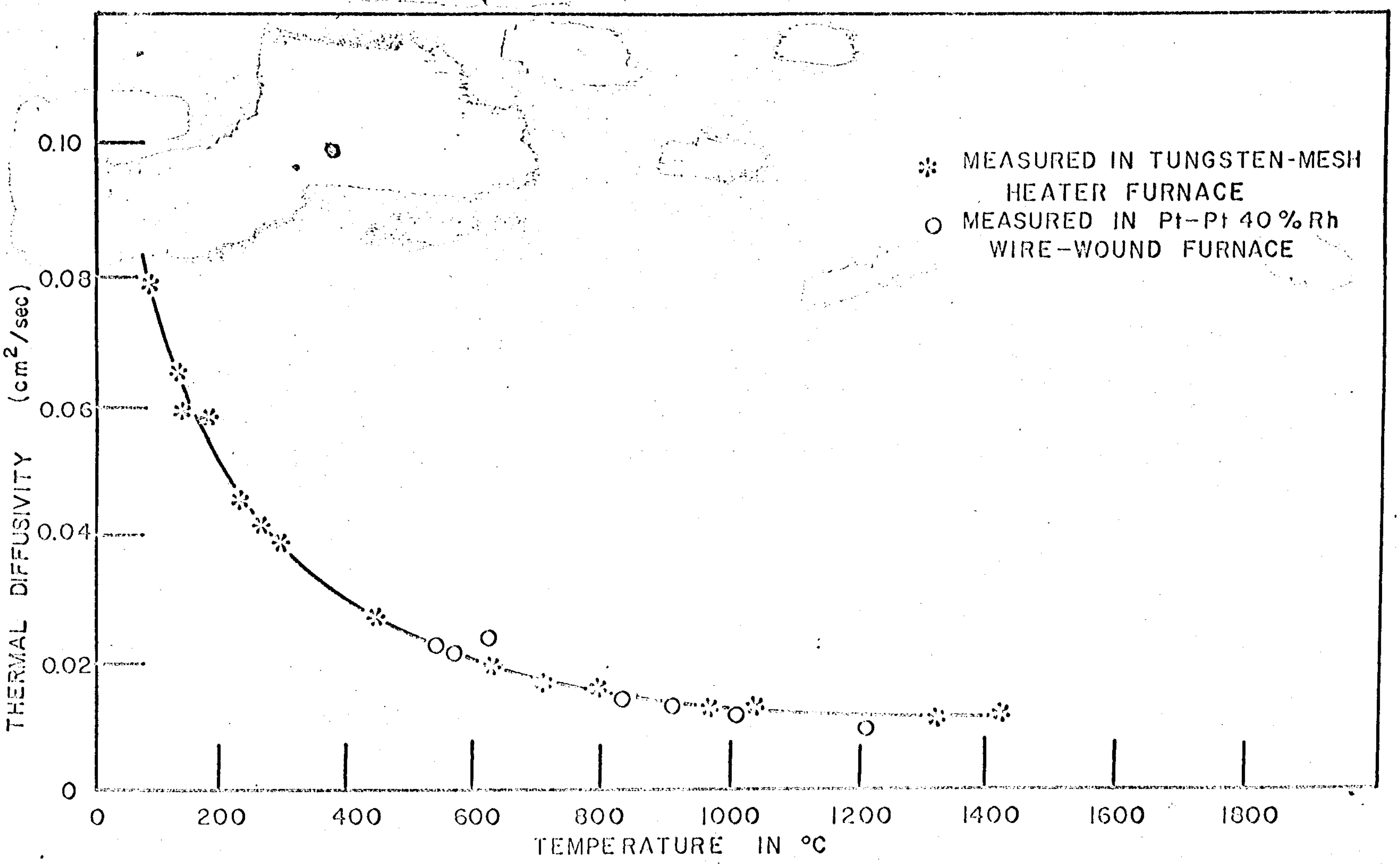
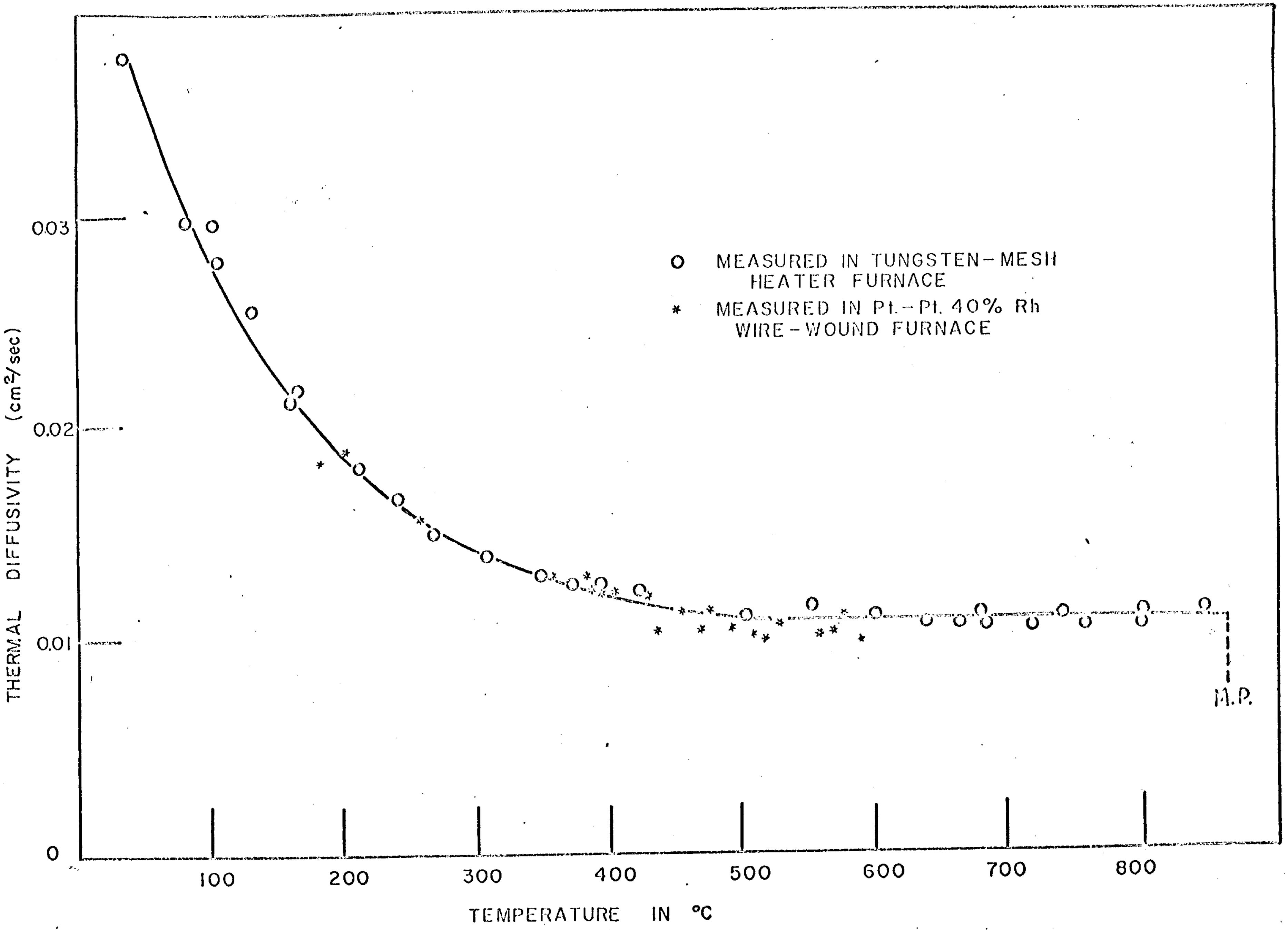
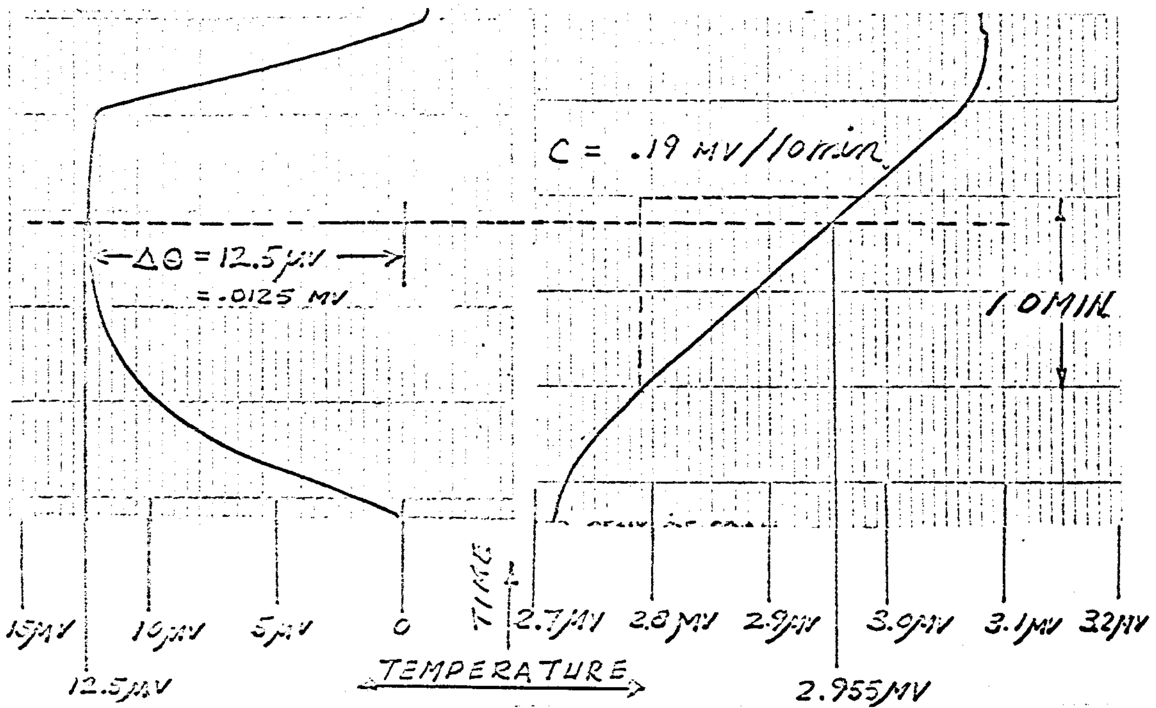


Fig. 7. THERMAL DIFFUSIVITY OF LUCALOX (Al_2O_3)



THERMAL DIFFUSIVITY OF LITHIUM FLUORIDE (LiF)

FIGURE 8



The difference in temperature rise, $\Delta\theta$, (millivolt) measured by Pt-Pt 10% Rh thermocouples located in holes 1 and 3 in Fig. 13.

The rate of rise of temperature, c, measured at the cylindrical surface of radius 2.25 cm. (hole 1 in Fig. 13)

$$\alpha = \frac{r^2 c}{4\Delta\theta}$$

$$= \frac{(2.25\ \text{cm})^2}{4} \quad \frac{(0.19\ \text{mv}/10\ \text{min.}) \times \frac{1\ \text{min.}}{60\ \text{sec.}}}{0.0125\ \text{mv}} = 0.0321\ \text{cm}^2/\text{sec.}$$

at 2.955 mv or 368°C

Figure 9. Typical Thermal Diffusivity Data of Lucalox Measured in Tungsten-mesh Heater Furnace

DISCUSSION

An unforseen difficulty needs reporting. The apparatus was designed to operate in a vacuum in order to eliminate heat losses and free convection effects. It was found, however, that this could not be achieved for the following reasons:

1. The contact resistance between the thermocouples and the specimen could not be eliminated in a vacuum. Many things were tried to bond the thermocouple tips to the specimens, but no successful bonding technique could be developed within the time period available.
2. The use of optical pyrometers would have eliminated this problem, but it was found that the optical problems involved were complex, and again, further study of this possibility is beyond the scope of the present study.

The eventual solution which was evolved was found to be satisfactory, even if not ideal. Helium gas was admitted to the chamber at a pressure at which its thermal conductivity was still high, but at which free convection effects were quite small. Thus, it was possible to minimize the contact resistance, and also to minimize the heat losses and resulting temperature non-uniformities.

Lucalox was studied primarily because several investigators have reported good data, so that it could serve as a check on the method and equipment. Reliable data on LiF were not available, and the results presented are new. Since LiF is seriously being considered as a potential TES material, this information is also important.

It can be seen from Fig. 8 that the measured value of the thermal diffusivity in the vicinity of the melting point is about 0.0105 cm^2/sec. McKinnon[15] et al report an average value for the conductivity in the neighborhood of the melting point of 0.0211 cal/sec-cm-$^{\circ}$C. Using the data of Refs. 17 and 18 to convert this conductivity to diffusivity, one computes a value of 0.016 cm^2/sec. It is believed that the results of this present investigation are more consistent with what would be expected, based on the fact that LiF should behave like LiH whose thermal diffusivity [18] would indicate a lower value than McKinnon's results.

The results on Lucalox (as seen in Fig. 10) are in excellent agreement with those obtained by Kingery et al [20] as well as with Rudkin's [12] data. The results of Fitzsimmon[5] et al are definitely in disagreement with the present ones.

CONCLUSIONS

The present work has shown the feasibility of the experimental method described in the text for application to temperatures up to 2000°C. plus.

It was found that operation in a vacuum is not essential.

Tests with Lucalox show good agreement with results of other investigators. New data on LiF were obtained and presented.

Future work will be concerned with modifications to allow the testing of liquids.

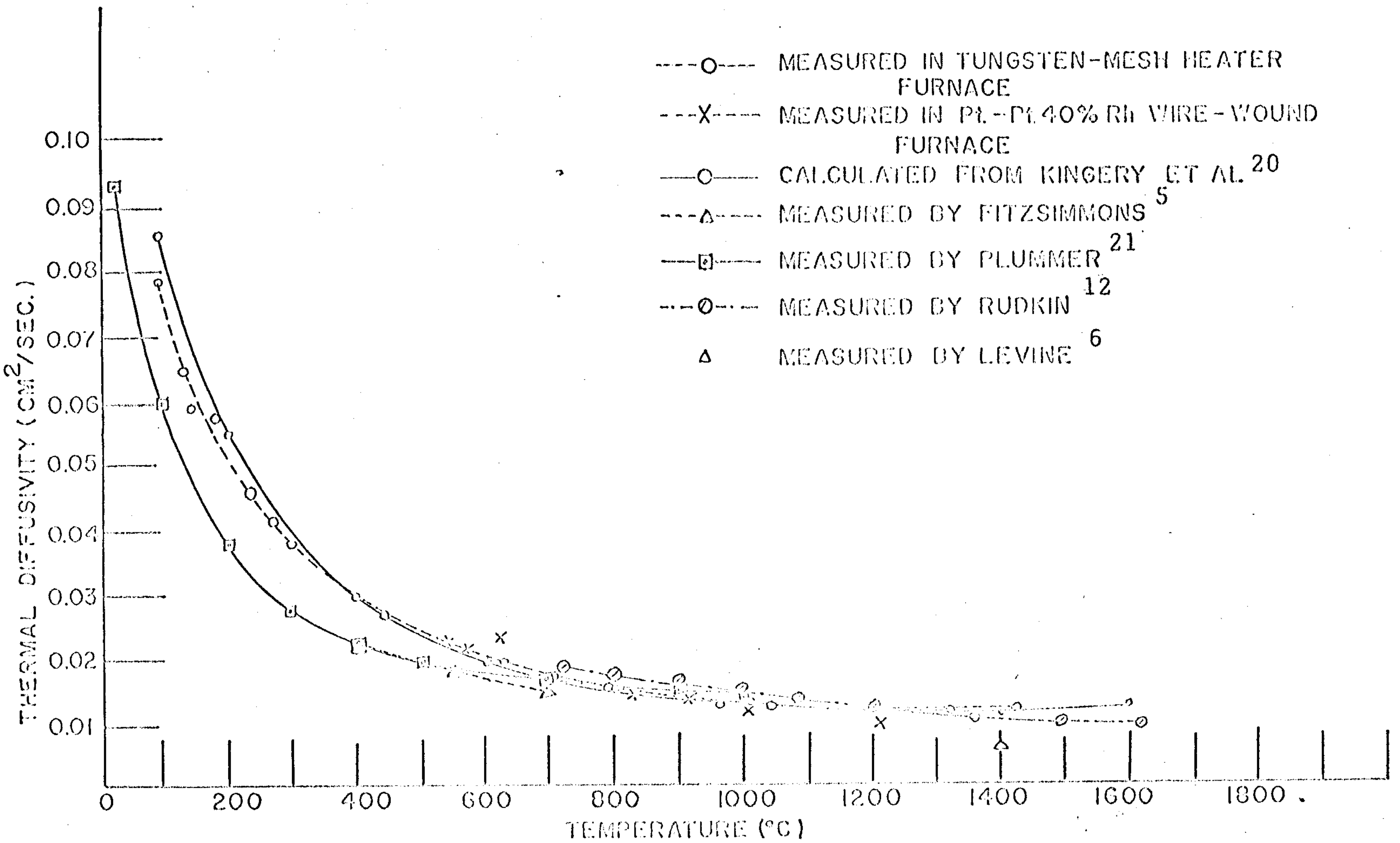


FIGURE 10. THERMAL DIFFUSIVITY OF AL_2O_3 REPORTED IN LITERATURE

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