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Thermal Conductivity of Beryllium Oxide From 40° to 750° C

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The thermal conductivity of beryllium oxide has been measured by an absolute method from 40° to 750° C. The apparatus employed steady-state longitudinal heat flow along a rod of high-fired beryllium oxide, surrounded by a tube with matching temperature gradient to minimize radial heat loss. The estimated accuracy of the measurements is about 3 percent. However, the values of thermal conductivity of the ideal beryllium oxide crystal are probably considerably higher than the values given because of the lower density (87 percent theoretical) of the sample used.

1. Introduction

Not only is beryllium oxide useful as a moderator in the utilization of atomic energy, but it has an unusually high thermal conductivity, much higher than other nonmetals and even higher than most metals over a limited temperature range. At room temperature, its thermal conductivity is about that of aluminum, whereas its electrical conductivity is extremely low. It was the purpose of this investigation to measure the thermal conductivity of beryllium oxide in the high-temperature range.

2. Sample

The beryllium oxide was originally fabricated by the Norton Co. by hot-pressing. A rough sample was taken from this material and machined, at the Oak Ridge National Laboratory, to a cylindrical rod about 0.5 in. in diameter and 6 in. long. From information obtained on this material, together with that obtained from a spectrographic analysis, it seems likely that its impurities (other than carbon) were less than 0.2 percent. It is possible that carbon was present in larger amounts in the sample, although it was white, with only occasional dark inclusions. The sample was fired in the NBS Mineral Products Division at about 1,700° C and machined to the form of a true cylinder having a diameter of 0.4524 in. at room temperature and an average density of 2.62 g/cm^3 (87 percent of single crystal). The method of original fabrication by hot-pressing may have caused a variation in density in the sample of several percent.

3. Method and Apparatus

The method and apparatus have been described briefly in technical reports [1, 2].[1] The method used was absolute in that the results were obtained without comparison with another material. A longitudinal heat flow was used to establish a temperature gradient in the sample. From the measured values of heat flow, temperature gradient, and the cross section of the sample, the thermal conductivity of the sample was calculated. Longitudinal rather than radial heat flow was used in order to obtain a reasonable temperature gradient in a conveniently shaped sample. The temperature gradient on the sample was determined by measuring the temperatures along the sample. The longitudinal heat-flow method, as applied to relatively long samples, has the inherent disadvantage that radial heat losses to the surroundings may reduce the accuracy of the results. However, in this case, the conductivity of the BeO was so high that radial heat losses did not seriously limit the accuracy of the results.

A scale diagram of the essential parts of the apparatus is shown in figure 1. Measured electrical heat, introduced in the "sample heater" at the top of the sample (BeO), flowed down the sample and its "adapter" to a heat sink. The sample heater consisted of six small helices of No. 38 Nichrome wire located in holes in the top of the sample. The adapter was used here to position the sample, as well as to fill in the needed length, because the apparatus was built to accommodate samples longer than 6 in. Anhydrous boric oxide was used to give good thermal contact between the sample and adapter, and between the adapter and the sink. This compound has a very low vapor pressure and has excellent wetting properties, maintaining good thermal contact at temperatures far below its melting point. The sink was cooled with either water or air, depending on the temperature range, and was equipped with a heater and thermocouple so that it could be automatically kept at a constant temperature.

The temperatures along the sample were measured with three thermocouples (No. 36 AWG platinum-platinum-rhodium) having reference junctions at 0° C and principal junctions on the sample at the three levels shown in figure 1. In addition to these three absolute thermocouples, a differential thermocouple was also used to ascertain directly the temperature difference between the upper and lower levels on the sample. All of the thermocouples on the samples were made with junctions peened into small holes (about 0.6 mm in diameter and depth) in the cylindrical surface of the sample. In order that the temperature gradient measured on the sample would correspond to the electric heat put into the top of the sample, precautions were taken to minimize radial heat loss along the sample. For this purpose, the sample was surrounded by a "guard tube"

[1] Figures in brackets indicate the literature references at the end of this paper.

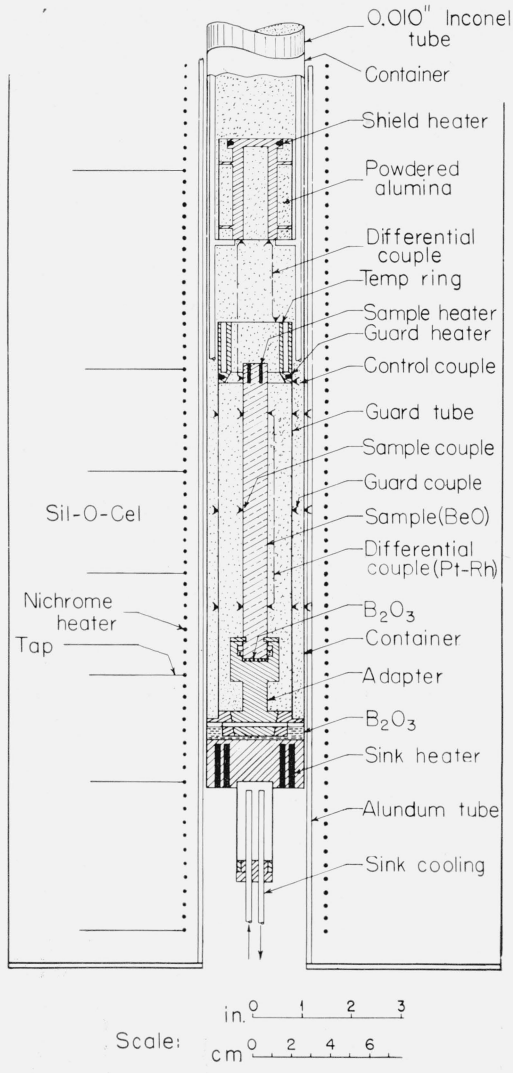

FIGURE 1. *Thermal-conductivity apparatus.*

(Inconel, 0.03-in. wall), with temperatures corresponding to those in the sample. Temperatures along the guard tube were measured with three platinum–platinum-rhodium thermocouples, with principal junctions attached to the guard tube at levels corresponding to those of the sample thermocouples. Three additional thermocouples, at the same levels on the guard tube but at different azimuths, were also provided. The top of the guard tube was heated by the "guard heater", and its temperature was controlled automatically, using the "control couple" close to the heater. Another heater with a control thermocouple (not shown in fig. 1) was provided at the bottom of the guard tube, but it was found unnecessary to use it when the furnace temperatures were suitably controlled. The top portion ("tempering ring") of the guard tube was made of thick nickel to which the electrical leads to the sample were thermally connected, so that it served as a tempering region for bringing the leads to the temperature of the top of the sample, and thus to reduce heat conduction along them. A "thermal shield" (nickel) and heater, placed above the sample and tempering ring, also served for this purpose and to prevent heat transfer upward through supports and insulation.

Although the apparatus shown in figure 1 has been used only for measuring the thermal conductivity of solids, it was designed so that it could be used also for high-conductivity liquids. For this application, the liquid would be contained in a thin-walled tube equipped with a suitable heater and a liquid expansion chamber extending up into the shield. To obtain the thermal conductivity of the liquid, it would be necessary to account for the thermal conduction in the container tube by making another experiment with the liquid replaced by a powder of very low and known thermal conductivity.

The whole assembly, supported by rods extending down to the bottom of the guard, was filled with fine aluminum oxide powder for insulation and enclosed in a 0.010-in. Inconel tube, which served to hold the powder. This tube, fitting closely in the "container" (0.035-in. Inconel tube) centered along the axis of the furnace, facilitated assembly. Thermocouple and heater leads were brought out from this container through an insulating seal at the top (not shown in fig. 1), so that the conductivity apparatus could either be evacuated or filled with argon. Because experiments with the apparatus had shown the electrical insulation to fail when it was evacuated at high temperatures, the apparatus was filled with argon after evacuation at moderately high temperatures to outgas the aluminum oxide. The thermal-conductivity apparatus was maintained at the chosen temperature by the surrounding furnace, which was equipped with numerous taps on the heater winding to give the desired vertical temperature gradients. Two automatic regulators, actuated by thermocouples in the furnace, were used to maintain constant temperatures.

4. Experimental Procedure

Two basic types of experiment were performed for each measurement of thermal conductivity. In the first type (called a *conductivity experiment*), the furnace temperature was controlled to the desired value, a known constant electric power was put into the sample heater, the temperatures of the guard and shield were adjusted to match those on the sample as closely as possible, and the sink temperature was adjusted to a constant value. After a steady state was obtained, several consecutive sets of observations of thermocouple emf and sample power were recorded. In the second type of experiment (called *calibration experiment*), no power was put into the sample heater, but in other ways, the experimental procedure was similar to the first. The purpose of this experiment was to correct for errors that did not depend on the power transmitted through the sample. Errors of this type are those due to differences, in thermocouples and those resulting from unknown heat leaks, which presumably were the same in both

types of experiments. Several other experiments were made to detect other errors and to determine their importance. Some of these experiments are described later.

Several hours were usually required to bring the various parts of the apparatus to the desired temperatures and to make sure that these temperatures were not changing significantly. The final data were usually obtained in a period of about 30 min, subsequent to an interval of about an hour during which the temperatures were observed to be constant. Automatic thermoregulators were used to control the temperatures of the furnace, guard, and sink.

5. Calculation of Results and Uncertainties

The conductivity values were calculated from the observed quantities by means of the equation

$$k=\frac{\dot{Q}\Delta X_0}{A_0(1+\alpha t_a)\Delta t},$$

where

k=thermal conductivity (watts cm^{-1} deg^{-1} C) at temperature t_a.
t_a=average temperature of sample between thermocouples.
$\dot{Q}$=heat-flow rate (watts).
ΔX_0=thermocouple spacing at 0° C (cm).
A_0=cross-sectional area at 0° C (cm^2).
α=coefficient of linear thermal expansion (deg^{-1} C).
Δt=temperature difference (deg C) between thermocouples.

This equation is valid for steady-state longitudinal heat flow over a small temperature interval. The determination of these factors in the conductivity equation, together with a consideration of their uncertainties, will now be discussed individually. The uncertainties referred to in this report are the authors' estimates (based on their judgment) on the basis that the observed quantity would have about an equal chance of being within that limit as being outside it, and that the sign of the uncertainty is just as likely to be positive as negative.

5.1. Heat-Flow Rate ($\dot{Q}$)

a. Electric Power

Heat was generated by direct current in the sample heater, and power was measured in a conventional manner, using a potentiometer in conjunction with a high-resistance volt box to measure the potential drop across the heater, and a standard resistor in a current lead to measure the current. The errors in these electrical measurements were almost negligible. In measuring the potential drop across the heater, the potential terminals were located to evaluate properly the heat that went to the sample. Because the sample heater was made with very high resistance (relative to that of the heater leads), the uncertainty in the location of the potential terminals resulted in only about 0.1-percent uncertainty in the measured thermal conductivity.

b. Heat Flow From Sample to the Tempering Ring

In addition to the electric-power input to the top of the sample, it is necessary to consider the heat transfer between the sample and its surroundings. Because of the excess temperature of the sample heater, heat flowed along the heater leads to the tempering ring; the uncertainty in this heat flow (taken to be 50 percent of the correction) averaged about 0.24 percent of the total heat flow in the sample. In addition to this heat flow, there was the heat flow between the top part of the sample and the tempering ring. The evaluation of this was difficult because of the configuration and the temperature distribution on the top part of the sample where the sample heater was located. Using differential thermocouples, observations were made of the temperature difference between the isothermal tempering ring and a point on the top part of the sample. The location of this point was determined by calculation so that the net heat flow from the top of the sample to the tempering ring would be proportional to the emf of the differential thermocouple. The height of the top of the sample relative to the guard was made so that the bottom of the sample heater was at the same level as the bottom of the tempering ring. In the actual experiments, the tempering-ring temperature depended on the power in the guard heater and was indicated by the differential thermocouple to be 2 or 3 deg higher than the temperature of the sample, making necessary a correction for the resulting heat flow. It was found convenient to evaluate this correction experimentally by making two conductivity experiments in which only the tempering-ring temperature was changed and the furnace temperature adjusted to maintain the match between the guard and sample thermocouples. Using the resulting change in sample gradient, the data were corrected to correspond to no difference in temperature as indicated by the differential thermocouple. However, there still remained an uncertainty in heat flow due to some uncertainty in the proper location of the thermocouple junction on the sample head. It was estimated that the uncertainty in the heat flow between the sample and tempering ring, excluding heat flow along leads mentioned above, resulted in an uncertainty in measured conductivity averaging about 0.5 percent.

c. Heat Flow Down the Insulating Powder

Even when the guard temperatures matched sample temperatures, some of the heat input to the sample necessarily went to maintain some of the longitudinal heat flow in the insulating powder between the sample and guard. The conductivity of the aluminum oxide powder with argon gas was determined approximately by a few experiments as it was used in the apparatus. The temperature distribution in the insulation had been previously estimated with a resistance analog computer, setting up boundary conditions corresponding to the configuration and assumed temperatures of the sample and guard. It was estimated from these results

that when the thermocouple on the top part of the sample indicated the same temperature as the guard ring, the sample heater contributed only 16 percent of the total longitudinal heat flow in the powder, the remainder of the heat being furnished by the guard. Under this condition, about 0.2 percent of the heat of the sample heater flowed down through the insulating powder. The uncertainty of this correction was estimated to give less than 0.1-percent uncertainty in the measured thermal conductivity.

d. Heat Flow Between Sample and Guard

Because it was found impractical to match the sample and guard temperatures exactly during all conductivity experiments, experiments were made that permitted calculation of corrections for imperfect matching. For each guard thermocouple, two thermal-conductivity experiments were made, varying only the difference between that guard thermocouple and the corresponding thermocouple on the sample. From the resulting change in temperature gradient on the sample in these two experiments, it was possible to estimate a correction for small differences in matching guard and sample thermocouples. It was calculated that there was an uncertainty of about 50 percent in correcting for heat flow between the sample and the guard. This uncertainty resulted in an average uncertainty in measured conductivity of about 0.3 percent.

e. Heat Flow Into Heat Capacity of Sample

If the temperature of the sample were changing with time, some of the heat input would go to produce this change, and the temperature gradient on the sample would not correspond to the heat input at the top of the sample. In all experiments, the rate of temperature change was less than 0.8 deg C/hr, corresponding to an effect of 0.5 percent in the calculated value of conductivity. The average uncertainty in the correction for this was negligible.

5.2. Thermocouple Spacing (ΔX_0)

The distance between the principal junctions of the upper and middle sample thermocouples was 4.97 cm at 0° C, whereas the corresponding distance for the middle and lower thermocouples was 5.05 cm; this gives 10.02 cm for the distance between the extreme absolute thermocouples. The distance between junctions on the differential thermocouple was 10.01 cm. These distances, taken as the lengths between centers of the thermocouple holes, were measured to better than 0.01 cm with a traveling microscope, but because the thermocouples were peened into holes 0.06 cm in diameter, the possibility of nonuniform thermal contact makes a tolerance of 0.03 cm appear more realistic. This tolerance corresponds to 0.3-percent uncertainty in the 10-cm spacing between the sets of thermocouples used in the conductivity calculations. The effect of thermal expansion on both thermocouple spacing and cross-sectional area is lumped into the correction $(1+\alpha t_a)$, which is described later.

5.3. Cross-Sectional Area (A_0)

The sample was ground to have a uniform diameter of 0.4524 ± 0.0003 in., corresponding to a cross-sectional area of 1.038 cm^2. The uncertainty in this area was estimated to be less than 0.1 percent.

5.4. Thermal-Expansion Correction ($1+\alpha t_a$)

Thermal-expansion changes the thermocouple spacing by the factor $(1+\alpha t_a)$, and the cross-sectional area by the factor $(1+\alpha t_a)^2$, resulting in the $(1+\alpha t_a)$ term given in the conductivity equation. The coefficient of linear thermal expansion (α) has been determined by White and Schremp [3]. At the highest temperatures (747° C) of the conductivity experiments, the correction for expansion amounted to about 0.6 percent, with negligible uncertainty in the measured conductivity.

5.5. Temperature Difference (Δt)

The accurate measurement of the temperature difference on the sample was difficult, requiring a number of tests to eliminate or evaluate certain errors. As described in section 3, two different thermocouple systems were used to measure the temperature difference over the 10-cm length on the sample. The two independent thermocouple systems served to check on each other, usually agreeing on the measured temperature difference to better than 1 percent.

All temperatures were measured with platinum–platinum-rhodium thermocouples of No. 36 AWG wires. A sample thermocouple was calibrated at several points between 0° and 1,000° C in the Pyrometry Laboratory of the National Bureau of Standards. No significant difference was observed between the thermoelectric power of this sample and that given in the standard tables [4]. Even though the thermocouples were all made from wire off the same spools, it was possible that they had slightly different thermoelectric powers. Although these differences might not be serious for measurement of absolute temperatures, they could be significant for measurement of small temperature differences at high temperatures. These differences were automatically accounted for by the calibration experiments mentioned previously, in which no power was put into the sample heater. These calibration experiments gave differences in thermocouple readings, which increased regularly up to 470° C, and amounted to as much as 7 μv at this temperature. At temperatures approaching 750° C, the differences became both larger and more irregular, so that after each conductivity experiment, a calibration experiment was made to evaluate the differences.

In addition to the differences described above, the evaluation of Δt was uncertain because of the occasional effect of high humidity on the potentiometer used to measure the thermocouple emf. This effect was observed as a reading on the potentiometer, even when the potential across its terminals was

zero. When the value of Δt was observed, using the differential thermocouple, the value was subject to the full potentiometer uncertainty (about 2 μv) because only one reading was involved. When the value of Δt was determined by using the two absolute thermocouples, most of this potentiometer uncertainty was reduced (to about 0.5 μv) because the value of Δt was obtained from a difference of two readings. It is for this reason that the uncertainties in the value of Δt, using the differential thermocouple, were larger than the uncertainties when using the absolute thermocouples, averaging about 1.5 percent as compared to 0.4 percent. That the two thermocouple systems usually agreed to better than 1 percent is evidence that the error due to the humidity effect on the potentiometer was not excessive.

6. Results

The results of the individual thermal-conductivity measurements on beryllium oxide are given in table 1. Values of observed conductivity (k) are given as determined by using each of the thermocouple systems (absolute and differential) at the average temperature (t_a) of that portion of the sample measured by the thermocouples. At the lower temperatures, where the thermal conductivity of the beryllium oxide changes rapidly with temperature, small corrections to conductivity were made for the curvature of the conductivity-temperature function. In this table, the quantities given are corrected for all known errors. In the previous discussion, each uncertainty has been estimated by the authors on the basis that the observed quantity would have an equal chance of being within that limit as being outside that limit. These uncertainties have been combined (square root of the sum of the squares) and arbitrarily increased by over a factor of 2 to give more realistic values of estimated error listed in table 1.

Table 1 indicates that the results, using the absolute thermocouples, seem to be reliable to about 2 percent. The experiments under 60° C are not as accurate as the other experiments. This is due to the smaller temperature drop in the sample, first because the limitations of the heat sink made it necessary to use lower power, and second, because the thermal conductivity of the beryllium oxide was so high in this low-temperature range. The larger error estimated by using the differential thermocouple in this low-temperature region is due to the humidity trouble mentioned previously. No results are given for measurements with absolute thermocouples above 500° C because of failure of their electrical insulation.

A smooth function of thermal conductivity was obtained graphically from the observed values in table 1, giving greatest weight to those values having the smallest estimated errors. Table 2 gives smoothed values of the conductivity at even temperatures as obtained from the graph. Figure 2 gives the deviations of the results (obtained with the two different thermocouple systems) from the smooth

TABLE 1. *Experimental results*

Date (1954)	t_a	Power	Absolute thermocouples			Differential thermocouples		
			Δt	Observed k	Estimated error [a]	Δt	Observed k	Estimated error [a]
	°C	w	°C	w/cm-°C	%	°C	w/cm-°C	%
Aug. 25	38.2	0.8960	3.95	2.19	5.4	4.11	2.10	19.2
Aug. 26	46.2	1.4452	6.84	2.04	3.2	7.03	1.98	10.7
Aug. 25	52.6	1.8894	9.41	1.94	2.6	9.61	1.90	7.9
Aug. 27	59.7	2.3620	12.15	1.879	2.1	12.33	1.849	6.2
Aug. 24	85.8	3.3179	19.16	1.674	1.6	19.28	1.661	4.0
Oct. 8	86.2	3.8007	21.88	1.679	1.7	22.09	1.660	3.6
Sept. 29	86.9	3.6073	20.90	1.668	2.1	21.07	1.652	3.9
Sept. 23	87.3	3.6084	20.86	1.672	1.8	21.08	1.652	3.7
Sept. 30	87.3	3.6138	21.01	1.662	1.7	21.17	1.647	3.7
Aug. 31	91.4	2.5903	15.20	1.647	2.0	15.10	1.655	4.9
Sept. 1	123.8	2.4474	16.26	1.454	1.7	16.33	1.446	5.4
Sept. 15	153.0	2.4894	18.27	1.316	1.7	18.36	1.308	4.1
Sept. 2	153.4	2.4795	18.27	1.311	1.8	18.37	1.302	4.2
Oct. 11	202.0	3.9603	33.80	1.131	2.0	33.65	1.135	2.6
Aug. 23 [b]	202.1	3.8745	33.29	1.124	1.6	33.10	1.128	2.4
Aug. 18	241.5	3.7312	36.20	0.995	1.7	35.87	1.002	2.3
Aug. 19	241.4	3.7396	36.51	.989	2.2	36.19	0.996	2.7
Aug. 20 [b]	251.4	0.8739	8.95	.942	2.2	8.85	.952	6.5
Sept. 20	287.3	2.3441	25.93	.872	1.8	25.54	.884	2.8
Sept. 17	287.7	2.3378	25.87	.872	1.9	25.71	.876	2.9
Oct. 7	379.5	3.5169	48.12	.704	1.5	47.77	.709	2.0
Oct. 5	380.4	3.5378	48.64	.701	1.9	47.60	.715	2.3
Oct. 12	439.2	3.3054	51.83	.614	1.2	51.82	.614	1.7
Oct. 18	517.2	2.9239	-------	-------	-------	53.63	.524	2.7
Oct. 19	578.7	2.5664	-------	-------	-------	51.54	.478	3.5
Oct. 20	646.7	2.7048	-------	-------	-------	59.94	.433	2.4
Nov. 2	747.9	2.6370	-------	-------	-------	65.84	.384	4.3

[a] Estimated error is the authors' estimate, considering only the various uncertainties mentioned in the text.
[b] The results on August 20 and 23 represent averages of 2 experiments on each day.

function. At 50° C and below, the observed values deviate markedly from the smooth function, giving values at 40° C that are 4 percent different. These deviations are probably due to the very small temperature difference of 4° C in the sample, so that errors in the measurement of this temperature difference have greater influence on the result. Figure 2 also shows the results at 251° C to be about 3 percent lower than the other results. It seems probable that this departure is also due to the lower temperature difference on the sample resulting from the lower power; the power here was only one-fourth the power in the other experiments in this temperature range. If it is assumed that there existed an unknown constant absolute error in either heat flow or temperature difference, the deviations of about 3 percent in the low-power experiments would indicate that the experiments with the higher power might be in error by about 0.8 percent. Consequently, the authors believe that the over-all accuracy of the results is more likely to be about 3 percent instead of the 2 percent indicated by the estimated errors listed in table 1.

TABLE 2. *Thermal conductivity of* BeO (*density=2.62 g/cm³*)

(Smoothed values)

t	*k*	*t*	*k*
° *C*	*w/cm-° C*	° *C*	*w/cm-° C*
30	2.155	400	0.666
50	1.967	450	.598
100	1.581	500	.541
150	1.328	550	.498
200	1.131	600	.462
250	0.972	650	.433
300	.845	700	.406
350	.746	748	.384

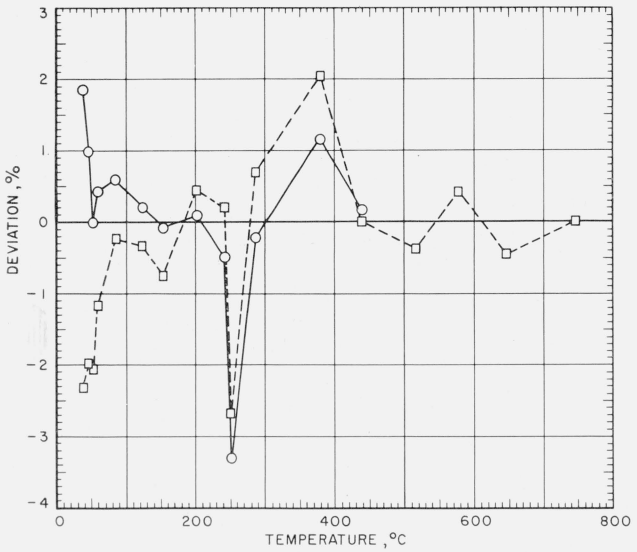

FIGURE 2. *Deviations of beryllium oxide thermal-conductivity data.*

Base line=NBS smoothed data.
○, NBS (absolute couples); □, NBS (differential couples).

No attempt has been made to correct the thermal-conductivity values for the NBS sample to correspond to zero porosity. Because the density of this sample was only 2.62 g/cm³ compared to about 3.0 for the ideal crystal, the thermal conductivity of the crystal should be significantly higher than the values given in table 2. From the investigation of Francl and Kingery [5], it would appear that the conductivity of the ideal crystal would be about 15 percent higher. However, the measurements of Powell [6] on beryllium oxide specimens (densities 1.85 to 2.82 g/cm³) would indicate a much larger correction. The authors feel that the correction for porosity is uncertain and that there are other factors beside porosity that also should be accounted for. One of these factors is the degree of bonding of the individual particles by the firing process.

7. Comparison With Other Results

Figure 3 gives a comparison of the results of the NBS measurements on BeO with the results of measurements at other laboratories on other samples. At the lower temperatures, the agreement with Scholes [7] is probably as good as the physical states of the two samples permit. Scholes used a sample having a density of about 2.97 g/cm³ as compared with the NBS sample having a density of 2.62. At higher temperatures, the results of Francl and Kingery [5] are consistently higher than the NBS results. They used a sample having a density of 2.86 g/cm³, but this would probably account for only a small part of the difference. The results of

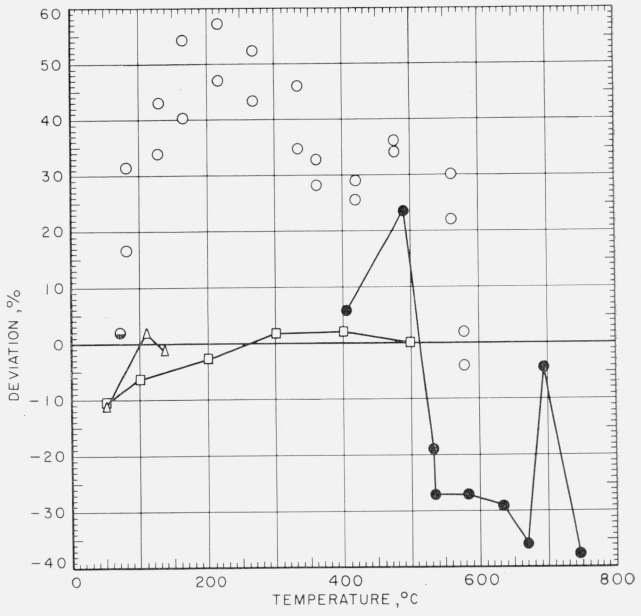

FIGURE 3. *Comparison of beryllium oxide thermal-conductivity data.*

Base line=NBS smoothed data.
△, Scholes (1950); ○, Francl and Kingery (1954); ●, Adams (1954); □, Powell (1954); ◒, Weeks and Seifert (1953).

Adams [8] are lower than the NBS results at higher temperatures, even though the samples had about the same densities (2.7 as compared to 2.62). Powell [6] measured the thermal conductivities of several samples of BeO having densities ranging from 1.85 to 2.82. His results, shown in figure 3, are interpolated for a density of 2.62 to compare with the NBS results; in general, the agreement is very good. Weeks and Seifert [9] determined the conductivity of a sample of BeO (density 3.0) at 70° C. Their agreement with the NBS value is probably better than the differences in the samples warrant.

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