NATIONAL BUREAU OF STANDARDS REPORT

9561

THERMAL CONDUCTIVITY OF A SPECIMEN OF PYROCERAM CODE 9606

by

D. R. Flynn
Environmental Engineering Section
Building Research Division
Institute for Applied Technology

Report to

General Electric Company
Spacecraft Department

NBS

U.S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS

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1. INTRODUCTION

This report presents results of thermal conductivity measurements in the temperature range 100 to 330 °K on a specimen of Pyroceram Code 9606 [1] which was fabricated from material cut from the same stock as, and adjacent to, a sample which was sent to General Electric Company, Spacecraft Department, Valley Forge Space Technology Center, Goddard Boulevard, King of Prussia, Pennsylvania

Pyroceram is the trademark used by Corning Glass Works for their family of fine-grained crystalline materials, made from nucleated glass by controlled crystallization. Pyroceram Code 9606 is available commercially and has been used mainly for missile radomes. Pyroceram Code 9606 is not the material used in making the heat-resistant utensils sold under the trade-name of Corning Ware. These are made from Pyroceram Code 9608, which has a different composition.

Some of the properties of Pyroceram Code 9606 and the criteria which have led to its selection as a candidate material for use as a thermal conductivity reference standard have been discussed by Flynn, Robinson, and Martz [1][2].

2. SAMPLE

Several years ago the National Bureau of Standards purchased seven 18-inch-diameter disks about 2-1/4 inch thick from Corning Glass Works for examination as potential thermal conductivity reference material. All disks were poured from a single melt, and were inspected at the factory by one of us for bubbles, seeds, or striations. In general, the disks were very free of flaws. There was a group of bubbles near the center of each disk, where the glass had been poured into the mold; in addition, there were a few bubbles located in other regions of the disks. The locations of all bubbles that could be seen were marked with a china-marking

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[1] A trademark of Corning Glass Works, Corning, New York.

[2] References appear under the heading 6. REFERENCES

pencil that would leave a permanent mark after ceramming. Photographs were also made of each disk. Examination of the unceramed disks under crossed polaroids indicated the existence of strains in the region within one or two inches of the edge of the disks. Since ceramming is done at temperatures above the annealing temperature of the glass, the strains were probably removed during the ceramming process. After completion of visual inspection, the disks were individually cerammed according to Corning's "Schedule B."

A total of thirteen disks were originally cast. These were labelled A through M, in consecutive order of pouring, by NBS. Several disks were broken at Corning, either mechanically or during the ceramming heat treatment. The disks received at NBS were those identified as B, E, G, J, K, L, and M. The specimen used in the present investigation was fabricated from material cut from the disk identified at NBS as G. No characterization measurements have been made yet on this lot of material.

3. TEST APPARATUS AND METHOD

The thermal conductivity of the specimen was measured by a steady-state longitudinal heat flow method in which the thermal conductivity was computed from the measured power input to a heater at one end of the specimen, the measured longitudinal temperature distribution along the sample, the measured thermocouple separation, and the cross-sectional area.

The apparatus used for these measurements is shown schematically in figure 1.

The specimen (A) was fastened to a heavy copper "cup" (C) which was thermally connected by three brass legs (E) to a liquid-nitrogen-cooled copper heat sink (F). A heater (I) wound around the copper cup (C) was used, in conjunction with a thermopile and controller, to maintain the copper cup at any temperature from about 90 to 330 °K.

A copper block (B) with an internal heater winding provided the measured heat flow down the specimen. The specimen was held between thin copper caps (shown in black) fastened to the variable-temperature cup (C) and the heater block (B). In order to achieve good thermal contact a eutectic alloy of gallium and indium (freezing point 15.7 °C) was used in the interfaces between the specimen and the thin copper caps. This assembly was pressed together by means of a loading screw, spring, and stainless steel spider, as shown at the top of figure 1. The spring was necessary to accomodate differential thermal expansion; it also provided considerable resistance to heat loss from the heater block.

A copper guard cap (H) and radiation shields (G) surrounded the specimen and heater block. Using the heater (J) on the guard cap, in conjunction with a thermopile and controller, the temperature of the guard cap (H) was automatically controlled to closely match the temperature of the heater block (B). The space between the specimen and the guard cap and radiation shield was packed with glass wool to minimize radiative heat losses. The entire apparatus was contained in a bell jar which was evacuated to less than 10^{-5} Torr to further reduce heat losses from the specimen or heater block.

Current leads from the specimen heater were thermally grounded to both the heater block and the guard cap so as to minimize conduction of heat along the leads. Because the temperature of the guard cap matched quite closely that of the heater block, the current leads to the specimen heater could be made large enough for heat generation in these leads to be negligible.

The temperature distribution along the specimen was measured by three butt-welded thermocouples pressed into transverse slits in the convex surface of the specimen. Additional thermocouples, designated by X in figure 1, were used to monitor other temperatures in the apparatus. All thermocouples were fabricated from 0.005 in. copper and constantan wire which had been calibrated against another lot of wire which had been calibrated by the NBS Temperature Section. Care was taken to thermally temper all thermocouple leads by keeping them in an isothermal region for several cm from the junctions. All thermocouple leads were wrapped around and cemented to a copper tempering block (D) to prevent heat from the room from being conducted to the measuring junctions. The thermocouple leads passed through epoxy vacuum seals to individual ice junctions from which copper wires led to the emf measuring circuitry.

Power to the specimen heater was provided by a regulated dc power supply operating in a constant voltage mode. The power input was determined by measuring the current through the heater, using a standard resistor, and the voltage drop across potential taps to the heater, using a high-resistance voltbox. Voltages from the standard resistor and the voltbox were read to five significant figures using a precision potentiometer. The emfs of the specimen thermocouples were read to 0.1μV. The emfs of other thermocouples were read to 1μV.

Each data point was obtained from two tests at essentially the same mean temperature but with different power inputs to the specimen heater in order to minimize systematic errors, especially in temperature measurement.

The thermal equilibriums obtained were such that the specimen temperatures did not drift more than 0.01 deg K over a half-hour period.

Several tests were run with a rather large temperature difference between the specimen heater block and the guard cap in order to evaluate the thermal conductance between these regions. Most of this conductance was due to the screw-spring-spider arrangement used to press the heater against the specimen. Using the thermal conductance values thus obtained, all tests were corrected for heat exchanges due to small temperature differences between the specimen heater block and the guard cap.

4. RESULTS

Thermal conductivity values were computed using the uppermost and central thermocouples on this specimen and also using the central and lowermost thermocouples on the specimen. The data involving the lowermost thermocouple were somewhat erratic. Therefore, only the thermal conductivity values computed from the uppermost and central specimen thermocouples are presented in this report.

The results of the thermal conductivity determinations are shown in figure 2 where the circles represent the data points and the solid curve is the fourth-order polynomial of least-squares fit to these data. Smoothed values computed from this polynomial are given in table 1.

Table 1. Smoothed Thermal Conductivity Values for a Specimen of Pyroceram Code 9606.

Temperature ° K	Thermal Conductivity W/cm deg	Temperature ° K	Thermal Conductivity W/cm deg
100	0.0593	240	0.0468
120	0.0590	260	0.0448
140	0.0578	280	0.0431
160	0.0560	300	0.0418
180	0.0538	320	0.0408
200	0.0514	330	0.0405
220	0.0490		

5. DISCUSSION OF RESULTS

The individual values of thermal conductivity plotted in figure 2 show only moderate scattering from the smooth curve. The extreme departure of an individual value of thermal conductivity from the smooth curve is less than one percent. The standard percent deviation of the data from the smooth curve is less than one-half percent. The uncertainty in the smoothed thermal conductivity values is believed to be not more than two percent.

The dashed curve shown in figure 2 represents the data reported by Robinson and Flynn [2] on a specimen of Pyroceram Code 9606 from a different lot of material. These data, which were obtained using a different apparatus from that used in the present investigation, are estimated to be uncertain by not more than three percent. Thus the differences between the two curves shown in figure 2 could be entirely due to experimental errors. However, it is felt that there probably is a real difference between the thermal conductivity of these two samples.

6. REFERENCES

[1] D. R. Flynn, H. E. Robinson, and I. L. Martz, Present status of Pyroceram Code 9606 as a thermal conductivity reference standard, Proceedings of the Fourth Conference on Thermal Conductivity, sponsored by U. S. Naval Radiological Defense Laboratory, October, 1964, paper I-F.

[2] H. E. Robinson and D. R. Flynn, The current status of thermal conductivity reference standards at the National Bureau of Standards, Proceedings of the Third Conference on Thermal Conductivity, sponsored by Oak Ridge National Laboratory, October, 1963, p 308 (also reprinted as NBS Report 8300).

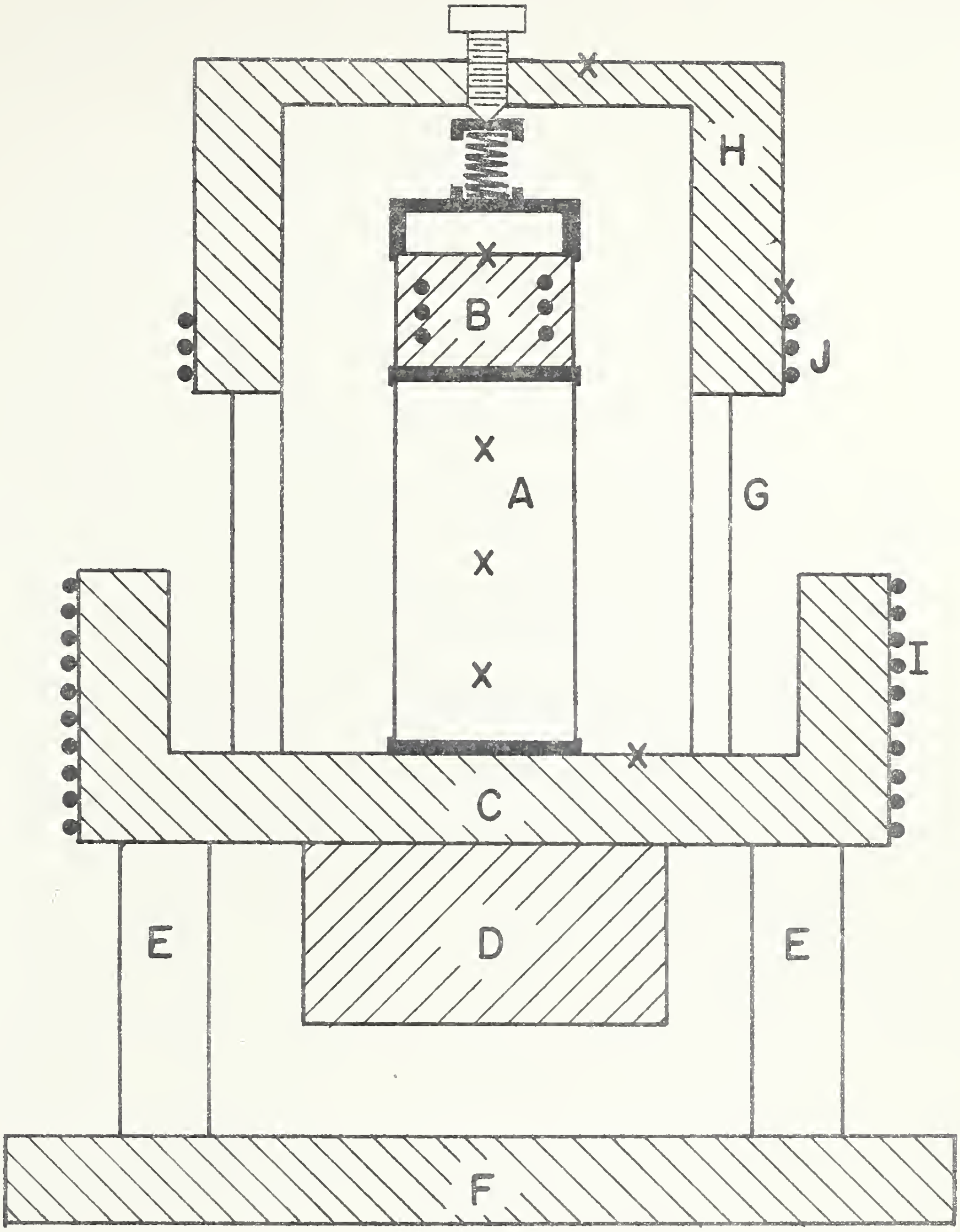

Figure 1. The apparatus used for the thermal conductivity measurements.

A. Specimen
B. Heater block
C. Variable-temperature copper cup
D. Thermocouple-tempering block
E. Legs used as heat leaks
F. Liquid-nitrogen-cooled heat sink
G. Radiation shields
H. Guard cap
I. Heater
J. Heater

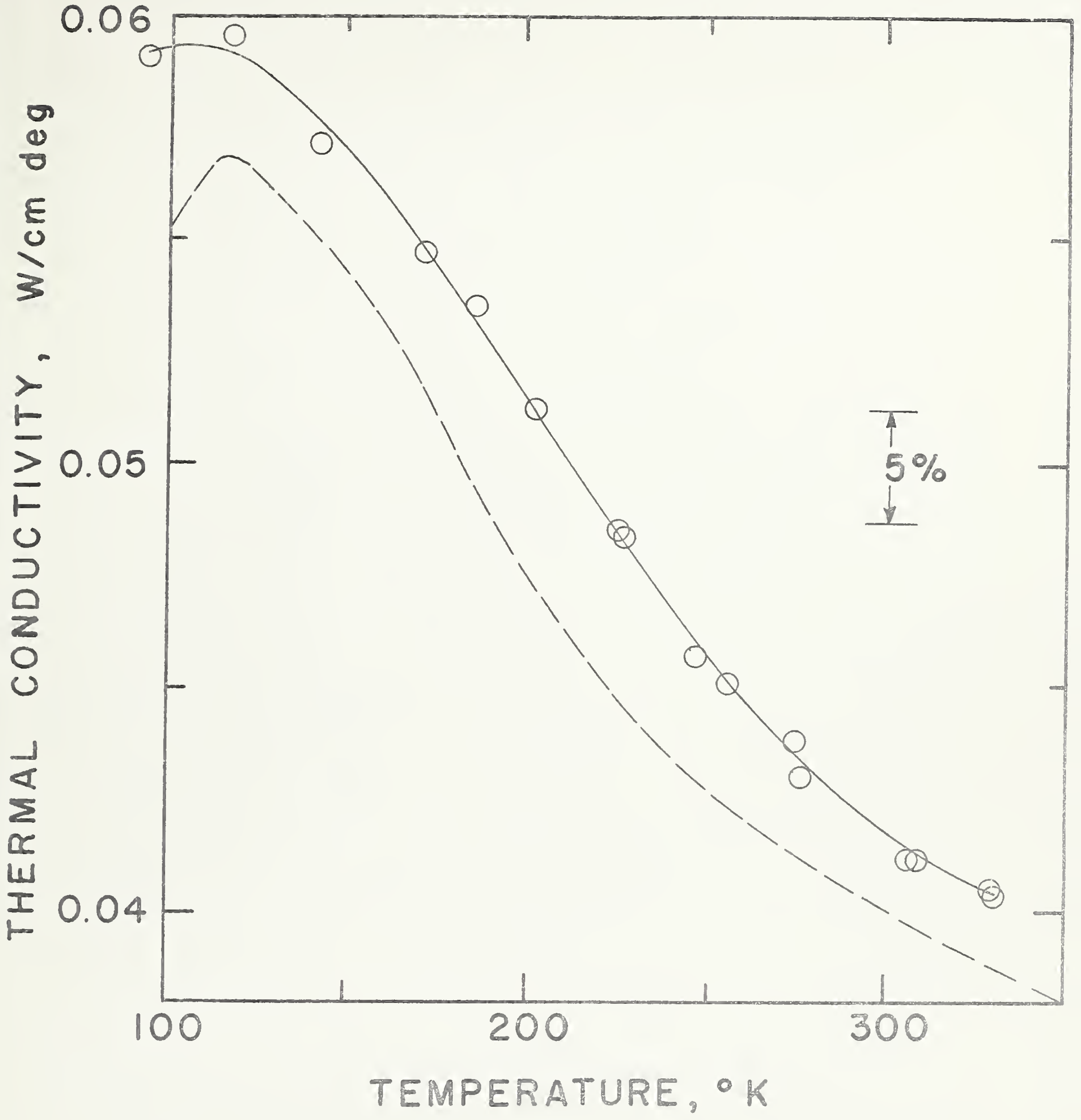

Figure 2. Thermal conductivity of Pyroceram Code 9606. The circles represent the data points computed using temperatures measured by the uppermost and central specimen thermocouples. The solid curve represents the fourth-order polynomial of least-squares fit to these data. The dashed curve represents the data of Robinson and Flynn [2]

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