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Technical Support Package

Measuring Thermal Conductivity of a Small Insulation Sample

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Technical Support Package

for

MEASURING THERMAL CONDUCTIVITY OF A SMALL INSULATION SAMPLE

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Measuring Thermal Conductivity of a Small Insulation Sample

Brief Abstract

A relatively simple device is described for directly measuring the thermal conductivity of very small test specimens that have very low thermal conductivities, on the order of the thermal conductivity of air. The size of the specimens whose thermal conductivity can be accurately measured using this device is significantly smaller than the size of test specimens used in other devices for the direct measurement of thermal conductivity. The device described in this disclosure will allow researchers who are developing new insulating materials to accurately assess the thermal conductivity of their new materials using small research-laboratory scale specimens.

Section I — Description of the Problem

General description of problem: Thermal conductivity is an important physical quantity that is often difficult to measure accurately. This is especially true when the conductivity of the material of interest is very low (i.e., comparable to that of air) and the problem is made even more difficult when small test specimens are used. On the other hand, researchers developing new highly insulating materials often wish, for a variety of reasons, to make specimens in the laboratory that are small in size. This often forces them to use indirect and possibly less accurate techniques to assess thermal conductivity, such as flash diffusivity. While flash diffusivity is very well suited for small specimens, it is an indirect method that is believed not to give accurate results for typical insulating materials that have a large radiative component to the thermal conductivity. Also, in order to convert thermal diffusivity to thermal conductivity additional accurate measurements of the density and heat capacity of the test specimen are required.

Typical direct methods require very large specimens, tens of centimeters or even up to a meter in size. There was one device reported in the literature in 1997 that was designed for 1-cm-sized specimens. However, this design was very complex and there is no evidence of further development of that.

Section II — Technical Description

Purpose and description: This invention is a device that allows relatively rapid, precise and accurate direct measurement of the thermal conductivity of highly insulating materials that have been formed into discs having a cross sectional area that is considered to be unfavorably small for such measurements.

Identification of component parts: The device described herein consists of an electric resistance heated copper disc — which in this specific case has dimensions of 1 in. diameter by 3/16-in. thick — and a second identically sized copper disc that is cooled using chilled water. Both opposing surfaces of the two copper discs are polished to a mirror finish using 4000 grit abrasive paper. A guard ring made from a strong, insulating foam known as Rohacell™ is placed between the heater and chiller discs. In this specific case, the ring dimensions are 1 in. outside diameter (or larger) / 0.75 in. inside diameter by 0.159 in. (4 mm) thick. The center of the Rohacell ring forms the test volume. The size of the test volume is

0.75 in. diameter by 0.159 in. thick. Matched thermocouples are used to measure the heater and chiller disc temperatures and the wall temperature.

The heater and chiller discs are each affixed to a larger disc of Rohacell that is approx 2.8 inch in diameter by 1.5 inch thick. Three holes are drilled near the edge of both of these Rohacell discs to accommodate Nylon threaded rods that are used to provide a light clamping pressure on the heater disc / sample / chiller disc assembly. A spring on each threaded rod helps to provide a uniform, gentle clamping pressure when clamped together. The assembly consisting of the two large Rohacell discs, the heater and chiller discs, and the Rohacell ring/sample volume when clamped together shall be referred to herein as the test assembly.

The test assembly is placed inside of an aluminum cylinder of approximate dimensions 10 in. long by 8 in. diameter by 1/8 in. thick. Copper cooling coils fed with water accurately maintained at a set temperature — typically 25 °C — are wrapped around the aluminum tube. The open ends of the aluminum tube are closed using foam and balsa wood and chill plates fed with the same source of 25 °C water that was discussed above that fed the chillplates.

One advantage of constructing a device with cylindrical symmetry is that it facilitates the use of efficient axisymmetric finite difference modeling. This modeling was essential for optimum design of this device and for understanding its operation.

A box constructed from an insulating material (such as corrugated cardboard) of size somewhat larger than the aluminum cylinder is placed over the aluminum cylinder. This produces a region around the cylinder, which is also maintained essentially at the set temperature (i.e., typically 25 °C). Future improvements will use a box where all six sides are formed from chill plates.

A second, larger box composed of an insulating material is placed over the first box. Dry air that is also of temperature essentially equal to the wall temperature (25 °C) is fed into the outer box.

A power supply provides the power to the heater disc. Two chillers are employed. One is set to 25 °C for the wall temperature and the other supplies chilled water to the chiller disc. Water-to-air heat exchangers (which may be referred to as radiators) are place in front of the air intakes for each chiller. These heat exchangers are cooled using the 25 °C chiller water. A pump built into the 25 °C chiller is used to flow the water through the aluminum cylinder and the cylinder-end chill plates. A small hobby pump provides the flow to the heat exchangers, and a peristaltic metering pump provides the flow of the chilled water to the chilled disc of the test assembly.

Finally, the room in which the device sits must have low humidity such that its dew point is less than the temperature of the low temperature chiller. Also, the temperature of the room should be controlled to within a few degrees.

Functional operation: The operation of this device involves fixing the wall temperature of the aluminum

cylinder to a value such as 25 °C and setting the heater and chiller disc temperatures to values that are preferably equally above and below the wall temperature. Typical values are 35 °C and 15 °C, respectively. Modeling shows that this symmetry in temperature combined with the symmetry in geometry leads to the greatest accuracy. This is because there is essentially no net gain or loss of heat from the edge of the guard ring when these conditions are met. The power required to bring the heater disc up to the desired temperature is recorded and the portion of the power that does not participate in the heating of the specimen is subtracted from the total power. The approach of subtracting off the power that does not participate in the 1D heating the specimen is fundamentally different from other approaches for this type of measurement. With most other approaches an attempt is made to eliminate the flow of all of the heat that does not go into the specimen and to correct for all heat leakages due to the imperfections in design.

With the present approach, the heat losses are determined via calibration using a test specimen of known thermal conductivity. While there are no suitable small, low conductivity calibration specimens available, the geometry of the device described herein allows the unique opportunity to use air as a calibration standard. The other hot plate thermal conductivity measuring devices that are available employ a significantly larger specimen volume and, because of that large size, air is not suitable as a calibration standard because a significant portion of the heat transfer will occur via convection rolls. However, it is well know that convection is not a major factor in the transfer for very small sample volumes of air. For the geometry of the present device, Finite volume modeling backed up by Grashof number correlations has shown that there is very little heat transfer through air via convection and that this very small difference is predictable so that it may be applied as a correction factor. Small corrections must also be made for heat transfer via radiation. However, it can be readily shown that radiative heat transfer between the two parallel discs can be minimized and accurately modeled if the copper discs are polished to a mirror finish.

The temperatures described above for the chilled disc, the wall, and the heated disc (15, 25, and 35 °C, respectively) are targets. However in practice it is not possible to precisely attain these temperatures experimentally. Therefore, the calibration must be performed over a range of temperatures about each target value. The temperature ranges may be kept rather narrow: viz: 25 ±0.2 °C and 15 or 35 ±0.5 °C. Statistical analysis of theoretical heat losses obtained from finite volume modeling of the experimental setup shows that it is feasible to express the heat loss in terms of a polynomial that is first order in terms of two parameters. The two parameters are (1) the difference in temperature between the hot disc and chilled disc and (2) the difference between the wall temperature and the average between the heater and chiller disc temperatures.

Once the equation for the heat loss correction has been determined, the conductivity of an unknown specimen may be determined. Before measuring the conductivity of an unknown specimen it first must be machined to 0.159-in. thickness or slightly less and preferably to 0.75-in. diameter. If the diameter is somewhat less that 0.75 in., it is possible to scale the conductivity assuming that the unoccupied space is filled with still air.

Alternate embodiments of the innovation: Alternate embodiments include geometrical dimensions, which are different from those described above. However, in order to continue using air as a calibration standard, care must be taken not to increase the sample dimensions to the extent where convection can begin to contribute significantly to the transfer of heat through air. Different materials of construction may be employed A different set of temperatures from those described above may be employed and different approaches to heat and or cool the discs and wall may be used.

The thermocouples may be replaced with differential thermocouples, which would directly read the required temperature differences, or RTDs may be used to measure temperature.

The copper discs may be coated with highly reflective coatings and further coated with dielectric coatings of the type used for copper-based mirrors for high power lasers.

An alternative configuration would involve the use of two chilled copper discs, one on each side of the heater disc. Two preferably identical samples, each having the Rohacell guard ring, would be tested at the same time. This configuration would result in an increase in the percentage of the electrical power that went into heating the specimens with the disadvantage of requiring two specimens and an increase in complexity.

Another alternative would be to use a heat flow meter design where suitable gauges are used to calibrate the heat flux along the central axis of the test assembly. Such a configuration may be less sensitive to test specimen diameter.

Different test atmospheres other than air could also be used.

Supportive theory: The theory of one-dimensional steady state heat transfer across an object with parallel major surfaces is well known and deceptively simple. The equation for 1D heat transfer is

$$Q = k/l\ A\ dT$$

In the above expression Q is the power, which in this case is provided by the electric resistance heating. The term l is the thickness of the disc, A is its cross sectional area, and dT is the temperature difference across the two major surfaces of the disc. The term k is the thermal conductivity.

The challenging tasks in thermal conductivity measurements involve setting up the geometry of the experiment so as to obtain essentially 1D heat flow across the test specimen and to block or, in this case, account for that portion of the heat that flows to locations other than across the major surfaces of the specimen.

With other approaches for steady state thermal conductivity measurement, an attempt is made to block the flow of heat into locations other than the test specimen by the use of guard heaters, which are set to precisely match the temperatures of the heater disc/specimen/cooler disc assembly. This type of technique is described in ASTM C177 "Guarded Hot Plate Apparatus for Thermal Conductivity". This test configuration also allows the heat flow across the specimen to be one-dimensional. In reality, these

experimental test configurations are imperfect and so corrections must be applied to account for these imperfections. For small test specimens of the type discussed herein these corrections are subject to grater errors that expected for the more usual larger specimens. There is one case in the literature where an attempt was made to develop a guarded-hot-plate device for very low conductivity, small diameter specimens. Preliminary results were presented in two papers from the late 1990's. See DR Flynn and R Gorthala in Insulation Materials: Testing and Applications, 3rd Volume, STP1320-EB 1997 p 337. For the apparatus described in this disclosure, approximately 1D heat flow across the specimen is obtained by using a specimen which is smaller in diameter than the heater and cooler discs. A ring formed from a strong low conductivity structural foam is placed around the test specimen. This ring has an internal diameter equal to the specimen diameter and an external diameter equal to or preferably larger than the diameter of the heater and chiller discs. Making the test specimen smaller than the heater disc sets up essentially 1D heat flow across the specimen. The finite difference models showed that this is essentially true. The ring is glued to one of the copper discs using a spray adhesive that according to its manufacturer does not penetrate into the porosity of the foam ring. This ring has the added advantages of protecting delicate test specimens from crushing. The ring also facilitates and simplifies specimen alignment. The heat that flows across the ring during an experiment is added in with the other sources of "lost heat" Another important result from the CFD modeling is the finding that the heat losses are unaffected by the thermal conductivity of the test specimen. Therefore it is valid to apply the heat losses determined from the calibration to test specimens of arbitrary conductivity.

Peripheral equipment: The peripheral equipment consists of a power supply, two chillers, a data logger, and a supply of clean dry room temperature air.

Maintenance, reliability, safety factors: The primary maintenance required is to polish the opposing surfaces of the heated and chilled copper discs, using 4000 grit polishing paper, as needed. There are no safety concerns.

Section III — Unique or Novel Features

Novel or unique features: The device described herein is unique in terms of its ability to accurately measure, using a steady state approach, the thermal conductivity of very small specimens, which have very low thermal conductivity — i.e., thermal conductivity on the order of that of air. Also, it differs from other steady state hot-plate devices in that no attempt is made to block the flow of heat in all directions other than into the test specimen. Rather than attempting to block the flow of this "lost heat" it is instead accurately accounted for using a calibrated test sample. Because of the small dimensions, this device is able to use air as a calibration standard as discussed above.

Advantages of the innovation: The applicability of this device to small specimens is a great advantage to developers of insulating materials who wish to prepare small batches of experimental material. Another advantage is that this device offers a relatively simple and relatively fast way to assess steady state thermal conductivities.

Test data and source of error: The experimental determination of the heat losses as a function of the set temperatures showed that the heat losses could be very precisely determined. The results of 8 measurements showed that the heat losses could be determined to a prediction interval of no greater than ±0.00021 watt, which is about ±0.15% of the typical value. The device was disassembled, then reassembled and the measurement was repeated. A result that was within the prediction interval was attained.

As discussed, above matched thermocouples were used for the three critical temperature measurements. The high precision was made possible because the temperatures were the average of at least 4000 sets of temperatures collected over an approximately 1.5-hour time span.

The impressive precision described in the above paragraph is attainable even though the data logger that was used measured to only ±0.1 °C. The averaging over 4000 points made this possible. It is interesting to note that for this approach to work the power supply must be sufficiently noisy so as to output an essentially Gaussian distribution of power about the nominal value.

Analysis of capabilities: As discussed above, the heat losses may be determined to with about ±0.15%. The resulting precision in determining the heat flow across the test specimen varies with the thermal conductivity of the test specimen. Specimens having higher thermal conductivity will require more heat to set up the desired temperature gradient. Since the lost heat is essentially constant, the fraction of heat flowing across a test specimen will be greater if its conductivity is greater. Therefore the precision of the test is related to the conductivity of the test specimen. For example for specimens having thermal conductivities of 0.5 that of air (0.013 W/m-K) mW and 1.5 that of air (0.039 W /m-K) mW the fraction of heat flow across the specimen is 12 and 28%, respectively. As a result, the precision of the measurement of thermal conductivity based on the errors in the determination of the lost heat would become about ±1.3% and ±0.5%, respectively. In reality other errors, such as errors in measuring specimen geometry would lead to somewhat greater conductivity measurement error.