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Home-built Apparatus for Measuring Thermal Conductivity of Glass and Polymer Materials

William R. Heffner[1] Shera Demchak[2], John Scruggs[3] and Ray Pearson[4]
[1]International Materials Institute for New Functionality in Glass, Lehigh University, Bethlehem, PA 18015, USA
[2]University of Massachusetts, Amherst, MA 01003, USA
[3]Tuskegee University, Tuskegee, AL 36088, USA
[4]Material Science & Engineering, Lehigh University, Bethlehem, PA 18015, USA

ABSTRACT

As a part of the IMI-NFG's series of low-cost experiments in glass science [1,2] we have developed a simple home-built apparatus for measuring the thermal conductivity of glassy materials, from polymers to oxide glasses, in the range of 0.1 to 1.5 W/ °C. Our apparatus is inexpensive, relatively easy to construct and accurate enough for students to use for quantitative measurements of their own glass or polymer samples. Standard materials are used to demonstrate good correlation with literature values. We also measured the thermal conductivity of a silica filled epoxy and showed a linear increase with fill fraction to 20%. This simple, low-cost method can provide students and researchers with a much broader access to this important property.

INTRODUCTION

Thermal conductivity (K) is an important property in the materials selection for many applications. Approximate K values for common engineering materials are widely available. However, for new materials, special formulations or composites, these data will not be available. This is especially true for polymers and inorganic glasses, where the compositions can be adjusted over wide ranges and the physical properties may also dependent on processing parameters. Filled polymer composites constitute another important class of materials where the K can be tailored over a wide range based on fill composition.
Standard measurement approaches for K, such as Modulated DSC [3, 4] or commercial Thermal Conductivity Meters[5], are expensive and generally not available outside of the specialized research lab. We present here a simple, home-built apparatus for the low budget exploration of thermal conductivity in polymers and glasses.

EXPERIMENTAL

The Low-Cost Student-Built Thermal Analysis Apparatus

Our apparatus is based on a straight forward application of the definition of thermal conductivity (K) as the ratio of the heat flow (Q) through a sample relative to the temperature difference across the sample (ΔT_{sample}), appropriately scaled by a sample geometry factor (thickness (t) divided by the area (A)), as illustrated in Figure 1. It is relatively simple to construct, consisting of a heated aluminum block placed on the top side of a flat sample, and a two-tiered pedestal base on the cold side. The pedestal allows an independent measurement of actual heat flow through the sample. Two embedded resistors (1/2 watt, 47 Ω in parallel with 5 v supply) provide

about one Watt of power to the top plate. The base pedestal provides a fixed, low thermal conduction heat path enabling us to establish a direct relationship between the heat flow (Q) through the pedestal and the measured temperature difference across the pedestal ($\Delta T_{pedestal}$).

$$Q = \alpha \, \Delta T_{pedestal}$$

The proportionality constant α is determined empirically in a separate calibration step.

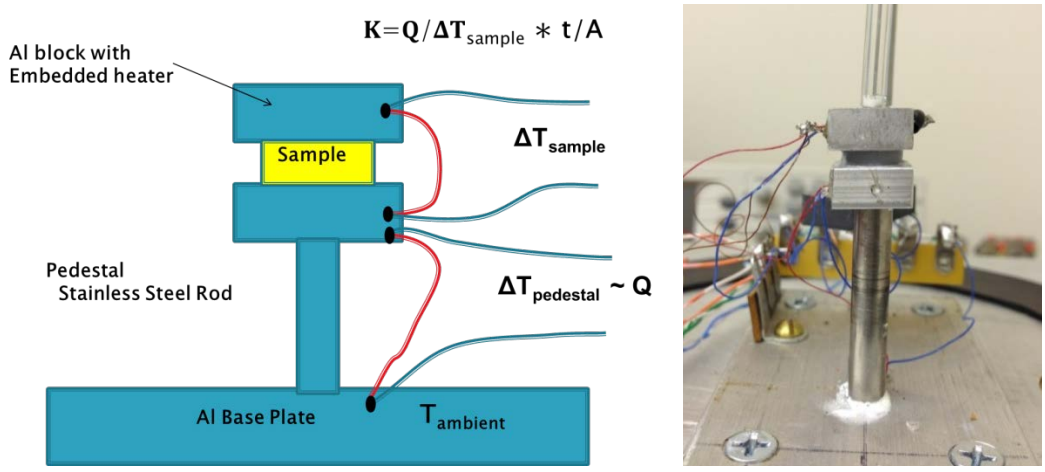

Figure 1: Sketch of the thermal conductivity apparatus. Top plate is heated by two embedded resistors delivering about one Watt of power. A hollow glass rod (not shown on sketch) provides pressure to the sample. An example of the actual device is shown on right. The width of the Aluminum base is 2 inches.

To differential Copper/Constantan thermocouple (TC) pairs are used to sense the temperature difference across the sample (ΔT_{sample}) and the temperature difference across the base pedestal ($\Delta T_{pedestal}$). Thin (0.010 inch) thermocouple wires are used to minimize the thermal conductivity through the wires. Two inexpensive instrumentation amplifier ICs (INA122 < $5 ea.) provide the gain required to convert the small differential TC signals into a voltage range appropriate for low-cost data loggers (~0-5 V). See Figure 2(a). In our case a gain resistor (R_G) of 100 Ω was used to provide an overall TC gain of 2000. Our data acquisition unit was programmed to record both of the ΔT signals directly into an Excel spreadsheet for real time monitoring.
Thermal grease is applied to both sides of the flat samples for good thermal contact, and a long, hollow glass rod with an attached weight applies pressure from above. While the glass tube does provide an additional thermal path for the heat to escape from the top block, its length and lower thermal conductivity (compared to the pedestal) result in only minimal contribution. The effect of such stray heat paths (including air and wires) can be accommodated through a calibration with known values of K. A simple plastic cover is placed over apparatus to reduce air drafts. Calibration of the heat flow through pedestal to the associated $\Delta T_{pedestal}$ voltage is determined by placing the heater plate directly on the pedestal (with thermal grease) and then measuring the steady state $\Delta T_{pedestal}$ associated with a known power to the heaters. If we assume that all the heat flows out of the heated block through the pedestal then we have a direct means of calibrating the relationship of pedestal delta T to actual heat flow. Our results show that this additional heat loss is small and can indeed be included in an overall calibration.

ΔT vs time measurements

Figure 2(b) shows the amplified TC voltage across the sample and pedestal as a function of time after the heating block is powered on. The initial rise from room temperature to its stationary value takes about 15 minutes for the sample shown below, providing a sample measurement time of about twenty minutes. Once warmed up the equilibration time can be even shorter, and repeat measurements made more quickly. In Figure 2(b) we removed the sample at 40 minutes and replaced it to show the repeatability of both thermal contact and measurement.

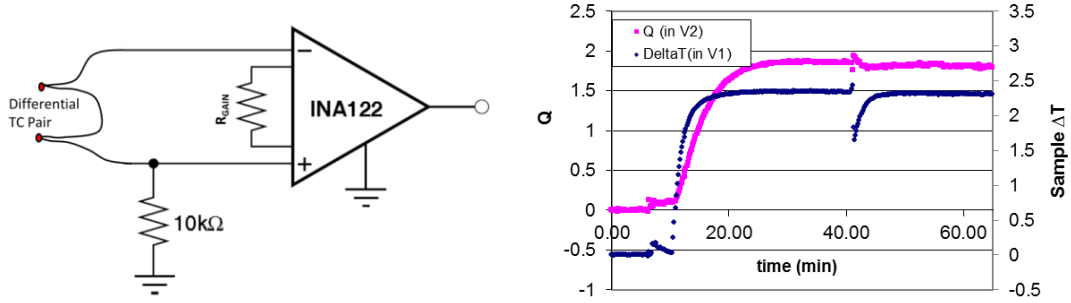

Figure 2. (a) Circuit diagram on left for the instrument amplifier (INA 122) used to provide the gain for the small differential thermocouple voltages. (b) On right, amplified voltages for ΔT_{sample} (blue) and $\Delta T_{pedestal}$ (purple) as a function of time as the heating block is powered on. Note that the scale is a voltage and not actual temperatures.

Samples and Preparation:

The apparatus requires flat samples and was designed to accommodate either a disc or rectangular sample no wider than about 1.2 cm across. Glass and plastic samples were obtained from sheets and rods of commercial material in the 2-3 mm thickness range. We chose Pyrex, high density poly ethylene (HDPE) and an acrylic material for our "standards" because of the availability of these materials and the consistency of their reported literature K values. The K values used cover a span from 0.18 (for acrylic sheet) to 1.1 W/ °C (for Pyrex), with HDPE providing a midpoint value (0.49). We should note that we only have approximate literature value ranges for our generic materials and the values listed here represent an average of the ranges found from multiple sources. All samples were polished enough to achieve a flat surface on both faces (to a smoothness of 300 grit).

RESULTS

Calibration with Known Materials

Figure 3(a) shows our measured K values for our "standard" samples of Pyrex, HDPE and acrylic plotted against their nominal literature values. Samples of approximately the same thickness (2-3 mm) and area (~1 cm^2) were used for this comparison. Teflon is also included to provide an additional, intermediate value (taken as 0.33 W/ °C from reference 4). Values for Teflon in the literature show a wide variation and thus we do not consider this sample as a calibration "standard", although it appears to fit our other data quite well. Good correlation is achieved for all four samples with our approximate literature values, with a slope of 1.15 and intercept of 0.10 W/ °C. The intercept is consistent with value obtained for a fiber glass sample (K ~ 0) of the same thickness. The standard error for our correlation is calculated as 0.04 W/°C.

Thermally Conductive Silica-filled Epoxies

Our apparatus was used to measure the thermal conductivity of series of nano-silica filled epoxies from 0 to 20% fill (by weight). In Figure 3(b) we show the measured thermal conductivities as a function of percent silica. The data exhibit a linear dependence on the fill fraction and the regression line extrapolates to a value of about 0.97 at 100% fill factor, in good agreement with what might be expected for a silica based glass.

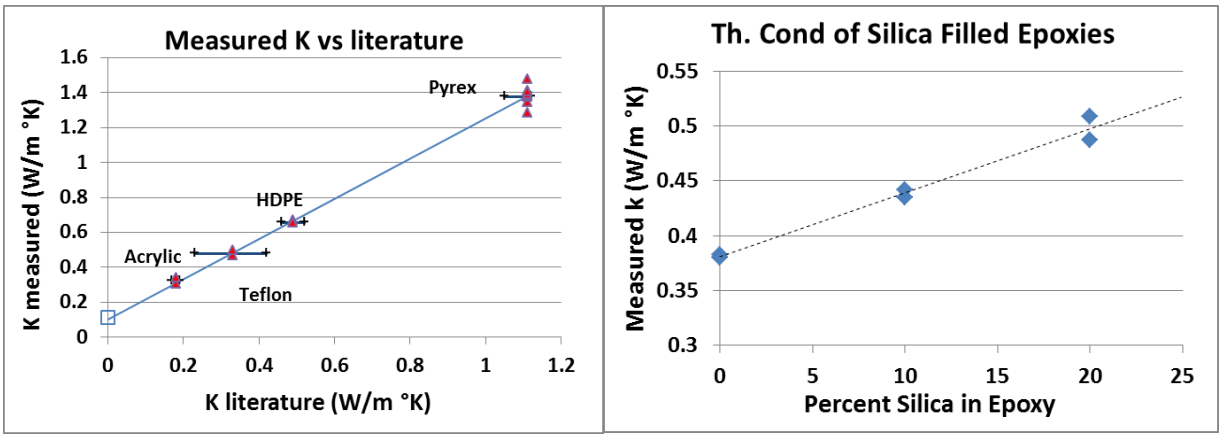

Figure 3. (a) On left the measured K for samples of Pyrex, HDPE, Teflon and PMMA samples are plotted vs. their nominal literature values. Horizontal error bars indicate spread in literature values found. We chose the most frequent, rather than a straight average. (b) On right the thermal conductivity of silica filled epoxies vs. the fill fraction.

Vacuum Measurements for Improved Accuracy of Calibration

In order to quantify and reduce the effect of stray heat loss through the surrounding air and thereby improve the direct calculation of K, we designed a home-built vacuum enclosure (for < $100 in parts, not including the pump) as shown in Figure 4. By simply calibrating the $\Delta T_{pedestal}$ to Q while under vacuum, we achieved a distinct improvement in accuracy of our calculated K values with the literature values.

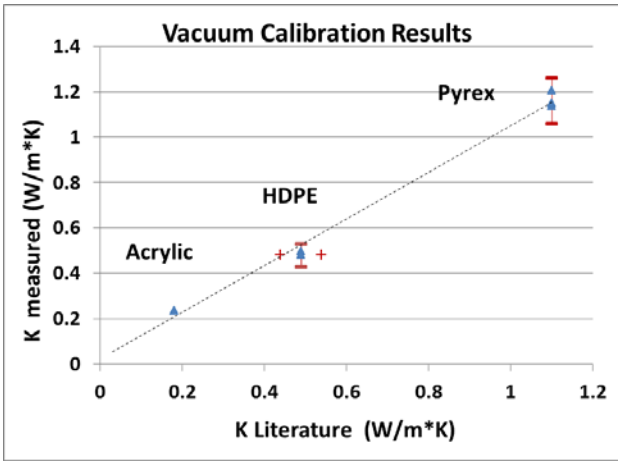

Figure 4. Home-built vacuum chamber on left and improved accuracy of correlation on right.

With the vacuum calibration factor (α) we achieve a near unity slope of 1.03 and a near-zero intercept of 0.02 W/°C. However, construction of the vacuum chamber introduces additional cost and complexity. Since correlation is excellent with or without the vacuum chamber, the benefit of the vacuum may not be considered necessary so long as a calibration with standards is performed.

CONCLUSIONS

We have developed an inexpensive instrument for exploring thermal conductivity of polymers and glasses. This apparatus can be built by the student or instructor, requiring only moderate machining skill and simple electronics. It is found to provide good correlation with literature values of the thermal conductivities and with excellent measurement precision, especially considering its low cost compared to the commercial alternatives. This new apparatus would be well suited for the undergraduate studies of thermal properties of polymer and glassy materials. The assembly has the added benefit of introducing the student to a variety of experimental design and construction techniques. For additional details and future updates please see the glass education page on our website at: http://www.lehigh.edu/imi/

ACKNOWLEDGMENTS

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REFERENCES:

1. William R. Heffner and Himanshu Jain, MRS Proceedings, 1233, (2009).
2. William R. Heffner and Himanshu Jain, “Low-cost, experimental curriculum in materials science using candy glass - Part 2: home-built apparatuses”, presented at 2013 Fall MRS Meeting, Boston (accepted for publication to MRS Proceedings).
3. ASTM Standard E1952, 2011, "Thermal Conductivity and Thermal Diffusivity by Modulated Temperature Differential Scanning Calorimetry," ASTM International, West Conshohocken, PA, 2011, wwwastm.org.
4. S.M. Marcus and R. L. Blaine, “Thermal Conductivity of polymers glasses and ceramics by modulated DSC”, Thermochimica Acta, 243, 231 (1994).
5. See for example the DTC-25 or DTC-300 Thermal Conductivity Meters from TA Instruments or their more expensive Flash Diffusivity Systems all described on their website at http://thermophysical.tainstruments.com/instruments/thermal-conductivity-meters/