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Low-cost, experimental curriculum in materials science using candy glass - Part 2: home-built apparatuses

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Low-Cost, Experimental Curriculum in Materials Science Using Candy Glass
Part 2: Home-Built Apparatuses

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ABSTRACT

We have been developing a collection of low-cost experiments for exploring the science of glassy materials through hands-on activities with sucrose based glass (a.k.a. hard candy). These form a mini-curriculum of glass science, consisting of inter-related experiments and home built apparatuses. It provides an environment to develop an understanding of glassy materials through active, prolonged engagement. Some of our earlier experiments were reported four years ago[1]. Since that report we have made substantial improvements and added new topics, including electrical and thermal conductivity, an improved DTA apparatus, and improved methodology for crystallization kinetics. All of our experiments are designed to be low-cost (typically <$100) and the apparatuses are designed for construction by students or teachers.

INTRODUCTION

Over the last decade US educators, policy makers and science advocates have become increasingly aware of the need to improve student achievement and interest in science, engineering and technology education [2]. They often attempt to promote the importance of science and engineering by emphasizing the potential opportunities within the “mega trends” and “big ideas” related to science, yet each new class of students seems to have less practical, experiential understanding of science. While our pre-college curriculum focuses more on standardized tests, and less on practical or hands-on experience, there is a strong and growing interest in “maker communities” [3] where young and old actively participate in hands-on science and engineering construction projects. Likewise, recent attention has begun to focus on both hands-on learning and the informal educational experience to the total educational experience of both student and adult learners [4].

These trends and our own experience suggest that building one’s own apparatuses and then performing experiments with them have an enormous power to spark enthusiasm and generate interest and intuition in science and engineering. Following this notion, we have developed a program to connect students with glass science through a series of hands-on activities centered on exploring the properties of glasses through sugar glasses, a.k.a. hard candy. This innocuous and easy to synthesize model glass provides an ideal vehicle for quantitative exploration of the material properties and behavior exhibited by both polymeric and commercial oxide glasses, but at much lower temperatures and with kitchen ingredients (sugar and corn syrup). Sugar glass serves as a common theme for our series of inter-related glass science experiments with home built apparatus to measure physical properties, constituting an experimental, mini-curriculum of glass science.

Several years ago we reported experiments on the synthesis, phase diagram, refractive index measurement, a fiber drawing tower, crystallization kinetics and a rudimentary DTA (differential thermal analyzer) [1]. Since that report we have made substantial additions and improvements to this collection, including experiments on electrical and thermal conductivity, improved DTA apparatus, and improved methodology and apparatus for studying crystallization kinetics. These new additions are described in this paper. All the modules are available and distributed through NSF's International Materials Institute for New Functionality in Glass (IMI-NFG) website at http://www.lehigh.edu/imi. The use of Internet allows us to include a variety of educational materials to support the learning experience, including tutorials, videos, project descriptions, student presentations and construction details. Likewise the website provides a means for reaching a large population of students and teachers, while providing a fast and flexible means to revise and add new content as it is developed.

NEW APPARATUS & EXPERIMENTAL ACTIVITIES

Improved Hot Stage for Crystallization and Devitrification Studies

Measuring the temperature dependent crystallization (or devitrification) rate from warmed sugar glass provides a simple yet quantitative demonstration of the crystal growth dynamics, including both nucleation and growth aspects, essential to understanding the nature of glass itself. The suggested sugar glass composition (2:1 sucrose to corn syrup ratio by weight) has its maximum crystallization rate at about 120º C [1], which is sufficiently high to produce a significant fraction of crystals within 30-60 minutes, consistent with a typical science laboratory period (1-3 hrs.). In our original implementation the sample "oven" consisted of a metal can heated by a light bulb, with a dimmer switch used to control the temperature. Whereas that approach was both simple and low cost, it had the disadvantage of poor temperature control, requiring a long time to reach stable temperatures and was subject to substantial temperature dips immediately after sample placement. Rapid temperature stabilization is essential for adopting the devitrification experiment into a standard laboratory time frame.

Fortunately, there are several low cost temperature controllers available on the market today. We mention two models. The simplest is the Autonics Analog Controller (TOS, available for under $40), although it has a load current limit of only 2 amperes. A step up is Autonics Digital Temperature Controller (TC3YT, $65), which has a digital display of the actual temperature and its control relay can handle up to 16 A, making it ideal for controlling via a standard hot plate heater. We use both of these devices to produce temperature controlled heating surfaces, large enough to hold multiple samples (typically, a drop of sugar glass between two 1x3" glass slides) at a time. An example of our home-built units is shown in Figure 1.

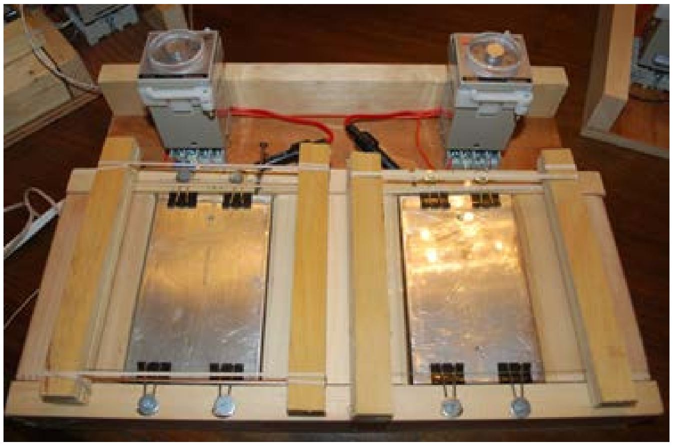

Figure 1. Dual temperature controlled sample “hot plate oven” made from 75 W strip heater ($30, McMaster-Carr) bolted to a ¼”x3”x5” aluminum plate and controlled with the Autonics TOS Analog controller. Samples are covered with a glass plate enclosure above the sample to simulate an oven (plate IS shown here).

Control with a ± 1° C temperature variation is typical with this apparatus. This and several related control options in our modules provide students with practice in making and using thermocouples and temperature control as well as the crystallization dynamics. Our website also contains other crystallization related experiments including moisture induced surface crystallization of sugar glass and thermally induced crystallization of a common glassy polymer, PET (polyethylene terephthalate).

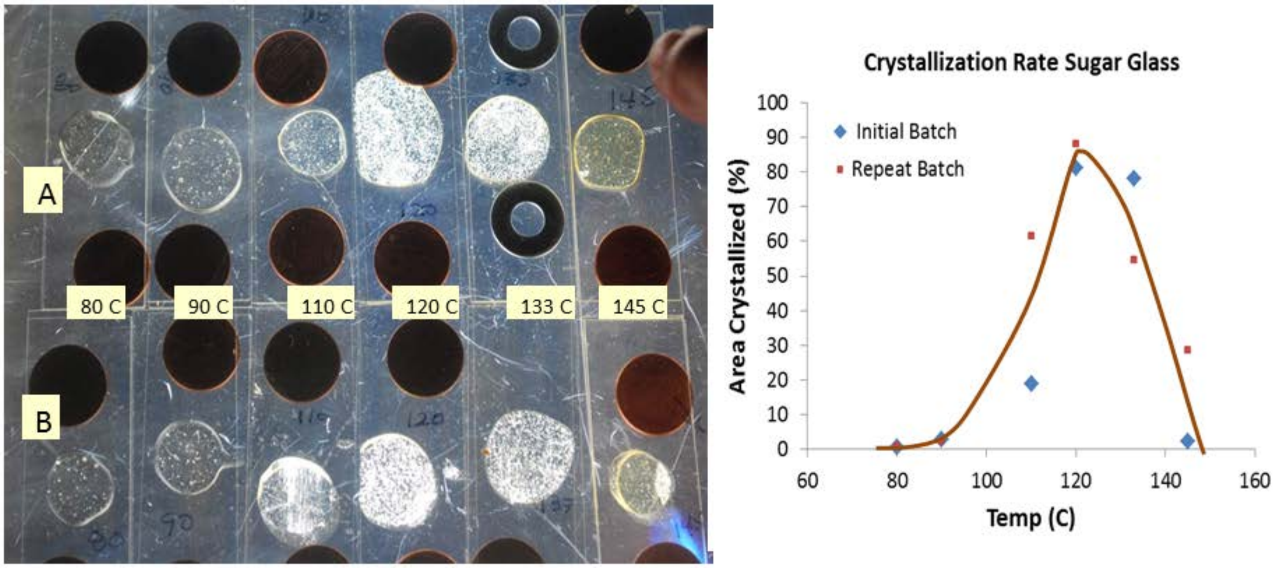

Figure 2. Example of results from a 2 hr. laboratory activity conducted with students at Tuskegee University. Note the growing crystals appear white against a dark background when viewed through crossed polarizers. ImageJ software [5] may be used to perform the analysis of fractional area crystallized.

Thermal Analysis and DTA

Any study of the glassy state would be incomplete without some consideration of the glass transition phenomenon. Whereas DSC (differential scanning calorimetry) is the common tool for measuring the glass transition and crystallization phenomena, such instruments are expensive and generally unavailable outside the dedicated research laboratory. To introduce basic thermal analysis to a wider range of students, we have developed a simple, home-built DTA apparatus, which provides essentially the same information as the DSC. Our DTA consists of monitoring the temperature difference between a glass sample and a reference material, placed in separate test tubes, while they are simultaneously heated in an oil bath. The oil bath consists of a beaker full of vegetable oil placed on a laboratory hot plate equipped with a magnetic stirring bar. The two test tubes are held in the oil bath by a simple wooden holder, and thermocouples are used to measure the bath temperature as well as the differential temperature between the two tubes. A sketch of the DTA is shown in Figure 3.

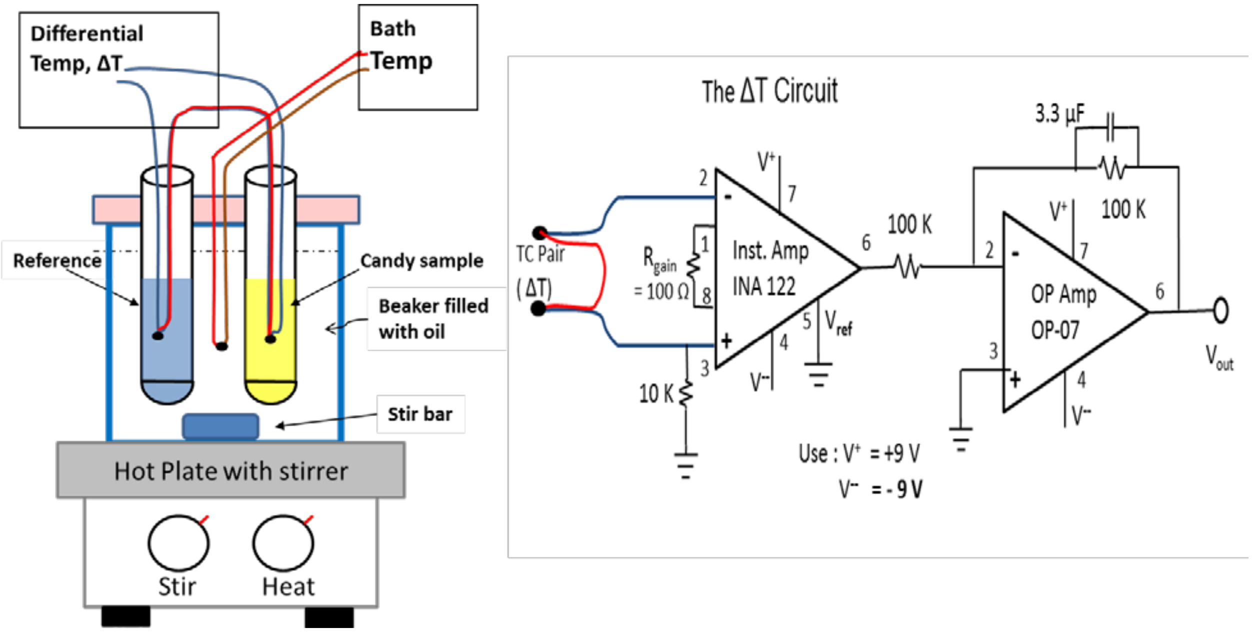

Figure 3. Sketch of the home-made DTA apparatus (left). The circuit used to measure the differential temperature is shown on the right.

For the differential TC pair, we make our own from a single piece of constantan wire soldered (PbSn is ok) at each end to two thin copper hookup wires (#24 gauge). Calibration data for this type T thermocouple can be obtained from standard tables. A low cost instrumentation amplifier IC (INA 122, ~ $5) provides the high gain (x 2000) needed to bring the small (microvolt) differential TC signals into a range reasonable for measurement with an ordinary DVM or data logger. The output is fed to a second filter stage to remove AC noise induced by the magnetic stirrer. This new circuit provides a substantial noise reduction in data over the previous apparatus [1]. The bath temperature is monitored using a standard type K thermocouple connected to an AD595 IC (cost ~ $15), The AD595 provides an internal, temperature compensated, ice point reference for the thermocouple and outputs a voltage calibrated directly to the temperature (at 10 mv. per ºC). The improved circuit for the differential voltage measurement is shown in Figure 3. Additional details on the construction are available at our website.

Computer assisted data acquisition is very useful, almost essential, for high quality capture of the various thermal events. For those experimenters who do not already have their own data logging platforms, we describe two very affordable options. The simplest, off-the-shelf option utilizes the DI-149 USB Data Acquisition unit (from Dataq Instruments at $59). A slightly more hands-on choice is based on Do-It Yourself (DIY) approach using a Basic Stamp microprocessor platform (the MoBo - from Parallax Inc. for under $100) [6]. Figure 4 shows results for the glass transition in a sugar glass recorded using this DTA apparatus. More examples were presented previously [7] and can be found on our website.

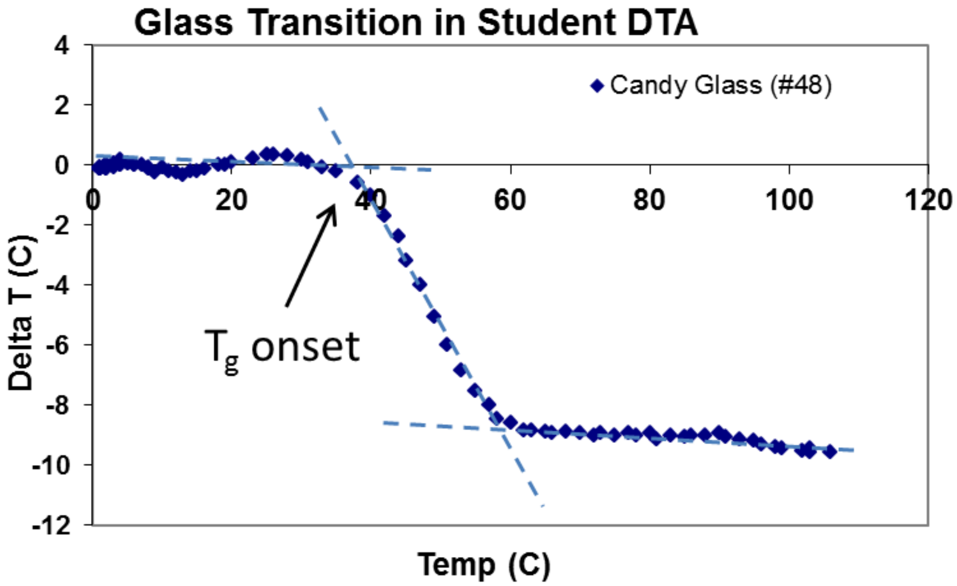

Figure 4. DTA trace for a sugar glass showing excellent Tg onset at 40º C. The sample and bath had been precooled in ice water for this run.

Electrical Conductivity of Glass and its Temperature Dependence

Common glasses are generally electrical insulators at room temperature, as are sugar glasses. The strong temperature dependence of electrical conductivity of glasses is of considerable at room temperature and engineers, as it determines the usefulness of glass in electrical devices, as well as reveals the nature of the amorphous structure and the transport of the conducting species [8]. Here sugar glass provides another opportunity to explore these issues and questions. However, measuring the extremely low currents associated with such insulating materials requires specialized equipment, such as an electrometer, not commonly available in a student laboratory. To facilitate hands-on engagement in this topic we have designed a simple, low-cost electrometer. It is based on an extremely high input impedance op amp IC (LM6081), capable of accurate current measurements in the sub pico amp range. Using this IC, a simple electrometer can be constructed for well under $100 (including all connectors and enclosure), one with sufficient accuracy to measure DC conduction in the 10^{-12} Siemens range. Specific details of such two-IC circuit were presented in a 2012 paper [9], and are archived on our website. Ultra-low current measurements are highly sensitive to unwanted stray currents, induced by surrounding electrical or magnetic fields, and good electrical shielding of the sample and electrodes is essential for low noise measurements of high resistances. The sample cell geometry has evolved considerably during the course of our studies. Its latest version is illustrated in Figure 5.

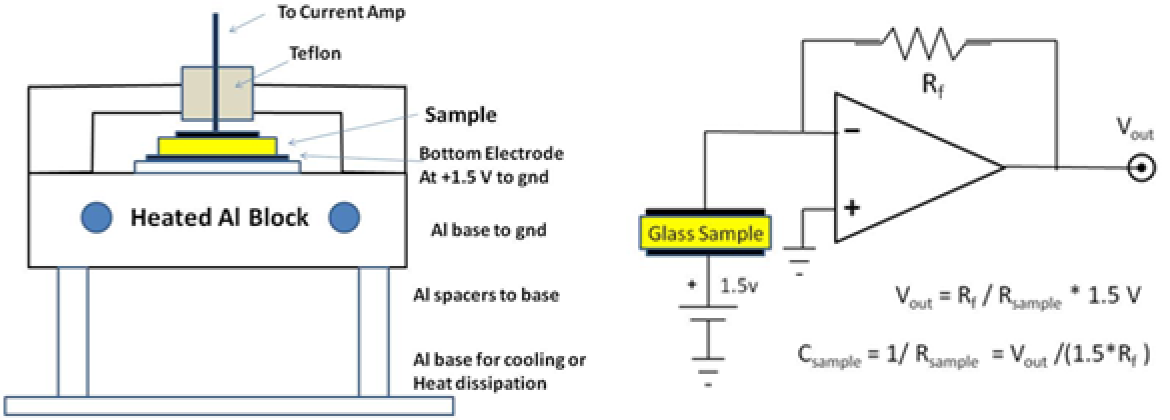

Figure 5. Sketch of the shielded sample cell with embedded heaters (right). Simplified diagram of the electrical circuit is shown on the left with its detailed version made available online [9].

This cell design combines excellent sample shielding with good thermal control. The sample and electrodes are completely surrounded by a thick, grounded aluminum enclosure. The bottom half of the aluminum enclosure can be heated with two embedded 30 W cartridge heaters to control the temperature; it is also connected to a lower cooling plate through four aluminum spacer rods. This later feature allows for sub-ambient cooling when the lower plate is immersed in an ice bath. Monitoring the current through the sample under a 1.5 V battery potential provides a measure of the DC conductance as shown in Figure 5.

Precision high impedance feedback resistors (to 1000 MΩ) are used to achieve the very high gain required at the lowest conduction ranges. While we are aware that DC conductivity measurements in ion conductors can be complicated due to depletion and polarization layers, this fortunately does not appear to be a significant issue with the experiments performed on sugar glass samples. Typical results for the conductivity of a sugar glass measured in air are shown in Figure 6.

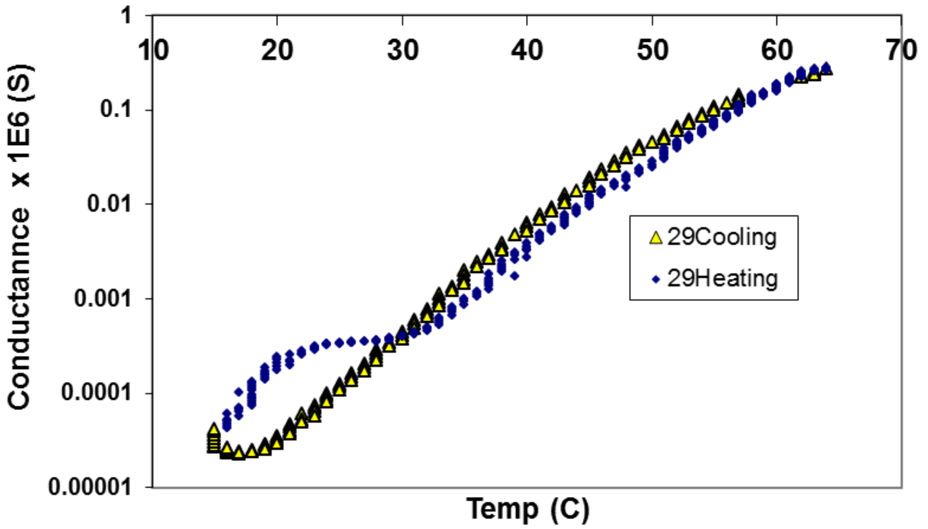

Figure 6. Conductance of sugar glass on cooling from well above Tg to well below, followed by reheating. The anomalous plateau in heating is due to moisture absorption below the dew point.

Here the sample was first cooled from well above Tg (~ 30-40° C, depending on amount of absorbed moisture) and then reheated. Notice both the upturn in conductivity on cooling below about 20° C and the plateau in the subsequent heating curve. This behavior is quite repeatable, but the anomalous upturn is believed to be due to absorption of moisture on cooling below the dew point of our ambient air. Runs made under dry nitrogen did not exhibit this anomalous behavior.

Notwithstanding the spurious effect from moisture absorption, the data show that the conductivity of sugar glass changes by four orders of magnitude over a 40° C temperature range. It provides the student with an opportunity to think critically about the cause for such large, unexpected results. The cooling data when plotted vs. 1/T [9], exhibit Arrhenius behavior over a wide range and provide an estimate of an activation energy at 190 kJ/(mole °K), quite close to the heat of vaporization reported for glucose of 194 kJ/(mole °K) reported by Oja [10] from vapor pressure data. This corresponds to the energy required to create a void or space sufficient for the charge carrier to move between sites, consistent with models for viscosity, diffusion and conductivity [8].

Thermal Conductivity Measurement Apparatus for Glassy Materials

Thermal conductivity is an important property in selecting a material for many applications that are subjected to temperature gradients and the generic values for many common engineering materials are readily available. Glasses are generally poor conductors of heat; their thermal conductivity is usually considerably lower than corresponding crystals (e.g. fused vs. crystalline quartz). For the material scientist making new formulations, such as glass-polymer composites, the thermal conductivity data may not be known and an experimental determination is necessary. Unfortunately, equipment to measure the thermal conductivity of insulating materials is not commonly available in most laboratories. Thus, we have developed a simple, home-made apparatus, which can provide students and researchers access to this important property for both polymers and glasses.

Our apparatus is based on a straight forward application of the definition of thermal conductivity 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 7.

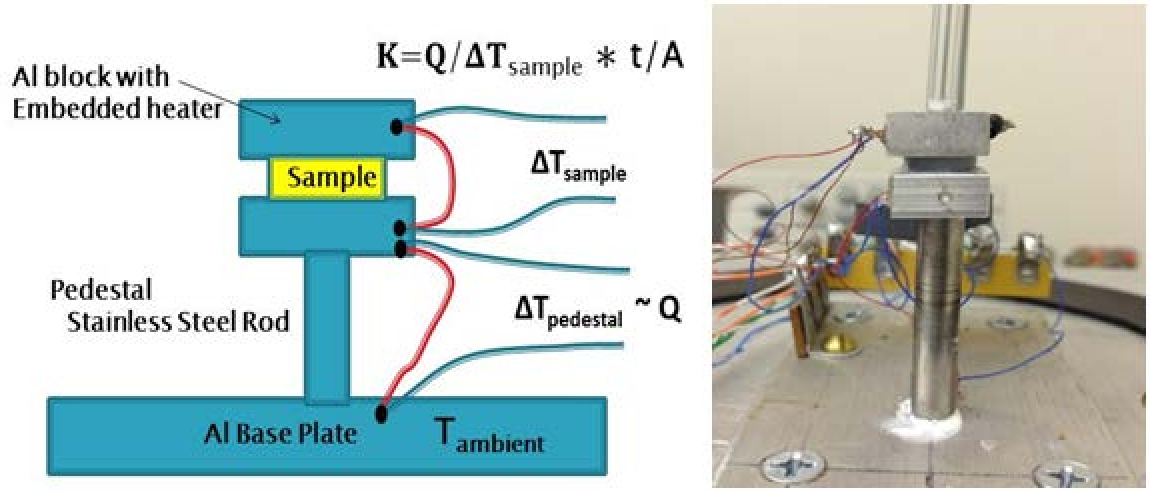

Figure 7. Sketch of the thermal conductivity apparatus. Top plate is heated by two embedded 47 Ω resistors wired in parallel and driven by a 5 V source. The actual device shown on left.

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, which provides an independent measurement of heat flow through the sample. Differential thermocouples pairs are used to sense both the temperature difference across the sample (ΔT_{sample}) as well as the temperature difference across the base pedestal ($\Delta T_{pedestal}$), the latter being proportional to the actual heat flow through the base. Two inexpensive instrumentation amplifier ICs (INA122), already described above, are used to convert the small differential TC signals into a voltage range appropriate for low-cost data loggers (~0-5 V).

The cost of parts for the basic apparatus is minimal (< $30) and requires only modest mechanical and electronic fabrication skills. Tests carried out in air with a collection of commonly available plastic and pyrex sheet material gave good correlation with their known thermal conductivity values over 0.15 to 1.1 W/m °K range. Recently, a home-built vacuum chamber has been added to quantify the effect of stray thermal conduction through the surrounding air. This has allowed us to refine calibration to accommodate for heat loss via air and obtain improved agreement with published values as shown in Figure 8.

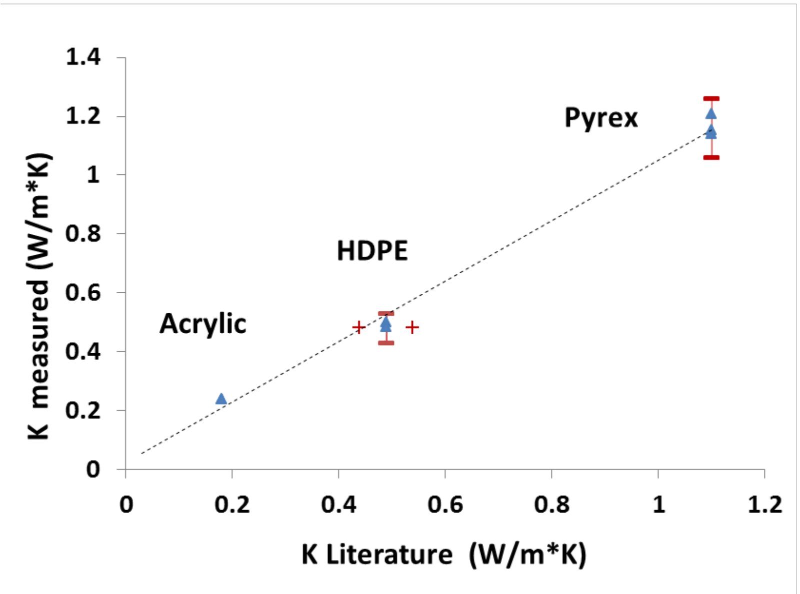

Figure 8. Comparison of measured vs. literature values of thermal conductivity for some common insulating materials. Good correlation is observed over the range of values examined.

We also measured the thermal conductivity of silica filled epoxies and showed a linear increase with fill fraction to 20%. Details on this apparatus are presented in a separate paper [11] and archived within IMI-NFG's online library.

CONCLUSIONS

In this paper we have summarized four recent additions to IMI-NFG's considerable collection of experiments designed for a low-budget, yet full engagement with glass science. Corresponding home-built instruments are described, which are suitable for the study of real glass science phenomena found in simple sugar glasses and other common materials. While home-built, the apparatuses are accurate enough for the student to carry out quantitative experiments in such core areas as devitrification, thermal analysis, and electrical conductivity of sugar glasses together with thermal conductivity on any polymeric or inorganic glasses. The interested reader is encouraged to consult IMI-NFG website for more detailed information. By combining some basic mechanical skills with some simple electronics, utilizing low-cost but powerful ICs, the student can construct instruments for sophisticated measurement of material properties and thereby pursue investigations based on his/her level of curiosity – entering into the awe inspiring aspects of real science. Once one has a material system under control, and a set of tools to make measurements and answer questions, there is no limit to exploring the world of glass science. We welcome any and all feedback, including additional experiments to include on our website.

ACKNOWLEDGEMENTS

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REFERENCES

1. William R. Heffner and Himanshu Jain, MRS Proceedings, 1233, (2009).
2. Committee on Science, Engineering, and Public Policy, Rising Above the Gathering Storm: Energizing and Employing for a Brighter Economic Future, (National Academies Press, Washington, DC, 2007).
3. See for example, Mark Hatch, *The Maker Movement Manifesto: Rules for Innovation in the New World of Crafters, Hackers, and Tinkerers* (McGraw-Hill, 2013).
4. Philip Bell, Bruce Lewenstein, Andrew W. Shouse, and Michael A. Feder, Editors, Learning Science in Informal Environments: People, Places, and Pursuits (National Academies Press, Washington, DC, 2009).
5. ImageJ is a public domain, Java-based image processing program developed at the National Institutes of Health. It is available for free download at http://rsbweb.nih.gov/ij/
6. "Temperature Logging and Control with the Basic Stamp Microcontroller", William Heffner and Jordan Davis, Am. Assoc. Physics Teacher Winter Meeting 2011, Jacksonville, FL, Jan. 9-12, 2011. Poster archived on IMI website at: http://www.lehigh.edu/imi
7. Ibid, "A Low-Cost Student Built DTA for Exploring the Glass Transition", William Heffner, 2010 Glass & Optical Materials Division Meeting, Corning, NY, May 16-20, 2010.
8. See, for example, the discussion in Chapter 14 of Arun Varshneya, *Fundamentals of Inorganic Glasses, 2nd Ed.* (Society of Glass Technology, Sheffield, 2004).
9. "A low-cost electrometer for measuring conductivity and glass transition in sugar glass", W. Heffner and N. Ward, 2011 Glass & Optical Materials Division Meeting, Savannah, Georgia, May 15-19, 2011. Poster archived at: http://www.lehigh.edu/imi
10. V Oja, and EM Suuberg. J. Chem. Eng. Data 44(1):26--29 (1999).
11. William R. Heffner, Shera Demchak, Raymond Pearson and John W. Scruggs, "Home-Built Apparatus for Measuring Thermal Conductivity of Glass and Polymer Materials", presented at 2013 Fall MRS Meeting, Boston (submitted for publication to MRS Proceedings).