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Building a Low Cost, Hands-on Learning Curriculum for Glass Science and Engineering Using Candy Glass

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ABSTRACT

We have developed a program to connect students, as well as the general public, with glass science in the modern world through a series of hands-on activities and learning experiences using sucrose based glass (a.k.a. hard candy). The scientific content of these experiments progresses systematically, providing an environment to develop an understanding of glassy materials within a framework of “active prolonged engagement” with the material. Most of the experiments can be assembled in a high school lab, or even in a home setting with minimal cost, and yet are appropriate for inclusion in an undergraduate materials lab. The cost is minimized by utilizing common, everyday materials and devices. Some of the activities included in our experiments include: synthesis, density, refractive index determination, glass transition, crystallization, kinetics of devitrification, thermal properties, etc. Temperature measurement, temperature control, and even automated data collection are part of the experience, providing an open path for the students to continue their own interesting and creative ideas.

I. INTRODUCTION

The 1983 publication of A Nation at Risk [1] identified the decline in the academic achievement of US students and the potential for failing to meet the national need for a competitive workforce. Since that time much social and political dialog has centered on the need to improve student achievement and interest in science, engineering and technology education in the US. Recently more attention has been brought to the significance of both hands-on learning and the informal educational experience to the total educational experience of both student and adult learners [2]. In response to this challenge, we have developed a program to connect students and the general public with glass science in our modern world through a series of hands-on activities and learning experiences.

Glass and glassy materials are important and ubiquitous materials in our everyday life. In fact, they are perhaps among the most common material in our everyday experience, from windows, doors, kitchen ware, eyeglasses, cameras, and insulation, not to mention the optical fibers empowering the information age. And yet, with this incredible body of experiential familiarity to relate to, students experience little or no introduction to this important material in any of their formal high school or college science training.

One of the problems in conducting any serious investigation of glass science for the younger students (especially in the high school or home setting) is the high temperatures required to make or form these materials, especially the commonly used oxide glasses, as well as the specialized equipment required to process these hard materials. However, a much lower temperature example of glass is found in the universally pleasant world of candies, the sugar glass, also known as hard candy. The sucrose-corn syrup-water system of candy glass mimics many aspects of commercial, soda-lime-silica glasses and these close analogies have been described in earlier papers [3, 4].

In this paper we describe a series of hands-on exercises for the student to experience both glass technology and glass science through explorations with candy glass. The experiments build on one another to provide a mini-curriculum of low-cost, hands-on activities designed to facilitate a rich experience of active prolonged engagement with glass science. The apparatus required for these experiments can be assembled from commonly available items from the home, hardware store or the high school lab and there is an emphasis on building one's own equipment (such as a light bulb sample heating oven). Our intent is to engage the students by allowing them to learn largely through discovery, building knowledge and interest through successive experiences around a common glassy material that they can make and modify as their ideas evolve. While the experiments in our series are simple enough for the students to do at home, they are quantitative in nature and allow students to explore "real glass science". Many are ideally suited for the student science project, while some are appropriate for a classroom demonstration (like the fiber drawing tower).

The materials in our candy glass curriculum are all 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 wide range of materials to support the learning experience, including tutorials, videos, project descriptions, student presentations and even 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.

II. BACKGROUND

While sugars and candy glass are not part of the traditional material science domain, they do certainly have a large and important place in food science and some of the best introductory and advanced material on sugar glasses can be found in their collections. McGee's very enjoyable classic, On Food & Cooking: The Science & Lore of the Kitchen [5] is highly recommended as a starting point for the student. For the more serious student looking for detailed information on sugars, there are two excellent up to date references from the food science community, one is Crystallization in Food by Hartel [6], and the other is Sucrose Properties and Applications edited by Mathlouthi and Reiser [7]. The latter are both available on Google books.

For the material scientist the behavior of the sucrose water system is best visualized through the binary phase diagram shown in figure 1. Data for the solubility as well as the freezing point depression for sucrose in water are available, from which the students could readily construct their own diagram [8]. Data for the glass transition temperature are likewise available [9]. The figure shown here has been prepared from these data using Excel and is in good agreement with the diagrams reported in other sources [10]. In our diagram we also include the boiling point data taken from the Food Industries Manual [11].

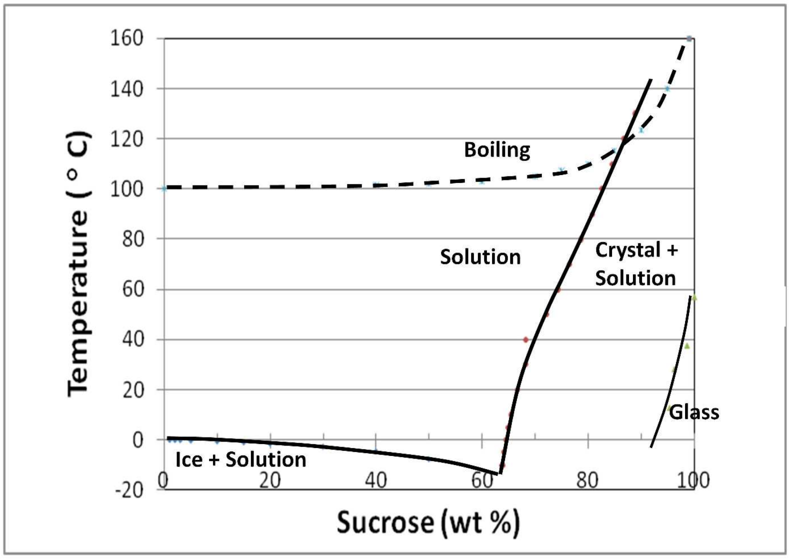

Figure 1. The sugar/water phase diagram for the sugar/water system with boiling point line included. Prepared from data referenced in the text.

The sucrose solubility vs. temperature curve (even included in some high school chemistry textbooks) defines the upper temperature of the two-phase region (crystal + solution). For temperatures above this line sugar crystals are unstable and will dissolve. Below the solubility line, sugar crystals are stable and can exist in equilibrium with the solution.

However, due to the high viscosity of the syrupy supersaturated solutions, crystals can be slow to form, especially in the absence of seed crystals or other nucleating sites. As a result high sugar content solutions have a distinct metastable region below the solubility curve. Supersaturation ratios as high as 1.2 are stable for a long period of time in sucrose/water systems, with spontaneous crystallization occurring at a ratio of about 1.3 [12, 13]. The supersaturation ratio, commonly referred to in the sugar literature, is defined as the ratio of the solution concentration (in wt. %) to the saturation concentration. Within the food industry it is well known that the addition of other sugars, in particular corn syrup (which contains glucose polymers), can greatly extend this metastable range [14]. In our experiments to follow we demonstrate this inhibition from corn syrup and utilize it in developing our own formulation for sugar glass.

The viscosity of the metastable syrup increases rapidly as the hot solution is cooled, eventually reaching the glass transition temperature. Once a glass is formed, the material is protected from subsequent crystallization due to the extremely high viscosity of this solid-like material.

In order to prepare candy glass, a mixture of sugar and water is first heated to dissolve the sugar crystals. For moderate sucrose content the solution will clarify prior to full boil as the sugar crystals dissolve. For high sugar content, the clearing point may occur after the solution begins to boil, as indicated in the phase diagram. By continuing to boil, the water content of the one phase solution can be gradually reduced, with a concomitant increase in the boiling temperature. Thus the boiling temperature can serve as a convenient measure of the water content and provides the simple monitor for determining when enough water has been removed to make a good room temperature glass. Boiling to 145-155°C is a typical end point for preparing a hard candy material. The moisture content of a hard candy is typically between 1-2 wt% [15, 16]. One can control the hardness by the temperature to which the mixture is cooked, but be aware that the temperature rises abruptly as you reach these lower water content region.

The solution temperature begins to rise abruptly as it approaches the requisite low water content and one must take care to not over cook the material. Sucrose begins to degrade (and turn yellow) starting at this temperature range. "Above 165°C the sugar is more than 99% sucrose and no longer boils, but begins to break down and caramelize" [17]. At 170°C it is hydrolyzed and splits into dextrose and levulose, or invert sugar [18].

The hardness of the candy (at room temperature) depends on the final boiling temperature (and thus the water content). This is reflected in the concentration dependence of the glass transition temperature curve, also shown on figure 1. A good hard candy should have a T_g of 40-50°C if it is to retain its hardness at room temperature. Candy with a T_g near room temperature will be soft and taffy like in texture. This relationship between T_g and water content forms the basis for the Cold Water Test also known as the Ball Test [19] used by old-time cooks to prepare candies long before thermometers were common in the kitchen. To perform this test, a small amount of the boiling solution would be dripped from a spoon into a dish of cold water, quenching the drops to near room temperature. Once cool the cook inspects the drippings to see if they are soft, hard or crack (brittle). Cooking to the soft ball state serves as the doneness test for fondants and fudge, while a hard ball is required for taffy and the hard crack for making good hard candies. For the interested reader McGee [19] provides a very clear discussion of this test and the various types of ball stages associated with different temperature ranges. We encourage all students to utilize these old time and highly intuitive monitors in conjunction with the solution temperature to develop a deeper understanding of the process.

While the two component sucrose-water system has the advantage of simplicity as well as a large body of well documented literature dating back to more than a hundred years, it has one serious limitation for actual candy glass making and student experiments. The sucrose-water only system is strongly susceptible to crystallization, very problematic for glass making. (Although controlled crystallization is actually required for the making of some confectionaries like fudge.) It is especially difficult to prepare good candy glass from a mixture of pure sucrose and water because these solutions have a relatively high rate of crystallization, especially at the low water content range required for candy glass. Frequently even stirring the hot solution can promote the onset of crystallization in simple sucrose-water recipes, ruining the preparation. As mentioned already, corn syrup has a large inhibitory effect on the crystallization rate and is almost always included with sucrose in the hard candy glass recipe. In one of the following sections we describe experiments which illustrate the effect of corn syrup to sucrose ratio on crystallization. For the bulk of our other experiments we have settled on a 2:1 ratio of sucrose to corn syrup as our basic recipe.

III. EXPERIMENTAL ACTIVITIES & MODULES

A. Synthesis of Candy Glass - Exploring the Properties and Applications of Glass

For our introductory activity in glass science we typically combine an interactive discussion of glass followed by showing students how to make their own sugar glass. Our version of the activity is typically delivered in a 90 minute period for a science camp or student/teacher workshop, but could easily be divided into two shorter periods to fit the class room schedule.

After a short discussion of the examples of glass in our everyday experience, we find it useful to encourage the students to list some properties of glass, based on their own experience. Typically the students will, with only minimal coaching, come up with a list which includes such attributes as hard, brittle, easily fractured, transparent, and flows on heating. By defining glass in terms of its common observable behavior, one can quickly establish the connection to hard candy as also representing a glass. The Jolly Rancher brand from Hershey Foods Corporation provides a convenient example of candy glass to include in the discussion. Focusing on properties provides an ideal spring board for a discussion of how glass is different from the other condensed matter phases (liquid and crystalline solid) and the structural differences associated with each of these states. Likewise the discussion of candy as a glass sets the stage and motivation for the making (synthesis) of hard candy glass.

There is an abundance of recipes for hard candy available from cook books or the internet. We also include a list of recipes in the glass making instructions. Our standard recipe for most experiments is a 2:1 (by wt) mixture of sucrose to liquid corn syrup with approximately 10% (by wt) water added at the start. The actual details of the recipe and the cooking procedure are provided on our website, with other relevant information. The cooking procedure involves monitoring the temperature with a low cost digital cooking thermometer as the glass making proceeds. The student is encouraged to note such phenomena as the onset of boiling, the temperature of complete dissolution (or clearing), as well as the emerging thickening ("stringiness") of the solution as the temperature rises and the water evaporates. The old fashioned techniques of testing for soft ball and hard crack, described in the previous section, are used to determine the progress of the preparation. By comparing such clear observables with the actual solution temperature the student begins to develop an experiential understanding of the glass forming process.

Once the hard crack state of the liquid is achieved (approximately 145-155°C), the syrup is removed from the heat, allowing a short rest period for the bubbles to escape. Next, the hot liquid is poured into molds, free form discs, sheets, test tube samples for experiments, or any number of shapes for consumption. To cap off this student activity we usually save some of the material for making candy glass fibers. Upon cooling to ~ 90°C the melt becomes ready for pulling long glass like fibers using wooden popsicle sticks. An on-line video shows a student pulling a candy glass fiber of more than 100 meters down a hallway [20]. In Section 3 below we describe a home built fiber drawing tower which provides a more quantitative extension to this initial exploration with fibers. After this first interactive lesson on glass and candy glass, the students are prepared to make their own candy glass at home and begin experimenting using some of the activities suggested below as a starting point.

B. Post Synthesis Activities

Once the students have learned to make their own sugar glass, a number of other interesting experiments can be explored. Two experiments that relate directly to the physics and chemistry classroom include density and refractive index measurements, while a fiber drawing tower provides an opportunity to explore an engineering based experience involving optimization of processing parameters.

1. Density

Density is an important property of glass, which often correlates well with other properties such as refractive index [19]. It is also a very basic property of matter introduced early into the science curriculum, so most young students will already have some familiarity with the concept. However, measuring the density of a water soluble, irregular shaped solid with standard laboratory resources provides an interesting challenge for the student to consider. The low cost method that we describe involves constructing a student-built pycnometer. The pycnometer is constructed from a small jar (e.g. 4 oz salsa jar) for constant volume, with a small hole drilled in the lid to allow for its filling with water to a fixed volume. The lid of the salsa jar is stiffened so that it would not deform (and thus change volume) by epoxying a steel washer to the top. This pycnometer can be constructed at very low cost (a few dollars at most for epoxy, washers and a jar of salsa or artichokes) and provides the students with a rich hands-on experience of actually constructing their own instrument.

Figure 2. Low cost apparatus for the measurement of density of irregular shaped solids, using Archimedes method.

To determine the density the pycnometer is weighed empty, filled with only water, and finally with glass of known weight and water. The weight of the glass divided by the weight of water displaced is the specific gravity. For density one must also include a factor for the density of tap water at room temperature (0.998). A simple triple beam centigram balance, available in most high school labs, will allow a density measurement to 4 significant figures.

Before tackling the candy glass density measurement, students should first explore the accuracy and repeatability of this method utilizing some standard oxide glass or other water insoluble material. By using a syringe to remove all air bubbles and to top off the water level to a fixed meniscus at the hole, we were able to achieve a standard deviation of 0.005 g/cm^3 for the density of pieces of a broken dish (oxide glass with nominal 1.68 g/cm^3 value). Achieving such accuracy requires careful attention to detail, including avoiding changes in room temperature. Once the accuracy of the method has been demonstrated for the water-insoluble materials, the student can tackle the density of their own candy glass. On the IMI-NFG website we include details for the density measurements made by one of our students on a collection of sugar glass samples made with varying ratio of sucrose to corn syrup. Our student investigator found no significant difference in density for the candies made to the same hard crack temperature over a wide range of sucrose to corn syrup ratio. By encouraging the student to consider some simple models for density of mixtures, the student should find this result quite plausible.

2. Refractive Index

Refractive index is another important property for a transparent, optical glass, and sugar glass provides an opportunity for the students to prepare their own samples as well as their own apparatus for measuring this basic parameter. We have examined two simple methods by which the student can measure the refractive index of sugar glasses made by themselves, using very basic apparatus. The simplest is based on Pfund's method [22], where only a laser pointer and a ruler are required to determine the refractive index of a thick, flat sample of a transparent material. If a transparent glass sample is illuminated with a laser pointer as shown in figure 3, then a change in reflected light will occur at the critical angle. The vanishing reflectivity at the critical angle produces a dark region of radius, r, around the center spot, if there is no air interface at the bottom surface. The refractive index and be easily calculated from the radius of the dark region and the thickness of the sample, h, using $n^2 = 1+ (2h/r)^2$. In the case where there is also an air interface on the bottom surface, such as a flat glass not adhering to the surface, then the light ring is observed in the center at the same condition.

The other, more accurate method, utilizes a student spectrometer and a prism of candy glass formed within a glass mold constructed by the student from glass slides. The minimum deviation method [23] allows the student to obtain the refractive index to four significant figures, while quantitatively exploring the nature of refraction. Each of these methods has been tested and described in detail by undergraduate students during IMI-NFG's summer REU programs at Lehigh University. Full presentations of their procedures and results are available on our website.

One of our students utilized the minimum deviation method to determine the refractive index of candy glasses over a wide range of sucrose to corn syrup ratio and found no significant variation of refractive index, consistent with the observed uniformity of densities. [We also include details on how to build spectrometer light sources of different wavelength from low cost LEDs, so that measurement of chromatic dispersion can also be explored by the student.]

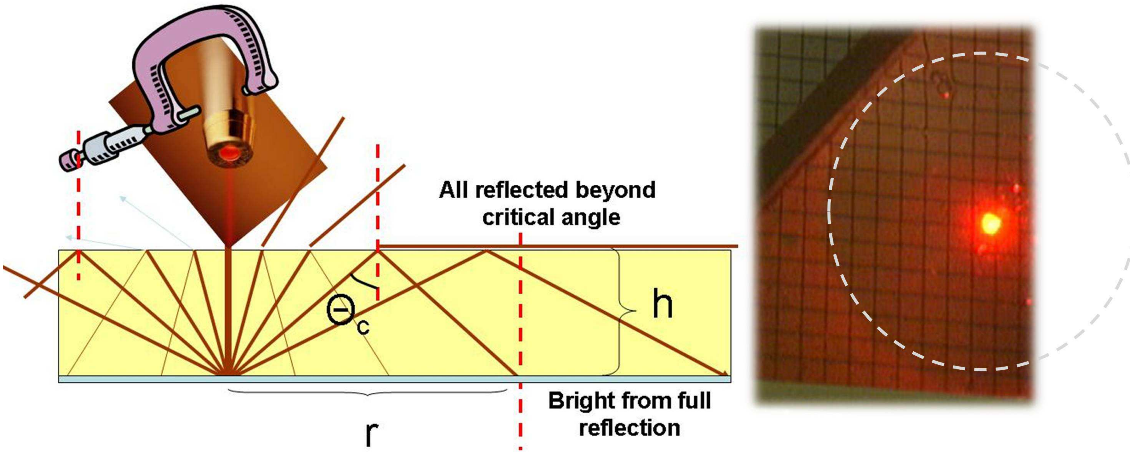

Figure 3. Basic concept of Pfund's method for measuring the refractive index of a transparent plate. Photo on right show darker region in the center of sugar glass slab.

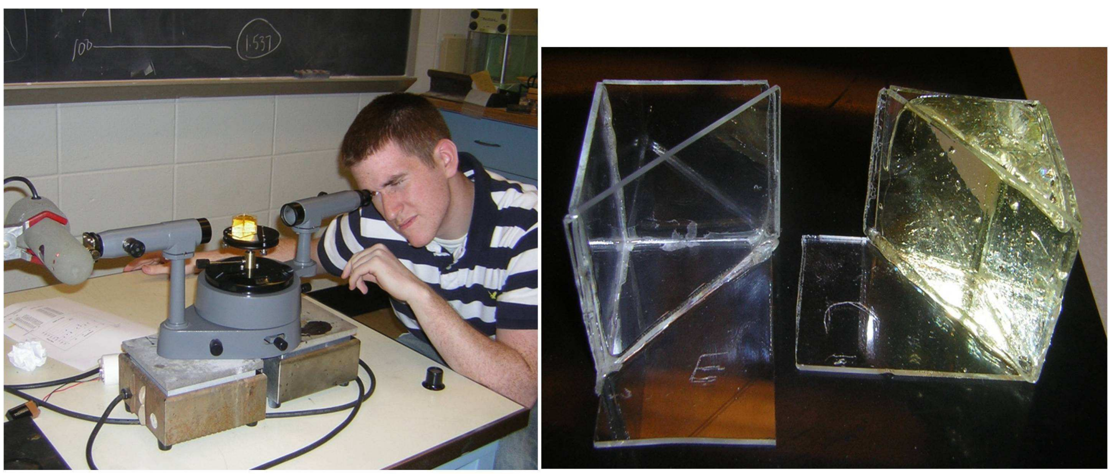

Figure 4. Left, an REU student measures the refractive index of a candy glass prism. Right, empty and candy filled prism molds made from microscope slides by the student.

3. Fiber Drawing Tower

Early on in the preparation of candy glass the student has the opportunity to experience the unique ability of glass to form fibers from the melt once the temperature is within an appropriate range. The fiber drawing property is noticeably temperature sensitive, providing an excellent opportunity for the student to engage in an engineering experiment to optimize and control the process parameters required for drawing candy glass fibers. To facilitate this inquiry we have designed a sugar glass fiber drawing tower which mimics many of the components of an actual drawing tower used for fabricating fibers for optical communication, but only at a much lower temperature and made again from low cost parts. Our candy glass fiber drawing tower utilizes one or two halogen light bulbs to heat a rod of candy glass preform (fabricated by the student). The temperature of the sugar glass is monitored using a low cost thermocouple, allowing him or her to quantitatively explore the optimal conditions for fiber drawing. The take up spools have been made from empty, plastic, peanut butter jars. As with the other activities, details with construction sketches are included on our website.

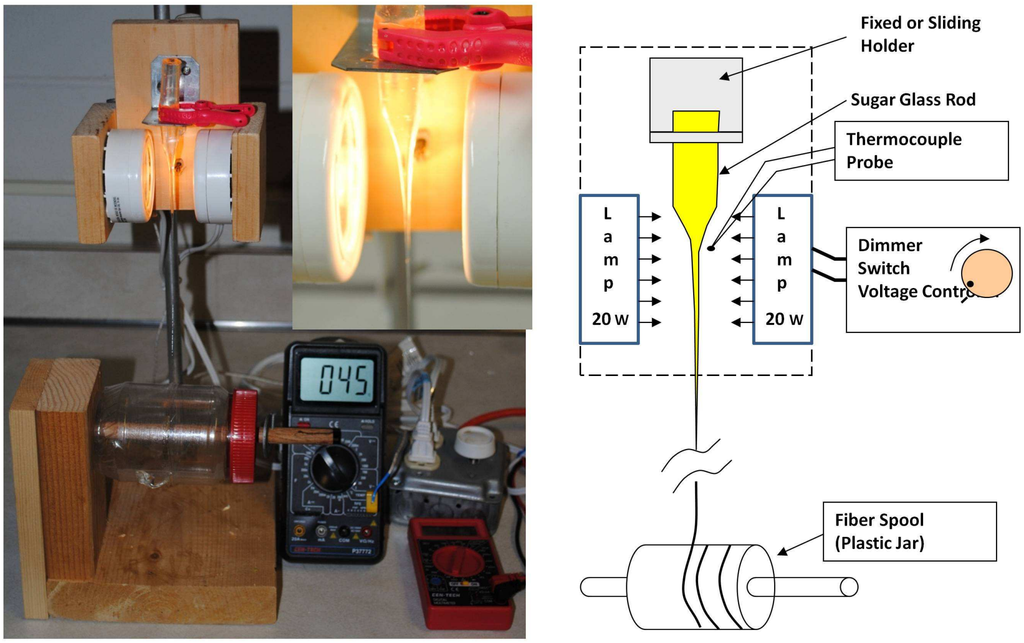

Figure 5. Photograph of our home-made fiber drawing tower for candy glass using two halogen lamps for the “furnace”. A sketch of the components for measurement and control is shown on the right.

4. Crystallization

As mentioned in the Background section, pure sucrose has a very strong tendency to crystallize in concentrated aqueous solutions even at high temperatures, making it difficult to form a glass with sucrose as the only sugar component. The addition of corn syrup greatly reduces the tendency to crystallize, which is why it is essential in most recipes, as well as ours. This addition of a third component provides an excellent opportunity for the student to explore the influence of the sugar to corn syrup ratio on the glass formation properties of this system. One of our early experiments was to examine this dependence of glass stability on composition in the sucrose/corn syrup/water system. A pseudo-ternary phase diagram for this system is illustrated in figure 6 below. Samples with varying sucrose to corn syrup ratio were all heated until the solution was at 145° C and then poured into paper containers to cool. Black paper was used for the base, so that scattering from any crystal formation would be easier to photograph.

On the high sucrose, low water side, the boiling solutions are very prone to crystallization as the water is driven off during cooking, making it nearly impossible to form a glass in a sauce pan from an all sucrose mixture. At 80-90 % sucrose to corn syrup ratio it is possible to form a candy glass on cooling, but the resulting material is very prone to subsequent crystallization within less than a day. A 70% or lower sucrose to corn syrup ratio is recommended to avoid severe crystallization problems. An insert in figure 6 illustrates how these crystals begin to form at the surface of these glasses. This tendency to form surface crystals was observed to be far more rapid during periods of high humidity, an observation suggesting that atmospheric moisture has a considerable influence on the crystallization dynamics. This early observation led to additional interesting experiments which were carried out by students to clarify and quantify this humidity conjecture. These experiments are described in the next sections.

On the high corn syrup side, glasses tended to be much softer than the high sucrose candy, and they displayed a strong tendency to become gummy or tacky under high humidity conditions. From these initial experiments we established that sucrose to corn syrup ratio of 2/1 would produce a relatively stable glass on cooling, and yet retain enough of a tendency to crystallize in time to provide a convenient system for subsequent studies of crystallization under high moisture and elevated temperatures.

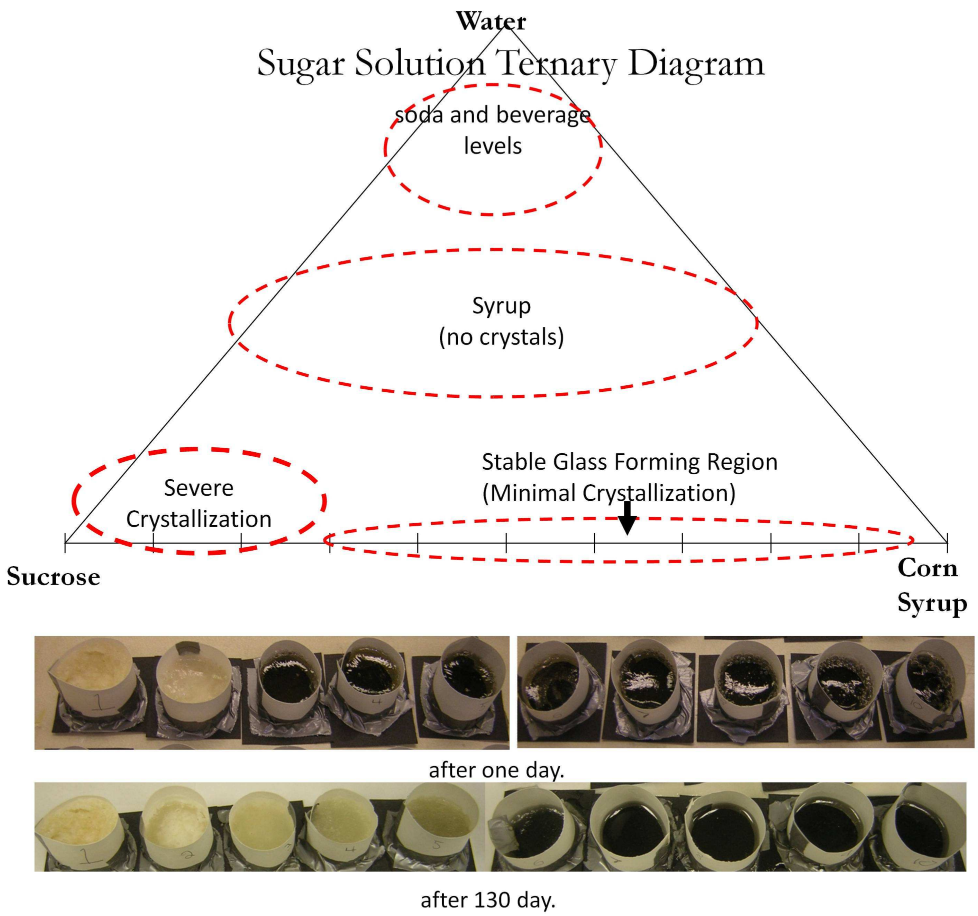

Figure 6. A pseudo-ternary diagram for the sucrose/corn syrup/water system at room temperature, illustrating the strong tendency to crystallize on the high sucrose side. There is no glass formation at room temperature until water is reduced to about 1-2 % by wt. Diagram is a cartoon and not to actual scale. Ratios on the horizontal axis are only approximately linked to the photographs. Below are photos of representative mixtures poured into paper molds with a black bottom to highlight the white crystals.

4.1 Quantitative Analysis of Crystallization

During the making of sugar glass, the experimenter (and cook) becomes painfully aware of both the tendency of the melt to crystallize as well as the importance of avoiding such crystallization in making clear, high quality hard candies. The simple glass system exhibits both moisture-mediated, surface crystallization at room temperature as well as thermally induced crystallization within the bulk, if heated sufficiently above the glass softening point. Sugar glass provides a rich opportunity for the student to explore quantitative experiments on both of these phenomena using relatively simple tools. We have developed experiments to explore both of these aspects with simple home-built apparatus. Our website includes tutorial material to guide the students with their understanding of fundamentals of crystal nucleation and growth.

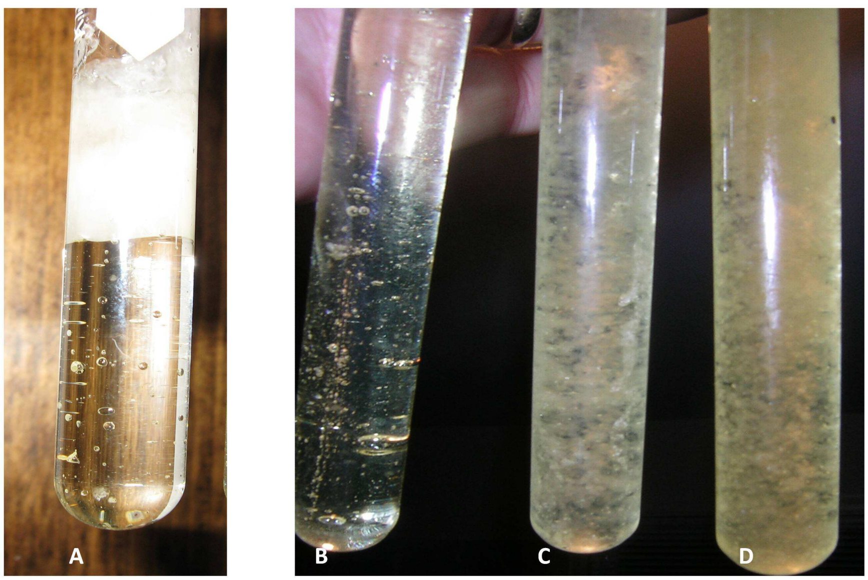

Figure 7. (A) Growth of crystals at the surface (top) of sugar glass in test tube at room temperature after several weeks of high humidity. The photo on the right shows the progression of bulk crystallization from an unheated clear candy glass (B), to one that is heated in a home oven set to 250° F (120°C) for approximately 20 minutes (C & D).

4.2 Effect of Humidity on Crystallization

Initial observations with high sucrose glasses, as well as our standard 2:1 glass, suggested a humidity dependence of the surface crystallization at room temperature. Motivated by the interest of a local high school student in doing a science project, we developed a simple approach for controlling humidity and for measuring crystallization rate. The samples for this study were prepared from circular globs of molten candy pressed between two glass slides and maintained at a uniform thickness by using two steel washers as spacers. Utilizing this sample geometry, only the outer edges of the circular discs of candy glass are exposed to ambient moisture. Thus all humidity-induced, surface crystallization occurs at the outer edge of the sample. The geometry also made it easy to examine and measure the progression of the crystal layer as it grows with time. Simple humidity controlled chambers were constructed from glass cookie jars with tight fitting rubber gasket seals. Three levels of humidity control were achieved using $CaSO_4$ desiccant for the dry chamber, water soaked paper towel for the high humidity chamber and saturated solution of $Mg(NO_3)_2$ for the intermediate (approximate 50%) humidity chamber [24]. Additional details of the method can be found on our website together with a copy of the student's final presentation of her experiments at the regional science competition (PA Jr. Academy of Science).

The results of this experiment were very instructive. Essentially no crystallization occurred on the sample maintained in desiccant, while samples from the 50% relative humidity (RH) chamber grew uniform thickness crystal layer, ideal for measurement. A standard digital camera (in macro mode) was used to record the layer thickness every few days. From a printout of the photograph the student could then use a simple scale (ruler) to measure the thickness of the growing layer. Calibration of the scale was achieved by also measuring the width of the glass slide (1") in the photograph.

The data in figure 8 show the measurements over a twenty-five day period for two different candy glass preparations (143° and 145°C final cooking temperature) including some replication (of the 145° preparation). All of the samples exhibited good repeatability, with crystal growth front (width) that grew at a uniform rate of approximately 0.4 mm per day.

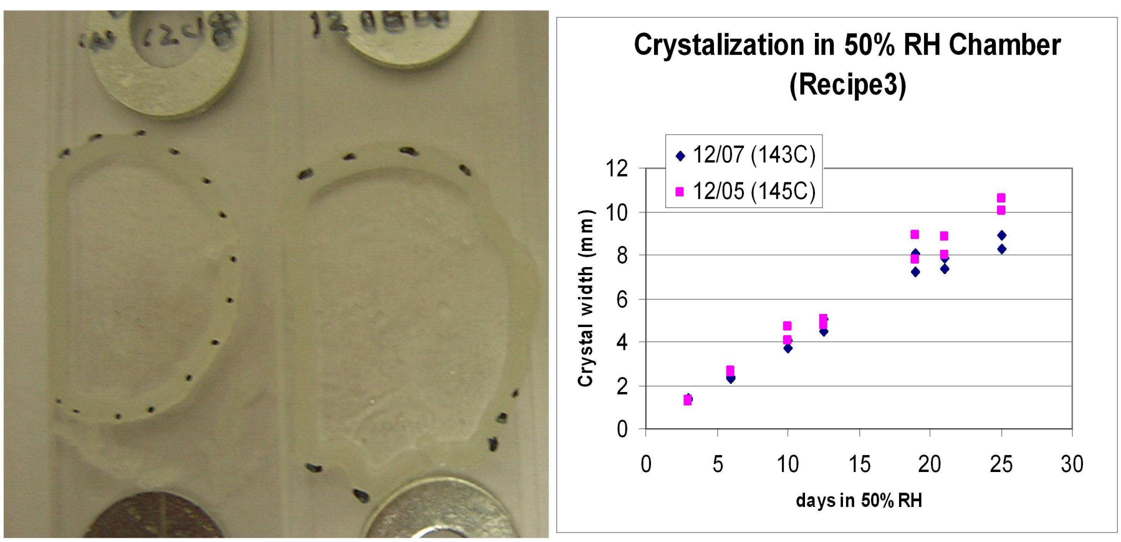

Figure 8. Photograph of crystal growth on outer edge of two samples taken on day 5 after placing in the 50% RH chamber. Note the uniform thickness of the crystal layer in both samples. On right, data show increasing width of crystal layer with time for a standard recipe glass. Two different batches are included (diamonds and squares). Note that the 145° C sample (squares) also includes a replicate.

Samples from the high humidity chamber (essentially 100% RH), did not exhibit the same uniform crystal layer at the outer edge as the 50% RH samples. Instead the outermost layer was clear and liquid in appearance with a thinner crystalline region at the inner growth front, suggesting a subsequent dissolution of the crystals with even higher moisture absorption. This later result was not expected and provides a good illustration of the wealth of interesting behavior for the investigator to discover in this simple system. From these simple experiments many additional experiments for examination come to mind, including the effect of composition.

4.3 Effect of Temperature on Glass Devitrification

Understanding the instability to crystallization underlying both the glassy state and the supersaturated solution from which it arises is an essential aspect of glass science. In glass crystallization is largely precluded by the enormous viscosity of the bulk. However, above the glass transition, the supercooled liquid becomes far more prone to crystallization as the viscosity falls rapidly with temperature, making it easier for the molecules to rearrange into the lower energy crystalline state. We have already discussed how moisture can mediate surface crystallization at room temperature, presumably by providing a lower viscosity path to rearrangement into the crystalline phase. Likewise, as the temperature is increased above the glass transition point, crystallization within the bulk is again possible.

The temperature dependence of crystallization rate is of fundamental importance and a central topic in material science. Sugar glass provides a very easy and accessible gateway to explore the temperature dependent transition from metastable liquid to crystalline state as the viscosity barrier is reduced with elevated temperature.

In order to investigate this temperature dependence, the first experimental obstacle is to achieve appropriate temperature control within the student budget. The home oven, while most convenient, is inadequate for such experiments as it is typically not designed for stable control at these low temperatures and has considerable hysteresis (over and under shoot). For students with access to a laboratory oven with good temperature control, we encourage this approach. However, in the spirit of empowering the student inquiry, we designed a low cost, home-built temperature controlled oven appropriate for this experiment. To minimize cost, a standard light bulb is used as the heat source for an aluminum base plate on which the sample is placed. Heat from the light bulb is controlled by a dimmer switch making low-cost temperature control possible. The temperature of the base plate is monitored by either a low cost digital cooking thermometer or a thermocouple probe. A Pyrex Petri dish provides the cover for the heating plates and the glass sample. A sketch of our apparatus including a plywood base plate and a #10 coffee can to hold the heating plates is shown in figure 9.

Details for the construction are included in our materials. The student is advised to characterize his/her oven by determining temperature vs. time at a range of voltages (e.g. every 10 volts), establishing both the heating time as well as a temp vs. voltage operating chart. This data allows the student to achieve the desired temperature more quickly with a little human assisted voltage ramping. With a little care the student can achieve a controlled temperature in less than about 10 minutes and a temperature uniformity of approximately ± 1°C is typical for this apparatus.

Figure 9. Sketch of the light bulb sample oven for the crystallization vs. temperature studies. (Right) Photograph of the complete apparatus with dimmer control, digital thermometer and digital volt meter (DVM) for monitoring voltage across light bulb heating element.

The samples used in this temperature experiment were the same type of samples described in previous section, i.e. circular glass glob placed between two glass slides (or a glass slide with a cover slit). Here spacers are very important since the material will become fluid at the temperatures required to initiate crystal growth, and without the spacers would ooze out of the glass slides. A calibration of temperature for the sample on the plates can be achieved by using an appropriate low melting standard such as stearic acid.

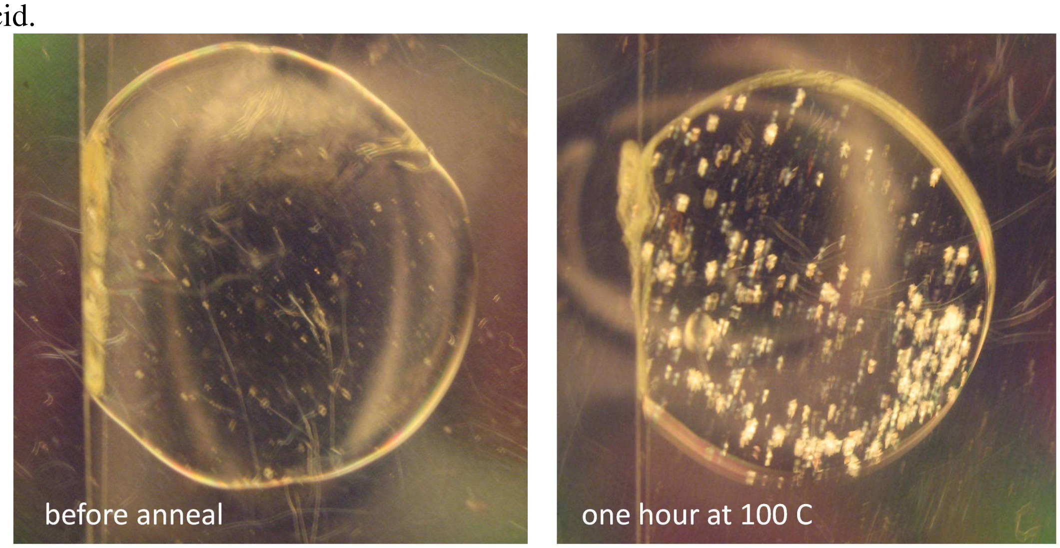

Figure 10. Photograph of crystal growth (right) in a sugar glass sample after one hour at 100°C. The sample on the left is the identical sample prior to heating. Photos taken with low cost Digital Blue computer microscope.

Once the oven temperature is stabilized, the sugar glass sample on microscope slide is placed on the aluminum heating plate of the oven, covered with the Petri dish and allowed to remain in the oven for fixed period of time (nominally 30 minutes). After this fixed period of thermal treatment, the samples are removed and allowed to cool back into the glassy state before examining. Temperatures at 10° increments between 80° and 140°C were found to span the range of interest for our sugar glass. With a T_g of less than 60°C, our material shows no appreciable crystal formation below 80°. Near 150°C (the final cooking temperature), the material begins to boil off water, so this provides an upper limit. Once the sample has cooled, the student examines the sample under crossed polarizers to observe the extent to which crystallization of sugar has occurred. We use a low-cost, computer based digital microscope, the Digital Blue QX5 (available online for under $100, http://www.digiblue.com/). This allows the student to both observe and record the nature and amount of crystallization that has occurred during the thermal treatment period. Any available laboratory microscope with transmission mode and low magnification objectives can be used instead of our Digital Blue. Simply place crossed polarizing sheets above and below the sample. The ability to take a digital photograph for subsequent comparison and analysis is very valuable in this experiment. When no such setup is available, we have found that good results can also be obtained using a standard hand held digital camera placed against the eyepiece of the microscope, while using the digital zoom to achieve a suitable image.

The extent of crystallization can be measured by estimating the area crystallized by eye or, preferably, by utilizing digital image analysis software, such as Image J, an easy to learn, public domain image processing and analysis software developed and distributed by National Institute of Health [25].

In this experiment we are primarily measuring the crystal growth rate, more conveniently expressed as a fraction of area crystallized. Observations of crystal growth with time in our samples show that the initially observed nucleation sites remain constant and that essentially all subsequent growth occurs at these original sites. This experiment provides a wonderful opportunity for the students to consider the distinction between nucleation and growth mechanisms and even come up with their own ideas on how they could establish which process dominates at a given condition. While one cannot see the initial critical nuclei emerge as they are only a few tens of atoms in size, one can often see the consequences of nucleation. These and other very early stage growth experiments in the sugar glass are also possible, but require a higher power microscope and are not part of our discussion here. Nonetheless, this experiment provides much opportunity to observe and ponder new questions and experiments on this topic.

The crystal growth vs. temperature experiments described in this section were carried out by a high school student for her science fair project using the simple method of spot counting for estimating the crystallized area. The experiment was also replicated by a college level REU student who included image processing (with Image J freeware) to measure the fractional area of crystals. In both experiments, the students observed a distinct maximum in the crystal growth rate near 120°C. Example data are shown in figure 11. Additional details for both of the student experiments are included on our website together with their presentations. We have also included a tutorial on nucleation and growth, discussing the topic within the framework of a student with only a high school background in chemistry. There the distinctions between homogeneous and heterogeneous nucleation are discussed as well as basic models for understanding the temperature dependence.

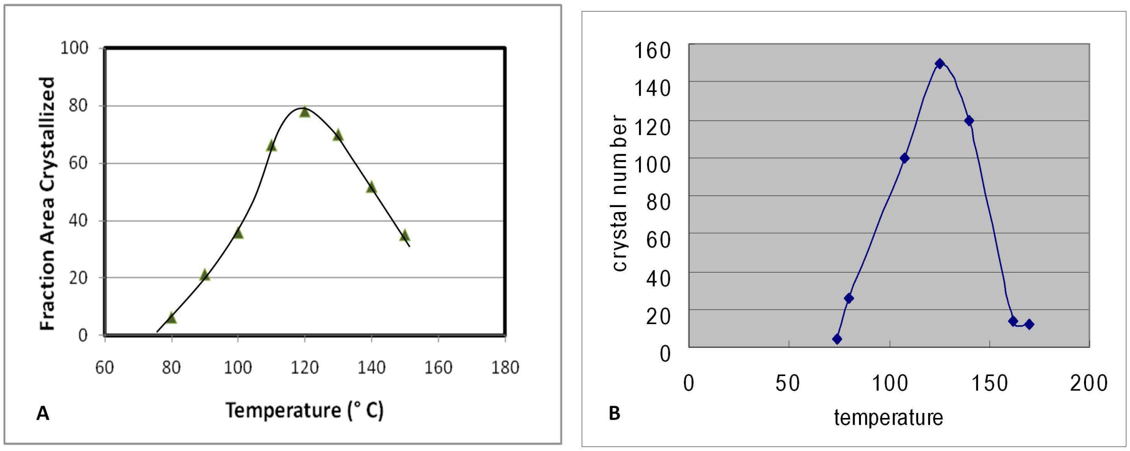

Figure 11. Examples of data measured in two different student experiments. Crystal growth rate vs. temperature in A) done by a college level REU student using Image J to extract the area from photos while B) performed by a high school student using the simple method of estimating the number of “crystal spots”. Note the distinct maximum near 120° C for both approaches.

5. Thermal Analysis

Any exploration of the glass state would be incomplete without some consideration of the glass transition phenomena. In a glass research lab today, DSC (differential scanning calorimetry) would likely be the most common method for measuring this property. However, such apparatus is expensive and generally unavailable outside of the research laboratory. Differential thermal analysis (DTA) is somewhat simpler to implement and provides a more straightforward understanding with essentially the same information. We have developed a simple DTA apparatus which can be constructed from items available in most high school laboratories. It consists of measuring the temperature difference between two test tubes, one tube containing the candy glass sample and the other filled with a reference material, while both test tubes are heated in an oil bath. The oil bath is simply a beaker full of vegetable oil placed on a laboratory hot plate equipped with a magnetic stirring bar. The test tubes are held in the oil bath by a wooden holder, which the student can construct, and thermocouples are used to measure the bath temperature as well as the differential temperature between the two tubes while the bath temperature increases. Since exact calibration is not important for the differential temperature (ΔT) measurement, we have constructed the differential pair from a single piece of constantan wire soldered at each end to two pieces of thin copper wire (#24 gauge used, available from telephone hookup wire). A sketch and photograph of our apparatus are shown in figure 13 and additional details on the construction are available on our website.

For the DTA we have chosen to introduce thermocouples as the preferred temperature sensor for three reasons. First, they enable a differential measurement essential to the method; second, they introduce the student to yet another method of temperature measurement used in the laboratory; and third, they allow the extension of this experiment to higher temperatures than the thermistor based digital probes can tolerate.

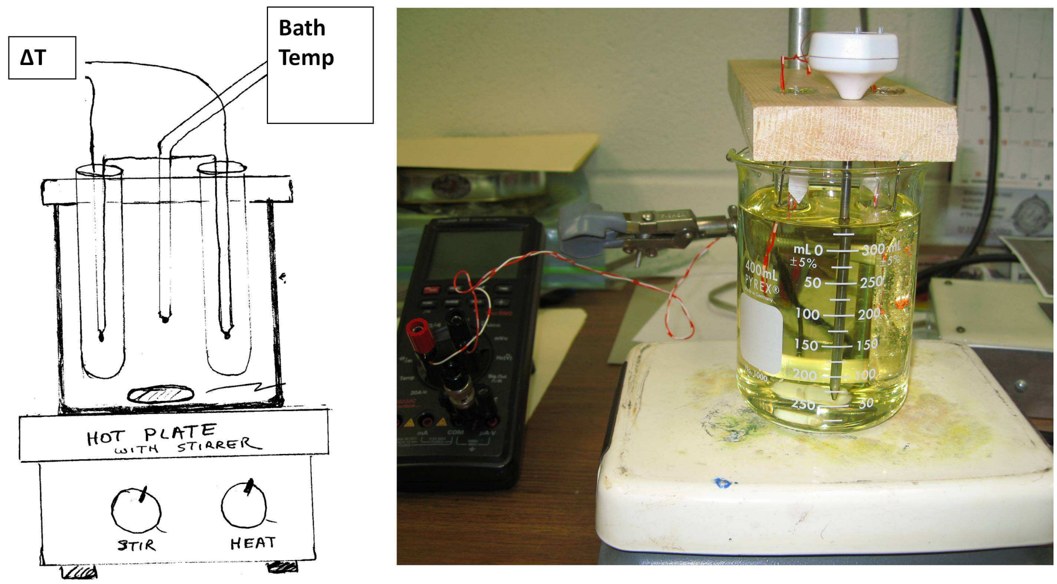

Figure 12. Sketch of the arrangement of a student-built DTA apparatus, together with the photo of a simple implementation with digital thermometer for bath temperature, and a single thermocouple meter for measuring the differential temperature.

In order to obtain a well defined T_g, it is important to quench the sugar glass prior to the measurement run; otherwise, you may not obtain a glass transition. Standard thermocouple meters are adequate to collect the data point by point, and low cost meters are available from about $30 (Harbor Freight). However, to minimize the tedium of such manual data collection, we also included automatic data logging to our DTA experiment. A simple approach would be to use one of the commercial or educational instrumentation (e.g. Pasco, Vernier) [26] available in high school and college labs. However, to remain consistent with our low cost and home-built approach, we also developed our own custom-built data collection instrument utilizing a relatively low cost microprocessor platform. The Basic Stamp Microprocessor (by Parallax, Inc.) was chosen for its low cost, ease of learning and wide popularity within both the educational and hobbyist communities. (There are many sensors designed to interface directly with the Basic Stamp platform, including a thermocouple module (based on DS2760 one wire interface chip) and a humidity sensor chip. There is also a substantial amount of educational material and support available from the Parallax website (http://www.parallax.com/). Although it adds some additional complexity up front, introducing the Basic Stamp for data collection allows the student to learn something about microprocessors, providing a much more flexible, easily adaptable tool for a variety of other experiments involving measurement and control. However, the choice between a commercial vs. home built approach to data measurement and collection will depend on the resources and skills available to the student investigator. Either way, automating the data collection, especially of temperature in thermal scans, enables the student to focus more on the observations and less on the tedium of collecting multiple data points. A more complete description of the details of our apparatus, including the electronic instrumentation, is included on our website.

Sample data for our sugar glass (standard 2:1 recipe) using our DTA are shown in figure 13. Here a very distinct T_g is observed with a step that commences just above 29°C that flattens by 55°C. Using a midpoint definition for T_g, we calculate a T_g of about 42°C for our standard sugar glass recipe. This value is in fair agreement with measurements made on a commercial DSC (TA 2920), where a midpoint of 38°C was measured. The small difference between these two values can be accounted for, at least partially, by differences in thermal history, heating rate, etc.

One should be aware that the details of the thermal behavior in the glass transition can be quite dependent on heating rate as well as the thermal history of the sample. We have had many frustrating opportunities to experience the dramatic variation in results that is possible when proper control of thermal history is not understood. For the measurements reported here, the sample was first quenched from the melt into a beaker of ice water and then allowed to stabilize overnight. Observing this variation of DTA scans with different sample history, while frustrating initially, provides a powerful opportunity for the student to really experience the meaning of fictive temperature and the truly non-equilibrium nature of glassy materials. For this reason it is advisable to have the experimenter begin first with a simple, non-glassy material, such as a low melting point crystalline solid to understand and calibrate the apparatus. Stearic acid was utilized to provide such a reference material. It provides a clear melting transition at about 70°C (and was used as our calibration standard).

The DTA provides the student with a relatively simple, hands-on, yet quantitative, access to the glass transition phenomena. Besides the advantage of low-cost access, our home-built DTA provides the student with a much deeper understanding of the underlying science and instrumentation inherent in the method. Likewise, the transparency of the design permits the student to actually peek and poke at the material as it goes through the various stages of the transformation. One can see the closure of cracks developed on cooling, the softening and subsequent melting of the glassy material and finally the evolution of water vapor bubbles as the material approaches its original preparation (boiling) temperature. Such visual access provides a wonderful stimulus for pondering many aspects related to glass science, indeed materials science.

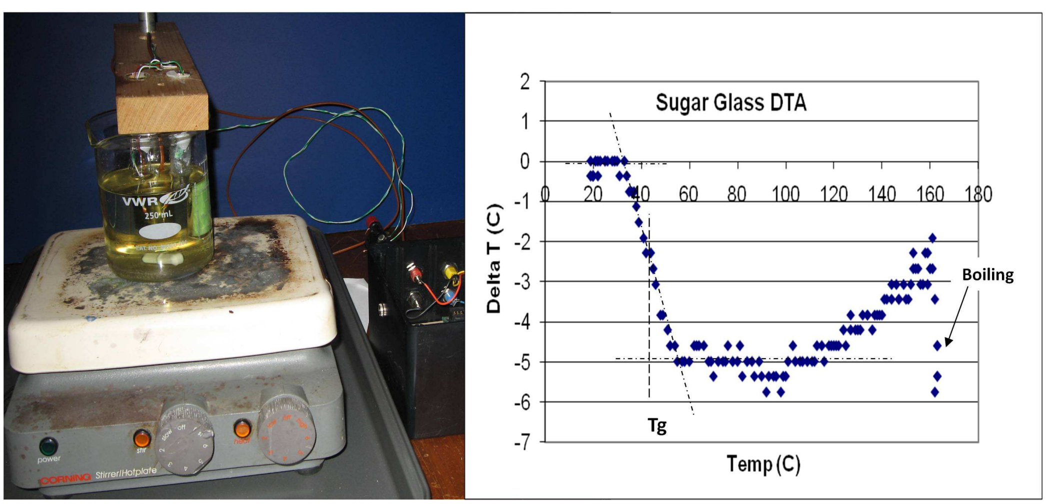

Figure 13. Photograph of the DTA apparatus with Basic Stamp microprocessor for temperature measurement and data logging. A sample DTA scan for the candy glass is shown on the right, with midpoint T_g indicated.

IV. SUMMARY

We have developed a hands-on, mini curriculum for exploring many aspects of glass science through experiments with sugar glasses. Combining a range of activities from material synthesis to measuring basic material properties with student-built equipment, we have developed an informal educational resource designed to facilitate interest and active prolonged engagement of the student with glassy materials in both a relevant and quantitative context. By utilizing a website to house and distribute our resources, we have established a means to reach a large student and teacher population while providing a fast and flexible means to revise and add new material as it is developed.

A complete description of the experiments presented here can be accessed at our website by any interested student or teacher. We include a broad range of materials in a variety of forms (video discussions, experimental procedures, tutorial modules, student presentations, laboratory resources, etc.). The information can be discussed and understood at varying levels of detail, including specific construction methods and procedures, not appropriate in a more formal publication format. Using a web-based approach for distribution of information also allows its regular updating. It is our intention that many additional hands-on activities and experiments will be added on an ongoing basis in the future. Hopefully, this resource will find application in the high school as well as college laboratories, and provide a resource for students and science enthusiasts to explore on their own in their home. We welcome any interesting and appropriate submissions from others in the glass community.

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26. Vernier (http://www.vernier.com/) and Pasco (http://www.pasco.com/) both produce data collection instruments commonly found in high school and college labs.