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Papers

A Glaze Process for Using Boron Trioxide (B_2O_3) to Reduce Adverse Effects of Alkali Vapor on Glass Cells

by

Graham Driver-Schroder*

ABSTRACT

An alkali-resistant layer can be glazed onto the inner surface of a glass cell by introducing an aqueous boron trioxide (B_2O_3) solution, evaporating the H_2O and then melting the residual biborate crystals into an interfacial glaze.

PROPOSITION

Glasses offer almost infinite possibilities to researchers and often are made to specific applications because of this; examples include Corning's Gorilla glass, Schott's BG series, and many others. These glasses have unique properties which make them valuable in the field. Sometimes the applications of these special glasses are offset by the cost of manufacturing them. There are a few factors as to why the glass that researchers need is not always cost effective to make which include scale of production, material costs, facility and energy costs, and general expenses laid out by the manufacturer. What this says is that new innovations are limited by manufacturing expenses associated to existing materials which could compromise the result of the new application. The solution to researchers could be coatings and glazes; offered as an example are ITO or Indium Tin Oxide coatings which provide optical, electrically transmissive coatings. The ITO glaze proposes a chance to make any composition of glass electrically conductive while maintaining the optical nature of the glass. The method for applying these ITO glazes is similar to the way the boron trioxide glaze is prepared. Figure 1 shows this process. The concept of improving properties of glass by the use of coatings and glazes offers researchers the opportunity to use a more standard and reasonably priced glass composition for their studies. Some applications demand a glass that can withstand certain volatile atmospheres; examples of this include SERF magnetometers and atomic clocks. These applications limit the glasses to be used in the system dramatically. If these requirements were applied to experiments being done by an underfunded group, then a glass composition which has heat and alkali resistance molecularly intrinsic would be out of the picture based on the cost of manufacturing. When this is the case, interfacial glazes can be used to meet certain requirements while maintaining a reasonable cost.

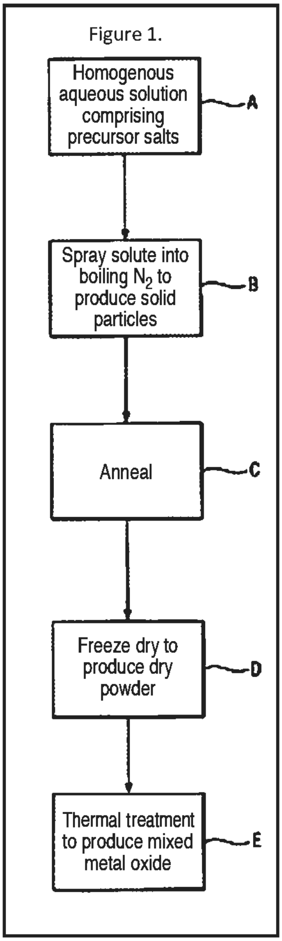

Figure 1. *ITO glaze routine*

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TASK

A boron trioxide based solution can be used as a glazing agent for glass that comes in contact with alkali as it is resistant to the darkening that occurs to most glasses that interact with alkali. Remaining transparent is the most important thing when dealing with alkali and glass because usually the glass must remain optically transmissive to work in these specific parameters. This is the case for High Pressure Sodium Light bulbs as when the alkali sealed inside the bulb darkens the glass, there is less light emitted. Similarly for SERF magnetometers, the cell used to house the alkali while in spin-exchange must retain optical quality to ensure that the laser used to induce the spin-exchange is not refracted in an unwanted way. What this means in application is that a cell that needs some level of alkali resistance could be treated with a boron trioxide glaze to reduce the darkening effects of the alkali on the glass. This reduces the overall cost of doing research with alkali and glass. The procedure for applying this glaze was broken down into four steps; introduction of aqueous B_2O_3, evaporation, removal of dense biborate crystal growth, and melting the residual biborates into an interfacial glaze. The first trials were done using a furnace as means of evaporating and firing the glaze. Cells would be placed into the kiln and gradually brought up to 550ºC.[1] This proved the theory, yet was excessive in time for small scale trials. It was found that for this scale of experiments, manually evaporating the solution using a bench burner and ring stand proved to be most appropriate and provided satisfactory results in comparison to evaporating the solution in a furnace. The same proved to be true for melting the residual biborate crystals into a glaze. In fact for the last procedure in this process, using the bench burner to manually melt the biborates into a glaze provided a more transmissive surface with fewer striae. Some cells towards the beginning of the trials were unusable due to cracks in the glaze itself, shown by hairline fissures reminiscent of interior surface cracking. This was solved by ramping the kiln down slower than a usual annealing cycle. What this says is that the glaze has an independent coefficient of expansion that should not be overlooked.

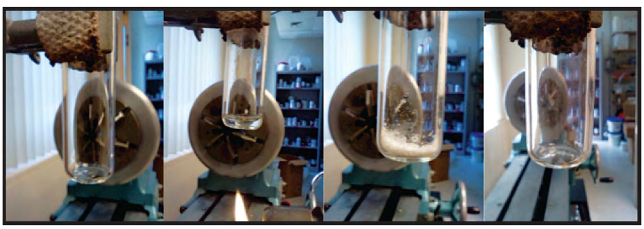

Figure 2. *B_2O_3 glazing routine*

PRIMARY RESULTS

The boron trioxide glaze was administered to vials of Corning's 4450 which were medium wall weight and 25 mm o.d. The majority of the cells remained totally transparent with few striations and a glaze thickness of four nanometers on average—as shown by SEM analysis. The solution being introduced into these cells was saturated with B_2O_3 according to the msds, that being 22g/L. The cells which were found unsuccessful in the glazing were so based on extreme differences in the biborate crystal growth and resulted in turn in a less transmissive glaze. Some of these inconsistencies can be attributed to intended variances in the percentage of boron in the solution however. Table 1 shows some data points from an early trial in further saturating the solution by adding anhydrous boron into the cells individually after having 1 ml of boron trioxide solution introduced; this was done to determine if elevated percentages of boron leave a more thick or even biborate deposit.

[1] Glazing temperature ranged from 550º-580ºC based on variances in the research. It was found that the closer to the slumping temperature the cell got, the more transmissive and even the glaze would be.

Cell	Solution	Procedure
A	1ml	Introduced 2.5 mg B_2O_3
B	1ml	Introduced 5 mg x 2
C	1ml	Introduced 10 mg
D	1ml	Control

Table 1. *Data table showing experimentation with elevating B_20_3 concentration*

What was found with this experiment was that more B_2O_3 added into the solution meant larger more dense crystals, not necessarily a thicker, more even coating. Cell 'A' cracked during evaporation, yet the biborate growth was still measured. After evaporation, cell 'B' had an additional 1 ml of B_2O_3 solution introduced and evaporated. This was done in hopes to create a thicker more sustainable optical glaze. Though the glaze was thicker, it was not as transmissive. Cell 'C' contained very dense biborate growth also full of striae. The control cell 'D' which just had 1 ml of B_2O_3 solution remained the most transmissive and evenly coated.

Control cell D remained most suitable for alkali resistant application due to its even coating and transmissive nature. It was decided after this experiment not to use innoculative anhydrous boron in producing these cells as it tends to lead towards an uneven growth of biborate crystals.

Cell	Solution	Procedure	Procedure 2
A	2 ml	.25 ml glycerin	.25 mg B_2O_3
B	2 ml	.5 ml glycerin	.5 mg B_2O_3
C	2 ml	.75 ml glycerin	.75 mg B_2O_3
D	2 ml	1 ml glycerin	1 mg B_2O_3
E	2 ml	1.25 ml glycerin	1.25 mg B_2O_3
F	2 ml	1.5 ml glycerin	1.5 mg B_2O_3
G	2 ml	1.75 ml glycerin	1.75 mg B_2O_3
H	2 ml	2 ml glycerin	2 mg B_2O_3
Control	2 ml	0 ml glycerin	0 mg B_2O_3

Table 2. *Data plot showing experimentation with glycerin in solution*

A trial was conducted before the experiments in which the concentration was altered: glycerin was added to the solution as a dopant in hopes to increase the solubility of the boron and to increase the viscosity of the solution. With a thicker more concentrated solution, it would coat the cell more evenly before evaporation and in turn deposit a more even layer of biborate crystal growth. With the glycerin, additional levels of B_2O_3 were added.

Though the solution did coat the cell more evenly, no matter how small the amount of glycerin in the solution was, it would leave a dark residue after evaporation. Adding glycerin to the solution also made the solution more sensitive exponentially. The solution had to be evaporated off much more slowly when glycerin was added.

CONCLUSION

It is possible to create an inexpensive alkali-resistant glaze using B_2O_3 that can be administered to borosilicate glasses. The samples produced had four nanometers of biborate glaze; this ensures that a glass surface remains optically transmissive under a volatile alkali rich atmosphere.

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An Extremely Efficient Vacuum Trap
A New Design

by

Georges Kopp*

ABSTRACT

This paper is divided in two parts: the first part dwells mostly on the proper and safe usage of traditional vacuum cold traps, the second part presents the genesis and the steps which led to the development of a radically and totally new design of a vacuum cold trap along with the steps of its fabrication. This design dramatically improves the efficiency of conventional vacuum cold traps.

INTRODUCTION

The subject of this paper is a new design for a highly efficient vacuum trap. Before going into details on this new design which greatly improves the efficiency of conventional vacuum cold traps, a review of the proper and safe usage of traditional traps will be made; this will include the purpose and functions of traps, safety considerations when using cold traps, and the proper use of cold traps.

PART 1

Introduction On Vacuum Traps

a. The Purpose and Functions of Traps

Traps are used on vacuum systems because of their ability to remove (or bind) condensable gases.[1] Traps protect both the pump from solvent that could damage the seals or by breaking down mechanical pump oils and they protect the user(s) from toxic vapors that could have gone though the pump.[2] (Exhaust must be vented to a fume hood.) Traps when used properly will even increase the vacuum in the system.

b. Safety Considerations When Using Cold Traps

Prevent air (Oxygen) from freezing within the cold traps.[3]

One of the more common laboratory accidents can occur when air (oxygen) is frozen in a cold trap. This accident is caused by placing a cold trap within liquid nitrogen while there is still sufficient air within the trap to be frozen. Later once the liquid nitrogen is removed (either intentionally or is boiled off), the frozen air vaporizes. The excess pressure created by this frozen air can result in an explosion of the entire line. To prevent oxygen from freezing in the cold trap, be sure that there is no air in the trap when pouring liquid nitrogen around the cold trap. One simple way to prevent explosions is to keep the line under continuous state of vacuum.

Limit the amount of moisture near the top of your cold trap.[4]

A common error when first starting up a vacuum system is pouring liquid nitrogen too high into a Dewar. People often overfill a Dewar in the early start up process because the liquid nitrogen boils off very quickly and there is a desire to overfill. The problem originates because

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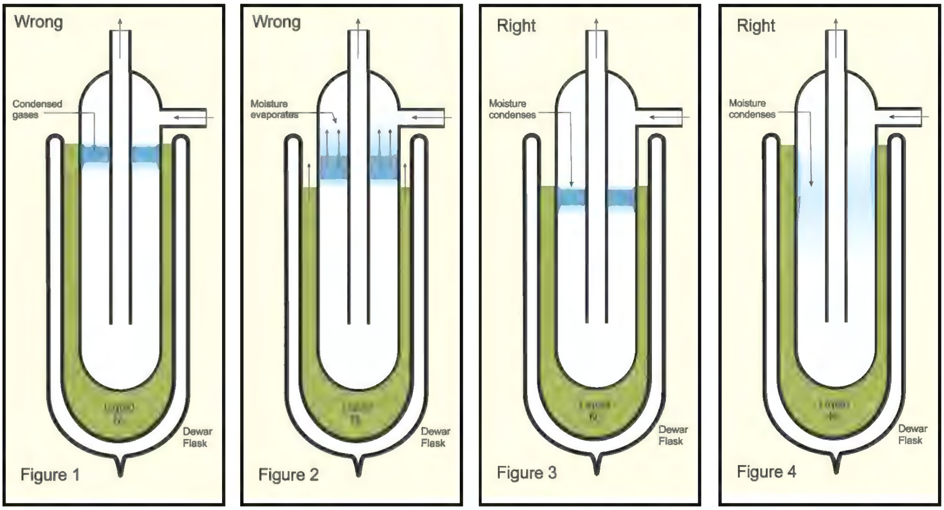
[1, 2] Gary S. Coyne, *The Laboratory Handbook*, 7.4.1 (Englewood Cliffs, New Jersey: Prentice-Hall, Inc., 1992) 328, 330.

[3] Coyne 333, 334.

[4] Coyne 333, 334.

the liquid nitrogen high in the Dewar freezes moisture in the upper regions of the cold trap. Later as the liquid nitrogen boils off, the moisture at the top of the trap evaporates and the pressure rises. This problem is easily prevented by maintaining a low liquid nitrogen level when starting the system. Later, by maintaining a high liquid nitrogen level, any moisture that is trapped will stay frozen. (Figures 1, 2, 3, and 4)

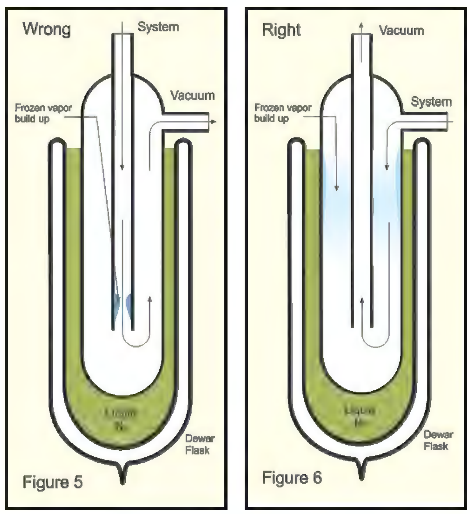


Figures 1-4. *Limiting the amount of moisture near the top of the cold trap.*

c. Proper Use of Cold Traps

Proper and improper orientation of cold traps.[5]

The trap orientation is not critical if the vacuum system operation does not generate copious amounts of condensable vapors. If the trap is attached the wrong way, there can be a build up of frozen material in the center tube resulting in a reduction of the trap's throughput or eventually a close-up. When the system is attached to the side inlet, the material will freeze on the side wall as it passes through the cold trap. There is no restriction of flow with this orientation. (Figures 5 and 6)



PART 2

A Highly Efficient Vacuum Trap

Steps in the Conception of a Completely New Design of a Cold Trap

Conception of a Completely New Design of a Cold Trap

A post-doctoral researcher from the McGill University Mechanical Engineering Department

[5] Coyne 332.

who was conducting research on new fuel efficiency approached me to design a special vacuum trap. Despite the fact that a conventional vacuum trap had already been built on his manifold to stop hydrocarbon samples from entering the gas analyzer, the researcher noticed that there were still some not being trapped. He asked me to see if it would be possible to improve the efficiency of the vacuum trap.

The trap used by the researcher was properly immersed in a Dewar flask full of liquid nitrogen and it looked as if there was not any way to improve the efficiency of the setup unless one used two traps in series or if one used a larger trap. Unfortunately, the lack of space under the analyzer was a limiting factor. Obviously the only way to improve the efficiency of the trap would be to increase the contact area with the liquid nitrogen in order to freeze the hydrocarbon either by lengthening the tube or by getting some liquid nitrogen inside the trap.

I began making drawings to see how that could be possible considering the space limitation. At first it looked impossible or much too complex in terms of glassblowing to make it practical. **Then I had an inspiration: why not simply get the liquid nitrogen to enter the trap through its bottom** so as to cool it down from inside as well. This way the gas would travel **twice** in contact with it and that would give it more time to condense. **That would double the efficiency of the trap without taking up any extra space.** After doing a few experimental drawings, I came up with a new design for a revolutionary vacuum trap.

This trap is built from three concentric tubes, two of them welded at their extremities in a Dewar flask style and an inner one welded only to the top. The assembly offers a double column of gas to be in contact with the liquid nitrogen. To me, the new design looked like it was going to solve the problem. Once a model had been built and tested by the researcher, it was proven to be perfectly adequate to stop the hydrocarbon gases from entering the analyzer without taking any more space than a conventional vacuum trap. (Figure 7)

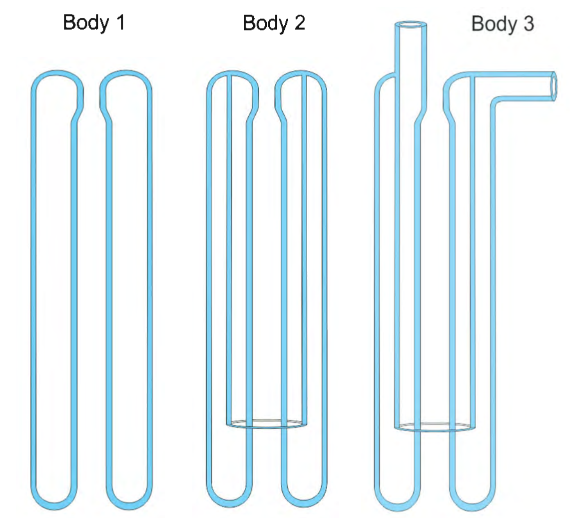


Figure 7

The new design not only has the advantage of being extremely efficient but it can also be connected in any which way since it is symmetrical. The user cannot make the common mistake of connecting it to the manifold the wrong way. Furthermore, the two tubes forming the trap are both quite wide, much wider than the inner tube in a conventional trap and are closer to the liquid nitrogen thus offering a better contact while minimizing the chances of blocking.

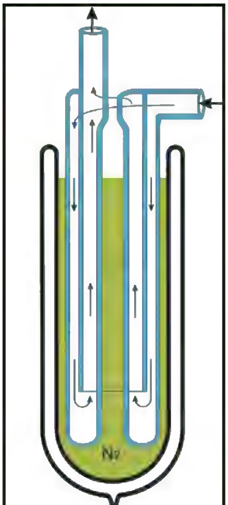


Figure 8

Last but not least, the trap is quite easy to build by using each tube in sequence so that the previous tube will hold the next one in place, no tools or jigs are needed to hold it. (Figure 8)

Construction of the Trap

In Figure 9, three tubes have been prepared on the lathe. Two of them have two sets of three small indentations done on the lathe to insure perfect fit, each one reaches the next inner tube to keep it concentric with the outer one that will hold it.

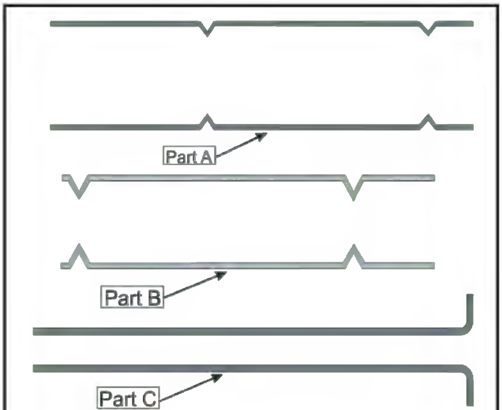
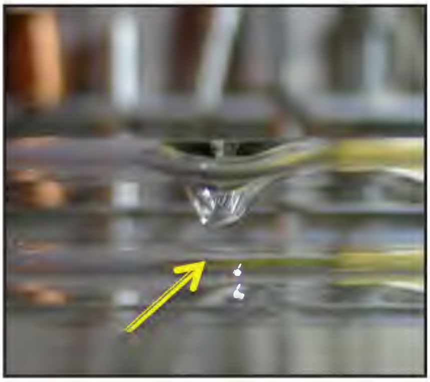


Figure 9

Picture 1

First step: the indentation is melted wide and partially pushed in only in order to build a thick layer that will be allowed to cool down a little before going to step 2. (Pictures 1 and 2)

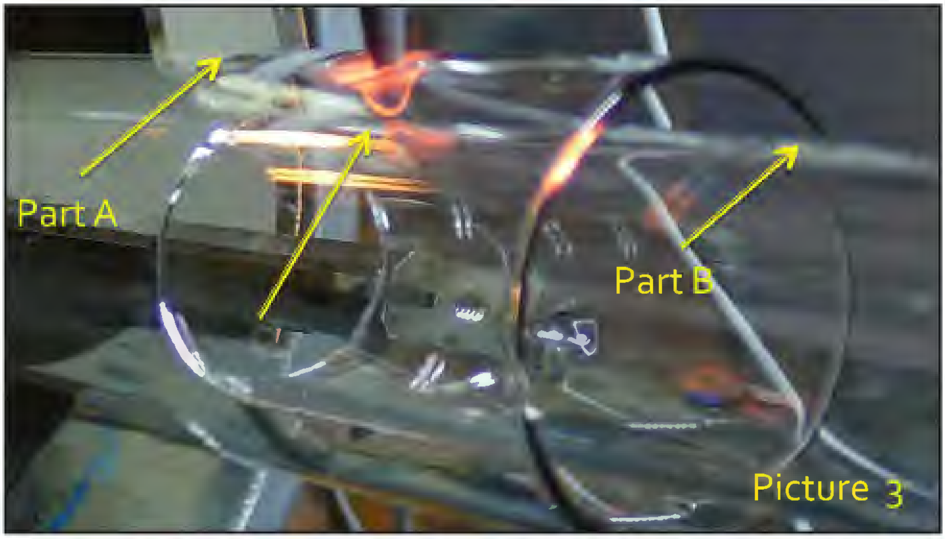


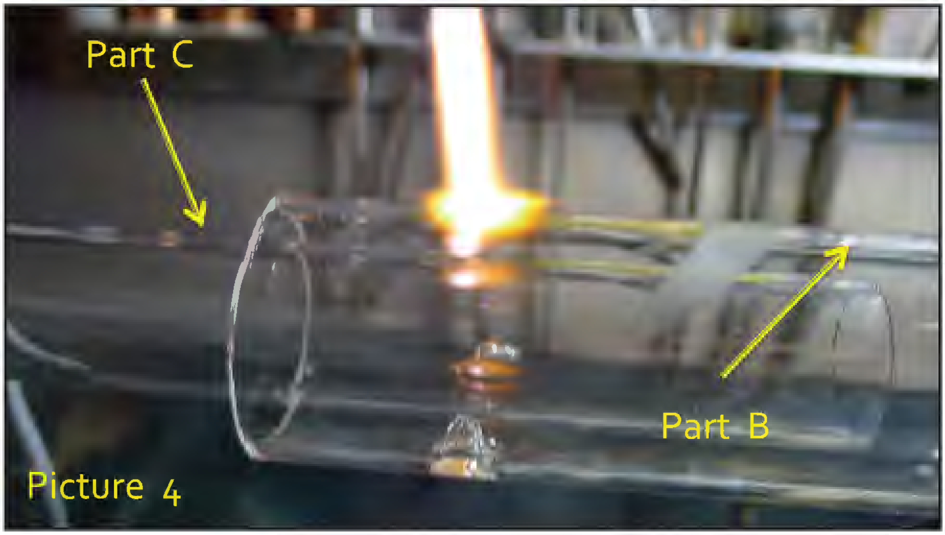
Picture 2

Second step: I melt only the outside and push in until it fully contacts the inner tube. (See middle arrow of Picture 3)

The same operations are repeated for part B. (Picture 4)

In Figure 10, the three tubes have been inserted into each other. They are kept in place and concentric by the two sets of three small indentations. The innermost part "C" had been flared to fit exactly into part "A."


Part A
Part B
Picture 3


Part C
Part B
Picture 4

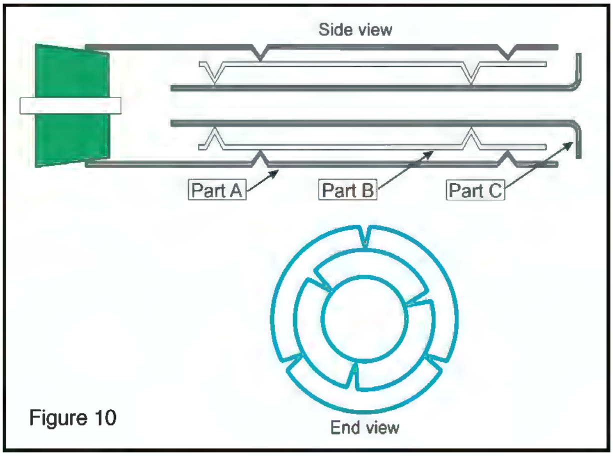

Side view
Part A
Part B
Part C
End view

Figure 10

In Figure 11, part “A” and part “C” will be fused together in the form of a Dewar flask seal.

Figure 11

Parts “A” and “C” in the process of being welded

In Figure 12, the Dewar flask type seal has been completed. The Dewar type seal has been blown in to increase the top area to give more space to attach the connection tube.

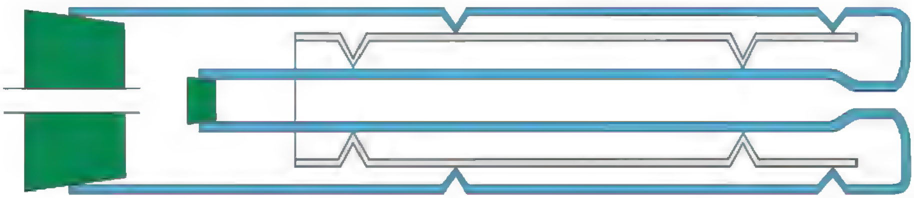


Figure 12

In Figure 13, a long enough piece of glass with a flare or flange is inserted in the trap while it is rotating on the lathe and melting the Dewar end; the piece of glass will push part “B” into the molten glass to make a good seal. Pay attention to avoid doing a “square” seal or it will crack later.

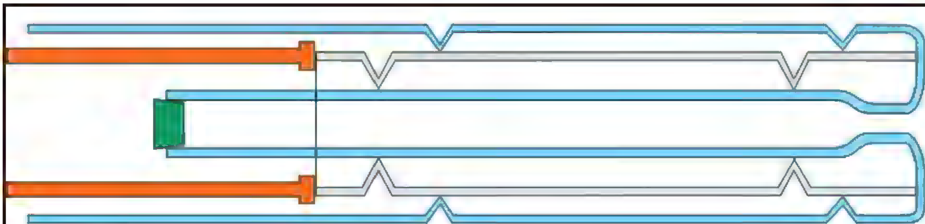


Figure 13

In Figure 14, the middle part “B” has been welded to the Dewar seal to complete the upper part of the trap.

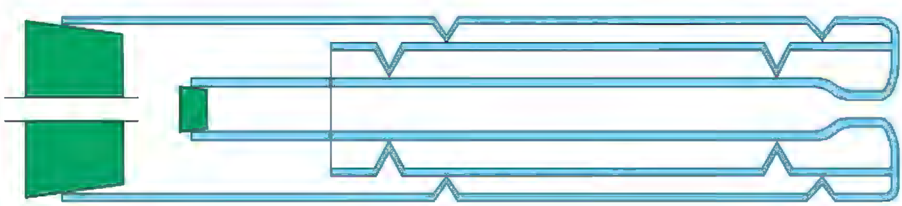


Figure 14

In Figure 15, the seal has been completed and the two arms welded in place. One is connected to the outer section of the trap and the other one to the inner section.

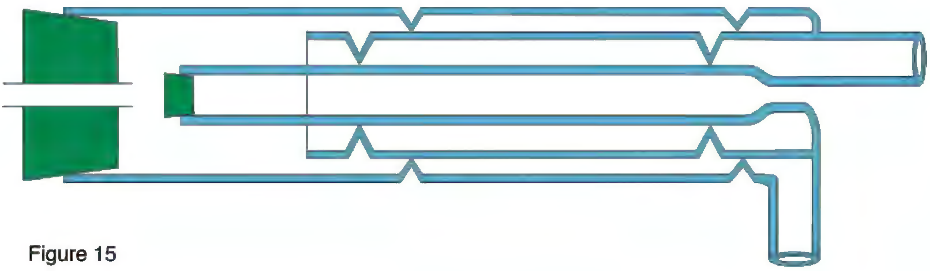


Figure 15

The trap, after being annealed, has been returned to the lathe but flipped over in the head stock. In Figure 16, the outside tubing has been trimmed to the desired length and the inner part cut to an appropriate length and flared to fit the outside wall as closely as possible.

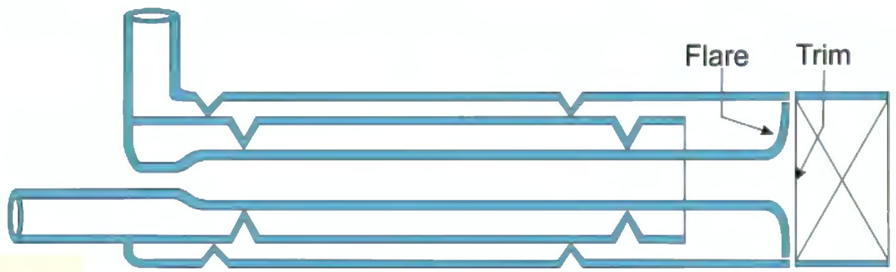


Figure 16

In Figure 17, the parts "A" and "C" have been properly welded together, the Dewar flask type seal has been completed. The trap is ready to be connected to a system after annealing.

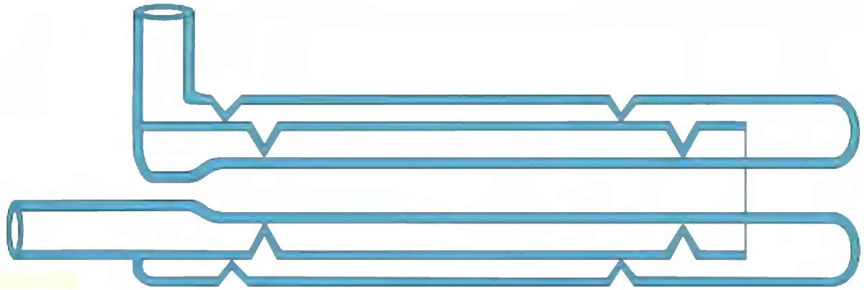


Figure 17

Cold Trap in Operation

On this final drawing (Figure 18) one can see the trap immersed in liquid nitrogen; the vapors will travel twice, down and up in the coolant. This design results in a much more efficient cold trap than a traditional one. This vacuum trap was connected to the manifold via two Cajon® Ultra Torr stainless steel connectors, they were chosen for their ease of connecting/disconnecting the trap from the manifold to drain it once the experiment was completed.

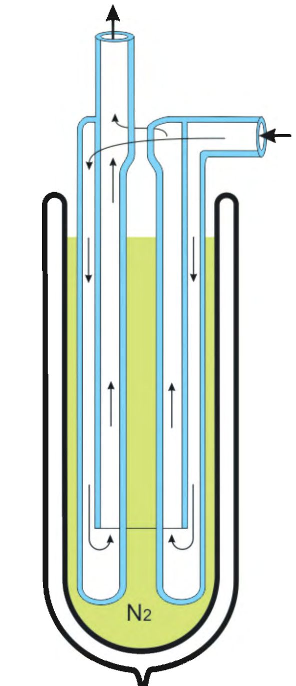


Figure 18

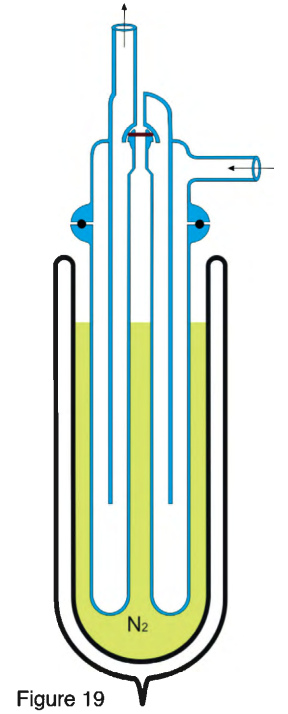


Figure 19

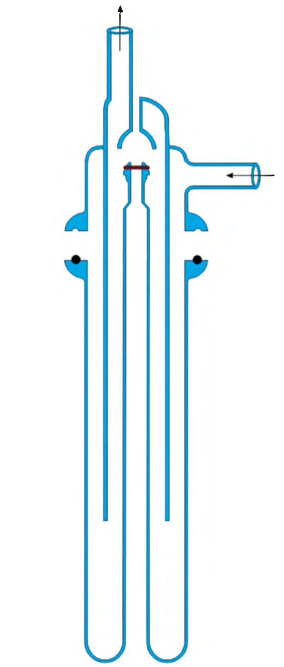


Figure 20

The model shown in Figure 18 could be made of two parts to enable disassembly. (Figures 19-20) If one wanted to have the same style of a trap permanently attached to a manifold it would have to be made of two parts. That can be achieved with the use of a pair of size #40 O-ring joints and a set of 18/9 ball joints with O-ring. One can see such a design in Figure 19.

Trap Being Disassembled

This design of cold trap does not present any special problems to build. It is made of two simple Dewar style seals. The only critical consideration is to carefully adjust the length of the outer member ball joint. I found that it can be held in place temporarily with Krazy Glue® over the inner member ball joint without the O-ring in place. The rubber O-ring is removed from the #40 joint while using the outer part as a support to seal the 18/9 joint in place. (Figure 20)

Construction of the Trap

The outer/inner most part of the trap is done as a simple Dewar style connection. (Figure 21)

Figure 21

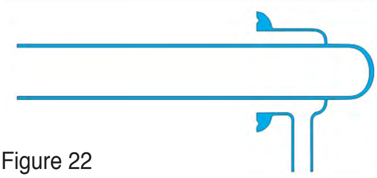


Figure 22

The outer / middle part of the trap is as well a simple connection, the side arm has been connected. It will be used later to blow into the trap to attach the outer member 18/9 ball joint. (Figure 22)

The outer / inner part of the trap is used as a holder to attach the outer member 18/9 ball joint to the upper part of the trap; the appropriate metal clamp for a #40 joint is used for that purpose. The outer part had a hole blown beforehand to speed up the connection. The outer joint is glued temporarily to the inner member with a little cyanoacrylate glue. The connection is to be done quite fast since the glue will not hold for long. Later the trap will be annealed and the glue will burn away in the oven. (Figure 23)

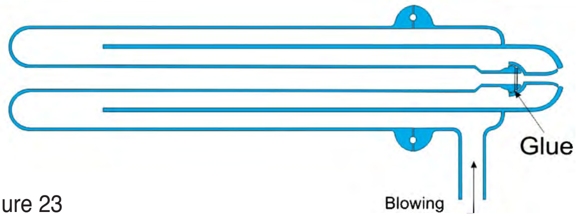


Figure 23

Once the outer member ball joint has been welded properly, a hole is blown and the second side arm is attached. The construction of the trap is complete. (Figure 24)

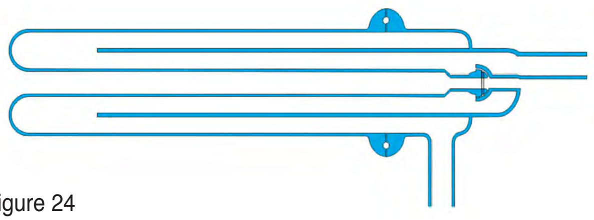


Figure 24

A size #65 stainless steel clamp is used to hold the 2x #40 Glass O-ring joints. (A in Figure 25) No clamp is necessary to hold the ball joint, the pressure from the #65 clamp is sufficient. (B in Figure 25)

Holding the Two Parts Together

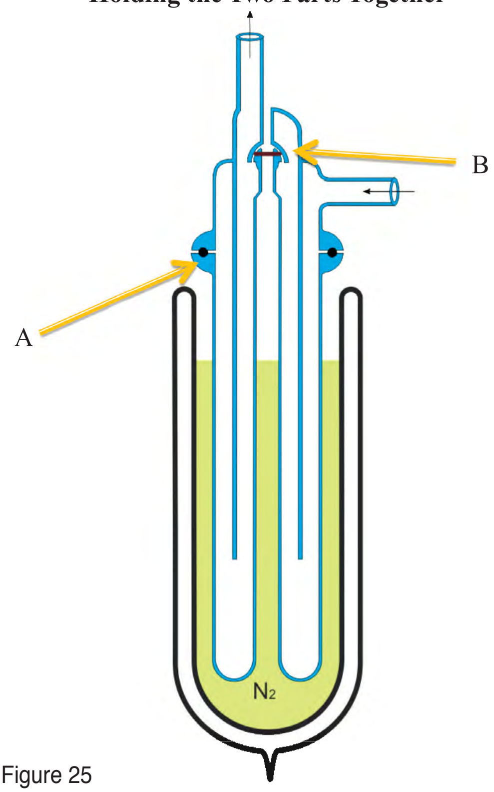


Figure 25

Constructing a Large Glass Vacuum Chamber for a Specialized Vacuum Deposition Process

by

Joseph S. Gregar*

ABSTRACT

This paper will detail the construction techniques for the fabrication of a large glass chamber. This chamber called the Chalice will be used for the vacuum deposition of exotic metals onto the surface of a glass cathode plate for a new prototype photo detector. The paper will also detail the technique that was used to get 21 electrical leads inside the vacuum tight chamber.

INTRODUCTION

This paper presents the construction techniques for the fabrication of a large glass chamber. This chamber, called a Chalice, is designed to be used for the vacuum deposition of exotic metals onto the surface of a glass cathode plate of a new prototype photo detector. The paper also details the techniques involved to incorporate 21 electrical leads inside the vacuum-tight chamber. (Photo 1)

Photo 1. *Chalice body*

CHALICE CONSTRUCTION

The Chalice is constructed from Schott Duran® #8330, borosilicate tubing 200 mm i.d. x 217 mm o.d. The wall thickness of 8.5 mm allows for its use in vacuum service without fear of implosion. The Chalice consists of four main body parts:

- A 200 mm i.d. top flange
- A 217 mm x 152.4 mm long center section
- A rounded lower section
- The tail section

The tail section is 50 mm o.d. x 5” long and consists of a graded seal of transition glasses to go between the borosilicate tubing and a Kovar 21-pin stem seal.

The following glasses were used in the transition between the Kovar sealing glass and the Chalice: Schott #8245, Kovar sealing glass, coefficient of expansion (coe) 5.1 is used for the 21 pin stem seal; Schott #8337B, coe 4.1 is the first intermediate grading glass; Schott #8487, coe 3.9 is the second intermediate grading glass; and Schott Duran® #8330, coe 3.3 is the borosilicate tubing.

There are two bulges integrated into the body of the chalice. The upper external bulge is 75

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mm from the top that allows the Chalice to rest on the support stand. An internal bulge supports a "glass tulip" that holds the electronics inside the Chalice at a specific height. Note that the precise location of the lower internal bulge is critical to make the required electrical connections.

The purpose of this Chalice is to process exotic metals onto the inside surface of a piece of Schott B-33 Borofloat plate glass which will be used as the cathode plate of a prototype, large area picosecond photo detector cell. Information on this photo detector can be found in the *2011 Proceedings of the American Scientific Glassblowers Society* on page 9.

This 21-pin connector was commercially made by ET Enterprises, a company with headquarters in the United Kingdom and a United States distributor in Sweetwater, Texas. They manufacture electron and photomultiplier tubes and fabricate complicated pin seals with an exceptional degree of quality and reliability. These Kovar metal pin seals are vacuum tight and allow for the feeding of electrical current into the Chalice to evaporate the desired exotic metals onto the cathode plate, which is positioned at the top of the Chalice. (Photo 2)

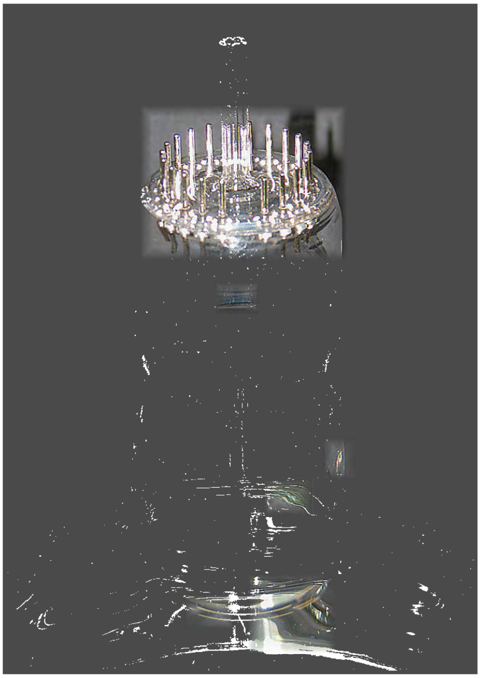


Photo 2. *21-pin connector*

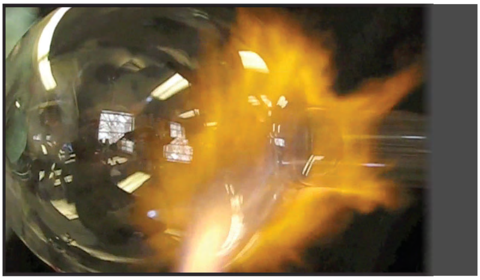


Photo 3. *Splice 50 mm o.d. tube onto a 5-liter round bottom flask*

To start the fabrication process of the Chalice, a length of 50 mm o.d. borosilicate tubing is spliced onto the bottom of a 5 liter round bottom flask. (Photo 3) After annealing, the flask is rotated in a glass lathe and a score mark is made at the location where the outside diameter of the flask is about 220 mm diameter. After the score is made completely around the flask, a very small sharp gas / oxygen flame is carefully placed on the score – in a tangentially orientated fashion – until the flask cracks apart. The flask is then set aside until needed.

A six-inch section of the 200 mm x 217 mm o.d. Schott Duran® tubing is cut using the hot wire thermal shock technique. The tubing and flask are chucked in 3-finger planetary chucks. A gas/air warming fire begins the heating of the parts, followed gradually by a bushy gas/oxygen flame before the sealing flame is turned on. A Litton, 10-fire, 7-jet tip ring burner connected to hydrogen and oxygen fuel supplies is used for this seal. This is a "no-blow" seal made by carefully controlling the rotation speed of the lathe. If the lathe rotation speed is too great then the ends of the tubing and the flask will flare outward.

Personally I always use the "peeling" technique to remove the score marks from the cutting

Photo 4. *Splice cut flask onto the 217 mm o.d. tubing section*

operations. The outer edge of the tubing wall is heated carefully and a warmed 10 mm borosilicate rod is used to "peel" the outer corner of the glass away leaving a nice clean edge. This technique is used on both the tubing and the cut edge of the flask. Both edges are then thoroughly heated to assure that the full thickness of the glass walls are softened to the "liquid" state before sticking them together. The joint area is heated with the hydrogen/oxygen ring burner from underneath and a gas/oxygen hand torch from above. Both of these are held directly on the joint to insure complete flowing of the seal; the seal is finished by holding a flat graphite paddle on top to achieve an aesthetically pleasing transition from the straight tubing wall to the spherical shaped flask. Careful and rigorous flame annealing is now done. Use a gas/air warming burner such as a Litton "Sun Spot" to heat the vessel while setting up for the next operation. (Photo 4)

NOTE: Other options were considered for making this lower portion of the Chalice. First, a longer length of the 217 mm o.d. tube could be heated in the center and pulled down to close off the tube and then blown to make the rounded bottom. However, this would use more of the very expensive 217 mm o.d. tubing, take more "fire power" and probably a lot more time. If successful, the only benefit would be that two domed sections would result. The second alternative involves sealing a thick flat borosilicate plate into the end of the tube and blowing it out into a shallow spherical shape. Not having a large plate available, this technique was not attempted.

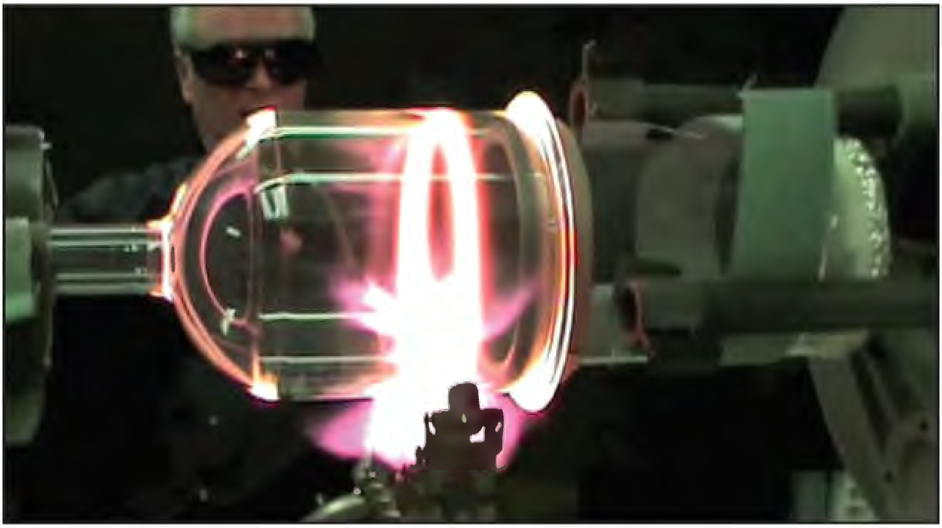


Photo 5. *Seal the 200 mm i.d. kettle flange onto the top of the vessel*

The 200 mm i.d. Schott kettle flange comes in a length of 75 mm. This gets spliced directly onto the top or second end of the 6" long 217 mm o.d. tube. (Photo 5) You must be certain that the flange is running very straight and true before splicing it. The flange also has to be snug on the holder so it does not tilt or walk off the holder during the splicing. This second or "top" splice is done with the same torch configurations as previously stated. It is done without blowing but the holder is set up for blowing during the next step. A short flame annealing is done at this point to widen and smooth out the strain pattern.

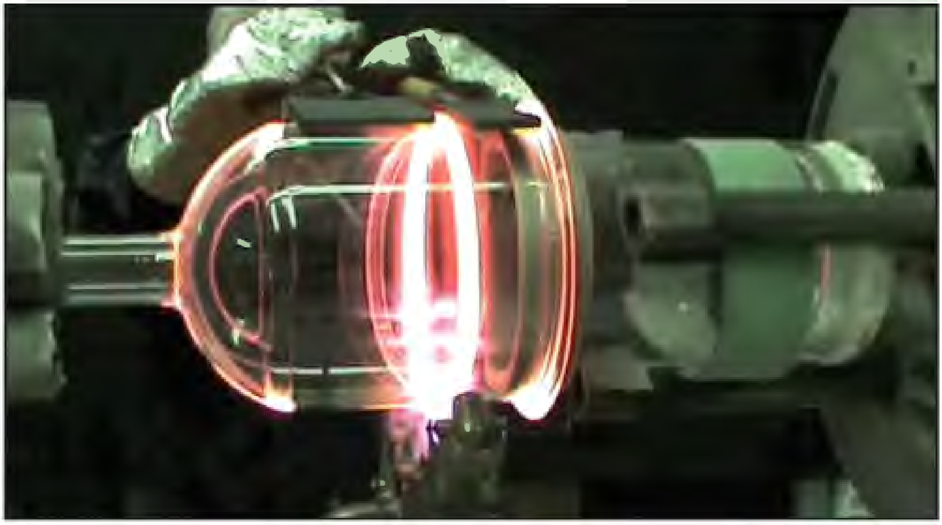


Photo 6. *Form the outer support bulge*

After completing the splice you must form an exterior bulge that is 10-12 mm larger than the o.d. of the tubing. (Photo 6) This bulge will be used for supporting the Chalice in a vertical position on a metal stand. This is accomplished by turning up the rotation speed of the lathe slightly, very gentle blowing through the headstock holder and, when needed, pressing downward on two flat graphite paddles to maintain a uniformly thick or wide bulge of about 10 mm. Again flame anneal after this operation with a bushy natural gas/oxygen flame.

FORM THE INTERNAL SUPPORT BULGE

After the flame-annealing step, quickly move your lathe ring fire 4" toward the bottom or spherical end of the Chalice to begin work on the internal or second bulge. (Photo 7) This bulge is produced by slowing the rotation of the lathe considerably. Add a little more oxygen to the lathe ring burner and heat until the glass glows red completely around the vessel. Heat until it penetrates completely through the wall thickness, turn off the ring burner by releasing the pressure on the foot control pedal, and gently turn the handle on the tailstock to draw out the glass about ½ inch. Reposition the ring burner directly in the center of the drawn out section and press the foot pedal control and heat the area again. Soften the flame by reducing the amount of oxygen and continue heating and gradually pushing down the heated glass with a 3/8" diameter graphite rod. This will form the internal bulge nicely. Because of the centrifugal force it may be necessary to use a flat paddle at the same time to maintain the outside diameter of the 217 mm tubing. Now it is very important to give the entire vessel a thorough flame annealing using a large bushy, bluish, natural gas/oxygen annealing flame. Heat the vessel until the glass gets that very shiny look where it is just about to take on a cherry red appearance. You will obviously feel a good amount of radiant heat on your face, arms and chest. You should wear a long sleeve cotton (not polyester)

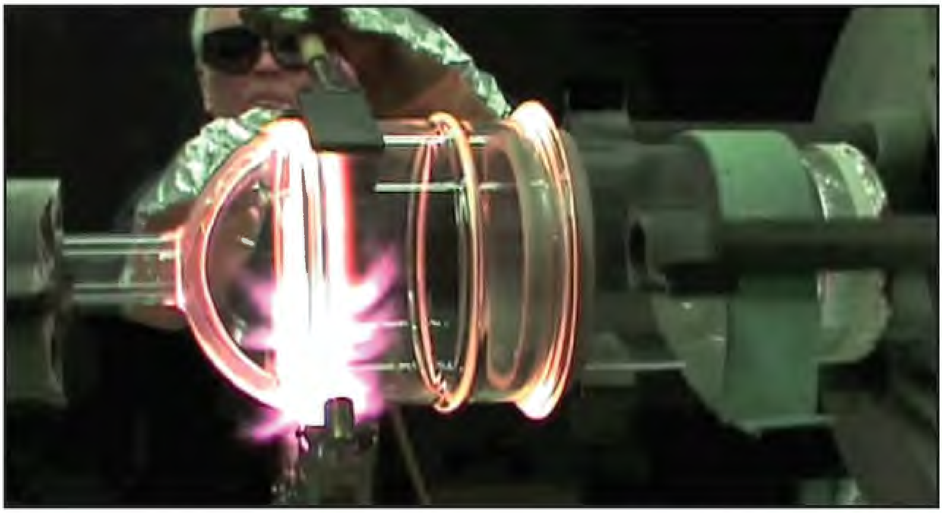


Photo 7. *Internal bulge*

shirt during these glassblowing operations. In addition, I wore long aluminized-backed Kevlar gloves and a gold reflective face shield. After the flame annealing is completed, run the vessel in an annealing oven overnight and pick up the final seals the next day.

Photo 8. *Add the 21-pin connector*

The last step in the Chalice fabrication is to splice on the 21-pin stem seal to the bottom. (Photo 8) I work about 2 inches out from the end of the spherical section. The 21-pin stem seal is made by a "pressing operation." That pressing is sealed into a 50 mm o.d. tube of Schott # 8245, Kovar sealing glass. This is a softer glass with some lead that allows the 21 Kovar pins to be sealed into it without the assembly breaking during cooling. There is a graded seal of several intermediate glasses that allows for a final seal into a 50 mm o.d. borosilicate glass tube. Neither the graded seal nor the 21-pin press can be overheated, so ideally it is best to work about 2 inches away and heat slowly. I was not given that 2-inch luxury, however, and had to work about three quarters of an inch from the graded seal. Also, chuck the 21-pin assembly gently and just tight enough to keep it straight during sealing. I favor fire cutting my tubes over any mechanical methods so both tubes

Photo 9. *Final flame annealing*

are fire-cut just before sealing them together. I use a Litton eight fire, single jet lathe ring burner with a hand torch over the top directed directly on the splice. Both torches are natural gas / oxygen flames. Work this seal as quickly as possible and do not let the flame heat too much area.

Immediately after this seal is completed, open the jaws on the tailstock and gently flame anneal the 50 mm tubing. (Photo 9) DO NOT overheat the graded seal or the 21-pin stem seal. Aim your annealing flame toward the spherical portion of the Chalice. Cool slowly, and the Chalice portion is completed. At this point the Chalice should not go into the annealing oven because of the Kovar pins and the different grading glasses used in the 21-pin connector.

Photo 10a. *The completed chalice*

Photo 10b. *Close-up of 21 seal*

Photos 10a and 10b show the completed Chalice. The 21 pins will allow up to 21 electrical, vacuum tight connections to be made inside the Chalice. These electrical connections will heat up and vaporize different exotic metals and deposit them onto a glass cathode plate. The next step is to build an internal array for holding the different exotic metals in position and to make the required electrical connections.

Photo 11. *Process oven setup*

"TULIP" CONSTRUCTION (INNER COMPONENTS)

The oven system in Photo 11 was used to process the cathode plates for the large area pico second photo detector. A high vacuum pumping system evacuates the inside of the Chalice and then a programmed sequence heats the entire vessel. Power is sent through the 21-pin electrical connections in the tail section where exotic metals are vaporized onto a glass plate inside the top of the Chalice that becomes the cathode plate. This process and the quantities of metals that are deposited on the glass cathode plate are critical to the success of the photo detector.

In Photo 12, take particular notice of these important components: the 21-pin stem seal connector at the bottom, a ½ inch stainless steel bellows to the glass vacuum pump-out port, the internal "tulip" assembly, and the 19 mm thick top window. A Kalrez O-ring is used between the 200 mm i.d. kettle flange and the top 19 mm thick plate to achieve a vacuum seal. The oven heats the entire Chalice up to 350 degrees C. The Kalrez O-ring is used because of the elevated temperature. The cost of a single Kalrez O-ring is $1,200.

Photo 12. *Close-up of the completed Chalice*

Photo 13. *Close-up of the 21-pin tail section*

In Photo 13, note the high temperature wires connected to the tail section via brass sleeve barrel connectors with heat resistant ceramic covered wires. Also note the ½ inch stainless steel bellows vacuum port, not part of previous photos, which was added as a later modification. It allows for expansion and contraction due to the elevated temperature and also dampens any vibrations from the vacuum pumping system. The ½” diameter also gives a larger pumping area than the smaller tube (6 mm o.d.) that was originally supplied on the 21-pin stem seal connector.

The “plug-in” electrical module is displayed in Photos 14a and 14b. The research team always wanted the internal electronics to be a “plug-in” component. This would allow for easy replacement of consumable materials, mainly the exotic metals that are consumed during the deposition process. They proposed many schemes that included attaching 21

Photo 14a. *The internal electrical assembly*

Photo 14b. *Close-up view*

separate metal electrodes through the wall and spaced equally around the circumference of the Chalice. The internal wires used for the electrical connections were long stranded wires that would allow them to "un-plug" the module and raise it up above the Chalice top flange. Then they proposed an internal pin array around the 8" diameter rings. These challenges were however simply not feasible. I determined that the best way to accomplish all these requirements was to use a pressed stem seal at the bottom of the tail section and create an assembly that could plug in and out easily to install and remove the electronics.

Photo 15a shows one of the first prototypes made to have a "plug-in" modular internal assembly. The Chalice has 6 glass blocks fastened to the inside wall using silver ink that were fired and bonded onto the internal glass wall at 500ºC. A glass plate was fashioned to fit inside the Chalice in between the glass blocks. The glass plate is notched so that, with just a slight turn, the inner assembly could be disengaged and removed. (Photo 15b)

Photo 15a. *First prototype of an internal shelf*

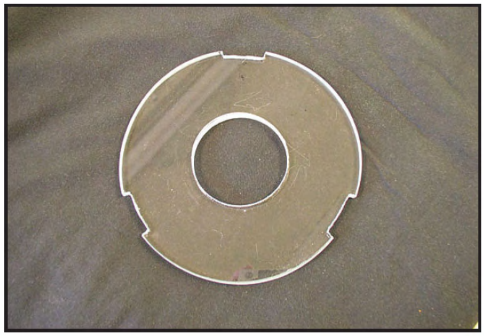


Photo 15b. *Removable shelf*

In Photo 16a, the internal glass blocks are shown fastened in place. Photo 16b shows that the blocks were formed to have the exact curvature to match the inside wall.

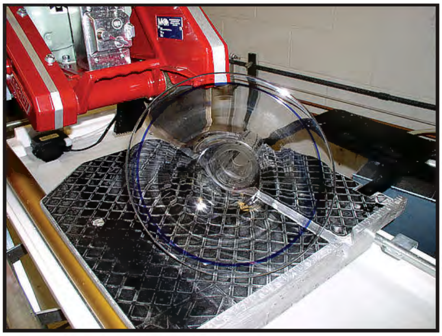
Photo 16a. *Shelf supports for prototype Chalice*

Photo 16b. *Curved supports*

From the notched plate, the "tulip" was born. The nicely shaped flare in Photos 17a and 17b was created by splicing a 4" o.d. x 4" long piece of heavy wall tubing onto a 2" o.d. x 2" long section of heavy wall tubing. This assembly was then spliced onto 45 mm o.d. x 10" long standard wall tubing. The 4" heavy wall tubing was flared up to about 9" diameter and the smooth flowing taper was formed along the section of the 2" heavy wall. The 4" o.d. heavy walled tubing was used to achieve the large flare and maintain a reasonably thick flared plate. Later the tulip's flat plate would be trimmed to 8" diameter. The tulip was then annealed before trimming.

Photo 17a. *The "tulip" design* **Photo 17b.** *Eight inches diameter*

The tulip was placed in a glass lathe and rotated. A circular line was marked on the flare for trimming with a permanent marker. The excess glass was cut off using a tile saw with a diamond blade (Photos 18a and 18b). The glass flare was then reduced down to the final diameter by using a wet belt sander. The tulip assembly was then washed and the ground edge was fire polished and annealed.



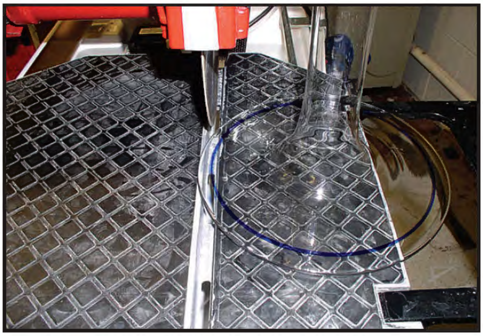


Photo 18a. *Tile saw for trimming the tulip flare* **Photo 18b**

Photo 19 shows the flared section of the tulip in a polariscope prior to annealing.

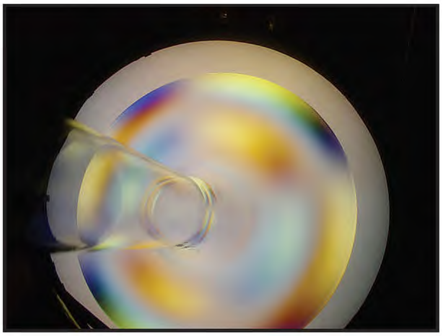


Photo 19. *Finished tulip in Polariscope* **Photo 20.** *Finished tulip in oven*

Photo 21a is a close up of the glass stem press seal with the 21 Kovar pins attached. In Photo 21b, the blue connector is the standard supplied connector but it would not survive the elevated oven temperatures. Therefore a connector had to be made to withstand 350ºC, which will now be described. This is the bottom of the tulip.

Photo 21a. *21-pin stem seal*

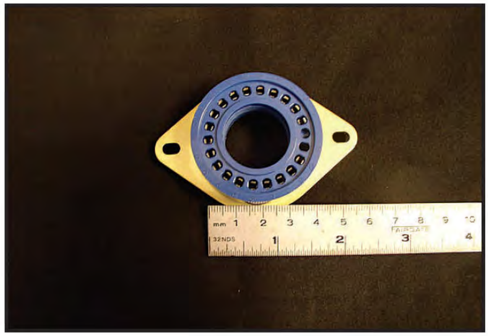


Photo 21b. *Plastic connector*

A 45 mm o.d. glass rod was not available so a 33 mm borosilicate rod was heated and gathered to 45 mm o.d. and used as the rod stock for the glass 21-pin connector. (Photos 22 and 23)

Photo 22. *Enlarged borosilicate glass rod*

Photo 23. *Close-up of enlarged 45 mm o.d. glass rod*

The new enlarged rod was cut into 19 mm thick segments using a diamond wet saw (see the 'hockey pucks' in Photo 24).

Using a glass lathe and a permanent marker make alignment lines on the hockey puck. Align a 7/8" o.d. diamond core drill on the drawn circles (for centering – see Photo 25b) and drill the hole to make the doughnut.

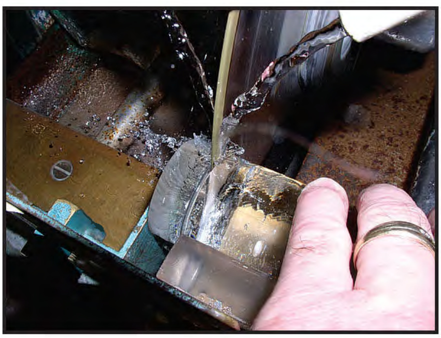


Photo 24. *Cutting the hockey pucks*

Photo 25a. *Mark hole location*

Photo 25b. *Drill the hole to make the glass doughnut*

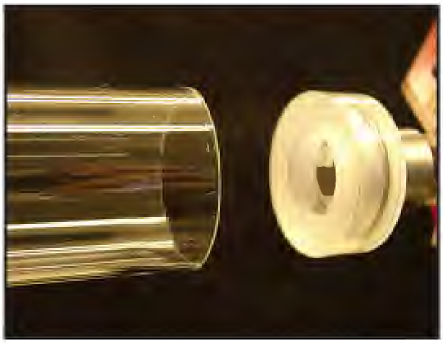


Photo 26a. *Splice the doughnut onto a tube*

Photo 26b

A 6" length of 45 mm o.d. standard wall tubing was attached to the end of the hockey puck. The 45 mm o.d. tubing spliced onto these doughnuts was then cut at 6 mm long making the pieces 25 mm in total height. (Photos 26a and 26b)

Photo 27. *Finished splice*

Notice that the holder is a modified (shortened) 24/40 inner joint with graphite tape wrapped around it to hold the doughnut for splicing. (Photo 27)

Photo 28 shows the completed doughnut blanks (a) before and (b) after the 21 holes are drilled into them.

Photo 29 shows the completed tulip after the 21 holes were precisely drilled into the end and sealed onto the tail of the tulip. Four 3/16" i.d. holes were drilled into the tulip

Photo 28a. *Completed doughnuts with tube extension*

Photo 28b. *The completed 21-pin glass connectors*

Photo 29. *21-pin connector spliced onto tulip*

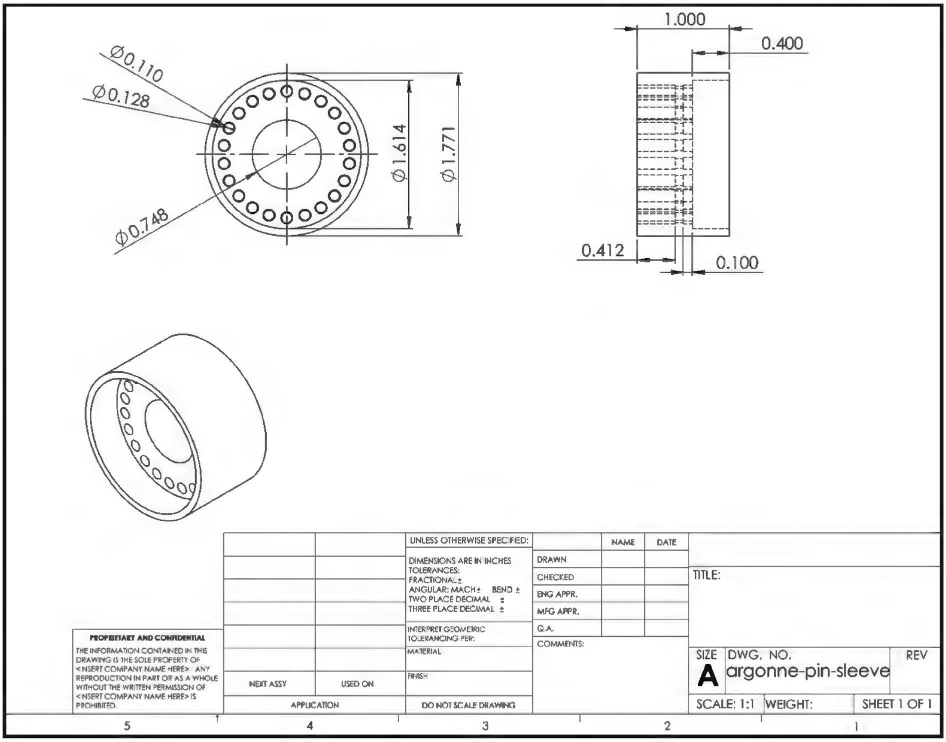


Photo 30. *Detailed drawing of the 21-pin glass connector*

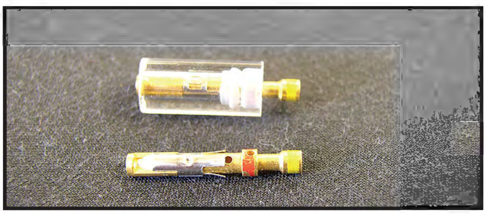


Photo 31. *Electrical brass barrel connectors*

flange for mounting onto the stainless steel electronics cage using stainless #8 bolts, washers, and nuts. Caution must be taken not to over tighten these bolts and crack the glass flange.

Photo 30 is the detailed drawing for machining the 21-pin glass coupling holes. The machining was completed by fellow ASGS member Ron Bihler of Precision Scientific Glassblowing of Colorado.

Photo 31 shows a sample of how the brass barrel connector is fit into the precision drilled bore of a capillary tube. Photo 32 shows the exact dimensions of the bore that

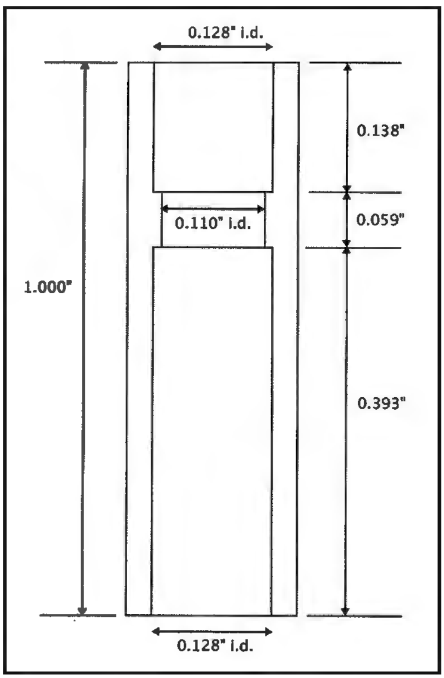


Photo 32. *Cross section view of 21-pin glass connector*

are required to make the connectors "click" into the correct position. This hole was then copied 21 times into the fabricated doughnuts. (Photo 33) Brass barrel connectors were then inserted into the 21-drilled holes to make the electrical connection.

Photo 34 shows a close-up of the glass 21-pin connector with the brass connectors inserted.

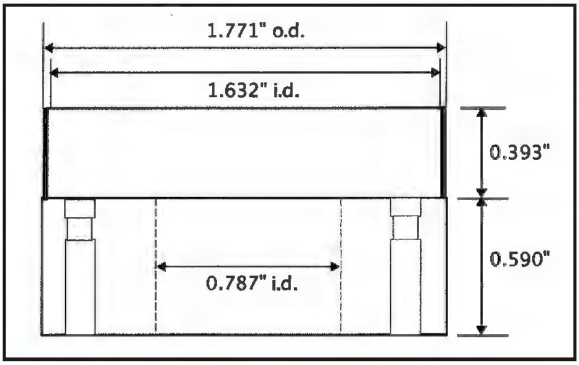


Photo 33. *Precision drilled hole specifications for pins detail*

Photo 34. *Tulip tail section with 21 brass barrel connectors installed*

These will plug into the 21-pin stem seal on bottom of Chalice tail section.

Photo 35 shows the tulip with the stainless steel wires inserted and running up to the electrical assembly.

Photo 36 shows a couple of early designs of glass wire supports to hold the stainless steel wires in position.

Photo 37 shows the most recent pin support design that seems to be the favored shape. These are more heavy duty and work very well.

Photo 38 shows the wires coming up from the 21-pin connector to the top of the stainless steel cage.

Photo 39 shows the completed Chalice positioned in the processing oven with all connections made, ready for vacuum pumping and electrical power up.

Close-up view of the electrical connections. (Photo 40)

Photo 35. *Assembled tulip*

Photo 36. *Glass insulating pins*

Photo 37. *Final pin design*

Photo 38. *Wired tulip assembly*

Photo 39. *Chalice complete*

Photo 40. *Wired tail section*

Photo 41. *Top removal tools*

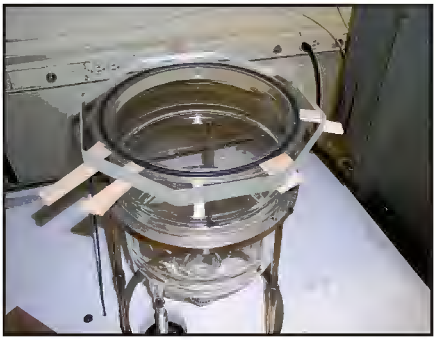


Photo 42. *Wooden shims*

Photo 41 shows some of the precision alignment tools used to remove the 19 mm top plate from the kettle flange. After the first heating of the assembled Chalice, the Kalrez O-ring had stuck to the top glass plate. The plate was successfully removed by using shims and a small hammer.

To separate the stuck plate, wooden shims were strategically placed around the circumference and forced between the flange and the top glass plate (Photo 42). Using thicker and thicker wooden shims around the flange the top plate finally released. The Kalrez O-ring was not damaged in this operation and was re-used many times without any change in the ultimate vacuum pressure.

ACKNOWLEDGMENT

This work was supported by the U.S. Department of Energy, Office of Science, Office of Basic Energy Sciences and Office of High Energy Physics under contract DE-AC02-06CH11357.

Cost Analysis: Economic Justification for a University Glass Facility

by
Steve Moder*

ABSTRACT

In the changing university economic environment, it is more important than ever for a glass facility to justify its existence based on cost analysis. Factors including annual productivity, valuable work practices, and outreach contribute to the overall analysis.

Universities are facing troubling economic times because of ever decreasing state financial support. The state support has been cut both in prosperous and recession years for a variety of reasons. As universities struggle with the loss of financial support, they have made painful cuts. Administrators now look to both support services and courses offered for cost effectiveness. Being an essential service to the university is no longer a guarantee of security without being cost effective. In the current economic environment, it is critical for the university scientific glassblower to evaluate the financial productivity of their facility.

In the past year, I was given the opportunity to justify my position using cost-effectiveness as a basis. What did the university save by paying my salary and benefits was the question, not what did I bill for the year. Project records for the year provided the means to answer their question, but more questions were growing in my mind about the overall value of a university scientific glass facility. In this paper, I will look at both the measurable cost savings and other contributions that enhance the department, college and university mission. Hopefully it will give you pause to reflect on your facility and the contributions you are making.

COST ANALYSIS

The need to address cost versus benefit issues involving the university glass shop requires knowledge of certain terms. For the purpose of this paper, I have adopted the definitions and explanations given by Sewell and Marczak, researchers from the University of Arizona, in their paper *Using Cost Analysis in Evaluation*.

For our purposes, there are three types of cost analysis used in evaluation: cost allocation, cost effectiveness, and cost-benefit.[1] All have specialized purposes and are useful at different times. Their rough definitions are:

- Cost Allocation: A way of setting up budget and accounting to determine unit cost. This information can be used in cost-benefit analysis and cost effectiveness analysis. The method is used to determine billing.
- Cost Effectiveness Analysis: A comparative method of examining alternative sources of a desired product. This can be used to determine the cheapest or most efficient way.
- Cost-Benefit Analysis: This is more for the human resource department or manage-

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[1] Meg Sewell and Mary Marczak, *Using Cost Analysis Evaluation* (Tucson: University of Arizona, 1997) 1-2.

ment, and can provide an answer whether economic benefits outweigh the cost of the service. This does not look at the intangible evidence like speed of service, etc.[2]

PUTTING THE COST ANALYSIS TO WORK

I strongly encourage the university scientific glassblower to work time in their day for cost analysis. At the completion of every project, a few extra minutes will give you cost allocation (unit cost). This is also the perfect time to do a comparative estimate (outside cost) of purchasing the piece off-campus. This cost effectiveness analysis accumulated over the course of the year will give you a thorough start on the final report when requested.

In order to aid you in your comparisons, I have provided data from a questionnaire filled out by a random sampling of university scientific glassblowers. The questions answered provide insight into types of work, estimates of annual productivity, backlogs, contact time, outreach, and other service possibilities.

The first question provides an overview of types of work fabricated and the percentage of torch time. The types of work were categorized as: prototype, expensive catalog items, routine catalog items, teaching lab glassware, repair work, and other. While percentages varied for the different glassblowers, the averages have been plotted in Figure 1.

The highest percent of time was spent on repair work. While this can be the most tedious and dangerous work, it may be the most cost effective work. Depending on the availability of independent glassblowers, this could be one of the most problematic to research. Having the scientific glassblower on site prevents these problems and should speed the repair.

The next most time-consuming task was prototype work. Prototype work highlights the need for a scientific glass facility. Without a scientific glassblower on campus, the researcher must first find a source, then work though consultation and design (possibly

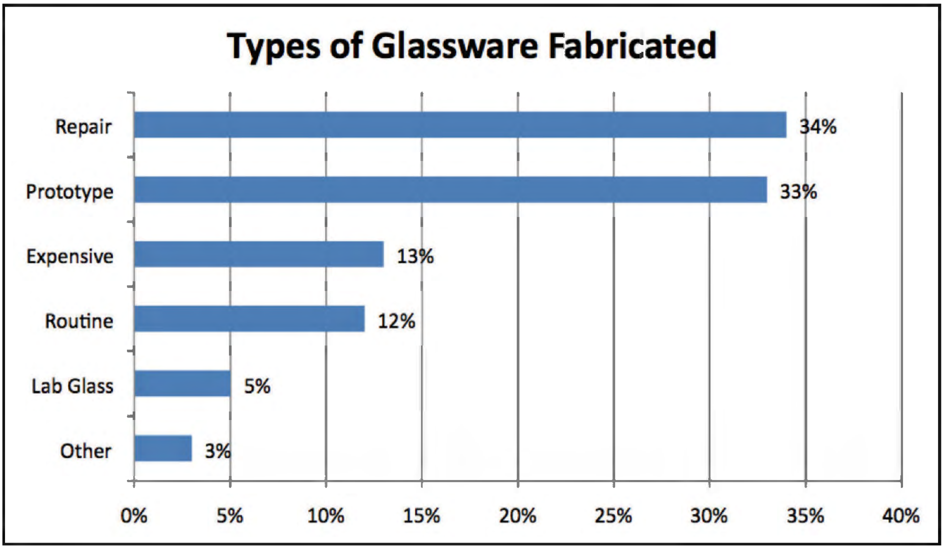

Figure 1

[2] Sewell and Marczak 1-2.

long-distance) and finally absorb costs from flawed prototype design. This work requires the most time in cost analysis.

Catalog items are valuable contributions to cost analysis. Expensive catalog items make up a substantial amount of cost-effective savings. These items can be cost-prohibitive to the researcher without the scientific glassblower on campus. Although routine catalog items may not seem to have an impact on an individual basis, over the course of a year, they result in substantial savings.

Teaching lab glassware may only take a small percentage of our time, but the effort is well-recognized in the era of shrinking budgets. From nicks and chips to fabricating organic lab ware and synthesis lab glass, a reasonable savings is realized. A good role in keeping the cost of education down also helps to justify the glass facility.

Last but not least is the other, at three percent of our effort. It is worthwhile to say these efforts should be cost-analyzed and savings recorded. While the work request may not be prestigious, will it save money? Do you have time to do it? These are good questions to ask yourself.

ANNUAL PRODUCTIVITY

The measurement of our productivity is given by the cost of the glassware fabricated in the glass facility. The cost of the glassware produced at the universities responding to the questionnaire was valued from approximately $100,000 to over $250,000 by the campus glassblower (see Figure 2).

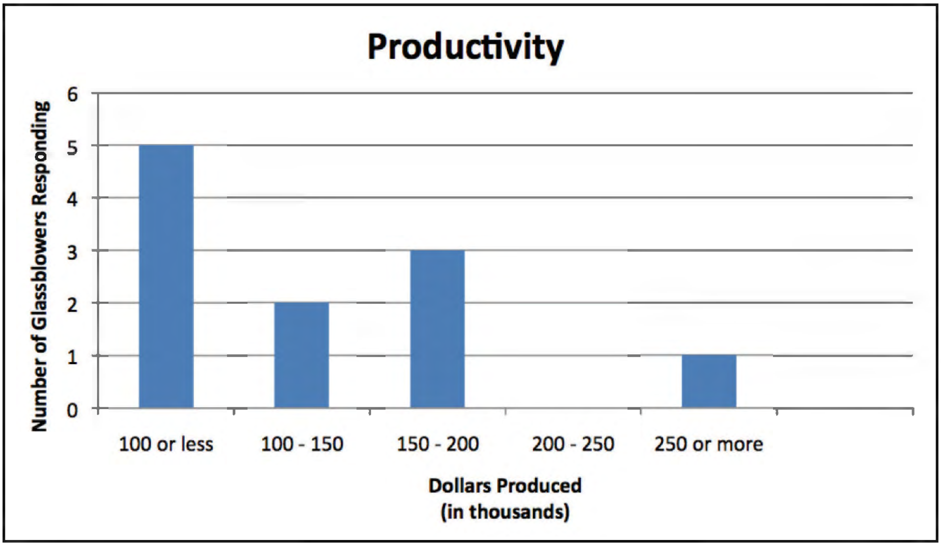

Figure 2

The university glass facilities responding to the questionnaire all had tangible savings when productivity was compared to salary and benefits, after removing charges for materials. The total saved did vary and this could result from the method used to assign value, location of university, and error in calculation, to name a few reasons. That being said, the savings seemed to range from 25 percent to over two times the salary and benefit amount.

VALUABLE WORK PRACTICES AND OTHER HELPFUL SUGGESTIONS

Above I have looked at cost analysis, dollars saved, and types of work that provide savings. Now we must look at important procedures and activities whose savings are not readily apparent in dollar value. The questionnaire was designed to look at backlog, contact hours, outreach, and other essential duties and services.

First, the dreaded backlog was examined. Backlog is dreaded by our customer because of lost productivity. The glassblower can quickly become a problem if this is too lengthy a time. While there are numerous reasons for a long backlog, it pays to keep this as short as possible. The respondents' delay times can be seen in Figure 3. It seems that the delay of around two weeks is average. Prompt attention to a work request pays huge rewards for both the customer and the scientific glassblower.

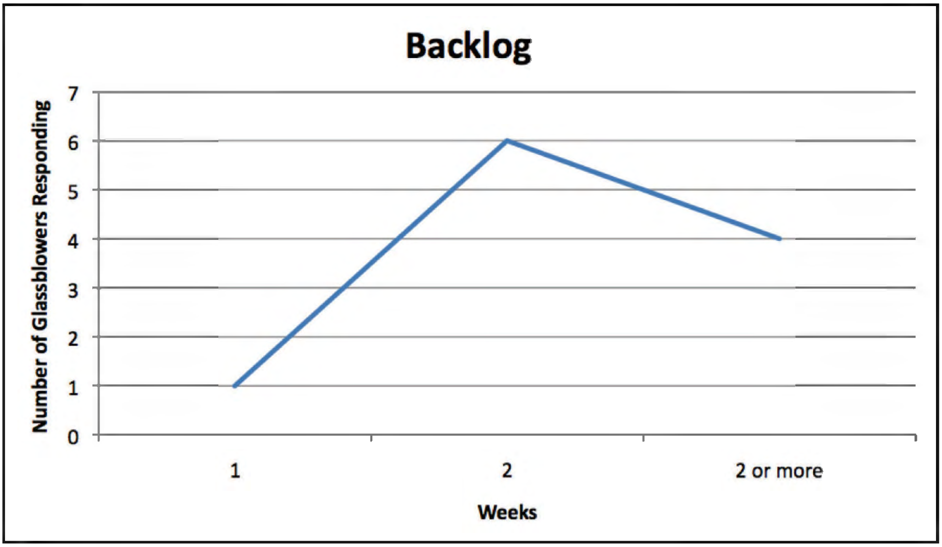

Figure 3

This questionnaire looked at the percentage of contact hours spent on consultation and design, informal glass sessions, formal scientific glass sessions, formal artistic sessions, and other. None of the respondents conducted formal artistic sessions. Consultation and design accounted for 67 percent of all contact hours with informal sessions at 20 percent, formal at ten, and the rest of the time in other. Customer contact hours are important to all facilities. Try to run the facility as a business with emphasis on the customer first. It is important to note that how we treat people will play a serious role both in whether you are kept at your position and replaced at retirement time.

Figure 4 provides us with a glimpse of how student contact hours are spent.

University glass facilities are the perfect venue for outreach. Just as the moth is attracted to flame, so are the curious artistic people. With very little effort, you can quickly design an outreach program for your facility. The questionnaire looked at outreach as student demonstrations, demonstrations for the general public, seminars for the general public, and other displays. The results were:

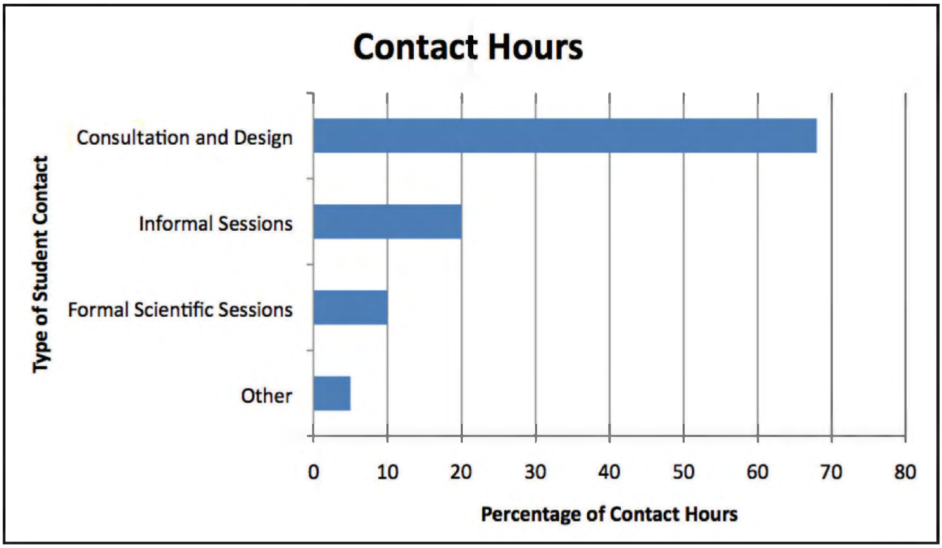

Figure 4

- Fifty percent of the glassblowers were doing student demonstrations. These are well-received by the public, and giving away the occasional trinket draws a positive response.
- Demonstrations for the general public draw numerous attendees and nicely support the campus recruiting process. 50 percent of the glassblowers provide these.
- Seminars for the general public are provided by 30 percent of the respondents. At the University of Toledo, we provide two-hour icicle making sessions to the general public free of charge. During the fall semester, we have around 350 people who enjoy this time. It usually takes five to seven days to fill the seminars with very little advertisement. The icicles make great presents and, as of today, have been gifted to people in 52 countries and 42 states in this country. I have found this to be a popular method of outreach, one that is appreciated by the university and the public.

Fifty percent of the glassblowers also do other forms of outreach. I look forward to hearing about these endeavors.

More than half of the respondents identified other essential services and duties they perform. I have made a brief list of their contributions:

- Serve on departmental committees
- Assist scientific materials manager
- Inspect glassware for safe usage
- Ensure speedy turn-around time
- Assist other facilities (stockroom, machine shop) as needed
- Train students in specific glassblowing procedures
- Assist building manager
- Provide glassware recycling, reclamation, and distribution services
- Provide safety oversight

All these contributions seem to have one thing in common: service to the department, college, and university.

CONCLUSION

As the trend of cutting state support for universities continues, opportunities exist for the university scientific glassblower. They can be realized by assessing your economic value for the administrators. This will be accomplished by cost-effectiveness analysis of your service. By taking a proactive approach, you have the opportunity to modify your service to increase its value to the university.

Services and outreach are supports for your cost analysis. Your quick product production timeline (backlog) and contact hours establish your value with the scientists just as cost effectiveness does with administrators. Outreach efforts bring your facility public recognition while supporting your department, college, and university mission. The opportunity to show your valuable contributions to research and teaching is in your hands.

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Jacketed 500 ml Multi-Port Flask

by
Doni Hatz[a]

ABSTRACT

A 500 ml round flask with multiple ports is jacketed inside a two-liter round flask to specific dimensions. There are four ports protruding outward around the widest part of the flask in all four directions. The two-liter flask is cut open and the small flask is supported inside and sealed closed. Each port is sealed into the larger outer flask. Threaded connectors are sealed to two ringsealed ports for temperature probes and 3/8" o.d. and 5/8" o.d. tubes for sampling ports. The jacket has two water connections using the GL14 threaded connectors sealed at a tangent and two 25 mm Chem-thread connectors for top and bottom ports.

Photo 1

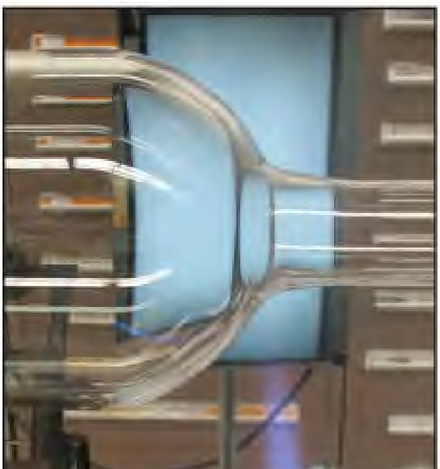

Photo 2

A 500 ml round bottom flask with multiple ports is jacketed inside a 2 liter round bottom flask to specific dimensions. The jacket allows for constant temperature control of the inside contents. There are four ports: two sample ports and two temperature probe inlets (Photo 1).

PART 1: The 500 ml Round Flask

The inside flask is prepared by pulling down 105 mm o.d. (outside diameter) SW (standard wall) tubing with a Carlisle CC Plus burner in the lathe. A 500 ml round bottom flask blank was considered but not for this project where the flask is slightly smaller and sometimes has an uneven wall thickness. While the 105 mm tubing is heated in the lathe, the side tubes are prepared at the bench Carlisle CC torch. A point is pulled on ½" o.d. MW (medium wall) tubing and trimmed to a 35 mm length; use the point as a handle. Another ½" tube section is prepped the same as well as a small section of 5/8" MW and 3/8" MW tubing and set aside.

Pull down a round bottom end on the 105 mm tube, blow out a hole and seal to 32 mm o.d. SW tubing (Photo 2). After shaping the round flask (Photo 3), a neck is tooled down to 32 mm o.d. and 35 mm in length. Melt and pull apart the large tubing but keep the neck closed off until the side arms are sealed to the flask (Photo 4). The 105 mm tubing is removed from the lathe and the flask is chucked into the head-stock of the lathe.

[a] The Procter & Gamble Company, 8700 Mason Montgomery Road, Mason, Ohio 45040. Email: Hatz.dj.@pg.com.


Photo 3


Photo 4


Photo 5


Photo 6


Photo 7

Once the flask is heated up to working temperature, use a hand torch to blow up four small bulges around the mid-section; this will help identify the alignment of the side seals. Remove strain after blowing up each bubble (Photo 5). Seal on each side tube, flow the glass, flame anneal and move to the next side seal (Photo 6). Check to see that the tubes are directly across from each other (Photo 7).

Open the 32 mm o.d. neck on the 500 ml flask and form a bead on the end of the tube for the ring seal; flame anneal and set aside.

PART 2: Prepare the 2 Liter Round Bottom Flask

A standard two liter round bottom flask is chucked into the headstock of the lathe. The flask is heated focusing on the round bottom end. A hand torch with a fish tail tip is great to blow open a large hole then seal to a piece of 38 mm o.d. SW tube. Heat the entire flask and remove the neck on the other side of the flask; seal on another piece of 38 mm o.d. SW tube. Flame anneal the flask and anneal in the oven before trimming at the wet diamond saw.

Support the 2 liter flask in the lathe with a light hold on the tailstock. A scoring knife is held tightly to one of the cradle burner heads while the lathe is turned on spinning at a fast

Photo 8

Photo 9

Photo 10

pace; press firmly on the flask and score all the way around the flask (Photo 8). Remove the scoring knife, wipe off the powder from the scratch and slow the lathe speed down to very slow. Hold the hand torch (National with piloted tip #4) on the edge of the score mark at a tangent with the fire facing upward (Photo 9). First you see the moisture from the surface of the glass burning off to the sides of the score mark. After a couple of minutes or less, the flask will crack apart (Photo 10); immediately turn off the lathe. If the score does not crack once the moisture burns away quickly, apply a droplet of water from a wet paper towel (Photo 10).

The flask was purposely cracked open with 2/3 flask on one side to 1/3 of the flask for the other side. This will allow enough spacing for the inside flask side arms to clear the flask while sealing the 2 liter flask back together.

The 500 ml flask is supported inside the 2 liter flask by sliding the 32 mm tube inside the 38 mm tube. Two inch wide masking tape works well for a support material wrapped on the 32 mm tube. Two sections of the tape are wrapped creating two balance points. The 32 mm tube is marked with a Sharpie® to identify complete revolution around the tube (Photo 11). The masking tape is wrapped several times until it fits tightly inside the 38 mm tube. A small 2 mm slit is cut into the tape for an air hole to blow into the outside flask (Photo 12).

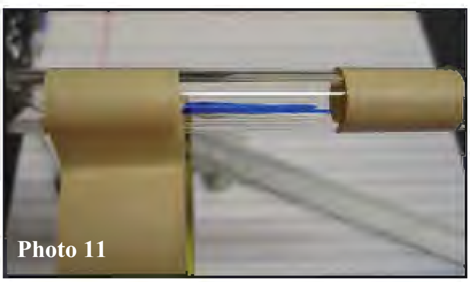

Photo 11

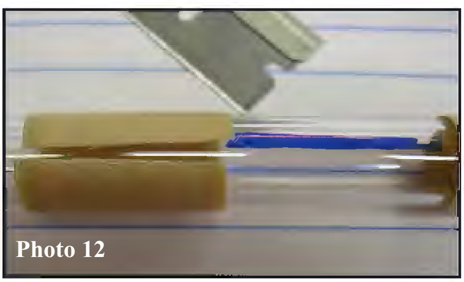

Photo 12

Slide the 500 ml flask inside the 2 liter flask and mark where the side arms need to be trimmed down to fit inside the 2L flask (Photo 13). Cut down the side arms to just barely fit inside the flask (Photo 14.)


Photo 13

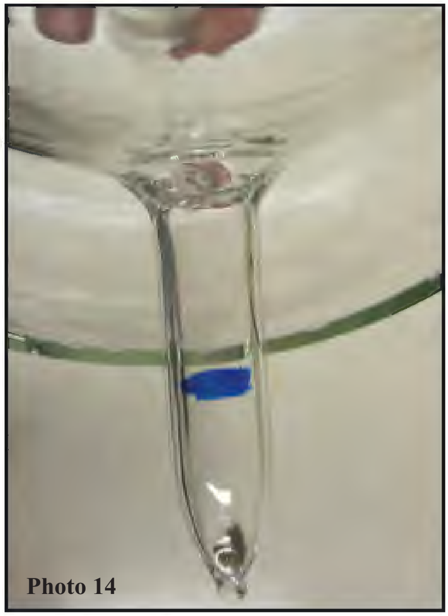
Photo 14

Photo 15

Once the flasks are set up, it is time to check the top piece of the 2 L flask. Consider 3-5 mm to flow the seal; if there is too much flask, trim another ½" of glass from top 1/3 section of the flask. The flask is marked with a Sharpie® in the lathe to confirm the exact line to cut. Let the mark dry completely or else the line will wash off. The flask is set up on the bed of the wet diamond saw and scored all the way around, continually turning the flask, until it is cut open (Photo 15). Clean up the flask and support in the lathe tailstock.

PART 3: Seal the 2 Liter Flask


Photo 16

Before sealing the flask together, the component parts need to be ready for sealing. Two side arms are pulled down, one of 3/8" o.d. MW tubing and the other 5/8" o.d. MW tubing (as mentioned before pulling a point for a handle) and trimming the tubes a couple of inches. Two #7 Ace threaded connectors are also set up supported with a 10 mm o.d. tube wrapped with graphite paper. The #7 Ace threads are trimmed short, about ½" below the thread. The two 25 mm Chemthread connectors are supported with a 25 mm o.d. MW tube wrapped with graphite paper then the glass thread is trimmed one inch below the thread (Photo 16).


Photo 17


Photo 18

The 500 ml flask is chucked into the headstock of the lathe and the top half of the 2 liter flask in the tailstock (Photo 17). The glassware is heated using the Carlisle CC burner, then switch to the cradle burner for the large seal (Photo 18). Work quickly with the cradle burner so as to not overheat the inside flask and cause it to slump. Once the seal is flowed in, flame anneal the flask then begin heating the 38 mm tube holder on the right side. Melt and remove the 38 mm tube and flow in the ring seal (Photo 19); then blow open a hole. Remove the tube, chuck the 25 mm Chemthread into the tailstock (Photo 20) and seal to the flask.


Photo 19

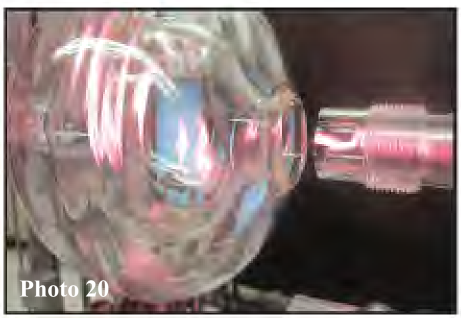
Photo 20


Photo 21

PART 4: Seal on the Side Tubes and Connectors

Heat the entire flask and flow in each ring seal blowing up a small bubble in the center of the seal. The small bubble is ready to blow open a hole for sealing on the tubes and connectors (Photo 21). Once all four ring seals are flowed in, start sealing on the 3/8” and 5/8” tubes (Photo 22) then seal on the #7 Ace threaded connectors (Photo 23).

Photo 22

Photo 23

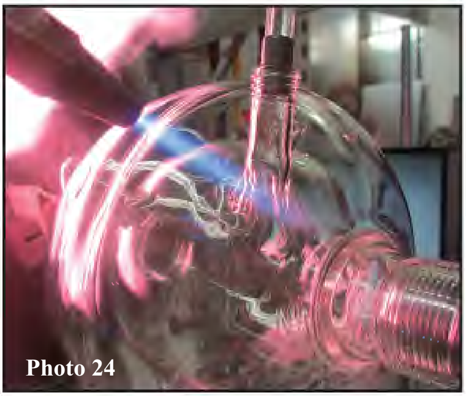

Photo 24

Photo 25

Photo 26

Next seal on a GL14 threaded connector on the right side of the flask near the #25 Chemthread (Photo 24). The GL14 thread is sealed on at a tangent. The tube holding the thread is heated and bent down towards the center tube to connect to the blow hose (Photo 25). All the while keep the entire flask hot. Hook up a "Y" connector to the blow hose on the right side between the flask and the blow hose swivel. Vacuum hose (heavy wall rubber tubing) is ideal between the flask holder and blow hose swivel so it does not pinch or collapse while blowing into the inside and outside of the flask. Regular rubber tubing is fine for the connection to the outside jacket (Photo 26).

Photo 27

Start heating the spot for the second GL14 thread on the left side. Blow up the area for the tangent seal (Photo 27). Heat the tubes on the left side of the flask sealing the 38 mm to the 32 mm tubing thinning and reducing the tubing next to the ring seal. The flask cannot be supported by the 25 mm Chemthread on the right side so the lathe is turned off and the 38 mm tube is melted. Pull away the tube carefully to keep the flask from slipping out of the 25 mm Chemthread; be ready with heated gloves to help secure the flask back on the 25 mm holder. Take the flask out of the lathe, hold the flask upward so it will not fall out of the holder and place into a hot oven; anneal.

Photo 28

Photo 29

Photo 30

Photo 31

Photo 32

Since the neck of the 25 mm Chemthread is too short to support the weight of the flask, a ring stand is set up for the next seal (Photo 28). A turntable or lazy-susan support can also be used but then you will have to blow into the top instead of the bottom. The lathe bed works well for a table top and a ring stand where multiple torches are set up, one in the back and a large Carlisle CC hand torch in front and a couple of hand torches for the small seals. Heat the top of the flask (ideal if the flask is still slightly warm from the oven annealing but cool enough to hook up rubber stopper with the blow hose and "Y" connector), clean up and remove excess glass (Photo 29) above the ring seal (Photo 30), and blow out a hole. Seal on the 25 mm Chemthread while keeping the flask warm with lots of fire power (Photo 31).

Seal on the GL14 connector to the outside jacket at a tangent on the same side as the other GL14 connector (Photo 32). Remove strain from the seal area and quickly place in a moderately warm oven about 350°C. Let the flask equalize in temperature for about 15 minutes then increase to annealing temperature of 565°C.

The flask complete top view (Photo 33) and side view (Photo 34).

Photo 33

Photo 34

Porous Wall, Hollow Glass Microspheres

by
William C. Sexton*

ABSTRACT

A research team at the Savannah River National Laboratory has developed thin, Porous Wall, Hollow Glass Microspheres in the one to several hundred microns range. These microspheres were originally developed for the storage of hydrogen and other gases. The use of these microspheres has expanded to drug delivery and, with the ability to control and alter the porosity, the uses are endless. The research team has recently been selected as a winner of the prestigious 2011 R&D 100 Award presented by R&D Magazine*.*

Hollow Glass Microspheres (HGMs) are not a new technology. All one has to do is go to the internet and Google™ HGM. Anyone can buy HGMs and they have a wide variety of uses. HGMs are usually between 1 to 100 microns in diameter, although their size can range from 100 nanometers to 5 millimeters in diameter. HGMs are used as lightweight filler in composite materials such as syntactic foam and lightweight concrete. In 1968 a patent was issued to W. Beck of the 3M™ Company for "Glass Bubbles Prepared by Reheating Solid Glass Particles." In 1983 P. Howell was issued a patent for "Glass Bubbles of Increased Collapse Strength" and in 1988 H. Marshall was issued a patent for "Glass Microbubbles."

If you Google™ Porous Wall, Hollow Glass Microspheres (PW-HGMs), the key words here are Porous Wall. Almost every article has its beginning with the research done at the Savannah River National Laboratory (SRNL). The Savannah River Site (SRS) where SRNL is located has a long and successful history of working with hydrogen and its isotopes for national security, energy, waste management and environmental remediation applications. This includes more than 30 years of experience developing, processing, and implementing special ceramics, including glasses for a variety of Department of Energy (DOE) missions. In the case of glasses, SRS and SRNL have been involved in both the science and engineering of vitreous or glass based systems.

As a part of this glass experience and expertise, SRNL has developed a number of niches in the glass arena, one of which is the development of porous glass systems for a variety of applications. These porous glass systems include *sol gel glasses* which include both xerogels and aerogels as well as *phase separated glass compositions* that can be subsequently treated to produce another unique type of porosity within the glass forms. The porous glasses can increase the surface area compared to 'normal glasses' on a 1 to 2 order of magnitude which can result in unique properties in areas such as hydrogen storage, gas transport, gas separations and purifications, sensors, global warming applications, new drug delivery systems and so on.

One of the most interesting porous glass products that SRNL has developed and patented is Porous Wall, Hollow Glass Microspheres (PW-HGMs) that are being studied for many different applications. The European Patent Office (EPO) just recently notified SRS that the continuation-in-part patent application for the PW-HGMs has been accepted. The original patent, which was granted by the EPO on June 2, 2010, was validated in France, Germany and the United Kingdom.

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The microspheres produced are generally in the range of 2 to 100 microns, with a 1 to 2 micron wall. What makes the SRNL microspheres unique from all others is that the team in Figure 1 has found a way to induce and control porosity through the thin walls on a scale of 100 to 3000 Å. This is what makes the SRNL HW-HGMs one-of-a-kind, and is responsible for many of their unique properties and their potential for various applications including those in tritium storage, gas separations, H-storage for vehicles, and even a variety of new medical applications in the areas of drug delivery and MRI contrast agents.

Figure 1. *The original SRNL microsphere team with flame former apparatus. The SRNL research and development team: Dr. George Wicks, Dr. Leung Heung and Dr. Ray Schumacher. Also on the team, but not pictured are Dr. Steven Serkiz and Dr. David Peeler.*

SRNL Hollow Glass Microspheres and subsequent Porous Wall, Hollow Glass Microspheres are fabricated using a flame former apparatus. Figure 2 is a schematic of the apparatus.

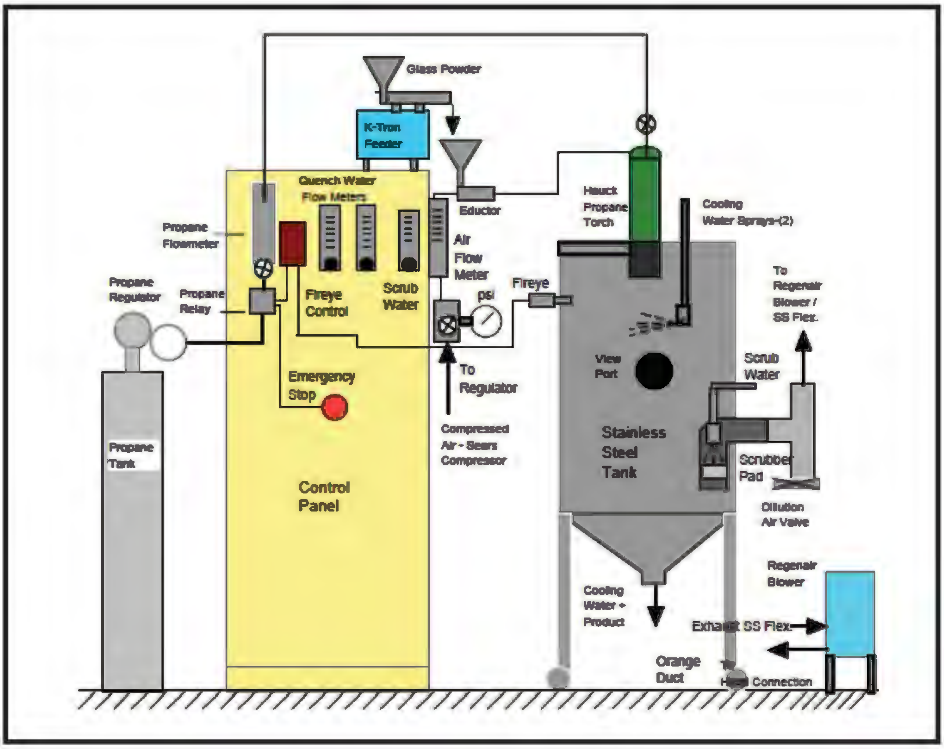

Figure 2. *Flame former apparatus*

The flame former apparatus shown in Figure 2 consists mainly of a control panel and a combustion tank. The portable control panel houses the water, gas and combustion controls. The stainless steel combustion tank contains the flame former, water spray and a collection basin. The apparatus is fueled by a portable propane tank and regulator.

The fabrication process starts by heating a glass powder (~20-40 microns) made up of silica, boron, alkali metal oxides, sulfur, and a small amount of various metal oxides along with a sulfate blowing agent. The heat zone is formed by a controlled gas-air flame from a modified Hauck torch. During this heating phase, the blowing sulfate agent becomes unstable and forms gas nuclei. As the spheres continue to be heated, they expand and form hollow glass microspheres. Next, the spheres pass through two water sprays that supply approximately 0.5 gallons per minute. These water sprays are used to quench the propane combustion flame to specific lengths, to act as a coolant for the stainless steel tank, and to capture the HGMs and glass particles in the quench water. The quench water exits from the bottom of the stainless steel tank and is collected in plastic pails. The HGMs are collected by floation. Figure 3 is a typical batch of SRNL microspheres. (Sizes 2-100μ with average diameter of ~50μ)

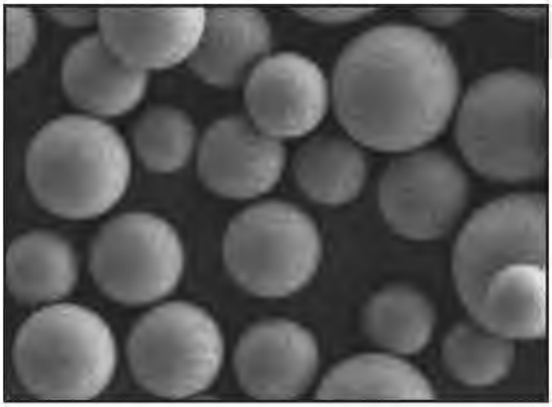

Figure 3. *Typical batch of SRNL microspheres*

The HGMs collected are heat treated at 620°C for 18 hours before they are leached in a 4 molar hydrochloric acid (HCl) solution at 80°C for four hours. The heat treatment produces phase separation. The importance of this process is that it actually produces two different glass phases, one rich in silica and the other rich in sodium borate. The sodium borate phase is an interconnected worm-like morphology so when it is removed by the leaching process, it produces interconnect pores or channels that extend from the outside of the shell to the inside. Figure 4 offers a schematic representation of a SRNL microsphere and wall porosity. This phase separation is called spinodal decomposition. Vycor® 7930 (thirsty glass) is also an example of this method. Figure 5 shows the similarities between the SRNL PW-HGMs and Vycor®7930.

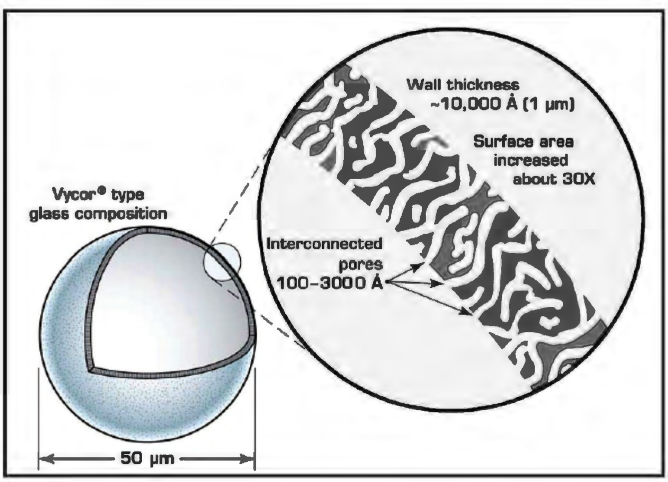

Figure 4. *Schematic representation of a SRNL microsphere and wall porosity*

Leaching removes the soluble sodium borate phase of the glass and creates porosity through the microsphere wall. Figure 6 is an electron microscope view of the porous wall of a microsphere.

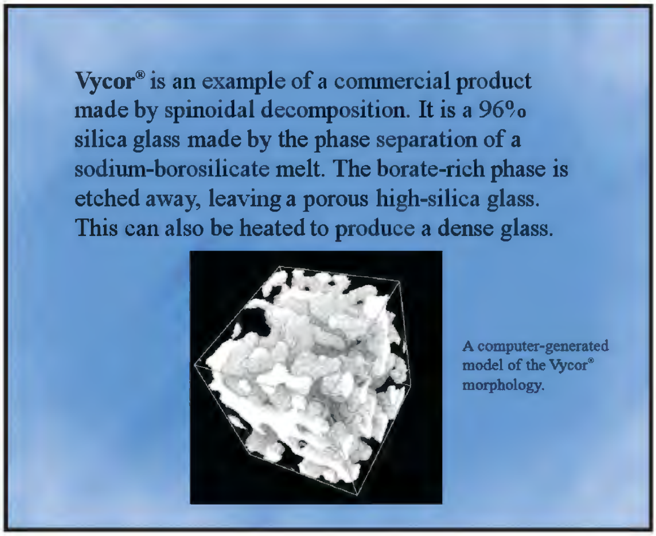

Figure 5. *Spinodal decomposition of Vycor®*
(Slide taken from presentation Spinoidal Decomposition and Phase Diagrams by Jason Bodson)

Porosity is the key and the research team at SRNL used this porosity to fill the PW-HGMs with special gas absorbents. Figure 7 is the first released micrograph of one of the microspheres after being successfully filled with palladium metal which is used in the storage of radioactive forms of hydrogen. The wall porosity allowed the microsphere to be filled.

As part of a joint collaboration with Toyota, SRNL conducted a test on filling the PW-HGMs with other special hydrogen absorbents. The test confirmed that very effective,

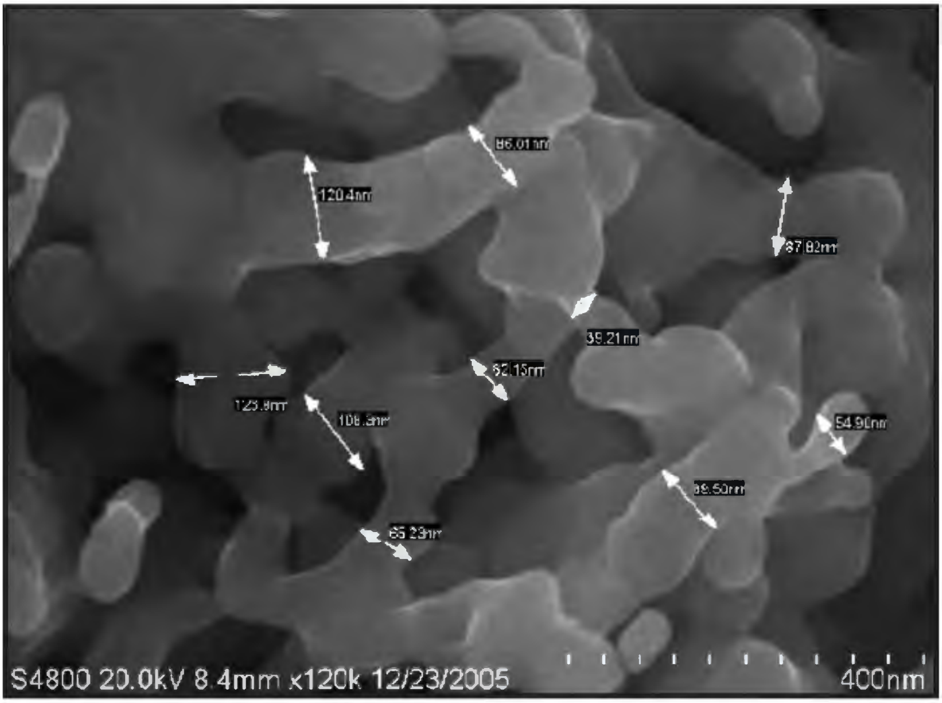

Figure 6. *Unique nano-scale wall porosity within the microsphere walls*

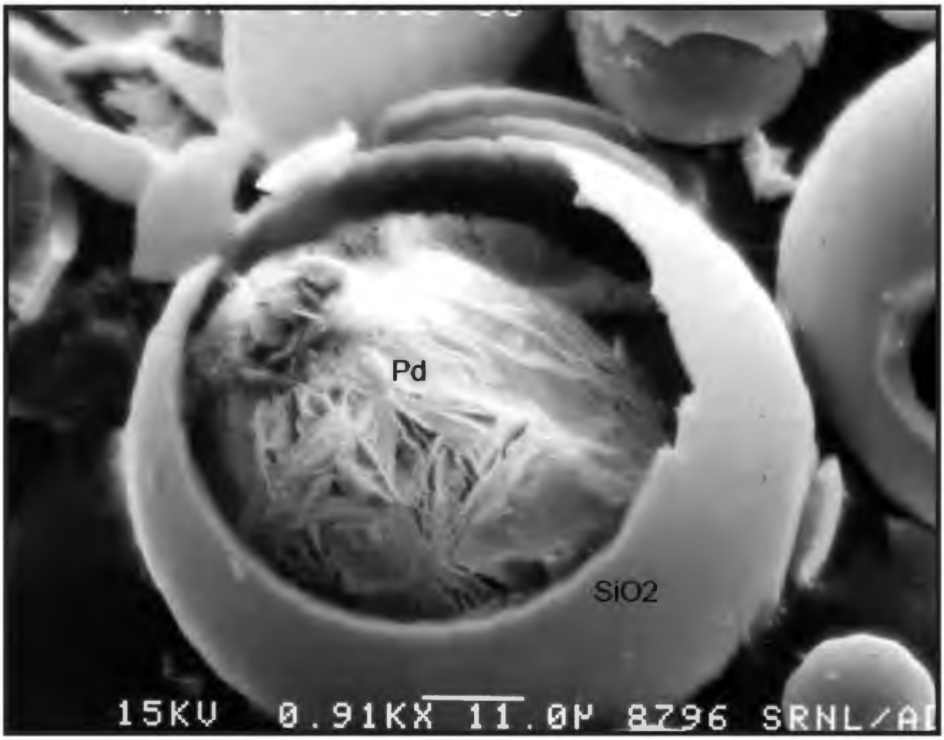

Figure 7. *SRNL microsphere filled with palladium (top of microsphere removed to view the inside)*

highly reactive absorbents could be filled and subsequently protected within the microspheres. This research could prove to be of major significance in the future of hydrogen fuel cells. In Figures 8 and 9 one of these nano-absorbent microspheres is shown for the first time. Bundles of nano-filaments were produced and noted, these bundles were observed on the outer as well as on the inner walls of the microspheres.

Further work is needed to clarify these unique findings. As noted in Figure 8, the nanostructures can be produced on the outside of the microspheres. This information is not only relevant to absorbents of gases but is also being investigated with the Medical College of Georgia using protein and fluorescent indicators for possible use in drug delivery systems and as new types of MRI contrast agents. An article published in the peer-review journal *Nanomedicine: Nanotechnology, Biology, and Medicine* discusses a possible application of the SRNL PW-HGMs for the delivery of anti-cancer drugs.

Figure 8. *Nano-structure absorbent grown outside PW-HGMs*

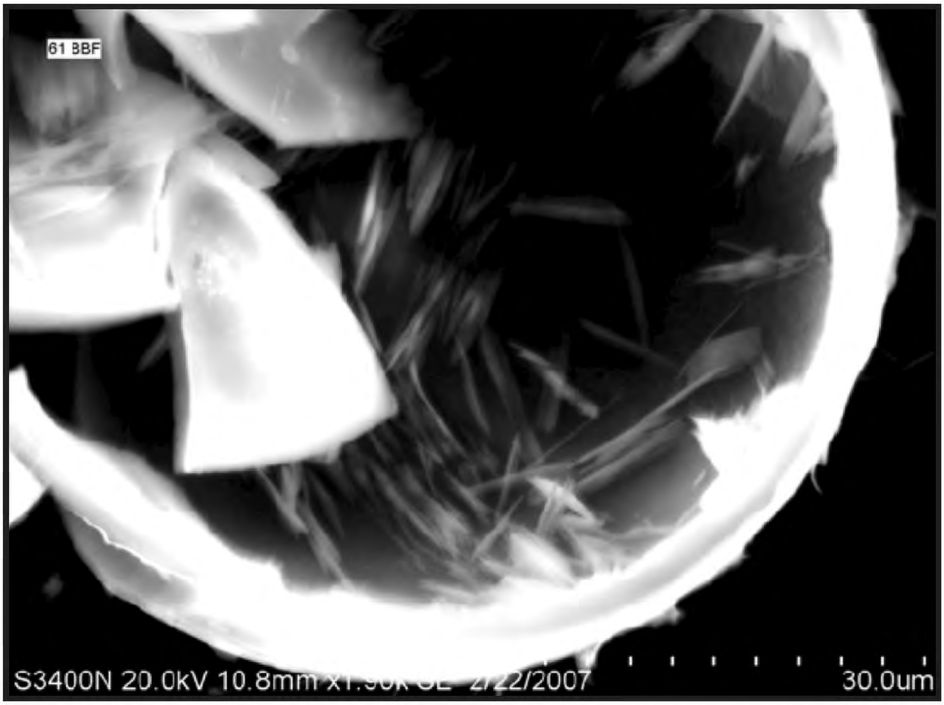

Figure 9. *Nano-structure absorbent grown inside PW-HGMs*

Porous Wall, Hollow Glass Microspheres have potential for use in targeted drug delivery, hydrogen storage, and other applications. SRNL's partners in this winning technology include Toyota, the Georgia Health Science University (GHSU), and Mo-Sci Corporation, a specialty glass provider who has been licensed by SRNL to manufacture and market the microspheres. My special thanks to Dr. George Wicks of SRNL for his help and guidance in preparing this paper.

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