

Proceedings

THE TWENTY-SECOND SYMPOSIUM
ON THE
ART OF GLASSBLOWING

1977

THE
AMERICAN SCIENTIFIC GLASSBLOWERS SOCIETY

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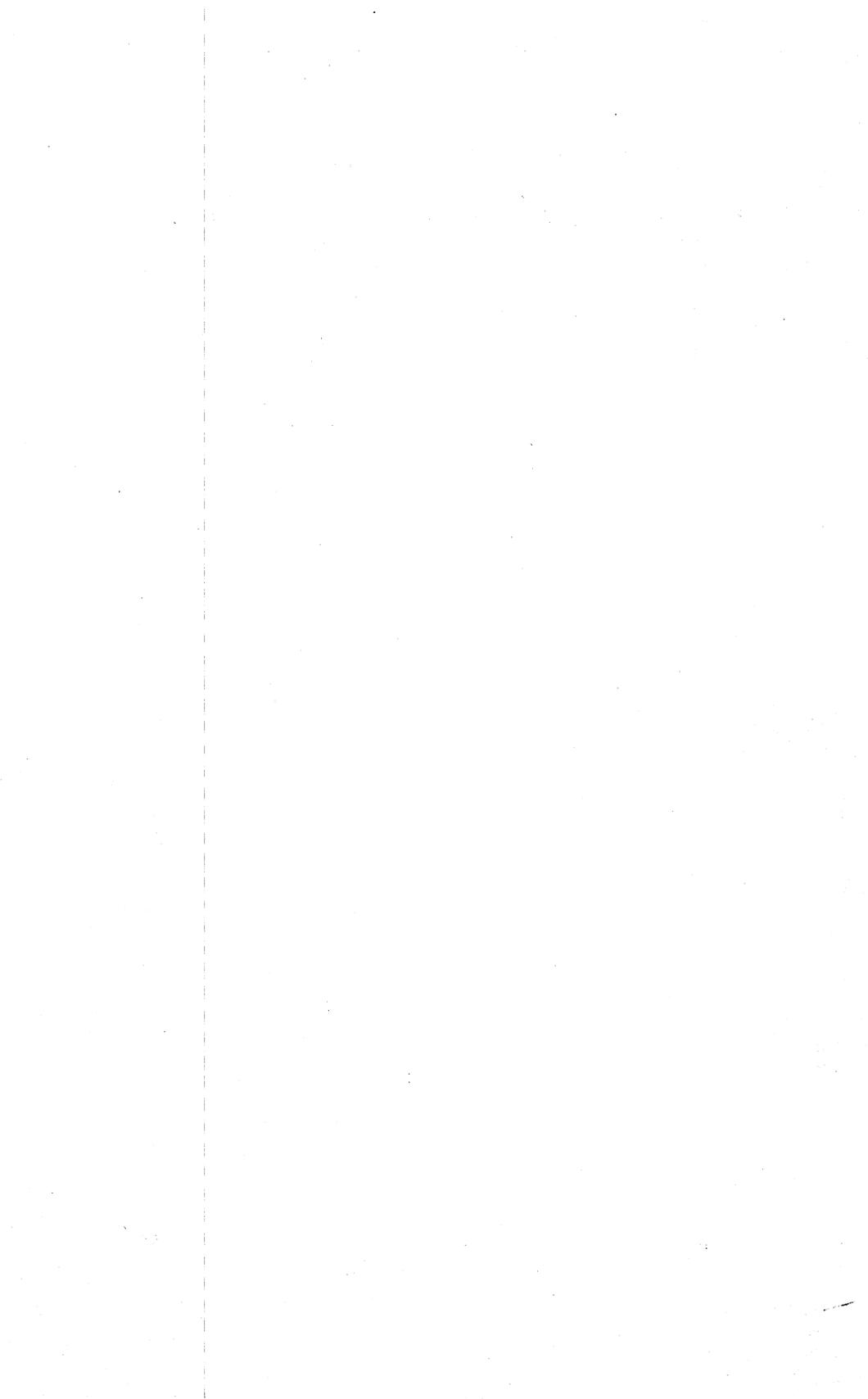
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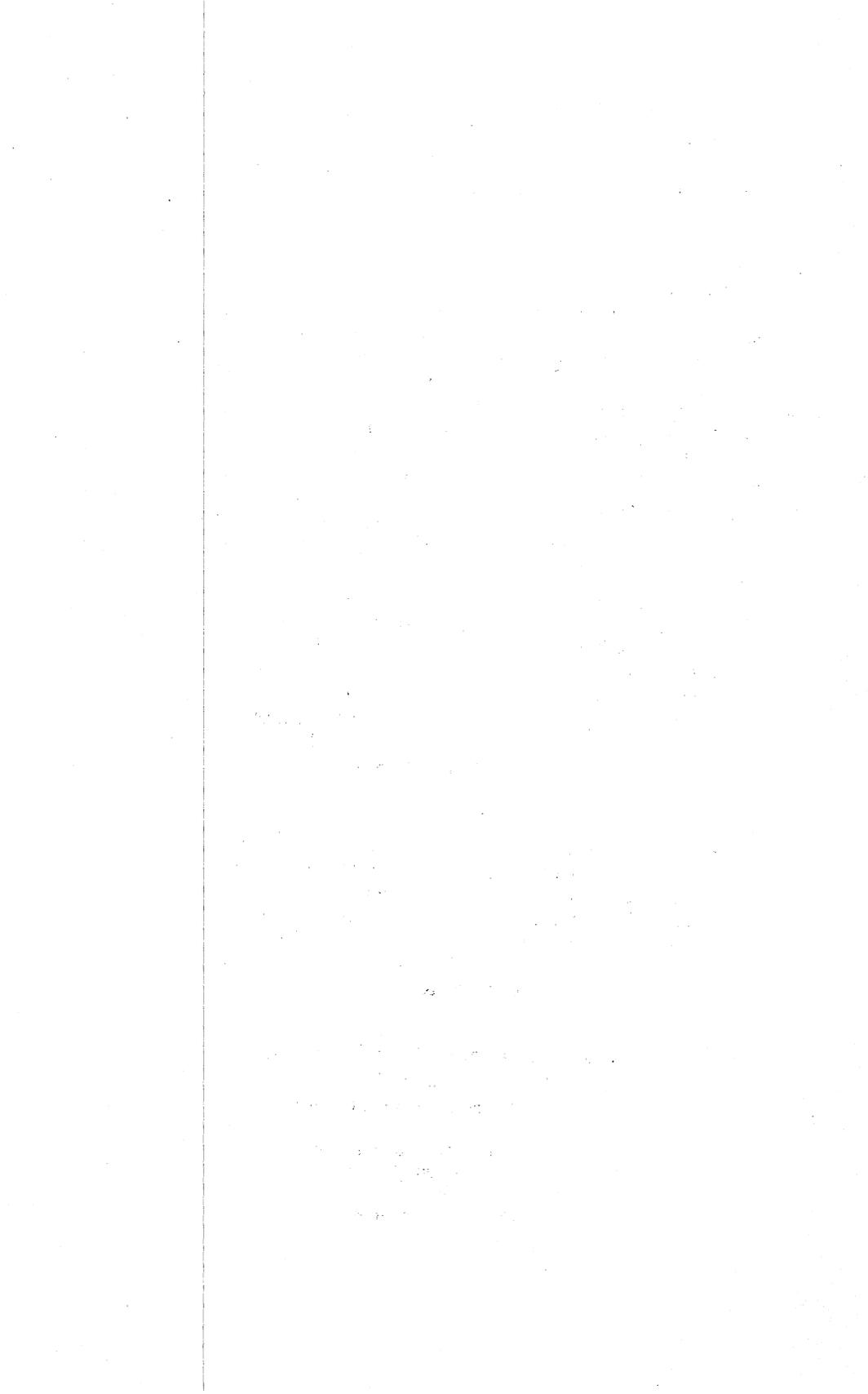
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A SURVEY OF ANCIENT GLASS TECHNOLOGY

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The intent of this paper is to survey early processes for the manufacture of glass objects by presenting information from early texts on glassmaking as well as results of the analysis of glass artifacts. A contemporary ethnographic example will be considered which supports archaeological evidence of prehistoric glass technology. In addition, a seventeenth century German text on glassmaking is cited as the first known description of lampworking.

The earliest manmade glassy material is a glass coating fired onto steatite beads used by members of the Badarian culture in Egypt at 4000 B.C.¹ We do not know how these beads were made. Steatite is a naturally occurring calcium-magnesium-silicate. The glaze contains soda, lime and silica. There are no marks from drying or firing on the glassy surface of the beads. From 2500 B.C. beads with a soda-lime-silica glaze coating a core of silica, either quartz, ground flint or sand, occur at many sites in Egypt, Mesopotamia and India. Three methods have been proposed for the manufacture of these beads. Alfred Lucas proposes forming the core material in a fired clay mold, followed by coating that part of the bead or amulet which will not contact a firing support with a soda-lime glaze, and firing to the appropriate temperature, about 1000°C.² Charles Binns experimented with the effluorescence of soluble salts from the core material, and he suggested this possible method of manufacture.³ Soda, lime and silica are mixed together with water, and molded. Upon drying, the soda which is soluble in water diffuses to the surface where water evaporates and the soda effluoresces on the surface. The molded object is then fired to form the glaze. If one examines the under-side of such objects, two distinctive types of marks occur, caused either during drying or firing. Where the object contacted a surface during drying no water could evaporate, thus no soda was deposited and no glaze formed. The other type of mark may occur during the cooling part of the firing cycle, where the solidified coating will adhere to kiln supports, particles often becoming embedded in the glassy surface. Firing marks can occur on objects made by the method proposed by Lucas as well as that proposed by Binns.

The third method involves a cementation process where no firing or kiln markings occur. Hans Wulff in surveying ethnographic crafts in Iran, described the making of beads in Qom which are used to decorate donkey harnesses.⁴ Ground quartz is mixed with gum, hand formed and dried. The beads are embedded in a powder of plant ash, lime and charcoal, fired to about 1000°C, above the melting point of soda and below the reaction point with lime. The molten soda draws away from the lime due to surface tension and coats the beads. Upon cooling, the powdery lime can be easily crumbled, exposing the beads. Figure 1 shows a replication of this process. Until recently, this ethnographic example was an interesting curiosity, but an earthenware pot containing a lime powder and beads, all sintered by over-firing, has been found in Egypt dating to the first millennium B.C.⁵ Further

analysis will be needed in order to understand the technology used for production of the earliest glassy materials.

Evidence from many first and second millennium sites indicates that a many step roasting, fritting and melting process was employed to refine raw materials for glass.⁶ Raw materials were probably fritted and melted in flat, dish-shaped crucibles. Upon cooling, the crucible was chipped from the glass ingot. Pieces of glass from the ingot may have been either heated and rolled or drawn into rods or simply flattened, or a solid mass of glass was ground in a manner similar to the treatment of stone. The earliest glass, whether used as beads, amulets, or inlay beside precious stones and gold in jewelry, furniture or masks, is opaque. The quality of intense, saturated color is believed to result from the intentional imitation of semi-precious stones. Examples were shown of a face carved and ground in profile and an inlay around an eye which was constructed from bent and joined rods and subsequently set into another material, such as stone or hammered gold sheet, with a bitumenous binder.⁷ At Tell el Amarna, and probably many other sites, the workshops for stone, ceramic and glassy materials were located in contiguous compounds near the palace.⁸ The proximity of workshops and potential for interchange of information between workshops may possibly explain the observed complexity of technology and diversity of artifacts.

The early production of glass beads and vessels, limited in quantity and intended as luxury goods, employed a technology based on the fusing of rods on a copper armature or on a friable core.⁹ The decoration was composed of rods of glass threaded onto the core much as one would wind thread on a spool, then combed into place and marvered, that is rolled against a slab, until a level surface was achieved. The vessel was annealed with the core in place. When cooled, the core was removed by scraping the porous core material. Marks of the tool often remain in the vessels, especially near the shoulder.¹⁰ Vessels formed on a core were made in limited quantity over about a 1500 year period until replaced by blowing during Roman times.

Although most early glass has been found in Egypt, it is quite likely that glassmaking and forming was introduced in the Near East (Syria or Iraq) because there is a greater diversity of technology present in the Near Eastern artifacts.¹¹ Glass is ground, or molded, or fused either as canes or as cross-sectional segments of rods, in addition to core molding. The diversity of beads which have been freely manipulated when molten is extensive. The numbers and states of preservation are poor due to corrosion. The glass has been buried in alluvial plains and has thus been seasonally washed with rain, as opposed to Egyptian glass in which careful burial in limestone cliffs above the Nile floodwaters promotes preservation. Another factor conducive to deterioration is that much Near Eastern glass has been subjected to heat, as for instance during the sacking and burning of a city. The earliest textural references to glassmaking are ninth century B.C. cuneiform tablets, which describe glass melting as a many stage roasting and smelting process carried on in small workshops.¹²

In contrast to the early production of glass as royal luxury goods produced by small workshops in limited quantity, is the large scale, high volume glass factory production and organization of Roman times. By 300 A.D.



FIGURE 1.

Beads which replicate Qom cementation process of glazing



FIGURE 2.

Egyptian core vessel

glass working had spread from Spain to Russia, from Britain and Germany to North Africa, and throughout the Near East.¹³ In excavation, a Roman level of occupation has many times become apparent by the existence of large numbers of glass fragments in refuse middens. For instance, small, inexpensive drinking vessels of the late first century A.D. were blown into two or three part molds, and the lip subsequently ground. For the most part, cups such as these were not punted. Decoration often imitates repoussé metalwork with friezes of gladiators in combat, circus scenes, or inscriptions such as toasts or the signatures of glassmakers. Another example is the molded square bottle used for the storage of oil, the shape of which facilitated packing of many bottles in a wooden crate. Here the bottle is blown into a square mold, empointed and the lip firepolished and rolled back upon itself for additional strength. Roman blown ware is quite thin, usually less than $1/32$ nd of an inch. Thus, annealing is not as critical a process as with the earlier Egyptian and Near Eastern core vessels, which average three

times greater in thickness. The beauty of much Roman glass depends upon its utilitarian quality as well as design which promotes production efficiency.

Romans were not without glass luxury goods. Examples include the cage cups or diatreta in which surface design is undercut and supported only by residual glass posts connecting the cup with the cage decoration. From the fineness of workmanship as well as the tremendous expenditure of time and effort required to complete a cage cup, one surmises that Roman glass production was divided among several specialized groups of workers. In general, glass workers are believed to have been craft specialists, divided into the vitrearii, or hot glass workers, who molded the glass blank for a cage cup and the diatretarii, or cold glass workers, who must have invested the time and skill to cut the cage into relief. Indeed, a law suit has been described in which a vitrearii is accused of providing a blank which, after much cutting, cracked.¹⁴

The first literary evidence of glassmaking technology in the west was written by a monk, Theophilus, in the eleventh century A.D.¹⁵ This text, entitled *On Divers Arts*, records information on the making of glass windows and objects necessary to complete a cathedral. Included in the text is an explanation of the cylinder method of window manufacture, a description of furnace construction, notes on the staining and gilding of glass as well as techniques for the making of rings, imitation gems and chalices. This text reveals the extent to which the loss of Roman organization and centralized distribution of raw materials affected glass production. A lessening in the degree of chemical understanding and control is exemplified by Theophilus' apparent lack of understanding of the relationships between oxidation state and decoloration with manganese dioxide.

Two other important and readable early texts are *The Pirotechnia* (1540) of Vaniccio Biringuccio, a glass factory owner and bronze founder, and *The Art of Glass* (1612) by Antonio Neri, a Renaissance entrepreneur.¹⁶ *The Art of Glass* was translated from Italian into English in 1662 by Christopher Merrett, secretary of the British Royal Academy.¹⁷ Each of these authors stress the importance of careful preparation of raw materials in the making of high quality clear glass. Neri and Merrett evaluate the relative merits of various sources of soda ash, sources from as far away as Spain, Egypt and Syria-Palestine. The information they present is a reflection of the technological history of glassmaking. With the rise of city states, commerce and a mercantile class in Europe during the late Middle Ages, the need for personal luxury goods becomes prominent. Examples of the complex and attenuated forms common in goblets were portrayed, as in Figure 4.

Another Renaissance development in technology is the use of a small heat source to produce fine detail or minute size in glass. The point at which it becomes more convenient or efficient to lamp work a piece of glass is a subject of debate.¹⁸ We suspect but cannot prove that some goblet stems may have been lampworked. A search for objects which would unquestionably require working over a torch was made. A group of early scientific apparatus is preserved in Florence from the late seventeenth century.¹⁹ Equipment which documents the development of digital and graduated



FIGURE 3.
Roman diatreta or cage cup



FIGURE 4.
Venetian-style goblet

thermometers by the academy of experimental science involves the sealing of fluid within the glass cavities by the use of a small heat source. Generally, a technological improvement precedes by some length of time the written documentation. The earliest textual reference to a torch is the German translation of *The Art of Glass* made by Johann Kunckel, a noted chemist and glass technologist, which was published in 1679.²⁰ Added to the translation is “. . . an original account on the little glassblowing which is achieved by means of a lamp.” Kunckel states that an oil lamp with a cotton wick serves as a heat source and air is blown with a bellows, through tin tubes which pass through a table (Figure 5). Another tube, which is not shown in the engraving, Kunckel says is fitted on the end of the tin tube and is bent at right angles. This tube has a small orifice “. . . so that the air can produce a small pointed and concentrated flame.” He describes using glass tubing and rods for the making of beads, little balls, small vessels and trinkets, such as figures and crosses. This account also includes the following prophecy: “He who understands can do in the glass what he



FIGURE 5.
Engraving of torch from text by Johann Kunckel

wants." "Other advantages each one is free to find out for himself." Subsequent improvements in furnace design, fuel sources and raw materials characterize the following centuries.

The major technological changes in the glass industry at the beginning of the twentieth century are the development of automatic forming machinery and the understanding of relationships between the chemical compositions of glasses and their physical properties. In Great Britain and the United States in the nineteenth century, an escalation of the size

and complexity of glass forming equipment occurred, as can be shown in the patent literature, in molding and pressing equipment and in the eventual automated production of bottles, patented in the United States by Michael Owens. In Germany, at the Schott Glass Works thousands of glass compositions were melted systematically and the elemental composition was related to standardized measurements of physical properties. Each of the developments described in this survey presage the enterprize of contemporary scientific glassblowing.

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FLAMELESS ELECTRIC HEATERS (SERPENTINE)

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INTRODUCTION

For many years industry has relied on gas flame and inefficient tubular heating elements for their Process Heating Applications. Presently the pressures caused by gas shortages, OSHA safety requirements, and higher productivity have created a need for an efficient electric heat source with inexpensive controls. Sylvania has developed a unique heating element whose configuration has extremely high heat transfer properties. This element is known as the Serpentine Coil.

CONSTRUCTION

Figure 1 shows the Serpentine Coil, a coil designed to expose the maximum surface area of wire to gas flow; this improves the heat transfer substantially over a conventional helical wound element. The Serpentine Coil is then mounted on a threaded ceramic core that supports each turn of the winding and the leads of the winding are crimped to a suitable electric connector with the assembly then being housed in an insulated tube. This forms the nucleus of a simple, efficient, inexpensive electric heater capable of heating gases to high temperatures (Figure 2). The heat transfer efficiencies of the Serpentine Coil and the helical winding are shown in Figure 3. Filament and gas temperatures are used to determine this "temperature efficiency" of an air heater. Thus 100% temperature efficiency would be achieved when the gas temperature equals the filament temperature. This indicates only the heat transfer efficiency and not the efficiency of total input power versus power required to heat gas to a given temperature.

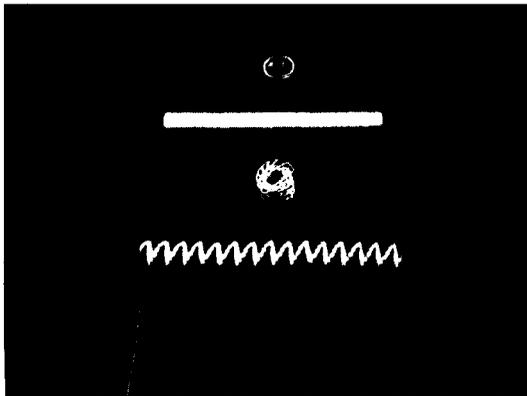


Figure 1

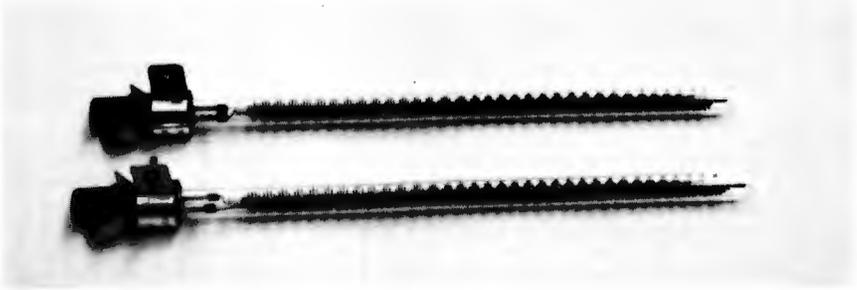


Figure 2

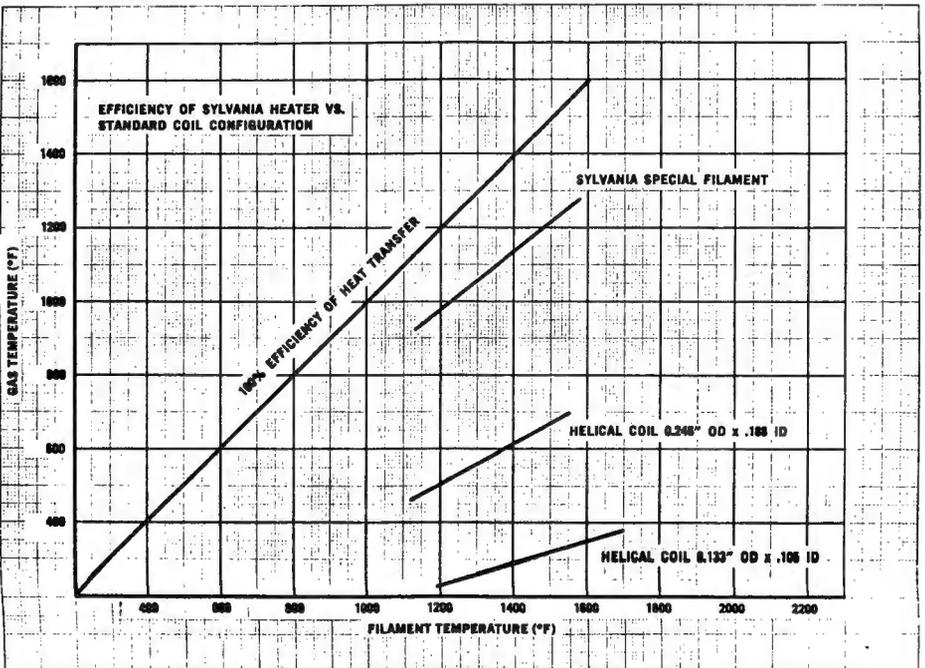
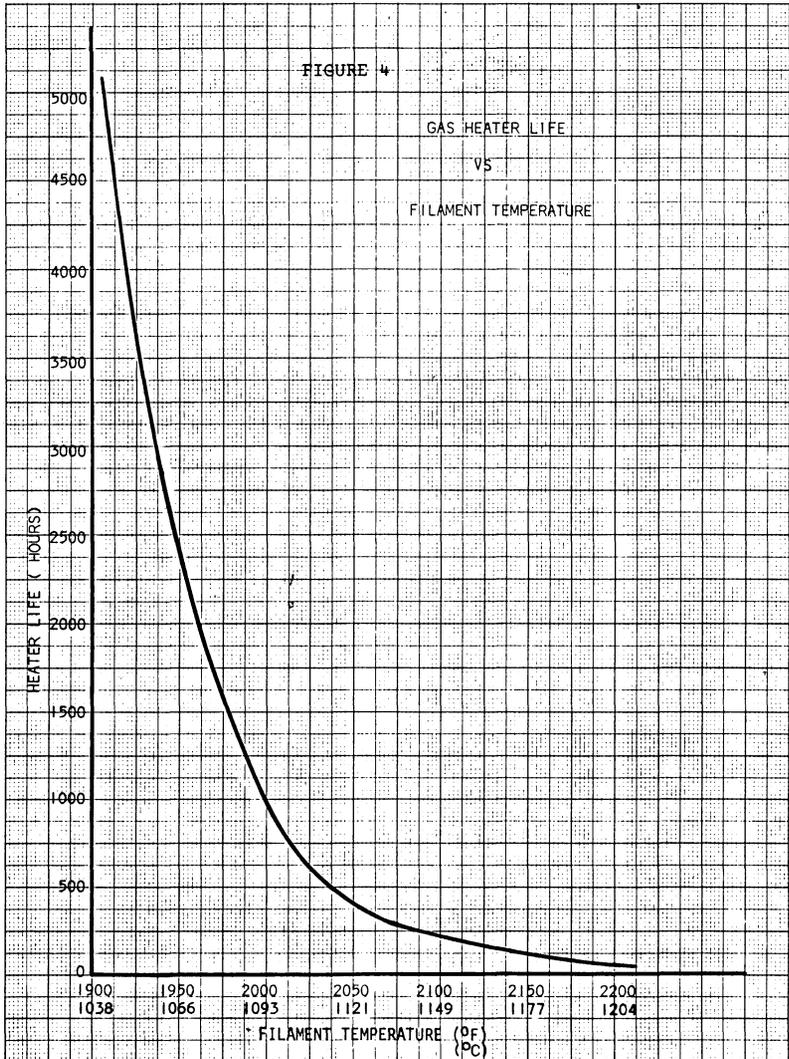


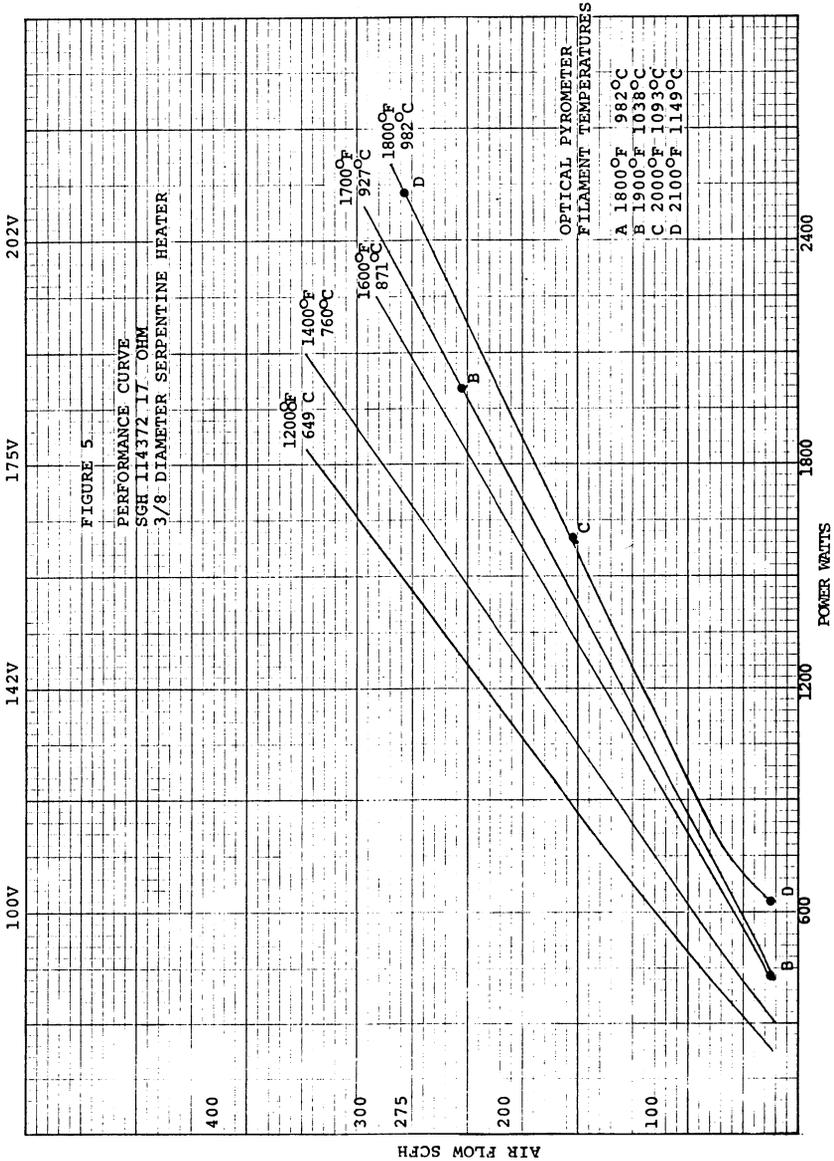
Figure 3

Conventional heaters normally have 40-50 percent thermal efficiency, while $\frac{1}{2}$ diameter Serpentine heaters operate at 80 percent. Consequently, the Serpentine is a more efficient filament and has a longer life since the filament temperature is lower than with other type heaters.

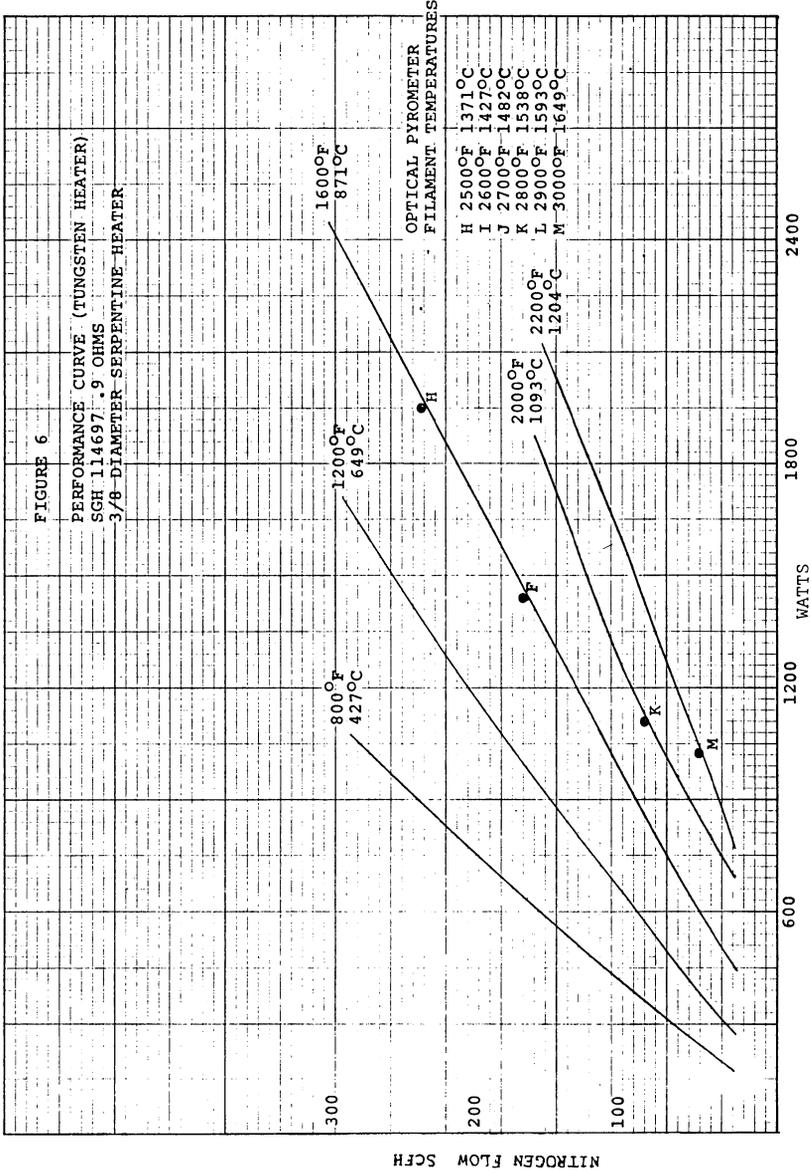
Figure 4 shows the life expectancy of a heater with the Serpentine configuration. (All heaters are life tested on a cycling basis [25 min. on, 5 min. off] over a range of filament temperatures from ambient to 2300°F/1260°C). A life in excess of 5000 hours can be expected when filament temperature is a maximum of 1900°F/1048°C. With a $\frac{3}{8}$ Diameter

Serpentine heater, gas output temperature would be 1700°F/927°C, or 90% efficiency. The maximum temperature output normally attainable using conventional coils is 1000°F/538°C — 1200°F/649°C. Life on such a heater is in the range of 1000 hours. Figure 5 shows typical performance curves on a nominal 17 ohm heater, Part Number SGH 114372 that has an inside diameter of 0.320 in. The curve demonstrates the efficiency and temperature capabilities of typical heaters. Example: with an air flow of 275 SCFH the air temperature at the nozzle is 1700°F/927°C with the filament temperature at 1900°F/1038°C.





The Serpentine heater can attain temperatures to 2200°F/1205°C by changing the heating element to a refractory metal (such as tungsten) and using an inert gas. The performance curves of a tungsten element heating nitrogen gas is shown in Figure 6.



A tungsten filament with an inert gas could operate at over 3000°C but the higher temperature of the tungsten heater is limited by the insulation materials used in the construction of the heater.

Tungsten heaters must be purged with an inert gas before being energized electrically and must be cooled to under 200°C in the inert atmosphere to keep the element from oxidizing. Unlike a ferrous alloy element whose resistance does not change when heated to high temperatures, the refractory (tungsten) element changes substantially. A “soft start” controller is used to increase the voltage in stages to allow the resistance change to stabilize thus avoiding high in-rush currents.

Some applications require higher flow rates that need higher wattage heaters.

One-half and One inch diameter Serpentine heaters are designed to allow for these higher flow rates. The larger diameter Serpentine heater with higher wattages heats greater flow rates at the expense of transfer efficiency.

Figure 7 shows the air temperatures of the three heaters with the filament at 1900°F/1038°C.

Figure 7

<u>Diameter</u>	<u>Part Number</u>	<u>Flow CFH</u>	<u>Gas Temp. °F/°C</u>	<u>Wattage</u>
¾	SGH 114372	275	1700/927	2300
½	CFH 117558	600	1200/650	3800
1	PGH 121700	1000	1000/538	6000

The transfer losses on the larger diameter heaters have been improved by using the basic Serpentine concept and enclosing it in a three pass heat exchanger housing. The ½” diameter is referred to as Serpentine II and the 1” diameter as Serpentine VI. (Shown in Figure 8)



Figure 8

The three pass housings are designed to have the entering gas flow cool the outer barrel, be preheated as it flows pass the inner barrel and finally superheated as it flows over the Serpentine coil. The radiation losses are eliminated with a significant improvement in heat transfer.

Figure 9 shows the data of the ½" and 1" diameter heater with and without the three pass housing with the filament at 1900°F/1038°C.

Figure 9

<u>Diameter</u>	<u>Part Number</u>	<u>Flow CFH</u>	<u>Gas Temp. °F/°C</u>	<u>Wattage</u>
½	CFH 117559	600	1200/650	3800
½	CHE 129762 with housing	600	1400/760	3600
1	PGH 121700	1200	900/482	6000
1	PGH 140291 with housing	1200	1300/705	6000

An increase of 200°F/110°C with a reduction of 200 watts is attained with the ½ inch diameter heater and a 400°/223°C increase is attained at the same wattage with the 1 inch diameter heater.

Tungsten element with the three pass housing delivers gas temperatures of 2651°F/1455°C with very good life. This concept offers an attractively alternative to gas flame for working glass. Excellent repeatability is obtained in sealing soft glass with significantly reduced strain: The heating elements for Serpentine II are 2000, 2800, and 3600 watts at 240 volts and are replaceable. Serpentine VI is made with a 5000 and 6000 watt replaceable element at 240 volts using the ferrous alloy elements.

The three major causes of premature element failure are:

- Compressor oils in the air line contaminating the heating element. The addition of an air filter and regulator before the heater will insure cleaner air.
- Air pressure loss with element being energized electrically. The addition of a normally open pressure switch before the heater could be set to de-energize the element with a loss of air pressure.
- Using the element above the recommended temperature. The proper balance of air flow and voltage should be maintained to keep the filament in the safe operation range.

Figure 10 shows a "Bench Top System" that has complete air and temperature control. The system operates on 120 or 240 volts and 5 PSI minimum air pressure, and is designed for the user to set the desired gas temperature on a phase angle fired temperature control mounted on the face of the system. A sensor positioned at the exist of the heated air stream will maintain the dial temperature within ±1%. If the air flow or dial temperature is changed substantially, the controller will automatically adjust the required power in seconds.



Figure 10

The system has a built in pressure switch set at 5 PSIG, an air filter, an air regulator and a circuit breaker.

The latest product that incorporates the Serpentine winding is shown in Figure 11 — “The Flameless Electric Burner”. The unique design operates on 120 volts, 1000 watts and 6 PSI of air to activate the Serpentine element.

Temperatures of 1100°F/595°C with the three pass housing keeping the barrel cool. The burner is designed for hand held applications and comes with a mounting bracket that can be secured to a table to hold the burner in a horizontal or vertical plane. The burner is gaining acceptance in industrial and laboratories applications due to the shortage of natural gas and the OSHA laws that do not allow combustible gases to be stored inside a public building.

FEATURES

- All the Serpentine heaters have the inherent feature of any open coil design; that is fast response to heating and cooling.
- The Serpentine is a low mass heater making its response to control faster than conventional open coils.



Figure 11

- Electric heaters are entirely flameless and are cleaner, safer and more controllable than combustible gas flames.
- Performance repeatability is excellent from cycle to cycle and heater to heater.
- The three pass housing reduces the potential for accidental skin burns. The unit can be permanently mounted and has easily changed heating elements.

APPLICATION

The Serpentine Coil has versatility and potential far beyond any gas heating concept of today. Element materials available allow for efficient gas temperatures in excess of 3000°C.

The Serpentine heaters are used in many industries in a wide variety of applications. The flameless heater can be used in any area where clean, fast, and efficient heat is necessary. Some of the unique applications are:

Packaging

Poly Carton Sealing machines have been redesigned to use the Serpen-

tine heater for faster carton sealing of ice cream, milk, juices, oils, and pastry.

Semi-Conductor

Silicon Wafer Drying — The Serpentine heater is used to heat nitrogen for drying silicon chips and wafers used in semiconductors.

Electronics

Soldering and Desoldering is accomplished with increased speeds and a minimum of oxidation. The high degree of control and direction of the heated stream allows a good quality solder connection without any surfaces being touched.

Glass

In glass annealing where temperature control is critical.

The strains produced in fire polishing, sealing and annealing are substantially reduced with the high degree of control and direction.

Annealing metal parts used in glass to metal sealing requires oxidation to insure a good seal. The Serpentine can be controlled to give this proper metal oxidation.

CONCLUSION

The shortage of natural gas is becoming more acute each year. Also the possibility of the federal government de-regulating natural gas prices will cause these prices to increase. OSHA laws also will affect the use of combustible gases adversely.

The Serpentine heater thus is gaining acceptance as a basic Process Heating tool limited only by the imagination of its user.

The high temperatures of the Serpentine heater with the inherent features of controllability, cleanliness, and safety are here today as a tool for most glass working processes.

THE OPTICAL WINDOW AND WHAT WE CAN DO WITH BOROSILICATE, QUARTZ AND SAPPHIRE

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BOROSILICATE GLASSES — Figure 1

Preparation

Chuck the graphite vacuum holder in the lathe and connect a vacuum hose with a swivel to a mechanical pump or house vacuum. Chuck the tubing in the opposite end of the lathe with a blow hose attached. Start vacuum pumping and position the window on the face of the graphite holder.

Preheating

Allow the annealing burner flame to form a carbon deposit on the window and the seal area. Continue the preheating until the carbon disappears from about one half of the window diameter. Avoid heating the graphite.

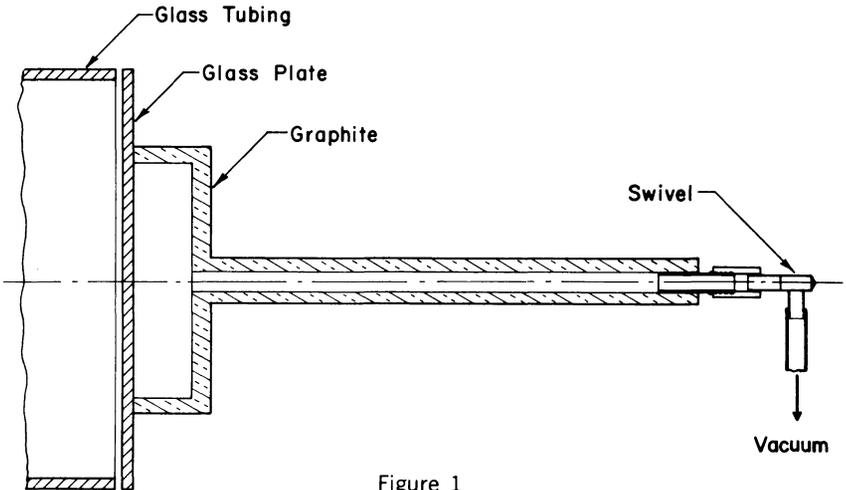


Figure 1

Sealing

Direct the lathe burners on the seal area with intense sharp fires until the seal appears complete. Use a paddle while blowing to make the seal uniform if necessary. Quickly remove the graphite from the window.

*The research described herein was supported by the Division of Physical Research of the U. S. Energy Research and Development Administration.

Annealing

Direct the annealing burner at the face of the window allowing the splash to also anneal the tube. Place the completed seal in an oven already at annealing temperature if possible. With care many seals can be brought to room temperature with the annealing burner.

QUARTZ

Quartz window seals can be made in the same manner. Preheating and annealing are of lesser concern but flame cleaning of surface devitrification is usually necessary after completing the seal.

QUARTZ WINDOW SEALS FOR OPTICAL CELLS AND DEWARS

Since this seal was described in detail in a paper I presented at the 1974 Symposium (pages 77-81 of the Proceedings) I shall only review it briefly. We also demonstrated the seal at Philadelphia and Chicago as well as London, England.

Preparation

The preparation of the tube end is of utmost importance in this seal. It must be ground flat with 600 or 1000 grit grinding compound on a lapping plate and then polished to a glossy finish on a nylon cloth using jewelers rouge or cerium oxide. This may sound like a time consuming procedure but the step can be accomplished in 10-15 minutes. When this is properly done and the window set in place on the tube end, interference fringes can be observed at the interface — Figure 2. The joining parts must be completely free of any dirt particles since they may cause a void in the seal. I use a 10% HF rinse followed by distilled water and air drying before the sealing operation. The window is then placed on the tube end and held in place with a slight vacuum or external pressure device.

Sealing

Using a #1 tip on a National Torch start the seal by heating one point on the perimeter of the window until the seal starts showing on the inner wall edge of the tube and follow this around the window. Remove the vacuum or pressure device and with another circling of the seal area complete the seal.

Annealing

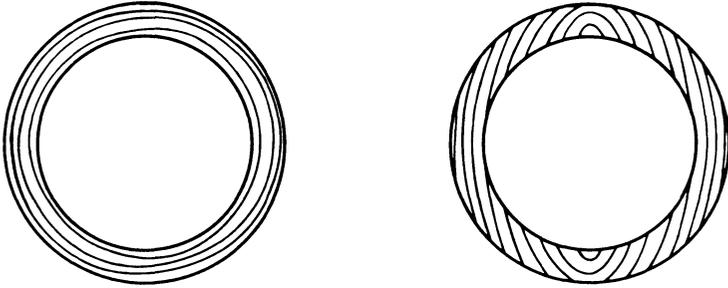
Anneal the seals at 1050°C

The advantage of this technique is that there is little or no distortion, parallelism can be closely controlled and there is no devitrification. These seals have been successful up to 3 inches in diameter.

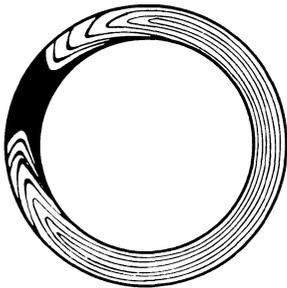
OPTICAL DEWARS

In regard to optical dewars the preparation of the shoulder for the window seal deserves some special consideration. In striving for a shoulder

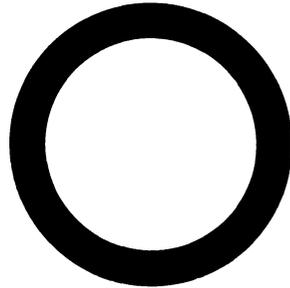
Interference Fringes



As they may appear before sealing operation (above)



Beginning of Seal



Completed Seal

Figure 2

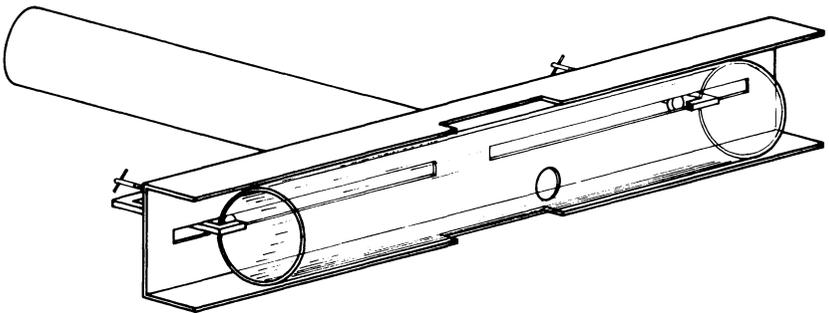


Figure 3

of suitable wall thickness it is very difficult to achieve by blowing since more of the glass is lost than necessary. It is also difficult to obtain a circular hole with a relatively uniform shoulder. Figure 3 shows a "T" holder that I designed for large "T" splices which serves well in preparing the shoulder.

Mount the tube in the holder, chuck the holder in the lathe and with the work piece rotating use a torch to pick a small hole of about $\frac{1}{4}$ " diameter. With the lathe burners tilted or a large hand torch heat the area surrounding the $\frac{1}{4}$ " hole and with a graphite reamer enlarge the small hole to the diameter of the window desired. This step should be completed in one quick operation to achieve the best results. Notice the cut-out section in the channel iron which allows for rotating the tube 180° and the reaming of the opposite shoulder. Now grind and polish the shoulders for the seal.

QUARTZ OPTICAL CELLS WITH THIN WINDOWS (0.2 mm)

There is too much flex with windows of this thickness so the method just described is not practical.

Preparation

Choose a quartz tube into which the quartz windows fit closely and attach a tubulation. The cell body, window and positioning tube should be mounted in a ring stand as shown in Figure 4.

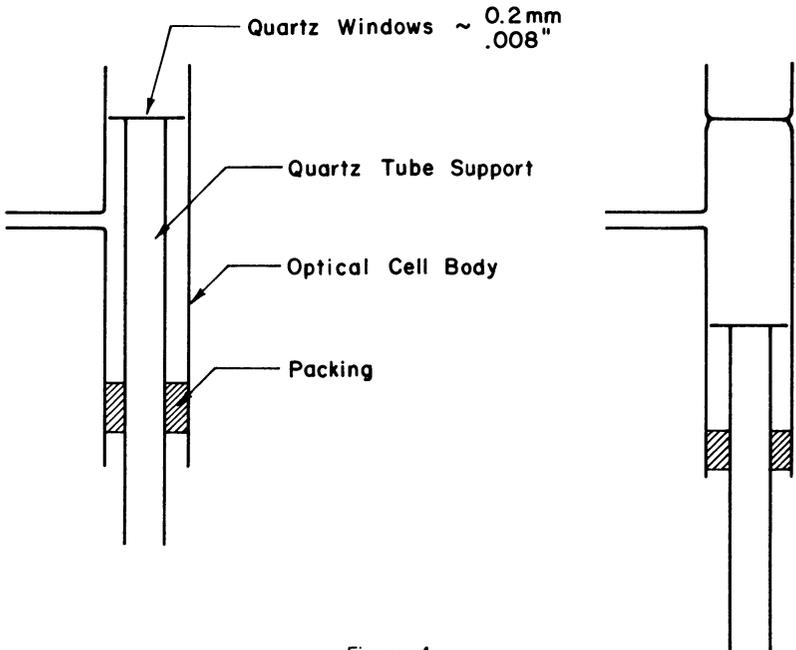


Figure 4

Sealing

The seal is started by localized torch heating of the tube until it meets and fuses to the window. Continue the seal by circling the tube with localized heating until fusion is complete. Now the positioning tube can be removed and returned with the second window resting upon it. The second window is sealed in the same manner. I suggest annealing the completed cell before removing the excess quartz tube on the glass saw.

A RECTANGULAR SHORT PATH LENGTH OPTICAL CELL

This is an optical cell where the path length required might be in fractions of a millimeter. The parallelism of the faces is controlled by a spacer, the distortion is minimal and little or no devitrification encountered. Figure 5.

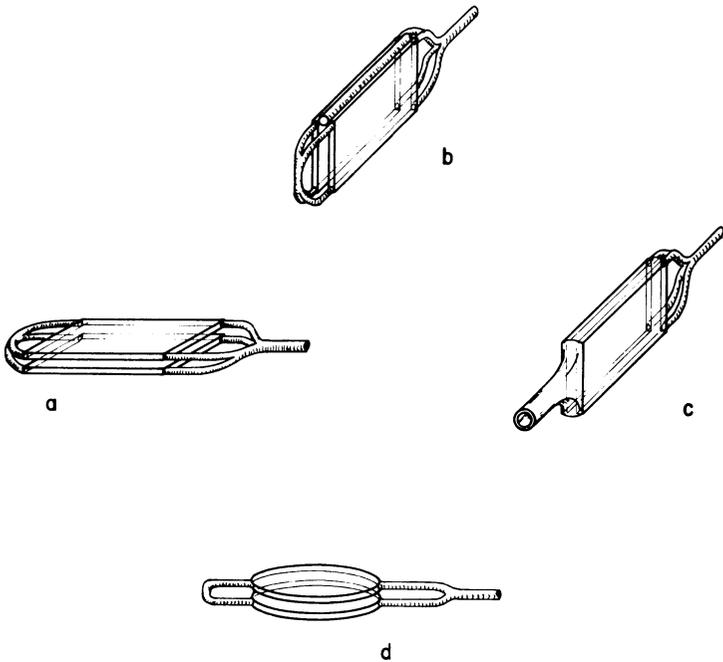


Figure 5

Preparation

The windows in this case are suprasil rectangles 5 mm wide x 50 mm length x 0.5 mm thickness. Short pieces of quartz rod are attached to all four corners of each rectangle and while a 0.2 mm spacer is held in place between the windows the rods are fused together and a tube or rod attached at one end to serve as a holder. Figure 5-A. The holder can now be clamped in a ring stand. Some suprasil rod is drawn of a diameter slightly larger than the spacing between the windows and cut to the same length of the cell. Position

the work piece such that the rod rests in the spacing of the cell edge. Figure 5-B. With a #1 tip on a National Torch move from one end to the other heating the rod until it fuses into the edges of the windows. I use a graphite rod to press lightly on the suprasil rod as it softens. Repeat this step with the opposite cell edge. Now remove the rod supports from the end of the cell opposite the holder and attach a tubulation. Figure 5-C. If it is a flow-thru cell, repeat this step at the opposite end of the cell.

This same method can be applied to short path length cylindrical cells. Figure 5-D.

SAPPHIRE WINDOW SEALING

Last but not least is a simple method of sapphire window sealing. Two of the more popular suitable sealing glasses 7280 and 7520 are no longer marketed by Corning but Kimble N51A and IN3 can still be obtained. Schott Glass of West Germany with a distributor in NYC manufactures a sapphire sealing glass #8436. Corning 7052 is not quite as suitable a match but can be used for smaller seals.

In this seal we must consider that sapphire is much more like a metal or a ceramic than a glass in the sense that when it is sealed to glass the seal is a bond as opposed to fusing. It has a coefficient of expansion of $\sim 65 \times 10^{-7}$ and a melting point $\sim 2050^\circ$. It is very sensitive to thermal shock from room temperature up to the temperature of the sealing operation. Therefore, any exposure to direct torch flaming can cause a fracture.

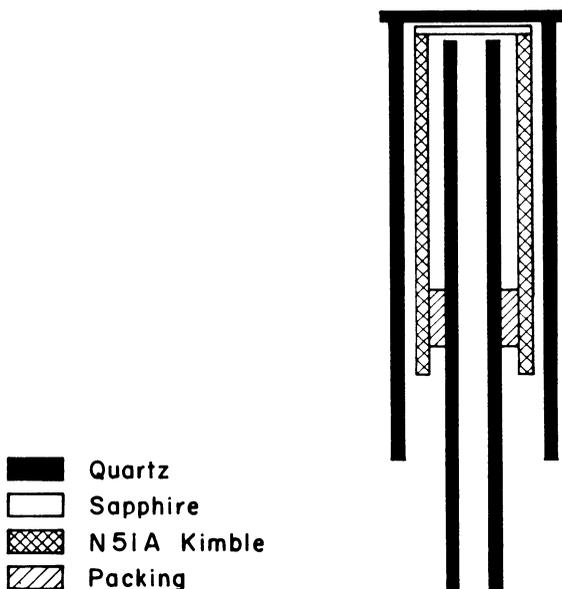


Figure 6

In the case of an optical cell with 2 sapphire windows you may choose the sealing glass for the complete cell with a graded seal on the tubulation or if only one window is needed, prepare a graded seal 774-3320-7052-N51A. The sealing surface of the N51A may be flame cut or flame polished after saw cutting.

Preparation

In a ring stand clamp the work piece and other parts as shown in Figure 6. The inner quartz tube acts as a resting place for the sapphire window after the glass softens, keeping it in perpendicular alignment to the tube. The outer quartz tube and plate allows for preheating, sealing and annealing without direct torch contact with the sapphire.

Preheating

With a #5 tip on a National Torch heat the surrounding quartz thinking in terms of bringing the temperature of the work piece up to the strain point of the sealing glass.

Sealing

With a circular movement of a sharply adjusted torch flame, heat the quartz plate directly above the seal area until the seal is complete with the sapphire window resting on the inner quartz tube.

Annealing

Reduce the work piece temperature by again flaming the quartz envelope or place in an oven already at annealing temperature (570°C is satisfactory).

MECHANICAL GLASSWORKING

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INTRODUCTION

This paper will, hopefully, have a two-fold purpose — first, to promote and encourage mechanical glassworking in more glassblowing shops, thus offering a more complete service to the scientist; secondly, to introduce to some glassblowers, unfamiliar with mechanical applications, some basic techniques and equipment for cutting, grinding, drilling and polishing of glass, ceramics and various other materials.

CUTTING FLAT STOCK — PLATE — PYREX — ETC.

To cut flat glass, such as plate or Pyrex®, the stock should be placed on a flat, felt-padded table. With a sharp glass cutter (Fletcher Terry) and a straight edge, make a mark at the desired location by holding the cutter with the angle side on the glass and making one stroke (with sufficient pressure to make a definite mark) for the full length of the desired cut. (Figure 1) Turn the glass piece over on the felt table, placing the marked side down, apply pressure with your thumbs to the back side of the marked area. (Figure 2) For removing narrow cuts of glass, a glass plier should be



Figure 1



Figure 2

used along the marked edge to run the crack smoothly along the mark of the glass cutter. (Figure 3)



Figure 3

For thicker pieces of glass and for cutting Pyrex, it helps to lubricate the glass cutter with turpentine. Quartz flat stock, of course, must be cut with a glass saw for good results. To cut circles, place a spring-tensioned glass cutter in an adjustable pivot bar. (Figure 4) Place the pivot in a drill



Figure 4

press, set for the desired radius and with the cutter set at a slight pressure on the glass, scribe your circle. (Figure 5) From the diameter of the circle to the edge of the glass, scribe lines with the glass cutter, about every one to one-and-a-half inches. (Figure 6) Tap the diameter of the circle with the end of a glass cutter; then break away the surplus glass and belt grind the edges smooth. (Figure 7)



Figure 5



Figure 6



Figure 7

Another method of cutting discs, washers or drilling holes is with a so-called biscuit or cookie cutter and a slurry of carborundum and water. (Figure 8) The cutter is made from thin walled brass tubing mounted on a shank that is compatible to the size of the chuck on your drill press. To prevent chipping when the cutter goes through the glass, it is advisable to wax the piece to be cut to an auxiliary backing plate of glass with red jeweler's wax or similar substitute. Fasten the material to be cut securely to the bed of the drill press. (Figure 9) Place a slurry of 150 grit carborundum and water on the glass and with the cutter rotating, apply slight pressure to the glass; release; feed more slurry; apply pressure; and so on until you have cut through the desired piece and into the backing material. (Figure 10) Allow about .010" to .015" material removal by the 150 grit slurry over the size of the drill. To contain the slurry to the desired location, a dam of Apieson Putty or similar material can be put around the area to be cut. (Figure 11) It is possible to modify a drill press with a variable speed motor and a reciprocating shaft so that with a cutter and a supply of slurry, the drill press can be set to cut discs or holes with no one having to be in constant attendance. (Figure 12)

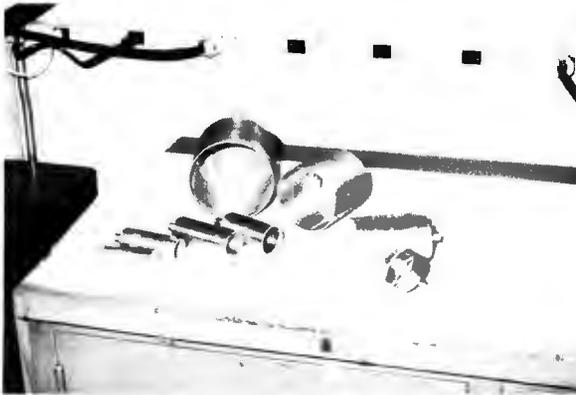


Figure 8



Figure 9



Figure 10



Figure 11



Figure 12

To cut holes in harder materials, diamond paste of different grit sizes can be used with nickel or brass tubing in place of a carborundum slurry. With this procedure, we have drilled holes in materials such as tungsten.

(Figure 13) Diamond core drills with a coolant head can be mounted in a drill press and used to core drill glass, ceramics, quartz or almost any



Figure 13

hard non-metallic material. Diamond drills will hold a closer tolerance and drill a straighter hole than using the tubing and slurry method.

(Figure 14) For faster drilling, routing, counterboring, etc., of hard materials where speed and accuracy is more important than cost, an ultrasonic machine is an asset to any glass shop. (Figure 15) This machine utilizes a cooled, rotating diamond tool in conjunction with ultrasonic vibration. We have found this machine to be extremely useful for working on very hard ceramics where other methods proved unsatisfactory. (Figure 16)

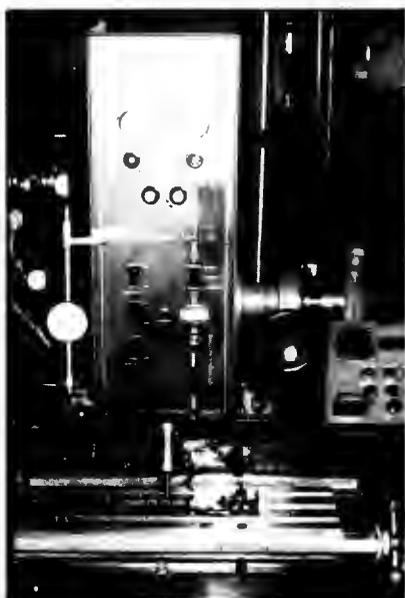


Figure 15

Figure 14



Figure 16

SHAPING

When shaping glass to unusual designs or patterns or cutting to a template, a starlite rotocator is very useful. (Figure 17) This machine uses a rotating and reciprocating diamond tool operated at high speed by high pressure air. (Figure 18)

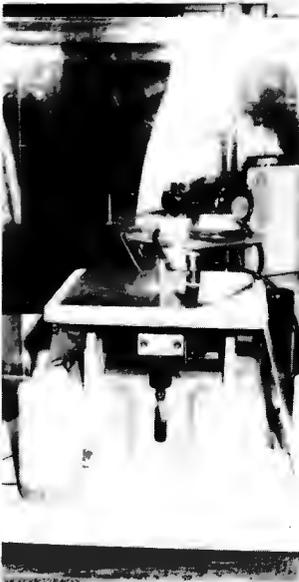


Figure 17



Figure 18

CUTTING — GRINDING — SLICING — SLOTTING TO CLOSE TOLERANCES

For cutting, grinding, slicing or slotting glass, ceramics, crystals or even moon rocks to close tolerances, we use magnetic chucks and dial indicators in conjunction with machines such as a Sanford or Do-all. (Figure 19) The material to be cut, along with a backing plate, should be waxed to a metal plate which can be held in place on the machine by the magnetic chuck. (Figure 20) Using a variety of either silicon carbide or diamond blades and grinding wheels, it is possible to cut slots as small as .003" and hold tolerances of .001". (Figure 21) Let me note here that the various manufacturers of these grinding wheels or cutting blades will recommend types; bonds, grit size and concentration to use on various materials.

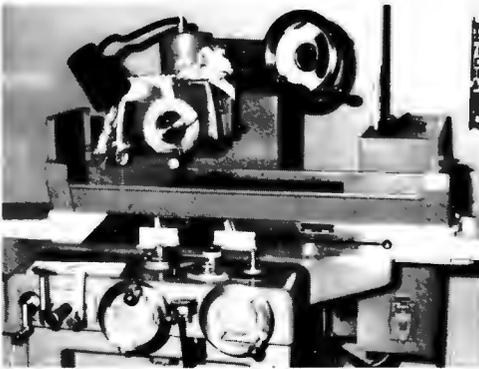


Figure 19



Figure 20

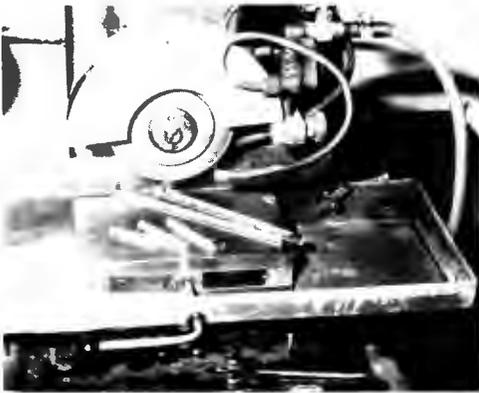


Figure 21

WIRE SAW

For cutting some materials or crystals, we use a wire saw as shown in the next figure. (Figure 22) These saws use either a loop of wire or as the one pictured, a hundred feet of wire on a capstan. The wires are either copper (used with a slurry of grit) or diamond impregnated wire which we lubricate with mineral oil or kerosene. Wire sizes used range from .002" to .015" in diameter.

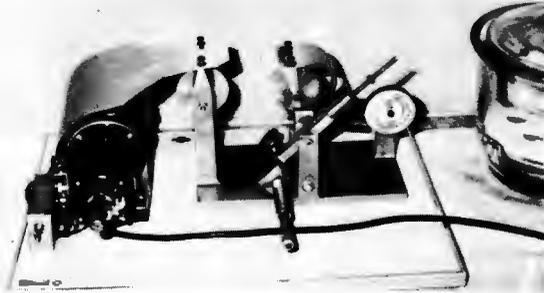


Figure 22

GRINDING PREPARATION AND POLISHING

Let me stress that when we talk of polishing or optical work, we are into a trade or art that can only be achieved by training, experimentation and practice. With this in mind, I will mention a few basic procedures we use to achieve a suitable polish on materials of different hardness. For softer materials, such as silicon or germanium, start with 600 grit (approximately 30 micron size) on a glass plate using a figure eight motion, if done by hand (Figure 23); or use a glass or cast iron plate if done on a lapping machine. Next, use 1000 grit (approximately 10 micron size), using the same procedures; and finally, use Linde A (.3 micron) on a Beewax Lap, either by hand or machine as shown in this figure. (Figure 24)



Figure 23



Figure 24

For Pyrex or quartz (depending on amount of material to be removed), start with 150 or 400 grit (50 micron size) on a glass, steel or cast iron plate. Next, use 600 grit on grooved glass or steel plates; then go to 1000 grit on grooved plates; and finally, to cerium oxide (about 1 micron) on a felt pad. (Figure 25) All of these steps can be accomplished either by hand methods or on a lapping machine.

Materials such as very hard ceramics or sapphire are best done on a lapping machine, using grooved copper laps charged with diamond paste. (Figure 26) Each grit size should be used with its own copper lap. Again,



Figure 25



Figure 26

depending on how much material must be removed, you can start with 45 micron, 30 micron or 15 micron diamond paste and then proceed through steps of nine, six, three and one micron paste, depending on the degree of polish desired. (Figure 27)

To prevent rolling of edges and to maintain flatness, extend your work piece with the same material or material of similar hardness. For example, if you are polishing a glass disc, drill a washer of the same glass; place the disc inside the washer and wax both pieces to a suitable holder. For lenses or curvatures, convex or concave laps with the desired radius of curvature can be machined or purchased.



Figure 27

TESTING OF POLISHED SURFACES

Using a monochromatic light source, such as a mercury or sodium vapor lamp (Figure 28), place a known optical flat onto your finished work



Figure 28

piece, press slightly, and allow the two pieces to equalize in temperature. (Figure 29) You will notice a system of what is called Newton rings or bands. Straight parallel lines indicate a flat surface, and rings or circles indicate a slightly convex or concave surface. The next figure (Figure 30) shows examples of pieces nearly perfect in flatness in #1 & #2; concave or convex in #3 & #4; and in #5 & #6, the same piece being both concave and convex on different areas of the same surface. Remember when testing with an optical flat, absolute cleanliness is essential, as a particle of dust can affect the fringe pattern.



Figure 29



Fig. 30 The use of the proof plate

Figure 30

CONCLUSION

To conclude, let me state once more that it is not the intent of this paper to give expertise in mechanical glassworking, only to show a few procedures and equipment to promote this type of work in more glass equipment shops.

ACKNOWLEDGEMENTS

The author of this paper would like to thank the five mechanical glassworkers of the General Electric Corporate Research and Development Center for their help and advice during the preparation of this presentation.

DEVELOPMENT AND TECHNIQUES OF TUBULAR QUARTZ TO MOLYBDENUM SEALS

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Gloucester, Mass. 01930

Bomco was incorporated in 1960 and from the beginning a significant part of our business has been making metal parts for companies making glass to metal seals.

In 1966 an engineer at Sylvania, Salem, Mass., David R. Dayton, came to us with a conceptual drawing of our present moly cup. I told him that in our opinion no one could do that economically, if at all. His response was that he had not come to solicit our opinion, but, rather, our willingness to try. We did, and six weeks later delivered our first parts, giving birth to the Bomco Moly Cup, which made possible the first modern 5 and 10KW tungsten/halogen studio lamps.

We subsequently applied for and were awarded two patents for our process of making feather-edged moly cups. Bomco was not the first to make feather-edged cups, but we were the first to make them with the proper metallurgical structure, dimensional tolerances, and, most importantly of all, at a low cost.

Dayton and his team developed the present method of sealing the Bomco Moly Cup into quartz based upon the Houskeeper principal developed at Westinghouse in the 1920's and expanded upon by Griener at G.E. and Thorne in England.

Sylvania's first lamps utilizing the Bomco cup were introduced in 1968. In the next several years other manufacturers of arc discharge lamps successfully made both short and long arc high wattage lamps for parking lots, stadium searchlights, and projectors using the Bomco Moly Cup.

Prior to our development, graded seals or pressed seals, which were costly, somewhat fragile and unreliable, expensive, and large were used. The latter were, in single foil construction, limited in current carrying capacity due to the i^2R buildup of heat and subsequent seal failure. Multiple pressed seals were costly and plagued with problems when the leads were spot welded to the foil ends.

The Bomco Moly Cup seal allows the unlimited passage of current into a quartz envelope at a reasonable cost with a high degree of reliability. The seal sees no current which is carried through a conductor of required size. This conductor, usually tungsten, is brazed or welded to a hole supplied in the moly cup bottom.

By 1974 we concluded that many manufacturers could not, or would not, make their own seals. We therefore contracted with a glass shop to make seals utilizing our cup for us to sell. By 1976 we set up our own shop, under the direction of Richard E. Ryan. As a result of having our own shop, we

were able to satisfy our customers' requests to develop a sleeve, or double-ended tubular quartz to moly seal. Again, at customers' requests, we developed a moly to stainless steel weld to produce stainless to moly to quartz transitions in short lengths. We are currently producing $\frac{1}{2}$ " diameter transitions in less than 1" lengths.

Moly was the logical metal for sealing directly to quartz. It has a melting point of 800°C above the required temperature for sealing quartz. Although it is expensive — over \$60 per kilo — it forms readily with proper control of tooling, lubrication, and temperature providing the moly sheet has the required grain structure, has closely controlled thickness, and, in general, is of superb quality.

We blank a circle of .020" thick moly, draw a cup, and trim the open end. We next redraw to a size giving a slight interference fit on our tapered spinning mandrel. After a final trim, the spinning process displaces metal axially until $\frac{1}{4}$ " of feathered edge is achieved. The last operation trims the cup to the required length.

The removal of forming lubricants must be carefully done to ensure successful sealing. We use two baths of chlorothene.

The cups, when trimmed, always have a small "burr" present. This must be removed prior to sealing to minimize stress when the seal is completed. We etch the cups in a straight solution of Clorox for approximately two minutes at 140°F. The cups are placed in a suitable container, open end up and not touching one another. Etching serves two purposes: First, it removes the burr; and, second, it reduces the end of the feather edge to less than .0015". Other chemicals can be used and can even be given an electrolytic assist. We have found that quartz "sticks" to highly polished moly just as well as to a matte surface.

The cups must be agitated before removing from the solution to wash away all residue of oxides. They are then rinsed thoroughly in water and then alcohol and then dried.

The techniques for encapsulating the moly cup in a quartz envelope and sealing it under vacuum have been known for a number of years. There are three critical areas to be considered to ensure a successful seal.

1. Absolute cleanliness of the cup itself. It takes only a very short time for oxides to form on the cup when exposed to air, especially if the humidity is high.
2. According to our experience, vacuum should be 60 microns, or better.
3. The presence of water vapor during sealing will result in faulty seals; therefore, warming of the capsule during pumpdown is recommended.

Some people hydrogen fire the cups after etching, but it has been our experience that this is not necessary providing that the cups were thoroughly rinsed and dried. After etching, we store the cups either in a warming oven or a desiccator.

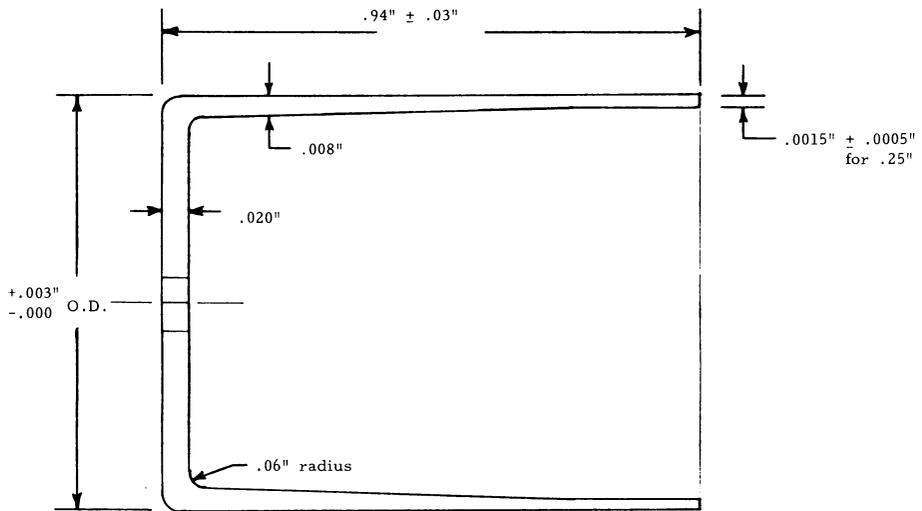
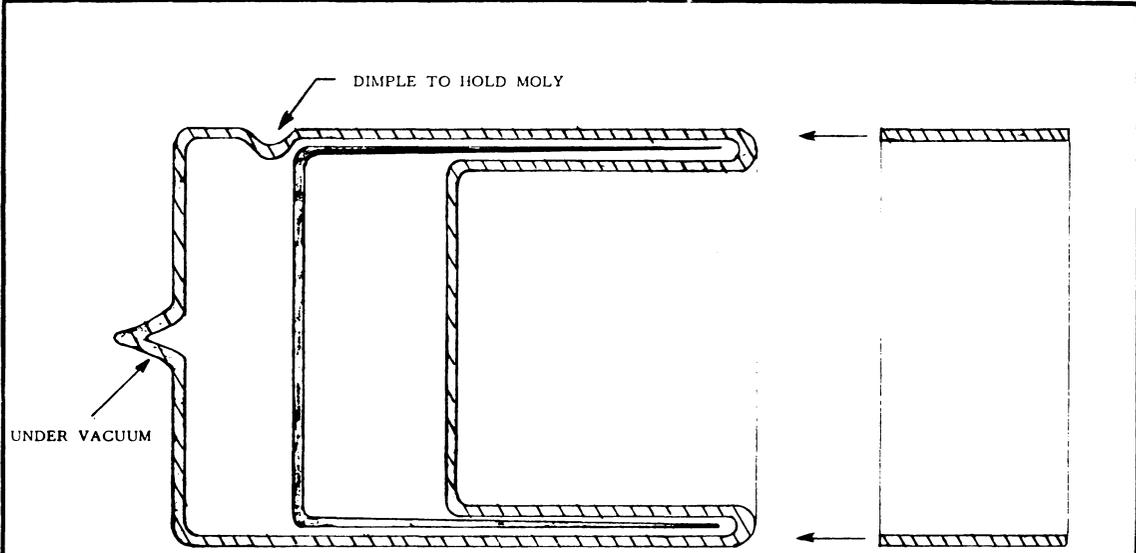


Figure 1

ALT.	DESIGN	DATE
bomco METALFORMING EXCELLENCE INC. Pt. 128, Blackburn Circle / Gloucester, Mass. 01930		
SCALE: NTS	APPROVED BY	DRAWN BY
DATE:		
Molybdenum Feather Edged Seal Element		
		DRAWING NUMBER



 QUARTZ
 MOLY

Figure 2

ALT	CHANGE	DATE
bomco METALFORMING EXCELLENCE <small>At: 178 Burlington Circle / Concord, Mass. 01930</small>		
SCALE	APPROVED BY	DRAWN BY
DATE	QUARTZ AMPULE WITH MOLY CUP	
		DRAWING NUMBER

During our pumpdown procedure we purge the warmed capsule and cup with a slight amount of hydrogen, which will remove oxides if they are present. We find that this procedure leads to more consistent sealing success.

Most of our seals are done on a vacuum fixture using "o" rings, but sealing a capsule off under vacuum is equally suitable.

The inside seal is made first, with a hand torch, making sure that the quartz adheres to the moly for at least the $\frac{1}{4}$ " of .0015" thickness. The fires are then directed to the outside, completing the Houskeeper seal. At this time an appropriate piece of tubing is added for later fusing to other apparatus.

It should be noted that the amount of sealed area on the outside of the cup is determined by the amount sealed on the inside. During the sealing process some provision must be made to prevent the moly from moving axially when the quartz reaches working temperature.

After the completed seal has cooled to below 350°C, it can be opened to air. We use a dry diamond wheel to cut away the excess quartz exposing the moly. This eliminates the need to wash away the grit when using a wet wheel.

The unsealed quartz on the inside of the cup is broken away using a metal drift pin. As violent as this sounds, we have never had a seal fracture from it. An alternate method would be to go down inside with a diamond core drill, but this requires precise control to avoid cutting the metal and is much slower.

We can supply the moly with the bottom of the cup removed for later brazing or welding to other metals. In this situation, a small piece of carbon should be machined to fit the open end of the cup and used to prevent it from moving axially during sealing and also acting as a stop for the inside piece of quartz.

The method of sealing the double tapered sleeve follows the same procedures as for a cup. Fixturing is much more elaborate because each end is sealed separately.

A properly made seal will withstand cycling from 800°C to liquid nitrogen temperatures.

Unprotected moly oxidizes rapidly at temperatures above 350°C. Bomco has developed an oxidation resistant coating which permits the moly to be exposed to 500°C temperatures indefinitely. At present the coating has to be applied to a completed seal, but we are working on a precoated cup which can be consistently sealed vacuum tight. The coating can be TIG welded or brazed. Brazing coated cups cannot be done in a hydrogen atmosphere as hydrogen degrades the coating. We suggest vacuum brazing as the alternative. All TIG welding should be done in a dry box. Argon is a satisfactory atmosphere.

Fusing of the quartz can be accomplished quite close to the seal with no ill effects provided the moly is kept below 350°C. A coating of wet asbestos on the moly has enabled us to fuse windows within $\frac{1}{2}$ " of the seal.

Research lasers use our sleeve seals as concentric electrodes operating at temperatures up to 1000°C. They are also used as concentric electrodes for flashtubes.

A seal with window, welded or brazed to a vacuum flange, makes a very short transition from metal-quartz for laser ends; typically a ½" diameter flanged sleeve with window is ¾" to 1¼" long.

As glassblowers become more familiar with tubular quartz to metal seals, the applications of the Bomco Moly Cup are limited only by your innovations and artistry!

DIFFUSION BONDING

K. D. EARLEY and GUSTAV ABEL

G.T.E. Laboratories
Waltham, Mass. 01970

In the next few minutes I would like to talk to you about a problem which often gives a headache to a glassblower. Namely, optically flat and clear surfaces!

As you know since the discovery of the laser beam, the demand for optically flat, clear windows is much higher than ever before. If you had to make such a device you very well know the difficulties of sealing a window to a tube or a flat piece to another without distorting it.

To get around the problems we tried different soldering glasses. These fritt seals were a big help but very limited; usually only soft glasses can be sealed together in this way.

Many have tried different types of epoxies. This method gives you the advantage of holding together different materials regardless of their expansion, but if there is any temperature involved they are not good either.

The last time the Symposium was held in Boston in 1965, the R. Z. 2 was introduced which was a soldering glass for quartz. It was available in thin rods and powder form. The seals were made at a low temperature, but because of a high copper content, many times the seals were not good for vacuum.

When I was at American Optical, we worked out a system to seal pieces together with a CO₂ laser beam. This was demonstrated in Albany during the Symposium. This worked well in certain cases but because of the larger expense involved, not too many shops could afford a CO₂ laser. So we arrived at diffusion bonding.

This is a method of joining glass by heating two or more optically contacted pieces at or near the annealing point of the glass and holding at this temperature long enough to cause them to become one piece.

Diffusion bonding is used generally with optical shapes. We have used the technique to make dewar flasks with optical windows, ion lasers that operate beyond the survival temperatures of O rings, gaskets, epoxies or grease, and special cells for optical measurements.

One such cell was made so the index of refraction of selenium oxychloride could be determined very accurately by the bureau of standards.

There are two conditions of paramount importance in diffusion bonding of glass surfaces: optical integrity and cleanliness. Optical integrity we will define as a surface free of scratches or sleeks with a figure approaching 1/10 of a wave. Cleanliness we will define as "acceptable" contamination by the last agent touching it.

Glass has a chemically active surface; it will combine with sulfates, nitrates, oxides, hydroxides and amines. Quartz is silicon dioxide and as

such has a surface rich in oxygen bonds ready to combine chemically with any molecule that wants to share a valence bond.

Quartz is acidic by nature and is stable in all acids except hydrofluoric. It is mildly attacked by alkalis and carbonates, but these generally at elevated temperatures.

The first step in cleaning glass is removal of gross contaminants with hot solvents. After solvent cleaning, the work should be put into concentrated sulfuric acid laced with about 5% nitric acid. These acids should be used hot, but if used cold, a protracted soaking is necessary — 10 to 12 hours at least. Demineralized water is used for rinsing work. Care must be taken in handling at this stage. Plastic tongs, rubber finger cots and such things will leave their own contamination on the glass. Platinum tipped holders if used carefully will not mark the glass.

Lens paper wetted withalconox or other suitable glass cleaner of the detergent type, should be used to lightly scrub the work for removal of acid digested debris. This debris, because of inertia, has to be physically removed by a scrubbing action. All cleaning agents, soaps, detergents and wetting agents remain on glass as a chemically combined film; it cannot be removed by rinsing, soaking or even boiling in water. It will dry to present a “black figure”, a term used to show ostensible cleanliness; water will form a continuous film, will not condense in tiny droplets as in the presence of dirt, therefore, shows no fogging when blown upon.

Rinsing with dilute acid will “crack” this film, a phenomenon made evident by violent surface turbulence if a single drop of dilute acid is applied. Acetic acid is preferred for this operation; it is an organic acid that is free of residue. It thus becomes the final “acceptable” contaminant left in chemical combination on the surface. The work is rinsed and blown dry with filtered air to prevent spotting.

Contacting should be done immediately. It can be done in two ways: parts can be placed together with lens tissues between and the tissue carefully removed while kept in alignment; or they can be contacted directly by placing one over the other and pressing out.

One thing must be kept in mind: when work is clean enough for diffusion bonding, the instant it is touched together it seizes and is very difficult to take apart. Pieces can be damaged by physical removal; attraction between pieces is so strong local adhesion takes place and ripping of surfaces occurs on separation. Thermal shock induced by rapid heating or cooling causes separation. Immersion in water can also be used.

The bonding operation is carried out preferably in a vacuum furnace. A vacuum prevents bonding growth from the periphery of the work inward sometimes resulting in unbonded patches seen as dull spots by the unaided eye.

Diffusion bonding is a time-temperature function and is, of course, exponential. Bonding has been effected at 1000°C for six hours with quartz glass; the same results have been accomplished in thirty minutes at 1150°C. It is obvious that the lower temperature, longer time period will result in better optical integrity of surfaces.

Glasses other than quartz have not been successfully bonded by us at temperatures under the annealing point. However, the best time-temperature relationship for a particular glass is best determined by trial and error on simple test pieces. These can be heavy walled cylinders of glass, one inch diameter, contacted to slightly larger windows. Rise time to bonding temperature should also be determined with these test pieces; too fast a rise time can act as a thermal shock causing separation.

I have a cell here with five surfaces which was made by the diffusion bonding technique. The first step, the triangle shaped bottom piece and one side, rectangular in shape, was contacted and sealed. We left the side piece longer, so after the first seal was made it was ground down to the right size and polished. Now the second side was contacted and sealed; the third in the same manner. Finally the top piece, which is a larger triangle shape than the bottom piece, was sealed to complete the cell. This particular cell went through four heating cycles without any ill effects to the material and stayed clear and optically flat.

The equipment used in diffusion bonding can vary from hand operated variacs and a potentiometer for temperature information, to a sophisticated SCR power source with cam operated controller. The most versatile set up for most laboratory needs would be a large tube furnace with cam controlled SCR; this can be operated as an atmosphere furnace or as a vacuum furnace. Just remember that vacuum needs are not critical. The cam will allow large leeway in up-time and hold time. We have never required more than six hours to reach 1000°C. This is less than 2° per minute and should accommodate very heavy parts indeed.

In closing it might be said that the furnace that has the most to offer is the atmosphere-vacuum furnace. We have found that taking quartz pieces to 500-550°C in air and then evacuating the tube gave us the cleanest work we have yet seen.

GLASS FLUORESCENCE CELL CONSTRUCTION FOR USE IN HIGH VACUUM LASER BEAM EXPERIMENTS

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In experiments utilizing infrared laser induced fluorescence, it is extremely important to place the detector as close to the excited molecular volume as possible. This constraint is due to both the weak intensity of the fluorescent radiation in the infrared region and to the possible reabsorption of the emitted fluorescence in the unexcited volume between the beam path and the observation window. Traditionally metal cells have been machined with critical tolerances to accomplish this goal; however, due to the corrosive nature of many gases, metal cells can present a chemical cleanliness problem in many experiments.

It was therefore decided to attempt to build a glass cell. This would solve both the critical tolerance problem with respect to window positioning and the chemical reactivity problem. A square glass tube measuring 40 mm x 40 mm was used for the cell body. "O-ring" joints were attached at

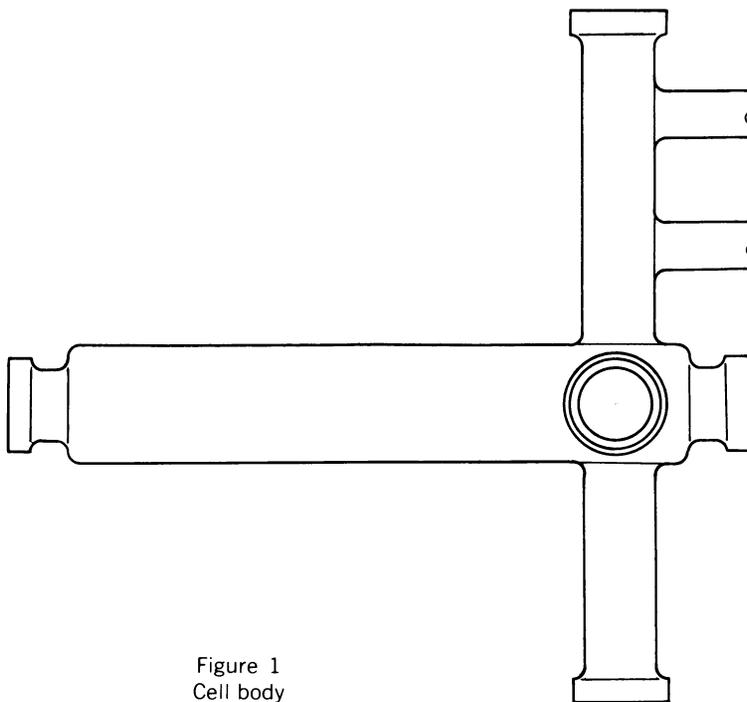


Figure 1
Cell body

either end to allow the laser radiation to enter and exit through windows positioned at opposite ends of the cell. The observation window was placed at right angles to the laser axis at a distance of 25 mm from one end. Two high vacuum stopcocks with ground glass joints were placed at the top of

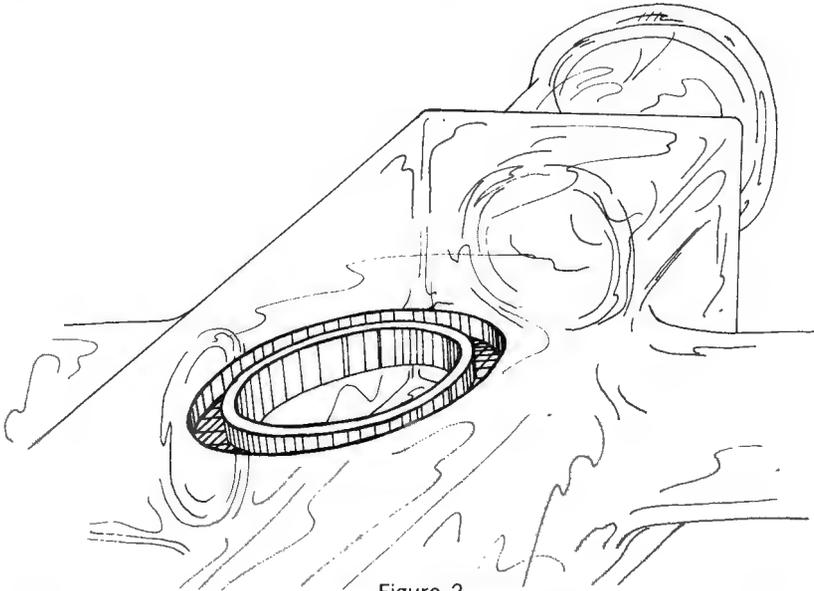


Figure 2

"O-ring" groove construction of observation window on square glass body of cell.

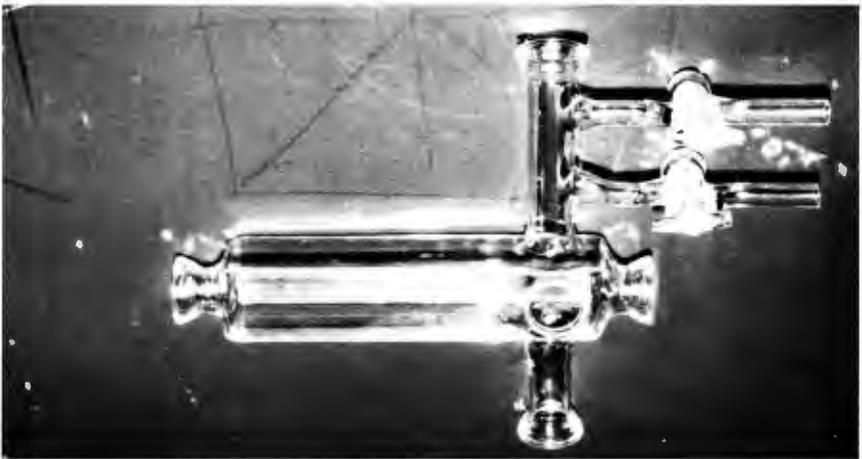


Figure 3

Completed cell

the cell to allow for pump out and pressure monitoring connections to the vacuum system. To make the window removable, and therefore interchangeable, "O-ring" construction was employed. In addition, since experimental conditions are typically at high vacuum (10^{-6} torr), no supporting flange was necessary at either the observation window or at the entrance and exit ports for the laser. A 32 mm diameter diamond core drill was used to cut the "O-ring" groove at the desired location to a depth of between $\frac{1}{2}$ to 1 mm. Subsequently, a second drill was used to drill a window of 25 mm at



Figure 4
Cell mounted in instrument

the center of the groove area. This sequence is preferable to drilling and cutting the groove later because centering becomes much easier. This type of construction has been utilized for a large number of cells and has been found very satisfactory for the following reasons:

- 1) Vacuum tight operation
- 2) Chemical inertness
- 3) Interchangeability of all windows
- 4) Utilizes existing square glass tubing
- 5) Cell window can be placed as close to the detector as existing metal cells without the relatively high cost
- 6) Glass cells are easier to clean and are not as reactive as metal cells

SCIENTIFIC GLASSBLOWING — TRAINING AND EDUCATION IN THE UNITED STATES

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PREFACE

The Technological Revolution of our age is established upon years of scientific research and development. Painsstaking laboratory experimentation is the necessary prerequisite for every small step in the process of discovery.

Because of the unique properties of glass, laboratory apparatus is constructed of this material. The scientific glass blower/technologist is the one who designs and creates the laboratory glass essential to the work of experimentation and consequently is a vital member of the research team. As we continue to expand our scientific efforts to understand man and to place him in harmony with his environment, the role of the scientific glassblower in the future will grow to even greater stature.

OUTLINE:

1. TRAINING PROGRAMS IN US (1900 - 1958)
2. SHORTCOMINGS
3. NEED FOR IMPROVED TRAINING METHODS AND FORMAL EDUCATION
4. DISCUSSION OF IMPROVED TRAINING METHODS AND FORMAL EDUCATION CURRICULUMS:
 - A. 2 YEAR TECHNICAL INSTITUTE
 - B. 2 YEAR COLLEGE
 - C. 4 YEAR COLLEGE
5. INITIAL PROBLEMS OF GRADUATES
6. ADVANTAGES OF THE IMPROVED TRAINING AND EDUCATION
7. REACTIONS OF A GRADUATE

Scientific glassblowing historically appeared in the United States prior to 1900 with the immigration of guild trained apparatus makers from Europe who served in four-year apprenticeship training programs of German origin. Science and technology at that time was very basic compared to the 1960's and 70's. Therefore, these early apparatus makers (lampworkers) were trained in manipulative skills and application tech-

DISCLAIMER — *This presentation represents the opinion of the author and does not reflect United States Department of Army Policy.*

niques. It was a logical and natural progression for scientific glassblowing training in the United States after 1900 to be based on the European guild systems. This early training was designed primarily for developing the traditional manipulative skills and providing application techniques that are so necessary for developing lampworking skills.

The generation of scientific glassblowers prior to 1950 had been taught by guild-trained peers who also served in similar apprenticeships, i.e., four years of training in fundamentals — later adding to skill and knowledge, each according to his ability and opportunity. What was taught as fundamentals was left to the discretion of the instructor. Those apprentices who were fortunate enough to have specialists available were taught by several teachers, that is, if the instructors were willing. Remember that under this type of guild system the secret knowledge and ability of each member was jealously guarded. This was, as a manner of speaking, instruction through a natural method of selection since the character of the apprentice was also being appraised.

It should be noted that the apprenticeship training programs for scientific glassblowing during this period (1900-1958) were as varied as the number of scientific glass producing companies, and research and development facilities in the United States. In fact, one very large corporation had an apprenticeship program that varied from one facility to another.

With such diversity in training programs, it would be difficult to estimate the cost of training 15 or 20 one-time competitors with the hope of retaining two or three with sufficient potential and desire to finish. It would be equally difficult to challenge the wisdom of not providing complete training for the apprentice by any one company.

Nevertheless, several privately owned scientific glass producing companies, along with several research and development facilities throughout the United States did successfully develop adequate apprenticeship training programs. I have selected the apprenticeship program developed in the early 1940's by Ace Glass, Inc. (manufacturer of scientific glassware), Vineland, New Jersey, as the "model" United States apprenticeship training program for scientific glassblowers prior to 1958. Table I lists the manipulative operations, hours devoted to each operation, and a product sample for each operation.

For example: Operation number one is fire polishing; pulling points, round bottoming and bending with 200 hours being devoted to making burette tips, pipettes, centrifuge tubes and manometers.

While training under this program, the apprentice lampworker serves 3.5 years or a total of 7000 hours, whichever comes first. It should be noted that the 7000 hours represents the average trainee's needs. A more gifted person might accomplish the same results in 4000 hours, which Ace Glass regarded as the minimum acceptable training time.

Due to the shortage of scientific glassblowers following World War II, the United States government subsidized the training of scientific glassblowers through the Veterans Administration. This Administration in turn, required that the course be approved by the State Department of Education. The New Jersey State Department of Education approved the Ace

TABLE I

MODEL APPRENTICESHIP PROGRAM OF INSTRUCTION*:

A. MANIPULATIVE TRAINING:

<u>OPERATION</u>	<u>HOURS</u>	<u>PRODUCT EXAMPLE</u>
1. FIRE POLISHING: PULLING POINTS; ROUND BOTTOMING AND BENDING	200	BURRETE TIPS, PIPETTES, CENTRIFUGE, TUBES, MANOMETERS
2. SOLID SEALS; SHAPING SOLIDS	300	STIRRERS
3. "STICK" SEALS	1000	STOPCOCKS, BURETTES
4. SIMPLE SMALL SEALS	1000	SIMPLE ADAPTORS, GAS MANIFOLDS
5. LARGE BLOW SEALS	1500	SMALL NECK FLASKS, GAS SAMPLING TUBES
6. LARGE TO SMALL SEALS } 7. SIMPLE RING SEALS }	100	TRAPS
8. HARDER RING SEALS	1000	CONDENSERS - ALL TYPES
9. USE OF PICK - (ADVANCED)	300	VIGREAUX COLUMNS, OTHER SIMPLE COLUMNS
10. LARGE RING SEALS (MORE DETAIL)	300	DISTILLING HEADS, SIMPLE TYPES
11. VOLUMETRIC WORK (INCREASED DETAIL)	300	RECEIVERS - DISTILLING AND MOISTURE TEST
12. ADVANCED WORK, AND/OR CONCENTRATION ON WEAKNESSES	<u>1000</u>	MISC. ITEMS - SPECIALS
<u>TOTAL</u>	7000	(AVERAGE APPRENTICE HOURS)

B. SELECTED LESSIONS:

1. PHYSICS
2. CHEMISTRY
3. MATHEMATICS
4. BLUE PRINT READING

* DEVELOPED AT ACE GLASS INC., VINELAND, NEW JERSEY, USA BY MR. CLYDE I. KRAMME

Glass apprenticeship manipulative training but it specified the addition of selected lessons in Physics, Chemistry, Mathematics, and Blueprint Reading in order to meet the program approval criteria. Ace Glass revised its apprenticeship program syllabus to include the required academic subjects and received New Jersey State Department of Education approval. Classes were taught three nights per week by Mr. Clyde I. Kramme, an Ace Glass employee who held a Masters Degree in Education.

In spite of the efforts of government agencies and private corporations to improve training for scientific glassblowers, the apprenticeship program's shortcomings became apparent in the mid-1950's.

APPRENTICESHIP TRAINING'S SHORTCOMINGS:

1. DIFFICULTY IN RECRUITING
2. DIFFICULTY IN RETAINING INTEREST
3. SCIENTIFIC GLASS INDUSTRY WAS TOO PRODUCTION ORIENTED
4. SENIORITY SYSTEM
5. LACKED STANDARDIZATION
6. DID NOT PROMOTE PROFESSIONAL STATUS

The scientific glass industry including the research and development facilities were becoming aware of the need to replace their aging scientific glassblowers. At the same time, it was becoming increasingly difficult to recruit high school graduates into apprenticeship type training due to increasing societal and parental pressures toward attending college. It was also becoming difficult to retain the interest of those who had been participating in existing apprenticeship training because labor unions were negotiating higher wages which were becoming competitive and potentially higher than glassblowers' wages. In addition, the 3.5 to 5 year glassblowing apprenticeship appeared to be a long, drawn-out process that provided no guarantee one would be successful. It was also too demanding. It required a tenacious determination and persistence to develop the necessary dexterity and skill which too few individuals possess.

The scientific glass industry was too production-oriented and customer demands were continually increasing. Many companies were working overtime, making it more difficult to fulfill the apprenticeship training obligation. Due to the high cost of training, there were attempts at teaching a beginner one or two basic glassblowing operations, then placing him on the production line where he may never be taught more — would become bored, then resign.

The seniority system tended to slow the learning process due to delayed opportunities for a young apprentice to experience challenging and difficult projects. On many occasions, unfortunately, the apprentice was not provided an opportunity to learn while in his prime.

Due to the fact that each scientific glass company, research and development facility and university independently designed its own apprenticeship training program for teaching scientific glassblowing, an obvious lack of standardization existed. This made it difficult for glassblowers and especially the apprentice to meet all-employer requirements, such as, quality, accuracy, interpretation of blueprints and sketches, time, acceptable tolerances, etc.

Also, apprenticeship training in the United States has historically failed to promote professional status for American scientific glassblowers that their European counterparts have enjoyed for decades.

NEED FOR IMPROVED TRAINING WAS INFLUENCED BY:

1. INADEQUACY OF HIGH SCHOOL EDUCATION
2. SPACE AGE DEMANDS
3. INCREASED COLLEGE ENROLLMENTS
4. ECOLOGY
5. ENERGY CRISIS

The need for improving training of scientific glassblowers was further influenced by the inadequacy of a high school education to provide psychological and economic security according to the existing social norms which require offspring to obtain fulfillment of the American Dream by graduating from a college or university.

Space exploration of the late 1950's and early 1960's presented new challenges for the research scientists, and these scientists in turn, placed greater demands on the scientific glassblower's creative ability and his knowledge of the fundamental, experimental and theoretical applications of glass technology. Space exploration created expansion of industrial research and development facilities. Paralleling this industrial expansion was the increase of college and university enrollments in Bachelor of Science Degree programs and advanced Science and Engineering degree programs, such as Masters and Doctorates.

THE BIERMANN THEORY:

THE DEMAND FOR TRAINING OF SCIENTIFIC
GLASSBLOWERS HISTORICALLY FOLLOWS A
TWENTY YEAR CYCLE.

This industrial expansion created an unanticipated need for additional technically trained scientific glassblowers. Historically, demands for such training occur in twenty-year cycles, that is, a glassblowing laboratory or shop employs 3 scientific glassblowers whose ages range from 40 to 50 years. Within 5 years the employer anticipates 2 may retire. A training program for teaching scientific glassblowing must be developed then implemented by recruiting possibly as many as 10 to 15 young persons over a period of several years in order to get 1 or 2 apprentices, who after 5 years or sooner, will be trained journeymen — then the employer's training program is terminated and hopefully will not be needed for another 20 years — when the cycle will probably repeat assuming no one resigns.

NEED FOR IMPROVED TRAINING WAS INFLUENCED BY:

1. INADEQUACY OF HIGH SCHOOL EDUCATION
2. SPACE AGE DEMANDS
3. INCREASED COLLEGE ENROLLMENTS
4. ECOLOGY
5. ENERGY CRISIS

Additional influencing factors are ecology and the energy crisis. Ecological research has established federal and state environmental quality requirements for industrial wastes and pollutants. The energy crisis has inspired scientists to search for new sources of energy and develop energy conservation methods.

The culmination of these influences in 1957 caused the New Jersey State educators, The Scientific Glass Industry and the community of Salem County (New Jersey) to realize the need for establishing career education in the technical field. This inspiration led to the foundation of the Salem County Technical Institute in 1958, Salem, New Jersey. Under the leadership of its Director, Mr. Herbert C. Donaghay, the institute quickly established a reputation for excellence in career education, particularly with its technology programs which included scientific glassblowing. Mr. Joseph Luisi from Ace Glass Inc., was the Institute's first scientific glassblowing instructor.



In 1960 the scientific glassblowing course was upgraded from a one-year curriculum to a two-year curriculum in scientific glass technology and in 1961 the Institute relocated from Salem, New Jersey to its present location in Penns Grove, New Jersey. This two year curriculum was the first of its caliber in the United States. Using the Ace Glass model apprenticeship program as a basis, and expanding the academic material by subject, the program was upgraded to a Junior College level. An advisory committee was created which included representatives from six scientific glass manufacturing companies and three state and local educational agencies who collectively developed the requirements for the scientific glass technology curriculum. This committee was given official status in the state of New Jersey, and its recommendations continue to become part of the scientific glass technology curriculum.

Table II contains the original two-year curriculum (diploma awarded):

SALEM COUNTY TECHNICAL INSTITUTE – 2 YEAR CURRICULUM

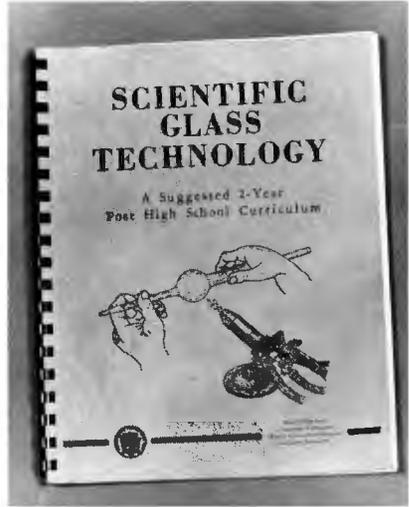
<u>FIRST YEAR (18 WEEKS)</u>	<u>HOURS PER WEEK</u>
SCIENTIFIC GLASSBLOWING	15
INDUSTRIAL CHEMISTRY	4
MATHEMATICS	4
BLUEPRINT READING AND DRAWING	3
TECHNICAL ENGLISH	4
 <u>SECOND YEAR (18 WEEKS)</u>	
SCIENTIFIC GLASSBLOWING	15
PHYSICS	6
MATHEMATICS	4
TECHNICAL ENGLISH AND REPORT WRITING	2
MACHINE SHOP	3
TOTAL CREDIT HOURS	60

DIPLOMA AWARDED

Note that the total hours devoted to scientific glassblowing was 1080 — 4 semesters x 18 wks x 15 hrs/wk compared to 7000 hours in the Ace Glass apprentice program. In addition, Salem County Technical Institute students were referred to local scientific glass manufacturing and research and development facilities during the summer months (June through September) following the first year, for employment as apprentice glassblowers. This employment opportunity added an additional 480 hours (12 wks x 40 hrs/wk) for a total of 1560 hours which may be applied against the 7000 hours required for apprenticeships in corporations that are committed to the labor union type guild system.

The primary textbooks used for the scientific glassblowing technology curriculum are:

Scientific Glassblowing, by Wheeler, and *Scientific and Industrial Glassblowing and Laboratory Techniques*, by Barr and Anhorn.



The scientific glassblowing student manuals (workbooks), one for each year, were compiled by E. Henderson, a consultant to the institute in collaboration with Mr. Harry Hunter, the institute's scientific glassblowing instructor. Both student manuals are based on the project method allowing each student to advance at his own rate. Each student's ability and knowledge is ascertained at the outset so that specific progress can be realized in definite steps and individually plotted. It should be noted that these workbooks or manuals include basic operations, providing the instructor flexibility to adjust the syllabus at his discretion; for example, he may desire to include more advanced projects to challenge the more gifted student.

In addition, a complete two-year scientific glass technology post high school curriculum has been compiled and written by the faculty of Salem County Technical Institute. This guide offers invaluable information to private industry, to colleges and universities who desire to establish a program for the training of scientific glassblowers. I must emphasize that this is a guide only, allowing for tailoring of subject matter to fit any group or individual. Only basic material is included — advanced material can be added to the syllabus as required.

As part of the institute's responsibility for providing continuing education which is equal in importance to full-time education — evening scientific glassblowing courses are offered: one of elementary level designed for chemists, laboratory technicians, science teachers, etc., and a second of advanced level designed to help the glassblower who finds himself bored with routine glass work and desires to improve his lot, and a third course designed to teach ornamental glassblowing to artistically creative persons of any glassblowing skill level.

WILMINGTON COLLEGE, NEW CASTLE, DEL, USA



In January 1971, Salem County Technical Institute became affiliated with Wilmington College, a four-year private college located in New Castle, Delaware. This affiliation enabled institute graduates to acquire a two-year Associate Degree in Applied Science by completing 15 credits from the Wilmington College Extension courses. These courses can also be applied toward a four-year Bachelor of Science Degree in Scientific Glass Technology.

Table III contains the Wilmington College four-year curriculum which reflects the third and fourth years' course requirements:

- Transfer of 60 credits from Salem County Technical Institute
- A "Core" of 9, 3 credit courses
- 6 liberal arts electives
- 3 business courses and
- 2 general electives

Recognizing the college level caliber of Salem County Technical Institute's programs, the Salem County Board of chosen free holders (an elected governing body) requested the New Jersey Board of Higher Education's approval to grant two-year degree awarding authority to the institute. In May 1972, approval was granted, and on September 3, 1972, the Salem County Technical Institute became Salem Community College. The scientific Glass technology curriculum was revised to include the liberal arts electives required for awarding an Associate Degree in Applied Science. This curriculum is illustrated in Table IV.

TABLE III

WILMINGTON COLLEGE – 4 YEAR CURRICULUM:

DIPLOMA COURSES ACCEPTED AS TRANSFER CREDITS	60
WILMINGTON COLLEGE COURSES	
CORE COURSES FOR TECHNICAL STUDENTS ¹	27
LIBERAL ARTS ELECTIVES	18
BUSINESS	
ECONOMICS II	3
STATISTICS I AND II	6
ELECTIVES	<u>6</u>
	15
<u>TOTAL CREDIT HOURS</u>	<u>120</u>

CORE COURSES FOR TECHNICAL STUDENTS

SOCIOLOGY	3
INTRO. TO PSYCHOLOGY	3
SURVEY OF MODERN EUROPE	3
PEOPLES & CULTURES OF ASIA	3
U.S. HISTORY I OR II	3
INTRO. TO POLITICAL SCIENCE	3
SPEECH	3
BEHAVIORAL SCIENCE ELECTIVE	3
HUMANITIES ELECTIVE ²	<u>3</u>
	27

¹CORE COURSES FOR TECHNICAL STUDENTS: EACH TECHNICAL STUDENT IS REQUIRED TO COMPLETE THE "CORE" OF 27 CREDIT HOURS.

²HUMANITIES ELECTIVE: STUDENT MAY TAKE HUMANITIES COURSE IN ART APPRECIATION, MUSIC APPRECIATION, LANGUAGE, PHILOSOPHY, LITERATURE, ETC.

BACHELOR OF SCIENCE DEGREE IN APPLIED/PHYSICAL SCIENCE IS AWARDED

Note that the total hours devoted to scientific glassblowing is 456 (semesters x 19 wks x 6 hrs/wk) compared to the 1080 hr technical institute curriculum and compared to the 7000 hr Ace Glass Apprentice program.

Students are given examinations in scientific glassblowing at selected levels of accomplishment in oral, practical or written form by the Scientific Glass Technology instructor. Examinations in the related technical subjects are given by the instructor of each specific subject.

In reference to certification of college examinations, it should be noted that colleges and universities throughout the United States apply for accreditation to the Middle States Association of Colleges and Secondary

TABLE IV

SALEM COMMUNITY COLLEGE – 2 YEAR CURRICULUM:

<u>FIRST YEAR</u>				
<u>FALL SEMESTER</u>		<u>CLASS</u>	<u>LAB</u>	<u>CREDITS</u>
MAT	101 COLLEGE MATHEMATICS I	3	0	3
ELT	101 INTRODUCTION TO ELECTRONICS	2	2	3
DRT	101 ENGINEERING DRAWING I	0	3	1
ENG	101 ENGLISH I	3	0	3
SGT	111 SCIENTIFIC GLASSBLOWING I	3	6	5
PE	101 PHYSICAL EDUCATION	<u>0</u>	<u>2</u>	<u>1</u>
		11	13	16
<u>SPRING SEMESTER</u>				
MAT	102 COLLEGE MATHEMATICS II	3	0	3
ELT	102 INDUSTRIAL INSTRUMENTATION	2	2	3
DRT	102 ENGINEERING DRAWING II	2	3	3
ENG	102 ENGLISH II	3	0	3
SGT	112 SCIENTIFIC GLASSBLOWING II	3	6	5
PE	102 PHYSICAL EDUCATION	<u>0</u>	<u>2</u>	<u>1</u>
		13	13	18
<u>SECOND YEAR</u>				
<u>FALL SEMESTER</u>				
CHM	101 COLLEGE CHEMISTRY I	3	3	4
PHY	101 INTRODUCTION TO PHYSICAL SCIENCES	3	3	4
SGT	211 SCIENTIFIC GLASSBLOWING III	3	6	5
	SOCIAL SCIENCE ELECTIVE	<u>3</u>	<u>0</u>	<u>3</u>
		12	12	16
<u>SPRING SEMESTER</u>				
CHM	106 GLASS CHEMISTRY	3	3	4
SGT	212 SCIENTIFIC GLASSBLOWING IV	3	6	5
BIO	101 INTRODUCTION TO BIOLOGICAL SCIENCES	3	3	4
SGT	213 GLASS FABRICATION & DESIGN SEMINAR	0	3	1
	HUMANITIES ELECTIVE	<u>3</u>	<u>0</u>	<u>3</u>
		12	15	17
<u>TOTAL CREDIT HOURS</u>				67

ASSOCIATE DEGREE IN APPLIED SCIENCE IS AWARDED

Schools. This independent organization established in 1887 strives to improve the quality of education and to strengthen the relations among secondary schools and institutions of higher learning. Once fully accredited, the college gains academic status and prestige and its students have better transfer ability to other colleges due to acceptance of quality credits and final grades.

INITIAL PROBLEMS OF GRADUATES:

1. NOT EASILY ACCEPTED BY PEERS
2. PEERS FEARED LOSING JOBS TO GRADUATES
3. PEERS BELIEVED THAT THE FORMAL INSTITUTIONS WOULD SATURATE THE MARKET WITH INFERIOR GLASSBLOWERS RESULTING IN LOWERING OF WAGES

I shall now candidly discuss several problems encountered by graduates: At first, they were not easily accepted by their peers who subconsciously feared losing their jobs to the younger generation. The older scientific glassblowers were further threatened by the misbelief that the formal college level training would saturate the market with inferior scientific glassblowers (due to less hours of manipulative training compared to earlier apprentice training methods) and result in lowering of wages.

Obviously there was little or no consideration allowed for the improved efficiency and improved effectiveness of the more formal training environment. Despite these problems, which have historical origins, (these problems are not new nor are they peculiar to graduates of formal training — they have been experienced for generations) there are definite advantages resulting from the improved training and formal education in scientific glass technology in the United States. They are:

ADVANTAGES OF IMPROVED TRAINING AND EDUCATION

1. CAN BE TAUGHT ADVANCED TECHNIQUES EARLIER
2. POSSESS TECHNICAL BACKGROUND
3. REFERRED TO EMPLOYER BY INSTITUTE/COLLEGE
4. EMPLOYER CAN HIRE AT LOW RISK
5. BASIS FOR PROFESSIONAL STATUS
6. STANDARDIZED TRAINING
7. IMPROVED JOB OPPORTUNITIES
8. FORMAL TRAINING

1. Graduates can be taught advanced techniques earlier and can learn faster with the basics mastered, provided they are given the opportunity.

2. Graduates possess a technical background in the applied and physical sciences that previously took their peers many years of experience to acquire — assuming there was a requirement for and also an opportunity to acquire such background.

3. Graduates are referred by the educational institution to the potential employer thus increasing the employer's ability to hire an apprentice with potential for improvement at a low risk.

4. Graduates are provided the basis for professional status since the American Society recognizes the college graduates as professionals, i.e., scientists, engineers, teachers etc.

5. Teaching of basic fundamentals is standardized so that the employer knows that the graduate's training up to that point is satisfactory.

6. The graduate has the proper background for advanced training, which is not only accepted but endorsed by large glass producing corporations and research and development facilities, thereby widening the graduate's job opportunities.

7. Opportunity exists for those who feel the need for more professional training and formal education, and who might otherwise not have the opportunity.

SPECIAL TECHNICAL ABILITIES:

1. APPLY SCIENTIFIC METHOD.
2. FACILITY WITH MATHEMATICS.
3. UNDERSTANDING OF MATERIALS, PROCESSES, EQUIPMENT AND TECHNIQUES.
4. KNOWLEDGEABLE OF PHYSICAL, CHEMICAL AND BIOLOGICAL SCIENCES.
5. EXERCISE INDIVIDUAL JUDGEMENT AND INITIATIVE.
6. COMMUNICATION SKILL.

I must emphasize at this point that each graduate should be thoroughly proficient with the following special technical abilities:

1. Proficiency in using the scientific method, to apply the basic principles, concepts and laws of physics, chemistry, and biological sciences, to scientific glass technology.

2. Facility with mathematics to use algebra and trigonometry as tools to develop, define, or to quantify scientific phenomena or principles: an understanding of, though not necessarily with, higher mathematics through analytical geometry, calculus and differential equations according to scientific glass technology requirements.

3. Thoroughly understand materials, processes, equipment, and techniques commonly used in scientific glass technology.

4. Extensively knowledgeable of a field of specialization, with an understanding of the underlying physical or biological sciences and their appli-

cation to the engineering, health, agricultural or industrial processing or research activities that distinguish the technology of the field. The degree of competency and the depth of understanding should be sufficient to enable the individual to establish rapport with the scientists, engineers and managers with whom he works and to enable him to perform a variety of detailed scientific or technical work using general procedures or instructions but requiring individual judgement, initiative, and resourcefulness in the use of learned techniques, handbook information and recorded scientific data.

5. Proficiently utilize communication skills i.e., to record, analyze, interpret and transmit facts and ideas orally, graphically, and in writing with complete objectivity.

In addition to the teaching/training objectives — an important and scarce ingredient that is required to objectively evaluate a program is feedback. I will attempt to provide some specifics with the following personal reaction as a graduate:

REACTION OF A GRADUATE:

1. TRAINING AND EDUCATION IN
RETROSPECT AS A STUDENT
2. TRAINING AND EDUCATION IN
RETROSPECT AS AN INSTRUCTOR
3. A PHILOSOPHY

As a scientific glassblowing student and apprentice, I continually asked questions: Why?, How?, I wonder if . . .?, always trying to reduce technique into basic components upon which to build. Fortunately, I had several instructors: Mr. Harold Biggs, Mr. Harry Hunter, and Mr. Frank DeCesare who realized that an answer or additional explanation was important — were patient — and took the time to respond. They set aside the traditional guild and seniority system attitudes, and created unlimited opportunities and made unlimited resources available for the development of a student's artistic and creative manipulative skill. While in his prime the student was allowed to advance at a rate directly proportional to his ability. As a result, the knowledge and experience gained during the past 16 years has enabled me to continually improve and refine my creative abilities like a Jonathan Livingston Glassblower.

As an instructor of scientific and ornamental glassblowing. I realized that as confidence is gained, one becomes more positive in his attitude — in his ability — and in his actions! Far too many people take the negative approach — It can't be done! There is no use in trying that! It's been tried before! Sure it's safer than making a mistake, but if you haven't made any mistakes, you haven't done anything!

Fortunately, fate provided me the opportunity of being a student during the implementation of the improved training and education in scientific glassblowing technology in the United States. I lived the transition, experienced the problems, felt the pressures give way to change, and can acknowledge the benefits.

In conclusion I must state that the college level training and education *curriculum must be flexible; designed to combine the best features of the guild method and the academic method*, providing well rounded basic training to small groups where group training will be advantageous, and individual training where specialized and difficult manipulative skills will be taught.

Finally, I would like to challenge you — The 22nd ASGS Symposium attendees, representatives of the scientific glassblowing companies, research and development facilities and universities/colleges throughout the 50 states with the responsibility to assure that scientific glassblowing curriculums and syllabuses are developed and continually updated to include the most efficient and the most effective training and teaching methods available. Each of us has something to offer. *Let's get on with it!*

DISCLAIMER — This presentation represents the opinion of the author and does not reflect United States Department of Army Policy.

Textbooks, students manuals, college catalogs, graphics and the complete two-year curriculum are available for additional information immediately following my presentation.

ACKNOWLEDGEMENTS

I would like to express my appreciation to several agencies, groups and persons for their contribution toward developing the curriculum and establishing the first institution (Salem County Technical Institute) in the United States which offers Scientific Glassblowing Technology as a two-year post high school program, and 12 years later developing cooperative arrangements with another college (Wilmington College) to offer the first four-year Bachelor of Science Degree in Technology:

- State of New Jersey Department of Education
- Salem County Board of Vocational Education
- Salem County Technical Institute, Administration and Faculty
- Salem Community College, Administration and Faculty
- State of Delaware, Department of Education
- Wilmington College, Administration and Faculty
- Scientific Glassblowing Advisory Committee

Clyde I. Kramme, Chairman, Ace Glass Inc., Vineland, New Jersey

John J. Gottko, Corning Glass Works, Corning, New York

Samuel Knisely, Mobile oil Co., Paulsboro, New Jersey

Nontas Kontas, Kontas Glass Co., Inc., Vineland, New Jersey

Daniel Pedroni, Lab Glass, Vineland, New Jersey

Edward Wheaton, Wheaton Glass Co., Millville, New Jersey

I would like to express special appreciation to the following individuals for their personal guidance and assistance and to whom I dedicate this presentation and publication:

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Herbert Donahay, Salem Community College, President
Champion Cole, Salem Community College, Evening Division Director
Jules Kahn, Wilmington College, Assistant Academic Dean and Evening Division Director
Nicholas Mazarrila, Ph.D., Wilmington College, Vice President of Academic Affairs
Charles DeWoody, Ace Glass Inc., Vineland, New Jersey

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NEW SOLAR ENERGY APPLICATIONS FOR GLASS

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There is one source of energy that does not need transportation to the user, no pipelines, no overhead wires, no train cars — no pollution and no depletion. That energy source is the sun. It delivers diffused energy of about 100 watts per square foot or 1KW per m². Because the sun's energy arrives at the location where it's needed, home, office, school or factory, the overall efficiency of systems used to convert the sun's radiated energy to usable forms should be compared with efficiency of production and transportation of usable energy from other non renewable sources. Cost effective use of solar energy as a direct replacement for other energy sources will require development of a very complex technology. Once solar energy equipment is installed it will have to operate with little attention over long periods of time. One type of solar energy collector is used to directly trap solar energy by means of an absorbing black surface. The tubular collectors of this type have been developed by Owens-Illinois, Corning, G.E. and Philips. Their construction is based on a glassblower's skill. But the most common form of the solar collector is a flat plate.

Figure 1 — The standard flat plate collector consists of a blackened metal heat exchanger preferably made of copper. There are one or two glass covers to insulate the absorber to prevent heat loss to the outside. Foam or fiberglass is used to insulate the absorber around the edges and the back.

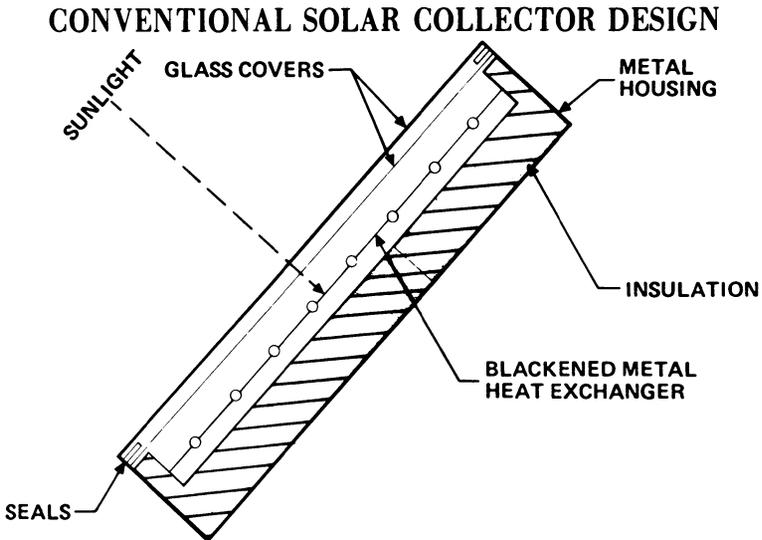


Figure 1

This design is simple and effective, but too expensive to be a cost effective replacement for other energy sources. The Boeing design (*Figure 2*) consists of glass panel structure containing vacuum cells across the width of the collector and liquid passages. The vacuum cells are sealed individually in the manufacturing process at the fusing temperature of glass. The vacuum pressure is about a quarter of atmosphere at ambient temperatures. The liquid passages are connected in a labyrinth fashion. This design forms very strong ribbed unitized structure. Sunlight passes through the vacuum cells and is directly absorbed in an infrared absorbing fluid. In case of power failure, the fluid may be drained under gravity to prevent overheating of the glass collector. The collector can be insulated in the back and around the edges by potting with foam insulation or by an additional row of glass vacuum cells.

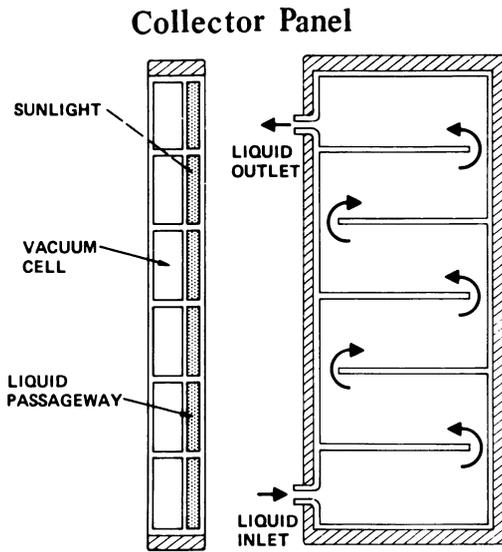


Figure 2

Figure 3 — The basic idea of the continuous forming process is to roll and fuse together three sheets of glass. The center sheet would be indented by rollers to form ribs of required height and thickness. Then the outside sheets would be fused to the ribs of the center sheet to form the vacuum cells and liquid passages.

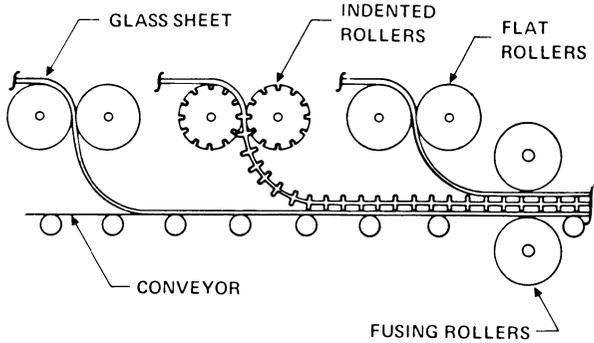


Figure 3

Simplified schematic of roll and fuse process for manufacturing of glass solar collectors.

Figure 4 — The actual process would be of course much more complicated. The top of the ribs have to be reheated before fusing. The tubulations for inlet and outlet have to be fused and the collectors have to be cut into panels. Considerable development effort will be required to develop the necessary processes and procedures. The speed of the process will be comparable to that for rolling and fusing armored glass which is about 2 meters per minute. A typical machine would produce about 1 square kilometer of collectors annually. Figure 5 shows a small collector model diffusion bonded from borosilicate glass. Tests on this model achieved an efficiency of about 50% at 90°C.

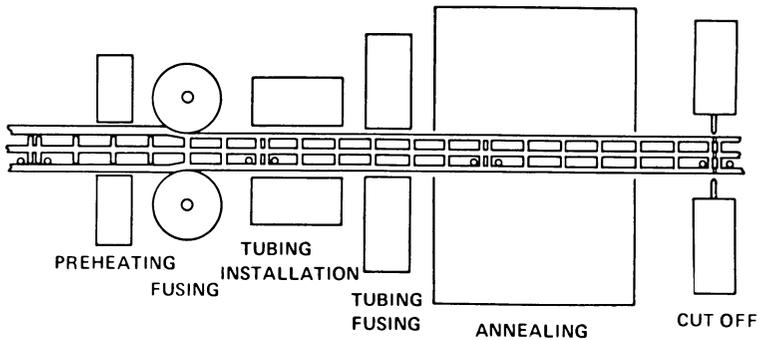


Figure 4

The farther steps in roll and fuse process of glass solar collectors.

By using black fluid developed under N.S.F. grant, the glass collector can be used the same way as conventional collector. In addition, since the panel structure is transparent, there are other possibilities. A white fluid would reject solar heat in summer while allowing some of the light in. A

fluid which passes the visible part of the solar spectrum while absorbing Infrared has much more interesting implications.

Figure 5 — This figure shows the distribution of the solar spectrum extraterrestrial (in space) and mass 2 — (on the earth's surface) with all the absorption bands. Almost half of the solar energy striking the earth is in the infrared region. A two and a half percent solution of cupric chloride will absorb all the infrared and transmit most of the visible radiation normally striking the earth. Photosynthesis (the process by which plants turn energy into food) requires light primarily in the visible and ultra violet wavelengths passed by the cupric chloride. (Cupric chloride is corrosive, and better fluid will probably have to be developed for mass application).

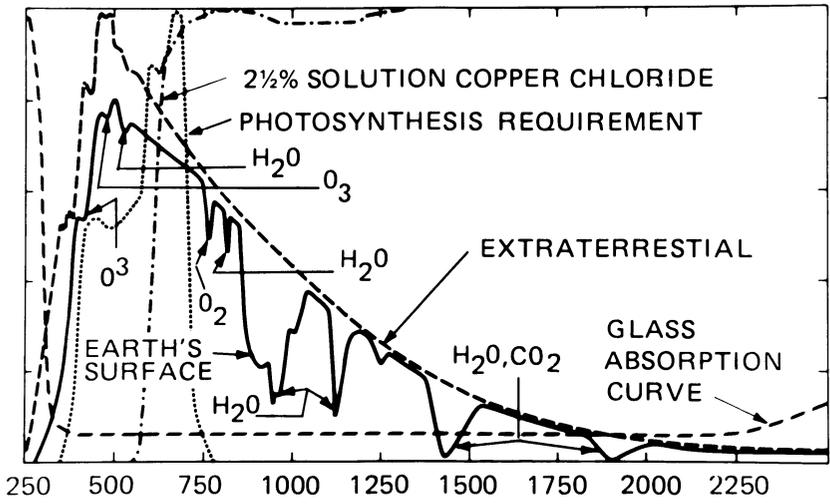


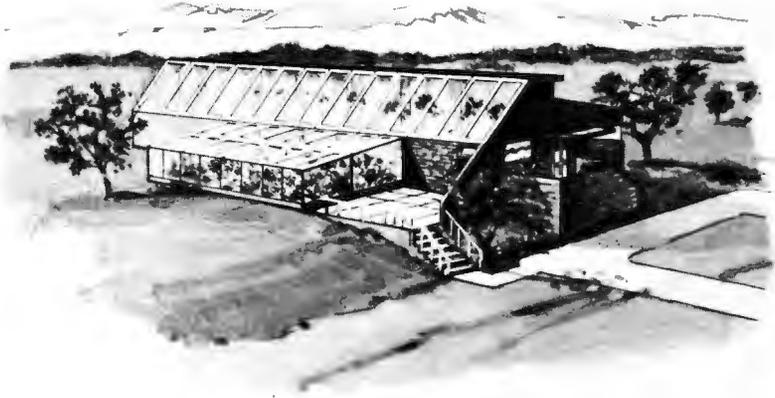
Figure 5

Elementary photo requirements of chlorophyllic plants in relation to the solar spectrum and to typical absorbing fluids.

Figure 6 — shows an artist impression of a home employing glass collectors as skylights and covers for an attached family greenhouse. The energy absorbed in the absorbing fluid can be used to provide heating and domestic hot water the same way as a conventional bank of collectors and in nearly the same amount, since all the infrared portion of the solar spectrum which is incident on the collector is absorbed by the fluid. The admission of light into the house for plant growing is a very attractive bonus from architectural point of view, as well as from an ecological standpoint. Also, additional layer of vacuum cell insulation would minimize the loss of the heat from the inside of the house.

The Environmental Research Laboratories of The University of Arizona are successfully operating a number of commercial greenhouses in

Tucson, Arizona, in Mexico, Venezuela, and on the Arabic Peninsula. The per acre crop yields for greenhouses in these sunny areas have been as much as ten times that open field production. The intensive solar heating is controlled by massive evaporative cooling (about 4 liters/m³/day). Many plants will not tolerate the resulting increased humidity. In addition, since large amounts of water are needed, evaporatively cooled greenhouses cannot be used where water is scarce.



THE **BOEING** COMPANY

Figure 6

Artist's concept of an integrated greenhouse and a family home utilizing all-glass solar collectors.

Figure 7 — This figure shows an artist impression of a commercial greenhouse equipped with absorbing fluid glass collectors, located in an arid region of the American Southwest. The energy removed from the incident solar spectrum by infrared absorption, will eliminate the need for evaporative cooling. The only water needed will be for plant growth. The heat collected can be stored in the form of hot water and recirculated through the collectors during the cold desert nights. The large amount of surplus heat can be used also for distillation of the brackish or salt water, for running air conditioning for crew quarters, or even for pumping water from deep wells and electrical power generation.

Another new glass application in the Solar Energy area is for heat absorbing glasses.

One existing application of infrared-absorbing glass occurs in light projectors. The application demands that the glass pass all the visible spectrum to preserve the true colors of the picture and absorb all the infrared to protect the heat-sensitive film. The heat absorbed in the glass is carried away by an electric fan. For this particular application, the Corning Company recently developed Code 4605 glass.

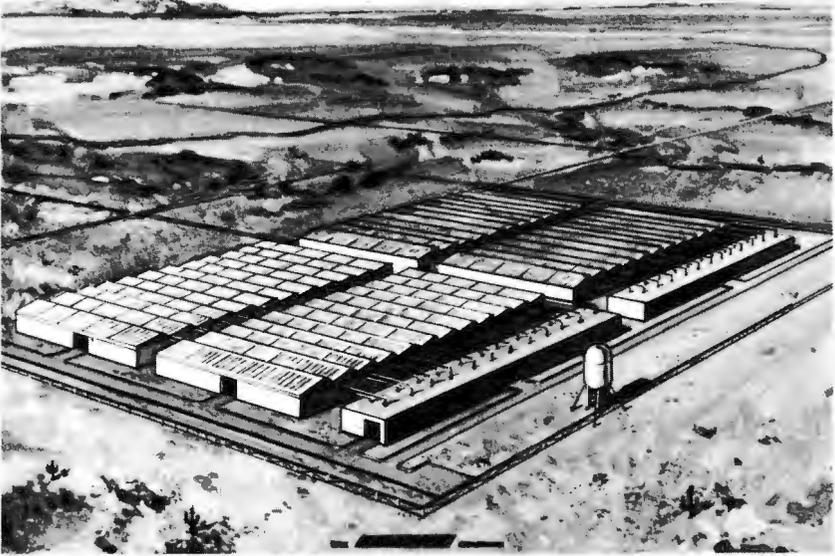


Figure 7

Artist's impression of a commercial farm with all glass solar collectors situated on non-arable land in the Southwest.

Figure 8 — shows the comparison in transmission between soda lime window glass and 4605 glass. The heat-absorbing glass passes nearly 75% of the visible spectrum and absorbs nearly all the infrared. Heat-absorbing glasses are basically phosphate glasses in which part of the silica composition is replaced by phosphorous oxide. An additional amount of iron oxide is used to control the final absorptivity. In the case of 4605 glass, a thickness of 6 mm is needed to absorb all the infrared of the solar spectrum.

The Shott Company manufactures a KG series of glasses which absorb the total incident infrared from the solar spectrum in a thickness of only 2 mm. *Figure 9* shows the transmission curves of KG 1, 2, 3, and 4 glasses. These glasses have much greater ultraviolet transmissivity than ordinary window glass. Their chemical durability (in existing composition) may not be adequate, however, for solar energy applications. The composition of heat-absorbing glasses can be tailored to a certain extent to fit a particular application; for example, a thickness of 100 mm or more can be used instead of 6 mm to absorb all the infrared.

A collector of similar construction to the one already described could utilize a heat-absorbing porous glass structure in place of the infrared absorbing fluid in the fluid-carrying passages. A fluid of a matching refractive index could be used to remove the heat from the glass. The total thickness of the porous glass structure, in the form of crushed glass particles or shapes similar to distillation column packing, would be sized to fully absorb the infrared portion of the incident solar radiation.

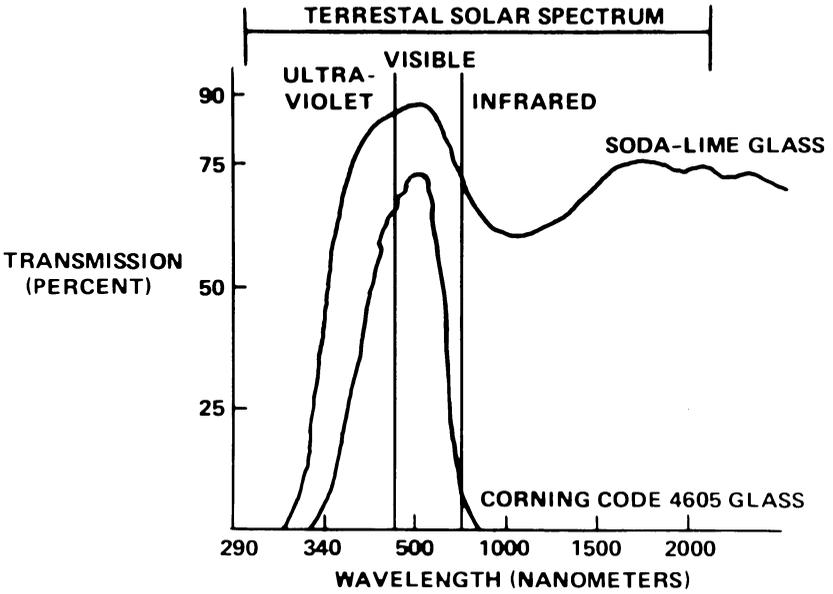


Figure 8

Transmission comparison between soda-lime glass and heat absorbing Corning code 4605 glass.

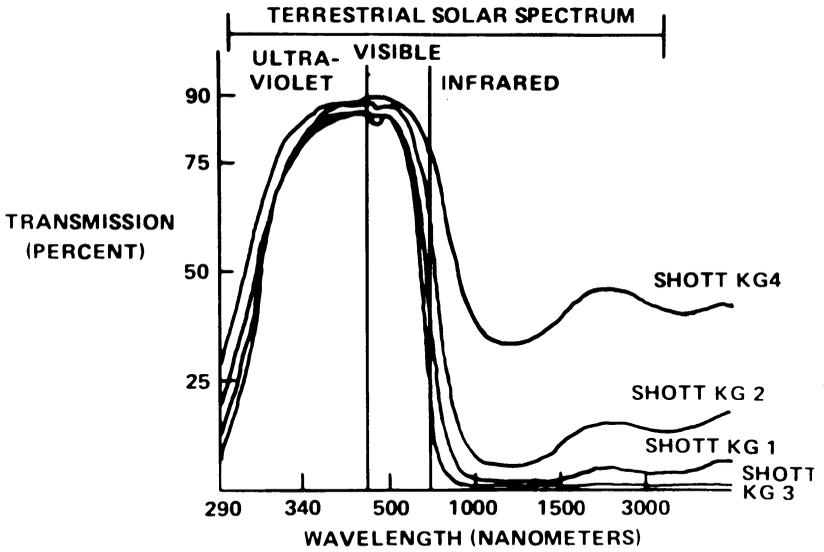


Figure 9

Transmission of Shott Company heat absorbing glasses, KG 1, 2, 3 and 4.

There are many solar collectors designs where the circulating fluid is air: Solar energy is absorbed in a metal structure coated with selective absorption material. By substituting heat-absorbing glass for the metal, transparent collectors can be designed and used as a skylight with a small loss of efficiency and significant gain in architectural acceptance. The easiest way to accomplish this, and one which requires the least amount of heat-absorbing glass, would be to use a venetian blind collector developed by Environmental Research Laboratories of the University of Arizona. *Figure 10* shows the venetian blind collector principle, the large area of absorbing surface (comparable to the glass window area) allows efficient heat transfer. Instead of using a black-painted metal venetian blind, strips of heat-absorbing glass can be installed. The natural draft ventilation will take the heat away from the heat-absorbing glass. This heat can either be directed inside the building for heating or vented outside for cooling. The hot air can also be directed by forced circulation to a rockbed heat storage unit. This type of collector would admit light to the building with very small losses. The heat-absorbing glass strips can be continuously rolled using existing equipment and then cut to the required length. Other types of air collectors would need large sheets of heat-absorbing glass, perhaps in corrugated form to increase surface area. This, however, would demand a greater capital investment, which could only be justified when a large solar energy market is established.

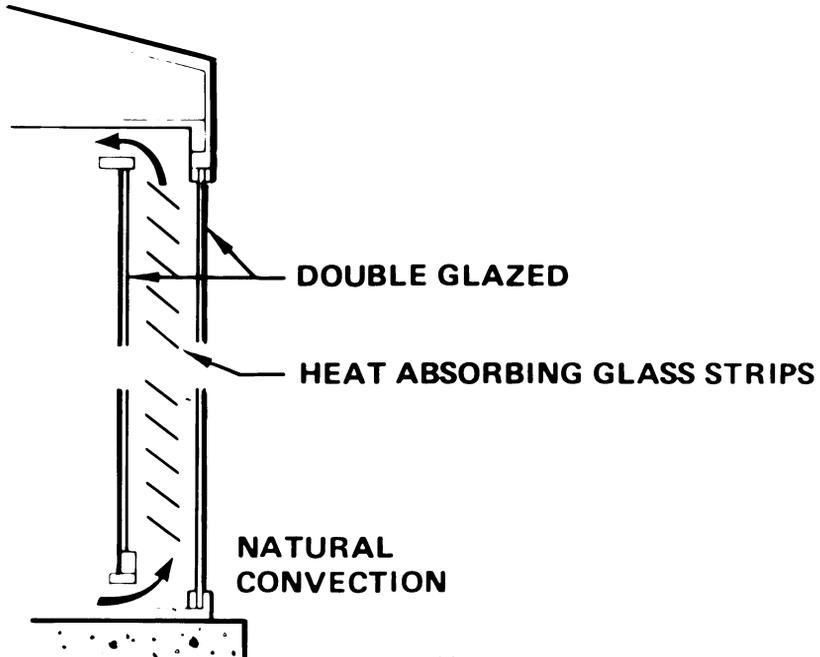


Figure 10
The principle of heat absorbing glass venetian blind air collector.

The principal of the Trombe wall, a passive solar collector, is based on a heat-absorbing black masonry structure that absorbs solar energy transmitted through a glass double window during the day. The heat is carried away during the night by properly induced convection currents. *Figure 11* shows the winter and summer operation of the Trombe wall. A Trombe wall made of heat-absorbing glasses would admit the light into the building while absorbing heat (*Figure 12*). By slightly changing the composition of the glass, the total infrared absorption could be obtained with a glass thickness of as much as 300 mm. This would allow the visible part of the spec-

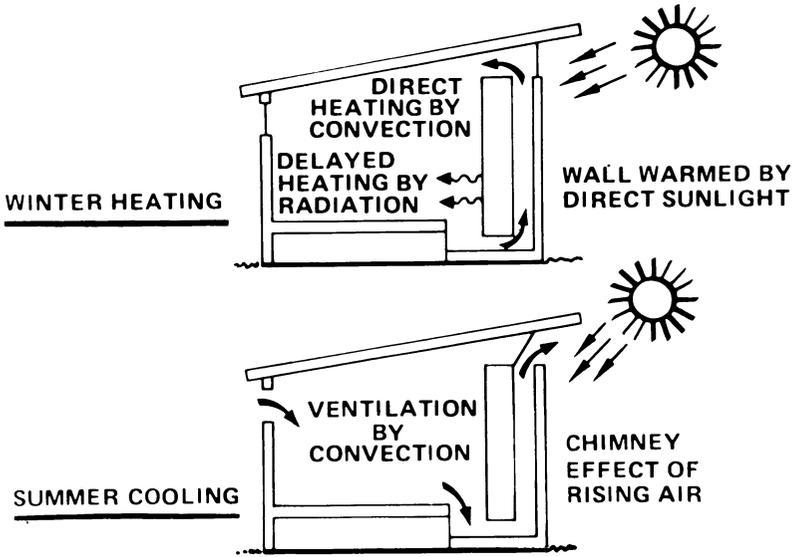


Figure 11
Principle of Trombe wall passive system: winter, summer.

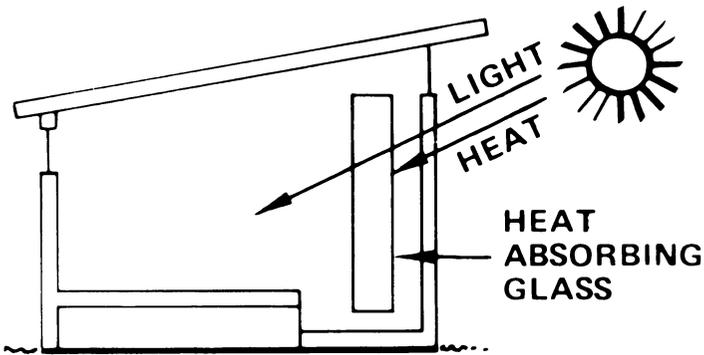


Figure 12
Heat absorbing glass structure.

trum to pass through the wall without sacrificing the heat capacity of the structure. The solid glass wall would be difficult to manufacture and handle, but casting of a large number of convenient size glass blocks can be done on existing equipment, with grooves and ridges pressed into them for the assembly of interlocking structure. Another advantage of a glass Trombe wall is that the infrared part of the solar spectrum is absorbed within the glass structure due to internal dispersion, whereas in a masonry Trombe wall the heat is absorbed on the surface and then conducted to the interior. A Trombe wall constructed of heat-absorbing glass can be temporarily installed in any room with southern exposure with only a small amount of light loss.

Another passive solar energy system application using heat-absorbing glass blocks involves greenhouses. There are a number of greenhouses being constructed with passive systems that use water ponds or rock piles in conjunction with reflecting surfaces. The blocks of heat-absorbing glass would offer an advantage for they transmit the light needed for photosynthesis. A number of configurations constructed out of glass blocks could be used to absorb the heat during the day and radiate it out during the night. The total mass of each structure should be sufficient to absorb enough solar energy during the day to protect the crops during the night. Additional directing of circulating currents can ensure an even heat distribution. The interlocking glass bricks could be prearranged according to the environmental needs of the particular crop and seasonal changes.

The properties of existing heat-absorbing glasses and certain flexibility with which the properties can be altered by changing their composition may give us glasses specially tailored to particular applications in solar energy utilization.

A description of glass for Solar Energy applications would be incomplete without a few words about the Solar Cells. Development of single crystal silicon solar cell makes Space Exploration possible. And solar cells are one of the most attractive means of utilizing Solar Energy on the Earth's surface, because they provide a direct, simple, no moving parts means of converting solar energy into electrical power. The only thing preventing their expanded use is cost. At present, solar cell arrays cost about \$2000/m². This cost must be reduced to about \$50/m² or roughly of 50 cents per watt. Glass promises an inexpensive substrate which lends itself to a fully automated process to bring the price down. One construction method is incapsulating silicon single crystal solar cells together with metallic interconnectors between two sheets of glass by electrostatic bonding, this is being developed in Burlington, Massachusetts. A large effort in many labs (including M.I.T.) is going into development of thin films of solar cells on glass, films such as amorphous silicon, cadmium sulphide and cadmium telluride, copper oxide and other compounds are being developed. Those films may not match the silicon solar cell efficiency which is already approaching 17%, but they promise inexpensive cells deposited on float glass, again a continuous automated process.

The sun is going to be around another 50 million years, glass which is made out of most abundant resource — sand, is going to be indispensable if not the only material which can be used to effectively harness its energy.

ISOLATION OF ALKALI METALS IN A VACUUM SYSTEM USING A HIGH CONDUCTANCE BAKEABLE MAGNETIC VALVE

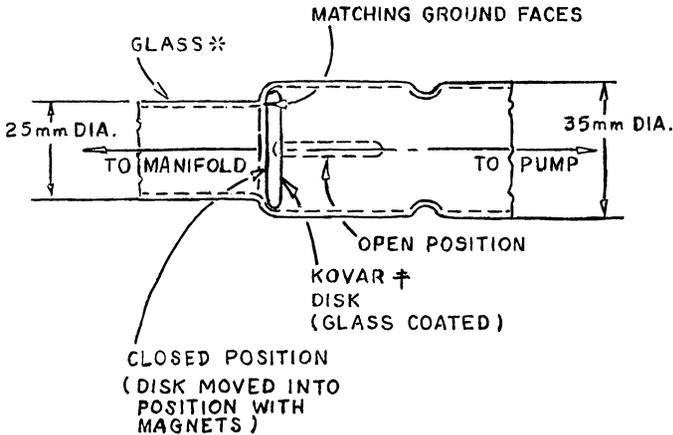
WILLIAM J. DEFLORIO, *Glassblower*

Gen Rad, Inc.
Bolton, MA

In the processing of rubidium lamps and cells we use small amounts of rubidium. A typical amount would be 5 milligrams sealed into 1720 alumino silicate break seals. The 1720 glass is used to allow the ampoules to be baked at higher temperatures without discoloration of glass or contamination of the rubidium. The break seal weight is also sealed in glass. After proper bake out and a suitable vacuum is obtained, the vacion pump valve to the manifold is closed. A Kovar disc that had been sealed between two plates of 7052 glass and ground flat is now moved into the closed position with a magnet, sealing off the manifold to the vacuum system.

The rubidium ampoule is now broken and then heated with a hydrogen torch to drive alkali metal into the 1720 precision blown lamps or 7070 cells.

Rubidium is now driven off of the kovar disc using a high intensity lamp. After the glass is cooled, the kovar disc valve is opened and the manifold and lamps or cells are re-pumped to an acceptable vacuum. The vacion valve is then closed. The proper amount of rare gas is let into manifold and lamps or cells and then they are sealed off. The kovar disc valve can be baked, is easy to build and operate, and isolates alkali metals without contamination. Figure 1 shows the vacuum system valve.



* CORNING GLASS † KOVAR IS REGISTERED
7052 TRADEMARK OF
7056 WESTINGHOUSE CORP.
7040

Figure 1

FAST EVACUATION OF HEATED DEWARs WITHOUT SILVER LOSS

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ABSTRACT

This paper discusses the problems encountered during evacuation of Dewar flasks at elevated temperatures. The insulating properties of silvered Dewars prepared in the manner described in this paper are excellent. Details of the preparation of the surfaces to be silvered, the silvering process, baking out, evacuation and the sealing off procedure are included. The processes are (1) Brashear formula for silvering with a slight modification, (2) London Laboratories Limited formula for easy silvering, (3) the method of preventing the silver coat from being spoiled when heating to a high temperature during evacuation, (4) baking out in two stages of decreasing temperature, (5) pumping off the gasses evolved while sealing off before completing the seal.

I

INTRODUCTION

It is well known that there are many ways of pumping and silvering Dewars. In this work a procedure is presented that produces higher quality silvered Dewars that are baked out at 500°C without silver deterioration.

Dewars made of Pyrex® glass are preferable to those made of Soft glass due to the added strength of the Pyrex. However, it has been difficult to obtain Pyrex Dewars that have insulating properties equal to those of Soft glass. With some modifications of the usual methods, my technique has been designed to produce Pyrex Dewars of uniformly excellent insulating qualities. The insulating properties of the silvered Dewars prepared in the way discussed in this paper are such that even in the wide-mouthed Dewars filled with liquid nitrogen near the top, there is no boiling. Rather, the change of the liquid nitrogen into the gaseous phase takes place entirely by surface evaporation.

II

PREPARATION OF THE SURFACE TO BE SILVERED

Time should be taken to select the tubing that will be silvered and evacuated. This tubing should be examined carefully for bubbles and striations that could hold parts of the cleaning solutions or silvering solutions. These imperfections could ruin the silver surface, or, worse, they could cause a leak to develop, creating a soft Dewar.

After carefully selecting the tubing for a Dewar, it should be washed with Alconox¹ and tap water, using as stiff bristled brush, rinsed and towel-dried. After cutting pieces with a wet carborundum saw, the tube should be rinsed with tap water and the cut edges cleaned with concentrated hydro-

®Registered trademark of the Corning Glass Works

fluoric acid on a cotton swab. Caution should be taken not to allow the hydrofluoric acid to stay in contact with the cut edge longer than fifteen to thirty seconds as this will cause the surface to become undesirably frosted. Rinse off the hydrofluoric acid with hot tap water and towel-dry the glass tubing inside and out.

The selected and prepared tubing may now be used in fabricating the Dewar by conventional methods. Once the Dewar has been constructed and prior to annealing, allow it to cool to room temperature. Take the Dewar and rinse the inside with a 10% solution of hydrofluoric acid in distilled or deionized water. It is important that the inside surface be completely wetted with this solution. Decant the 10% hydrofluoric acid solution and rinse the Dewar carefully and thoroughly with distilled or deionized water. The inner surface should be totally wetted showing no grease spots. If any grease spots or water droplets appear, repeat the 10% hydrofluoric acid solution and rinse again.

III SILVERING PROCESS

The silvering solution is prepared as follows: (1) one gram of stannous chloride crystals is dissolved in 10cc of distilled or deionized water. (2) After the stannous chloride is completely dissolved, 0.2 cc of this concentrated stannous chloride solution is added to 1000 cc of distilled or deionized water. This solution has a shelf life of not more than three hours. Pour enough of this diluted solution of stannous chloride into the Dewar (approximately 2/3 full) to wet the entire surface by shaking or rolling the Dewar. After all the surfaces have been wetted, drain the Dewar and rinse twice with distilled or deionized water. Allow the Dewar to drain while the silvering solution is being prepared.

The Brashear Process, as described in *Scientific Glassblowing* by E. L. Wheeler, is one of the easiest and most reliable liquid plating procedures and all materials are readily obtained. The process is as follows:

Silvering Solution for the Brashear Process

Three solutions are prepared in the following proportions.

- A. 2 l. of distilled water, 50 g. silver nitrate.
- B. 2 l. of distilled water, 90 g. potassium hydroxide.
- C. 800 ml. of distilled water, 80 g. cane sugar plus 100 ml. of ethyl alcohol and 3.5 ml. of nitric acid (specific gravity 1.42).

Solution A should be kept in a dark place or in a dark bottle. Solution C should be aged at least 30 days before using. If it is to be used at once, boiling for 30 minutes will have the same effect as 30 days aging.

The solutions are used in the proportions, A : B : C = 64% : 32% : 4% by volume, or see Table I. The solutions are prepared for use as follows: concentrated ammonium hydroxide is added to solution A, drop by drop, while vigorously shaking the flask until the precipitate, which first forms, completely disappears. To this is added half of the required amount of solution B causing a dark brown or black precipitate to form. If insufficient ammonium hydroxide is added before solution B is

added, the precipitate will be a yellowish green. If this does occur, the solution should be discarded and the process started again. Ammonium hydroxide is added again, drop by drop, while shaking until the black precipitate is almost, but not completely, dissolved. Gradually the remaining half of solution B is added, while continuing to clarify the solution with ammonium hydroxide. The ammonium hydroxide should be added more slowly toward the last, since the reaction is slow and there is danger of going past the end point. The final solution should not be completely clear; rather it should be a slightly cloudy solution which if allowed to stand for 30 minutes, will become clear except for a few black flakes which settle to the bottom. The solution may be used immediately or it may be allowed to stand for as long as an hour without affecting the results.

Cooling Silvering Solutions. After solutions A and B have been prepared by the addition of ammonia hydroxide, it is advantageous to cool them to at least 15°C. For strip silvering, a temperature of 10° to 12°C. is recommended. The lower temperature allows time for decanting the solution into the vessel after the addition of solution C and before the actual plating out begins.

SILVERING PROCEDURE

Enough solution should be prepared to fill the vessel about two-thirds full. This solution may be decanted into the vessel, the required amount of solution C added, and the solution mixed by shaking. The vessel should be rolled or shaken during the entire plating operation. The time required for the solution to react and deposit silver on the walls depends on two factors: (1) the temperature of the solution (the lower the temperature, the slower the reaction); (2) the age of solution A (the older the solution, the slower the reaction). The time of completion of the silvering process can be determined by occasionally removing a little of the solution and noticing the character of the precipitate. When the solution becomes flocculent the silvering is completed and the solution should be removed to prevent the formation of a gray deposit or bloom. With practice, the condition of the solution inside the vessel can be determined by feel. When the solution is spent, the precipitate forms a heavy sludge that can actually be felt moving from one end of the vessel to the other as it is tilted end to end.

If a heavy coat of silver is desired, a second coat may be deposited over the first after removing the spent solution. Brighter and thicker silver coatings may be obtained with a given amount of solution by silvering twice with half-strength solutions (obtained by diluting with distilled water) than can be obtained by silvering once with the full-strength solution.²

Another silvering process is the London Laboratories Limited Process. This solution has an advantage in that the company ships you concentrates that only need to be diluted with water. No sludge has been observed with this process. Of the two, the London Laboratories Limited Process is the one recommended. The procedure follows:

Use Instructions For Specialty Item Silvering

These CONCENTRATES are diluted as follows:

Sensitizer (a)

Dilute 0.2 cc RX in one liter distilled, or deionized water. This is now both the sensitizer and the after rinse solution, at use strength. This solution has a life of only 3 or 4 hours, after which it should be dumped and a fresh solution made up.

Silver & Alkali (b)

Dilute 20 cc NS-1 in 480 cc distilled water in a one-liter beaker. Dilute 20 cc NS-2 in 480 cc distilled water in a separate beaker. Mix the 500 cc dilute alkali solution (NS-2) with the 500 cc dilute silver solution (NS-1), while stirring, to give one liter of dilute silvering solution.

NOTE: *Under no circumstances* should you allow the NS-1 concentrate to come into contact with the NS-2 CONCENTRATE, as this mixture at this concentration becomes EXPLOSIVE.

Reducer (c)

Dilute 20 cc NR in 980 cc distilled water to make one liter of dilute reducer solution.

SILVERING PROCEDURES

A — *Cleaning:*

1) To silver a closed vessel or bottle on the inner surface, rinse the inside of the vessel with an approximate 10% hydrofluoric acid solution. Decant the acid solution, and rinse the vessel thoroughly with distilled or deionized water to remove the hydrofluoric acid. Caution: Use rubber gloves when handling the hydrofluoric acid.

2) To silver flat glass surfaces, cleaning is best accomplished by scrubbing the glass with a slurry of cerium oxide in water, and a nylon bristled brush. One-half ounce of cerium oxide per liter of water should make a satisfactory slurry.

B — *Sensitizing:*

1) Pour into the clean bottle enough dilute sensitizer solution so that the entire inner surface is completely wetted by rotating or shaking the bottle. About 25% of the volume of the bottle should be ample.

2) Decant the sensitizer solution and rinse the bottle several times with distilled or deionized water to remove the excess sensitizer.

C — *Silvering:*

1) Pour equal volumes of dilute silver and dilute reducer solutions into a clean beaker. The volume combined should be sufficient to slightly more than half fill your vessel or bottle. Stir with clean glass or teflon stirring-rod to combine the two solutions. These steps should be done SWIFTLY.

2) Quickly pour the mixed silvering solution into your vessel, or bottle, and rotate it or shake it for at least two minutes,

until the silvering reaction is completed. The proper reaction temperature should be roughly 80° Fah on the silvering solutions. An acceptable thickness for the silver film must be determined by inspection as to opacity or other standards that you may wish to achieve, after the reaction is completed.

3) Decant the spent silvering solution.

D — *Final Rinse After Silvering:*

1) Repeat sensitizing step No. 1 listed above, making certain that all silvered surfaces are wetted with the dilute sensitizer solution.

2) Decant the sensitizer solution and blow dry with clean oil free air or heat at slightly above 100°C (212°F).³

After the silvering is complete, the Dewar is drained. If there is any sludge left inside of the Dewar, a little distilled or deionized water should be added to rinse it again. After draining the water and sludge, fill the Dewar approximately 2/3 full with the diluted stannous chloride solution. Roll or gently shake the Dewar until all the surfaces are wetted. Drain this solution and rinse once with distilled or deionized water. The Dewar is drained for one hour then is ready to be evacuated.

IV

BAKING PROCESS

The silvered Dewar is sealed onto the pump-out stem inside an annealing oven. *Note: the Dewar has still not been annealed.* The Dewar is now evacuated with a mechanical roughing pump.⁴ Check the seal for leaks with a tesla coil. The Dewar is then isolated from the mechanical roughing pump by means of a three-way stopcock in the system outside the oven. This allows the mechanical roughing pump to run and, at the same time, the Dewar is brought up to atmosphere. With the Dewar at atmosphere and sealed to the pumping stem, the oven heaters are turned on and the annealing oven runs through its regular annealing cycle. After the temperature decreases from 565°C to 500°C, it is held constant and the vacuum pumps started.

V

EVACUATION AND SEALING

The silvered Dewar is held at 500°C for a period of one hour. During this time the mechanical roughing pump is used in conjunction with a three stage Mercury Diffusion Pump.⁵ The temperature is lowered to 400°C and the pumping continued until the best vacuum possible has been achieved. The Mercury Diffusion Pump can now be isolated and the Vac Ion Pump⁶ turned on. This pump achieves a vacuum of 1×10^{-8} mm. in a matter of one hour. After the vacuum is as hard as it will get, the stem should be slowly sealed off. Care must be taken in sealing to allow the hot gasses to be pumped away. Using the vacuum gauge as a guide during the sealing, it is possible to seal off the Dewar without losing the hard vacuum. If the evolved gasses produce a lower vacuum reading, the sealing off process

should be halted until the vacuum gauge indicates a hard vacuum. After the Dewar is sealed off, it is removed from the annealing oven and allowed to come to room temperature. Once at room temperature, it is wrapped with tape for safety.

VI CONCLUSION

This procedure allows the glassblower to fabricate, silver and evacuate a Dewar in one day. It not only is simple but eliminates the worry of silver deterioration during the heating process. It also allows the Dewar to be annealed and outgassed for evacuation at the same time. A very high quality Dewar is the finished product.

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- ⁴Mechanical Roughing Pump — Sargent-Welch Scientific Company, 7300 North Linder Avenue, Skokie, Illinois 60076 Model No. 1376 300 L/M
- ⁵Mercury Diffusion Pump — Allan B. Brown, University of Connecticut, Storrs, Connecticut 06268 Three Stage — approximately 60 L/M
- ⁶Vac Ion Pump — Varian Eastern Service Center, Room 221, Terminal Building, Greater Pittsburgh Airport, Pittsburgh, Pennsylvania 15231 Model No. 911-5011 15 L/S

VERSATILE MULTIPLE OUTLET SEMI-BALL AND SOCKET VALVE

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The multiple outlet semi-ball and socket valve described in this paper was initially designed at the request of bacteriologists who needed a device that could deliver germ-free medium from a large reservoir into a number of growth chambers and could, at the same time, maintain sterile conditions. The valve designed to meet these specifications has unusual versatility for a number of applications.

VALVE DESCRIPTION AND OPERATION

The multiple-outlet valve assembly, shown in Figure 1, is designed to direct flow from a primary conduit into any one of a number of secondary conduits, as well as to direct a similar but reverse flow. The valve includes two mating hemispherical sockets that each rotatably receive a spherical valve plug. The valve plug is attached to the primary conduit and includes diverging passageways from that conduit to a plurality of ports. Each of the ports is alignable with one or more of the glass joints sealed into the lower hemispherical socket. The upper hemispherical socket includes a slot for the primary conduit arranged in such a manner that the motion of the conduit along the slot, and the rotation of the spherical plug about the various axes, will position the valve-plug ports relative to their secondary outlets.

Both the upper and lower hemispherical sockets are modified Pyrex Ace Glass Co. polished joints size 28/15. The valve plug is a 28 mm diameter Teflon sphere that uses a 12/2 capillary socket joint as its primary conduit.

Figure 2 illustrates an exploded view of the valve, and Figure 3 illustrates an enlarged cross sectional view. The numbers below refer to parts indicated on both figures. As shown, the valve body includes a glass upper hemispherical socket (11) that is compressably sealed and rotatably connected to a corresponding lower glass socket (13) so that the two enclose a sphere. The spherical volume is occupied by a Teflon spherical plug (15) which has passageways (17) terminating in ports (19). A clamping device (21a and 21b) holds the two hemispherical sockets together against a sealing and lubricating ring (23), to permit relative rotation of the two sockets in a sealed condition. The ring and the spherical plug fitting into it are composed preferably of a material such as Teflon and the sockets of glass, to permit lubricative sealing engagement.

A number of luer joint conduits are sealed to and penetrate the wall of the lower hemispherical socket and terminate with openings (27) on the internal surface of the socket. These openings are alignable, as shown, with one or more of the ports within the spherical valve plug. Such alignment is achieved through rotation of the valve plug.

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Figure 1
A scale photograph of the multiple-outlet valve assembly.

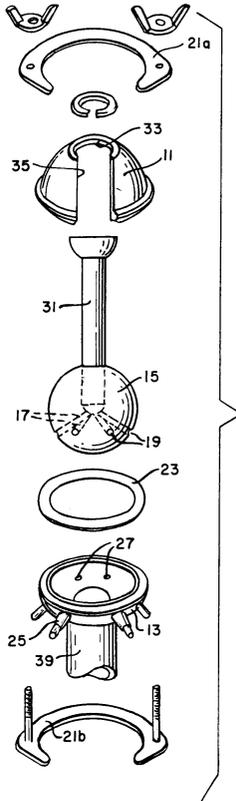


Figure 2

An exploded perspective view of a variable-port valve assembly.

Valve plug 15 is provided with a primary conduit passageway (31) that is pressed and sealed to the upper portion of the valve plug so as to interconnect with the plug passageways (17).

The primary conduit extends through the upper hemispherical socket by way of the socket apex top opening (33). This opening continues into a slot (35) which extends from the apex of socket 11 to its lower circumference. Movement of conduit 31 in slot 35 is accompanied by corresponding rotation and positioning of the spherical valve plug.

Such positioning is illustrated in Figure 3 in the alternate placement of the primary conduit. Rotation of the upper hemispherical socket along with the spherical plug permits great flexibility in the alignment of the ports of the valve plug with one or more of corresponding openings (27) into secondary outlets (25).

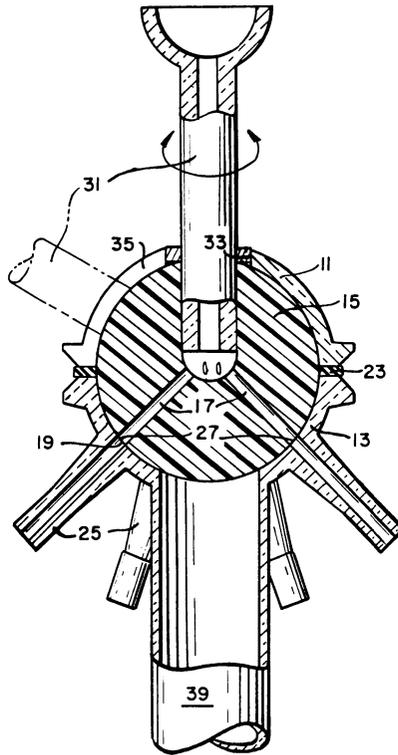


Figure 3

A cross sectional view of a variable-port valve assembly illustrating one alternate position.

Lower hemispherical socket 13 is illustrated with a somewhat larger outlet (39). This outlet is not ordinarily aligned with one of the ports in the valve plug. When the plug is rotated so that primary conduit 31 moves downward and is positioned in slot 35, any of the ports can be aligned with conduit 39. Conduit 39 can thereby be employed as a diversion or "dump channel" for any desired material.

Figures 4a and 4b illustrate a somewhat altered upper socket (50) and a lower socket (60). In Figure 4a a plan view of the upper socket includes a flange (45) positioned about the lower part of the socket base. This flange includes a number of U-shaped grooves (47) equally spaced around its circumference. A slot (49) is included from the apex of flange 45 to the socket base. Indexing grooves (51) of U shape are likewise provided along the edge surfaces of the slot 49.

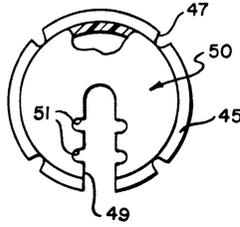


Figure 4a

A plan view of a hemispherical socket including indexing means for use in a valve assembly similar to that of Figure 1.

Figure 4b illustrates an alternative lower hemispherical socket (60) that is adapted to engage socket 50 through sealing ring 59. This second socket includes a flange (53) of somewhat greater width than flange 45 of the upper socket. The top surface of flange 53 is provided with spring-loaded balls or pins which are normally extended but which can be recessed within suitable openings to a level below the surface of sealing ring 59 on application of pressure. These pins are arranged in a circumference corresponding to the flange 45 in the hemispherical socket 50 as shown in Figure 4a. On rotation of the upper socket 50 in respect to lower socket 60, the pins (57) and grooves (47) act as indexing means to assure particular valve position when in corresponding alignment. Similar indexing can be accomplished through the U-shaped grooves (51) in cooperation with pins, balls, or probes that can be carried on the primary conduit (31) or sealing ring (23) shown in Figures 2 and 3.

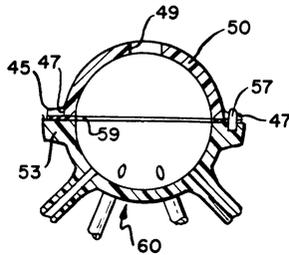


Figure 4b

A side view in section of a second hemispherical socket in engagement with the socket and indexing means of Figure 4a.

Figure 5 illustrates an exploded view of a multiple port valve that includes a floating hemispherical sleeve (65) provided with a number of radial passageways (67). In this particular configuration, the spherical valve plug 69 is sized to fit within the hemispherical sleeve 65, which in

turn nests within the lower hemispherical socket 61. Sleeve 65 is attached to a sealing ring (73) equipped with indexing tabs for independent rotation. Abrupt off-on programming can thereby be performed merely by rotation of hemispherical sleeve 65 while maintaining other valve components in position.

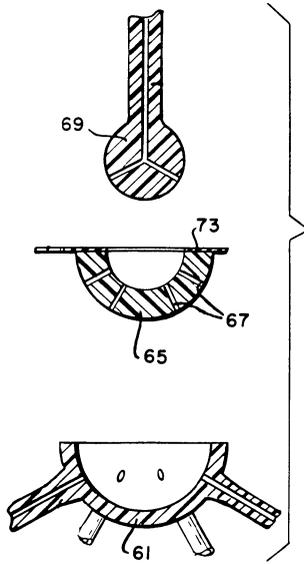


Figure 5

An exploded view of selected valve components illustrating the use of a hemispherical sleeve within a variable-port valve assembly.

Figure 6 illustrates the multiple outlet valve as an electrical switching device. The three main body components of this valve are made of a non-conducting material. A number of insulated electrical conduits are sealed to and penetrate the lower hemispherical socket and terminate with metal contacts on the internal surface of this socket. A valve plug is provided with proper electrical conduit passageways. A primary electrical passageway is sealed to the valve plug and all its electrical passageways are properly interconnected. An upper hemispherical socket similar to that shown in Figure 4a is used in this switching device. Indent mechanisms are provided so that rotation of the upper hemispherical socket and the valve plug will engage properly with this lower socket, and will also serve as a means to assure a particular valve position of correct electrical contact alignment.

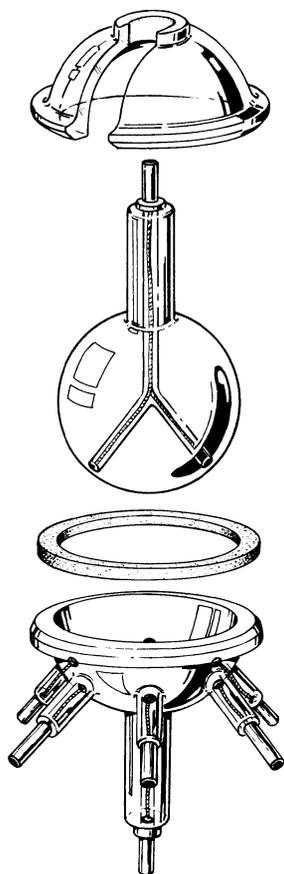


Figure 6

An exploded view illustrating the valve components as an electrical switching device.

VERSATILITY

Although this valve was designed to deliver fluid from a common reservoir to a number of chambers, it is adaptable to a large number of applications. It can be used to feed a single gas, liquid, or slurry supply from one inlet to one or more of a number of outlets or, alternatively, to introduce fluid flow from one or more of several inlets into a single outlet. The valve can be employed as a fraction cutter in liquid and gas chromatographic separations, or during sample elution from ion exchange resins. The valve can easily be adapted for vacuum service through suitable use of Viton o-rings as a sealing means. This adaption can also be used for gas or plasma flow transfer and/or in conjunction with a mass spectrometric analysis.

In addition, this valve can provide an electrical switching device for both AC and DC electric currents. It can operate an electrical switch that will control a gas or fluid transfer device in an arrangement in which both the switch with multiple contacts and the valve with multiple ports are of similar construction.

CONCLUSION

In its present form, this valve has proven successful in distributing germ-free media from a single source to a number of bacterial growth chambers. Its usefulness in a number of different types of applications is indicated above. Further applications are possible with other valve configurations, and different lubricants may further improve its operation.

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IN ATTENDANCE

The following are on record as having attended the Twenty-second Symposium on the Art of Glassblowing held at the Sheraton-Boston Hotel, Boston, Massachusetts, June 19-23, 1977. As fully paid registered participants, these persons are entitled to a copy of the Proceedings.

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