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ON THE
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PHOSPHORIC ACID/COPPER II OXIDE CEMENT FOR SEALING TUNGSTEN ELECTRODES TO QUARTZ CAPILLARY PLASMA TUBES

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INTRODUCTION

A simplified method for sealing electrodes to small-bore quartz capillary plasma tubes was needed to aid in the study of the plasma. This report will describe a simple method which has been successful in our laboratories.

DISCUSSION

Placing small electrodes in plasma tubes enables one to take electrical measurements, such as electron density, and is an aid in the study of spectrochemical excitation sources. In order to achieve these measurements in the study of microwave-excited argon plasma and eliminate the more complex techniques (1) used in the past or the use of borosilicate glasses (2) having much lower thermal properties, the following method was devised.

The quartz capillary tube is first cut to the desired length. It is then ground flat on opposite edges to provide a flat surface for mounting and for drilling. The tube is then mounted to a piece of plate glass with epoxy cement. This unit is then epoxied into a metal container, Figure 1, so that the tube will be submerged while drilling. The shallow metal container is made heavy enough so it could be firmly attached to the bed of a vertical mill for fine adjustment while drilling (other precision drilling equipment may be used).

The plasma tubes fabricated required a series of electrodes in line spaced 1.00 cm apart. Holes were drilled in the submerged quartz using a 0.031 in. diameter diamond plated solid drill (Lunzer Industrial Diamonds, New York, N.Y.). The tube was then removed from the mounting plate and cleaned thoroughly. The ground surface opposite the holes was then ground and polished to provide good optical properties. The tube is now ready to have the electrodes sealed in place.

The electrodes in this case are made from tungsten rod .020 in. in diameter and ground on the end to fit the holes in the tube with minimal clearance so the cement seals at the surface of the tube and does not flow into the center. Electrodes were cut to a length of 1 cm to allow enough to silver-solder copper lead wire to the electrodes without reheating the cemented seal.

In order to allow the electrodes to penetrate a specified depth into the center of the tube, a length of tungsten wire of the appropriate diameter is placed into the tube and the electrodes are set in the holes to the depth required. The electrodes are then sealed into place using a high temperature cement made from a mixture of phosphoric acid and copper (II) oxide.

The cement is made by slowly adding phosphoric acid to copper (II) oxide until a paste sufficient for pouring is formed. This is then poured into a small test tube, 1/8 in. to 1/4 in. inside diameter, and allowed to dry for 24 hours. The cement is then removed from the glass in rod form, and is ready to use. The above technique was used in this case but any form could be used to form strips or rods for use. Simply pouring the paste out on wax paper or sheet glass in small strips and allowing it to dry works well.

The seals are now cemented into the quartz tube by placing the electrodes in place as previously described and wiping the cement on in an operation similar to soldering. Using a hand torch with a small oxygen-gas flame, the quartz, tungsten, and cement are heated simultaneously and a small amount of cement is deposited at the base of the electrodes, Figure 2. Although the cement has a very low conductance it is best if the cement seals do not touch, Figure 3. I suggest that a few test pieces be done to perfect the technique before actual application. Caution must be used not to overheat the area allowing the cement to flow into the center of the tube. The cement will withstand temperatures in excess of 900^{o}C and flows easily at around 1200^{o}C allowing it to bind well with borosilicate glass as well as quartz.

In this application the quartz temperatures were in excess of 450^{o}C for extended periods, and the plasma reached temperatures of 2500^{o}K in the center of the tube, Figure 4. The seals proved to be quite stable structurally and able to withstand the thermal stresses during operation and while silver soldering the copper leads to the electrodes, Figure 5. It is also possible to seal other metal electrodes such as platinum and silver to various glasses with this cement. Although it was not required in the application described above, the seals proved to be vacuum tight when tested to pressures of 1 x 10^{-6} mm of Hg.

CONCLUSIONS

Although this method worked extremely well for the case described, it is not implied that it will be a replacement for other proven methods. It is a low cost, relatively simple alternative.

ACKNOWLEDGEMENTS

The author would like to express his thanks to Dr. Scott R. Goode and to Dr. David C. Otto, Chemistry Department, U.S.C., for their cooperation and advice in developing this method and help in preparing this paper.

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2. R. Auni and J.D. Winefordner, *Spectrochim. Acta.*, **30B**, 281 (1975).

Figure 1

Plasma Tube Mounted in Metal Tray for Drilling

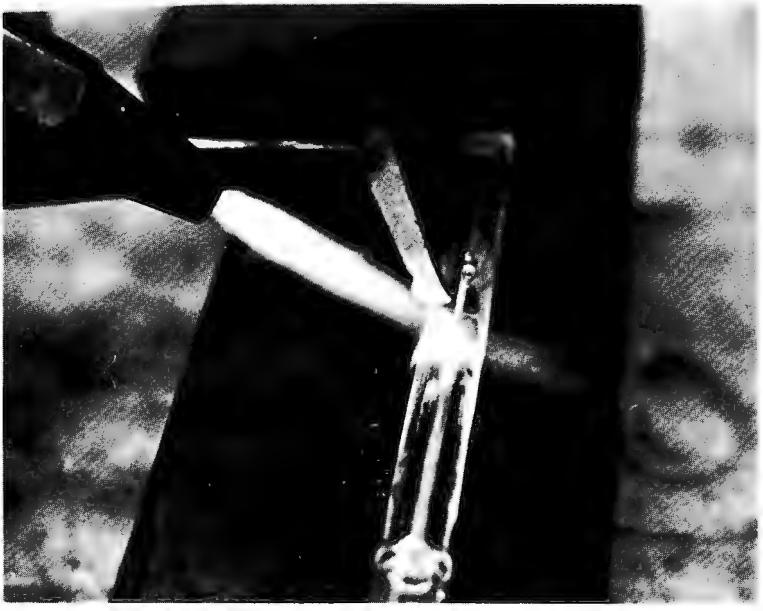

Figure 2

Application of Cement to Plasma Tube and Electrodes

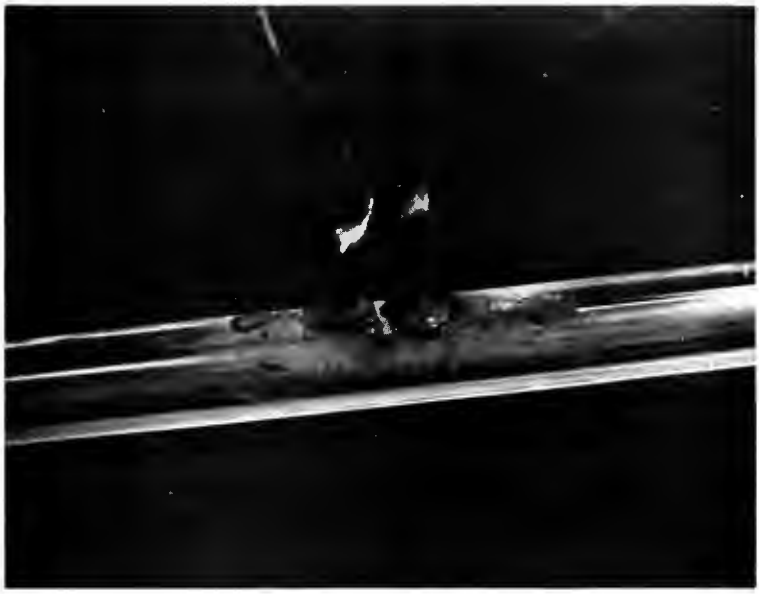

Figure 3

Finished Tube and Seals Indicating Separation of Cement Seals

Figure 4

Plasma Tube During Operation

Figure 5
Silver Soldering Leads to Electrodes

IMMERSION INTERFEROMETRIC STUDIES OF GLASS SEALS

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1. INTRODUCTION

The primary goal in glass-glass sealing is achieving a mechanically strong seal manufactured consistently and economically. To achieve this goal, it is helpful to understand the influence of the processing variables on the boundary conditions of the glass seal. The usual examination of the final stresses alone will seldom lead to information on the real causes for the weak glass seal. A multiple beam immersion interferometry examination is useful in providing data on refractive index variations across the whole junction from outside to inside sealing edge. It is the purpose of this presentation to demonstrate the practical aspects of the interpretations of the interferograms in making good, strong, uniform glass seals repeatedly.

2. IMMERSION INTERFEROMETRY

The normal interferometry can be modified in a described way to determine the refractive index changes preferentially to the topographical features of peaks and valleys.

The sample under examination is immersed in a liquid of the same refractive index and this liquid in turn fills the space between two optical flat mirrors. A diagramatic cross-section of the assembly is shown in Figure 1. The sample assembly called interferometric cell is surrounded by a heating chamber with a resistance wire enclosed in an air-tight space. Temperature of the fluid is measured by a thermocouple. Three screws with five threads provide a precise positioning of the mirrors. The optical scheme of this method is illustrated by Figure 2. Light from a mercury vapor lamp source passes through a condenser, then through a filter for monochromatic line of 0.5461 micron wavelength to a pinhole. The emerging beam is collimated to a mirror and then reflected to the interferometric cell and finally is viewed by a microscope. The interferogram has lines (= fringes) connecting points of identical refractive index. In contrast, the normal interferometry has lines connecting points of the same elevations. In immersion interferometry, the homogeneity of the transparent solid is indicated by a series of straight-line fringes. On the other hand, any small inhomogeneity is detected by fringe deviations from the straight lines. These deviations are converted to the refractive index changes by a relatively simple mathematical relation.

3. INTERFEROMETRY OF GLASS-GLASS SEALS

The glass seals can be divided conveniently into three groups for further discussions.

Type 1 - Two Glasses of the Same Composition

Type 2 - Two Glasses of Different Compositions

Type 3 - Two Glasses of Different Compositions Sealed by a Third Glass (Solder Glass)

3.1 Interferograms of Type 1

The good, strong seal of Type 1 will show an interferogram with continuous straight lines extended and interconnected through the seal interface. The straight lines indicate no changes in refractive index. This condition can be observed when all process variables are under control. In this case the diffusion of glass A into glass B is exactly of the same order as glass B into glass A, as shown in Figure 3.

The actual seals will deviate somewhat from this ideal condition. They will have interferograms with lines of varying degree of deflections from the straight lines in the direction of decreasing refractive index as indicated by Sign -, in Figure 3.

The greater these deflections, the greater the differences of refractive indexes in relation to the ideal, good seal. Numerous independent studies indicate that the refractive index decreases are due to volatilization of alkalies from the two to be sealed glass surfaces caused by preheating burner flames. These deflections are more pronounced in seals made by higher temperature and longer time of preheat cycle. These deviations were drastically reduced by employing preheat flames of medium temperatures and by shortening the preheat time. The final sealing cycle, on the other hand, can be increased for improved seal. By controlling these basic variables, strong seals were achieved with helpful guidance from interferograms.

3.2 Interferograms of Type 2

The ideal seal of the two glasses with different compositions is theoretically expected to have fringes connecting glass A with glass B without interruptions. Figure 3, middle diagram, shows a typical interferogram of Type 2. In this case, the glass A has higher refractive index than glass B. The signs + and - indicate direction of increasing (+) and of decreasing (-) index.

The real seals will show several deviations from this ideal fringe configuration, as will be discussed below in the next section 4.

3.3 Interferograms of Type 3

Interferograms of Type 3 are more complex as indicated by the last diagram of Figure 3 representing Color Television Glass Tube Assembly. Two glass parts (A = Front Panel; B = Back-Funnel) are sealed by a third glass C, a low melting solder glass. The diagram has two boundaries, one on each side of glass C. At these boundaries, the fringes of glass A are connecting to fringes of glass C which are shifted to the direction of an increased refractive index. The sealing glass C has the highest refractive index in this assembly. Basically, the Type 3 diagram is a combination of two Type 2 diagrams connected by the sealing glass C. In the ideal strong seal, the fringes are continuous without any breaks and any sharp deviations. Here again, the real seals show different degree of variations from these ideal seals. These variations are suggesting the proper adjustments of the controlling variables. By applications of the information of these interferometric studies, strong seals were made on a routine basis in the Television Industry.

4. POOR AND GOOD SEALS

Three examples of Type 2 seals were selected to further illustrate the practical applications of the immersion interferometry. Type 2 seal appears to be more

appropriate for further discussion because two of these make Type 3 seal, and the Type 1 seal was already covered in some detail above in Section 3.1.

4.1 Poor Seal

Interferogram of a poor seal is divided into two parts by an interfacial dark band as indicated by Figure 4. The section above this band is relatively homogeneous glass A. The section below this band is less homogeneous glass B with fringes of several deflections observable in the middle section of this seal. The refractive indexes vary from -0.0185 to +0.00242. These variations occur within 450 microns from the interfacial band. In the same area, the measured stress of 2250 psi compression indicates composition changes which can decrease the thermal expansion. The dominating refractive index change was a relatively drastic decrease. Both types of information suggest increase in silica above the initial starting composition B. This observed increase in SiO_2 can be due to a cord (stria), to loss of alkalies, or a combination of both. The actual measured increase in silica was only +0.8%. Examining the fringe deviations, we can appreciate the high sensitivity of this method to the small composition changes.

4.2 Improved Seal

Several variables of the sealing process were adjusted; namely, the preheat flame position was moved slightly away from glass B, the preheat cycle and the flame temperatures were reduced. These adjustments led to an improved seal as indicated by the next interferogram (Figure 5). The homogeneity of glass B is substantially improved and it approaches the uniformity of glass A. The small region with slight deviations can be noticed on the right side of the photograph where the final closing flame was directed during the last seconds of the sealing. This improved seal is approaching the theoretical postulate for the ideal seal of Type 2 described before, but it does not completely satisfy this requirement as shown by the tabulated refractive index changes as a function of distance from the interface.

Table 1

IMPROVED SEAL

Refractive Index Changes as a Function of Distance from Interface

Distance Microns	Glass A Index Change	Glass B Index Change
0	-0.0034	+0.0028
18	-0.0021	+0.0017
54	-0.0013	0.00
72	0.00	

4.3 Good Seal

Further improvements in the sealing process were achieved by adjusting the flame distance from glass B, by directing the preheat flame uniformly toward both glasses, by decreasing the preheat time and finally by increasing the final seal cycle. The total sealing cycle was extended by about 12 seconds to improve the poor seal and to manufacture the good seal.

The interferogram of the good seal is shown in Figure 6. The fringe deflections are uniform without breaks connecting glass A with glass B. The boundary band is missing and the interfacial conditions are excellent, indicating a good control of the sealing process. The refractive index variations are in agreement with the theoretical postulates for an ideal seal as indicated by tabulated data of Table 2.

Table 2

GOOD SEAL

Refractive Index Variations with the Distance from the Interface

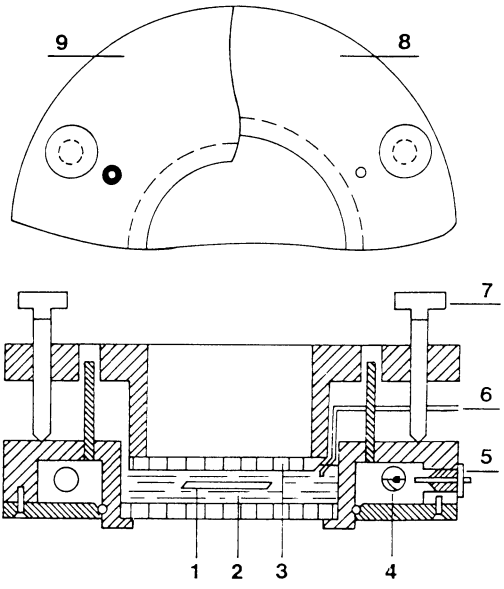
Distance Microns	Glass A Index Change	Glass B Index Change
0	-0.0024	+0.0024
22	-0.0016	+0.0016
44	-0.0008	+0.0006
72	0.00	0.00

5. CONCLUSIONS

5.1 Immersion interferometry gives useful information on the boundary conditions of glass-glass seals.

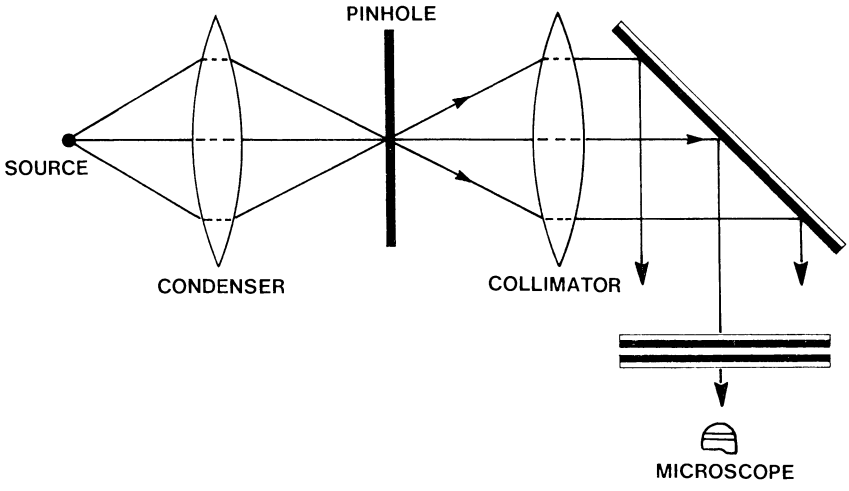
5.2 Immersion interferometry is recommended for studies of production sealing processes.

5.3 This presentation covered the practical applications of immersion interferometry in making good, strong glass seals.



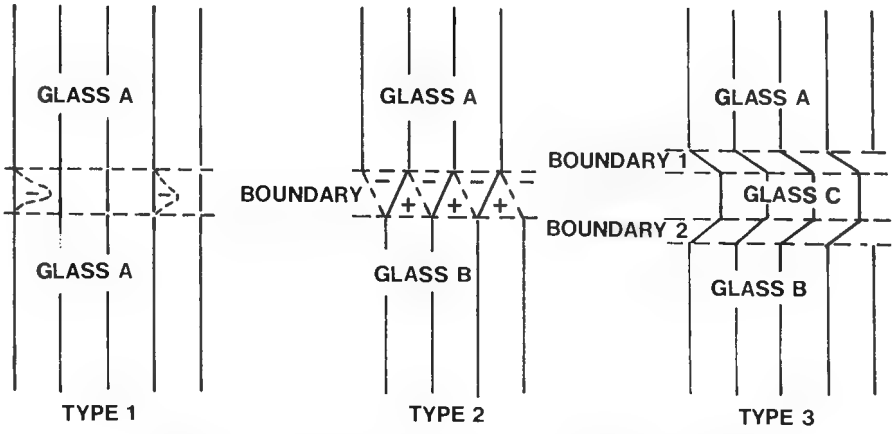
Sketch of interferometer cell.

Figure 1



The optical system. Cell is shown as two parallel plates.

Figure 2



Ideal Interferograms of Three Types of Glass-Glass Seals

Figure 3

Figure 4

Figure 5

Figure 6

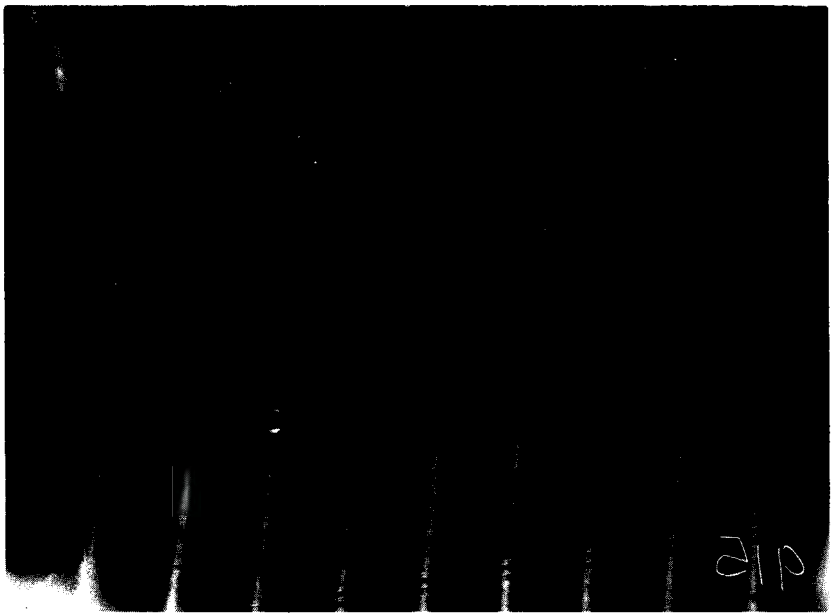


Figure 7

COMMERCIAL BUILDING AND INDUSTRIAL APPLICATIONS FOR SOLAR ENERGY

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Presented by Dr. Y.K. Pei

Commercial building and industrial process heat energy requirements in this country are large. Boilers utilizing fossil fuels provide the bulk of energy required to do heating, cooling, and hot water for commercial buildings as well as process energy in the industrial area. It has been stated that if solar could do as much as 20 percent of energy requirements of this area by the year 2000, it would save many barrels of oil and contribute significantly to the energy independence of this country.

Considering the scope of the commercial building and industrial energy applications, not all of this energy from a quality standpoint can be provided by solar. Further, one should separate the various forms of solar, for example, solar thermal from photovoltaic and other forms. For this paper, we have limited the solar discussion to the thermal area only. It has been shown through the various demonstrational programs that solar thermal can be made to function efficiently providing energy up to 250^{O} F. There are projects showing higher temperature applications; however, as one increases the temperature of the operation, decreases in efficiency are realized.

The basic solar thermal equipment available today falls generally into three categories. Flat plate solar thermal collectors generally apply to lower temperature applications. Typically 150^{O} F is an upper temperature range of this equipment. Utilizing selective coatings and vacuum as insulation, evacuated tubular collectors have shown great promise up to 240^{O} F. These type of collectors generally can convert about 50 percent of the light energy striking the collector surface to thermal energy over this temperature range. Various forms of concentrating collectors have been shown to provide energy as high as 700^{O} F. As noted earlier, inefficiencies in the conversion process and the transfer process to the load are present, operating at the higher energy levels.

Many commercial buildings and industrial process applications require large amounts of energy for relatively constant loads as a function of their operating mode. Solar energy is a variable source as it strikes a particular surface. Its variability comes from the sun being located in a different position throughout the day relative to the collector surface. In addition to that, variations in cloud cover and atmospheric interference such as particulates, pollution, etc. affect the amount of energy and the flux rate striking a collector surface. Interfacing this variable source to a large and constant demand is a formidable design problem. Evacuated tubular solar collector technology has some of these interfacing considerations incorporated in its design.

REVIEW OF THE EVACUATED TUBE TECHNOLOGY

The basic solar receiving device is an element called an evacuated collector tube. The tube, consisting of two hermetically sealed cylinders of borosillicate glass, measures about 4 feet in length and about 2 inches in diameter. A vacuum is created

between the two walls. A cross-sectional view of this element (Figure 1) describes the internal components. Noting that light strikes the outer wall and passes into the collecting element, the transmissivity of this outer glazing of glass is 0.92. This indicates that 92 percent of the light energy striking the cover tube surface is transmitted through the glass and is available to be absorbed. Between the cover tube and the inner tube called the absorber tube a vacuum is formed with a pressure of about 10^{-4} torr. This level of vacuum insures that conduction and convection losses from the absorber surface are negligible. The significance of this is that the solar collector can operate virtually independent of the outside conditions of temperature and wind.

On the surface of the inner glass layer a selective coating is applied. The properties of this coating are shown to be absorptance α=0.86 over the solar spectrum and emittance ϵ=0.05. It is at this surface that the light energy passing through the vacuum is converted into thermal energy. Further, thermal energy is transported from the absorber wall to a working fluid which carries the energy into the solar system. Inasmuch as one end of the evacuated tube is sealed, a tube shown as the feeder tube is used to provide for a fluid path in and out of the evacuated tube element.

Several designs of evacuated tubes assembled in a manifold have been developed. Each has its own characteristics and because of its design finds certain application. A design where 24 tubes, 12 above and 12 below, a centrally located manifold connected in series is referred to as the SUNPAK series solar collector. This was one of the earliest models of solar collectors designed into demonstration projects. More recent development to add drainability to the basic model has been to incorporate the evacuated tubes in a single-sided manifold. In this design, upon system command, the fluid in the collector loop can be drained and stored until operation is resumed. Domestic hot water applications are primarily addressed by this design. While we can describe two different manifolding or plumbing techniques of the basic evacuated tube, we see the future having additional types. For example, there is no reason why the same tube cannot be plumbed in a way so that air can be manifolded in and out of the basic collecting element. Our research and development efforts along these lines show an air collector to be extremely attractive. Air as the heat transfer medium in the collector can operate at higher temperatures. The absorber surface can operate as high as 600^{o} F.

The previously mentioned energy transfer techniques pertain to different applications and each has its distinct operating characteristics. We do not expect these to obsolete the others, rather we see others being developed for specific applications. The basic energy collecting device--the evacuated collector tube--will probably remain much as it is today.

COLLECTOR PERFORMANCE

Figure 2 illustrates the performance of the SUNPAK series solar collector. The data presented shows the typical instantaneous efficiency as a function of the parameter $(T_{in}-T_a) \div Sp$ where T_{in} is the inlet temperature to the collector, T_a is the ambient temperature and Sp is the insolation in the plane of the collector. The performance for SUNPAK is shown along with that of SUNPAK without a reflector as well as that of the flat plate solar collector with double glazing and a selective coating. It is possible that the intercept of the flat plate curve is higher than the SUNPAK; however, the slope of the SUNPAK performance curve is flatter, indicating lower thermal losses and higher operating efficiences as described by the parameter $\Delta T \div Sp$. In daily operating conditions the value $\Delta T \div Sp$ is greater than 0.4. In higher temperature

operations, such as those found in commercial building energy systems and industrial process heat, the parameter $\Delta T \div Sp$ can be as high as 0.8. One can see that the SUNPAK evacuated tubular collector outperforms flat plate collector by a significant magnitude.

The instantaneous efficiency curve, at times, is misused for system design application. For building systems and industiral process applications, one should integrate the instantaneous efficiency curve over the solar operating day. Rearranging the performance on a daily basis as a function of ambient temperature is shown in Figures 3 and 4. In these figures also, the daily performance of the evacuated tube collector is compared to a flat plate collector as a function of ambient temperature. Note that the energy output is relatively insensitive to both operating and ambient temperatures for the evacuated tube. Referring to Figure 3 in the uppermost plot, the temperature $T_{in}=150^{o}F$ is typical of space heating and service hot water applications; the temperature $T_{in}=180^{o}F$ could be useful for processing hot water; the temperature $T_{in}=240^{o}F$ would be applicable to air conditioning by a lithium bromide absorption unit. The daily insolation of 2150 Btu per square foot in a collector tilt plane represents a clear, sunny day on which solar noon insolation is about 300 Btu per square foot per hour.

These plots illustrate the insensitivity of the evacuated tube collector to ambient temperature inasmuch as there is less than 10 percent decrease as the ambient temperature drops from $100^{o}F$ to $0^{o}F$. On the other hand, the energy output of a two cover, selective coating, flat plate collector drops significantly with the ambient temperature due to a higher loss coefficient. The $150^{o}F$ case in probably representative of winter system operating conditions. It can be seen that both collectors produce about the same amount of energy at ambient temperatures of $100^{o}F$. However, in the region $T_{a}=0^{o}F$ to $40^{o}F$, which is typical of winter ambient, evacuated tubes are capable of producing more than twice as much energy per day as a flat plate collector.

Plots similar to those in Figure 4 are useful for comparing low daily insolation levels that bracket the range of long term averages typically found in many cold climate locations. On an 1100 Btu per square foot day, solar noon insolation is about 150 Btu's per square foot per hour while on a 530 Btu per square foot day, the solar noon insolation is about half this value. It can be seen for cold climates that SUNPAK is still capable of delivering about 50 percent of the available energy whereas the flat plate collector is either near or at cutoff.

It should be emphasized that the better performance of SUNPAK shown in Figure 4 under cold, cloudy conditions is due to a low loss coefficient. SUNPAK is generally thought of as a high temperature collector, but good performance at high temperatures on sunny days is essentially equivalent to good performance at more moderate temperatures on cloudy days. In short a good high temperature collector is a good low insolation collector.

COMMERCIAL BUILDING APPLICATIONS

FEDERAL OFFICE BUILDING, SAGINAW, MICHIGAN

One of the earlier installations of the SUNPAK solar collector was the Federal Office Building in Saginaw, Michigan. This installation is the largest one as of this time. The collector size is about 7,000 square feet or 6,000 SUNPAK evacuated solar

collector tubes. The building serves about 200 people and covers an area of about 60,000 square feet. Solar energy to this building provides about 50 percent of both the heating and cooling loads. Operation of the collector in the winter at this location has shown it to be a highly effective energy gathering device.

TERRASET ELEMENTARY SCHOOL, RESTON, VIRGINIA

Another commercial building, located in the suburb of Washington, D.C., is Terraset Elementary School. Its basic design is unique inasmuch as most of the building is inlaid in earth. This school serves about a 1000 people daily. The building design incorporates energy conservation by its design. Solar collectors, about 5,000 square feet, provide about 30 percent of the heating and cooling to this building. An additional feature in this school is microprocessor control of the solar and the building energy. The microprocessor controller has been an extremely effective solar control element as well as an effective energy management tool.

The previous two examples of commercial building solar projects probably can be classed in the area of unusual or modern design.

EL CAMINO REAL ELEMENTARY SCHOOL, IRVINE, CALIFORNIA

This next commercial building is also a school as can be seen by the courtyard view of the facility. From street level one would not think of this elementary school as a "solar" building. Behind the parapet of this one story building is located 5,000 square feet of SUNPAK solar collector. The collector in this southern California building provides about 50 percent of the air conditioning load. Simplicity and layout are key features of this solar array on the flat roof. Note the minimum of piping in connecting the solar loop with the building system loop. Viewing a schematic, Figure 5 of this project, illustrates the simplicity of the solar system design. Basically, the collector is in a loop interfacing the building system through a heat exchanger. It should be noted that no storage other than what is in the solar loop is present in this system. In applications such as this commercial building or in industrial projects where the loads of the building are large and present daily, the need for storage is eliminated. The collector in this design is turned on by a clock which is set at the threshold level of insolation. As the temperature builds up to a level useable for this application, energy is taken from the solar loop through the heat exchanger. Typically, energy levels of 180^{O} F and higher are delivered by the solar collector. This project was funded by the U.S. Department of Energy and has extensive monitoring equipment. Data being generated will provide future designers of solar systems inputs of both radiation in the area as well as solar collector operation.

TROY-MIAMI LIBRARY, TROY, OHIO

Another DOE funded project is this public library in Troy, Ohio. While the library appears to be ideally suited and designed for utilizaiton of solar, it is a retrofit project. The addition of the collector to this sloping building came several years after the building was completed. Three thousand (3,000) square feet of SUNPAK solar collectors on this facility provide for most of the heating load of this building. It also is monitored by the Department of Energy's computer.

YELLOW FREIGHT TERMINALS
MIAMI, FLORIDA, AND GREELEY, COLORADO

Industrial buildings providing for heating and cooling are from their systems very similar to the public buildings. Yellow Freight truck terminal in Miami, Florida, utilizes solar collectors to provide some of the energy to its building needs. In this application the collector provides energy to absorption machines which provide air conditioning to the office facility for most of the year. Data from this installation suggests the SUNPAK solar collector to be an extremely effective collecting device in intermittent sunny climate such as southern Florida. To explore heating application in extremely clear, bright sky, Yellow Freight installed in SUNPAK solar collector on its terminal building in Greeley, Colorado. Radiation levels in this part of the country are often measured above 300 Btu per square foot per hour flux levels. The climate in the wintertime is typical of much of the northern United States in both temperature and snowfall. In both of these industrial applications, the thermal mass of the solar loop fluid aids in the integration of the energy into the system. Water is typically used in both of these applications as well as the other projects. Freeze conditions are handled by system control.

BONNEVILLE POWER ADMINISTRATION, VANCOUVER, WASHINGTON

In contrast to the previous slide of a solar installation in an extremely high solar flux area, is the installation in one of the lowest solar flux area in the United States; that is, Vancouver, Washington. Cloud cover and rain are a matter of common occurrence in this part of the country. The SUNPAK solar collector in this installation will provide data on the energy collection in this high diffuse radiation area. Preliminary data suggests effective collection and conversion of the building load. In this industrial building solar is integrated into a system utilizing a heat pump, an absorption machine, and a heat exchanger from reclaimed transformer energy. Of special note also is the retrofit aspect of adding solar collectors on top of this "older" industrial building.

ANHEUSER BUSCH, JACKSONVILLE, FLORIDA

Industrial applications of solar to process energy are similar in some respect to some of the previous commercial building solar installations.. Specifically, industrial installations generally have large flat roof areas for solar collector location. Typically and most economically, a collector arrangement in a sawtooth fashion is preferred. Assembly of the collector arrays in clusters can minimize system's piping.

Installation at the Anheuser-Busch, Jacksonville, Florida, plant is one such example. The system shown here was designed and installed by Barry-Wehmiller Company. It is the first brewery process heat system in the world. Solar generated heat is used in the beer pasteurization process. The system integrates 4600 square feet of SUNPAK evacuated tube solar collectors with a patented Thermelt phase change thermal storage subsystem. The collectors are seen in the foreground with two of the four storage houses immediately behind the collector array. The system is entirely roof mounted except for the process heat exchanger and piping connections.

The storage concept in this particular project is unique from the previously discussed system. A Thermelt capacitor panel contains a phase change material (cavity area) and the heat exchange circuit (tube pattern) in a single modular element. The phase-change material has a melting range of 187^{o} - 199^{o} F and a latent heat of fusion

of 89.6 Btu per pound. In operation, excess heat over and above process requirements generally above 160° F is stored by the circulating solar fluid to the tube pattern melting the phase-change material in the center. Later the heat is retrieved by the same fluid loop circulating directly from the process heat exchanger through the storage device solidifying the phase-change material and recovering its latent heat of fusion.

In the Anheuser-Busch installation four capacitor houses or clusters containing 54 Thermelt panels are used. The house walls are well insulated to an "R" value of 50. Each house has its own control valving so that it can be cut in and out of the solar loop. The storage houses were installed over vertical roof support columns. None of the system installation necessitated any structural bracing of the roof.

Alternate methods of locating such phase-change material storage could be in the space below the collector frame. This arrangement would make use of space that is normally unused in a sawtooth solar design.

In the Anheuser-Busch project a Sunkeeper microprocesser is used to control the solar system and operating modes. A mode-display console shows real time temperatures throughout the system. Typically, the microprocessor operating the system turns the collector circuit on at insolation levels greater than 80 Btu per square feet per hour or 8:00 a.m.

System Turn-On:	$I > 80$ BTu/ft^{2}hr or 8:00 a.m.
Capacitor Charge	T coll.$>180^{\circ}$ F
Capacitor Discharge	T coll.$<160^{\circ}$ F
System Turn-Off	$I < 60$ BTu/ft^2 hr and $T_{HX}<160^{\circ}$ F

The following illustration shows a typical temperature history generated by the Anheuser-Busch system during a day's operation. The maximum collector outlet temperature normally exceeds 200° F during the central part of the day. Active collection durations depend upon the season; however the solar-derived heat supply period is usually extended at least 4 hours beyond the active collection period through the use of the Thermelt storage system. On a typical day in August, the system supplied the pasteurizer with heat until 2 a.m. although the active collection phase terminated at 7:30 p.m. This is an extension of about 6-1/2 hours. The automatic operation of the system has been virtually troublefree. The collector operates at daily efficiencies of over 50 percent and system efficiencies of about 90 percent.

SUMMARY

Our experiences in the solar area, from the discussed solar projects, have provided much valuable data about solar integration into commercial buildings and industrial process applications. Briefly, they can be listed as follows:

1. A solar energy system can be designed into commercial building heating and cooling system and industrial process heat applications.

2. Commercial building and industrial process energy requirements up to 240° F have successfully been demonstrated.

3. User acceptance of solar as an energy source has been favorable.

4. Maintenance requirements for solar are not unusual or different from conventional energy process equipment maintenance schedules.

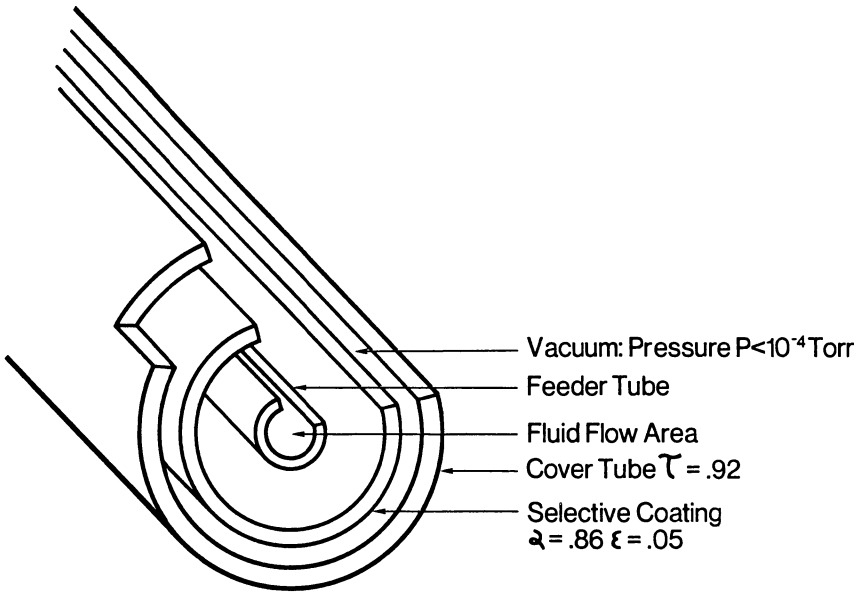

Figure 1

Collector Cross Section

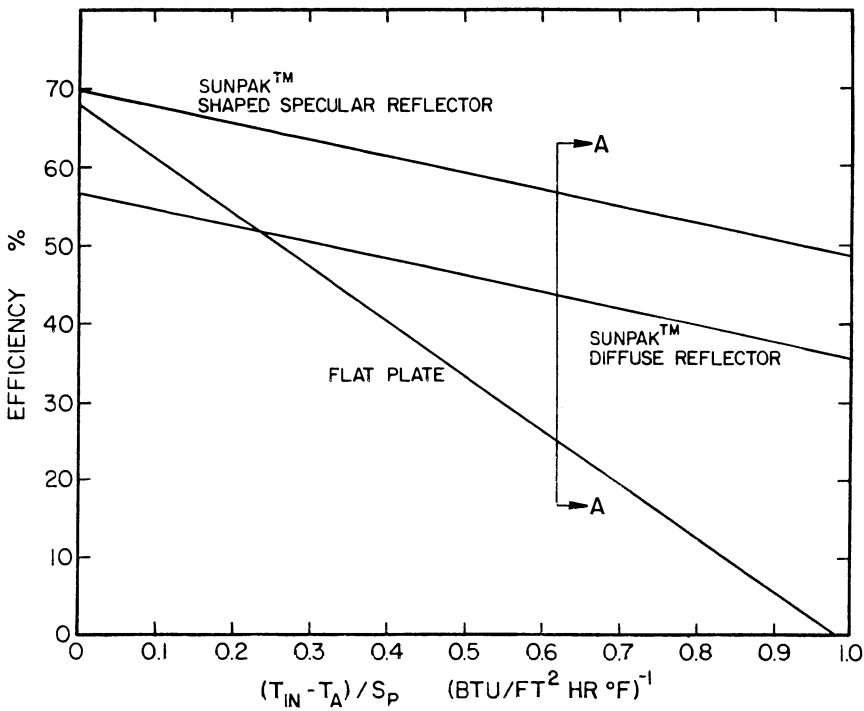

Figure 2

Instantaneous Collector Efficiency

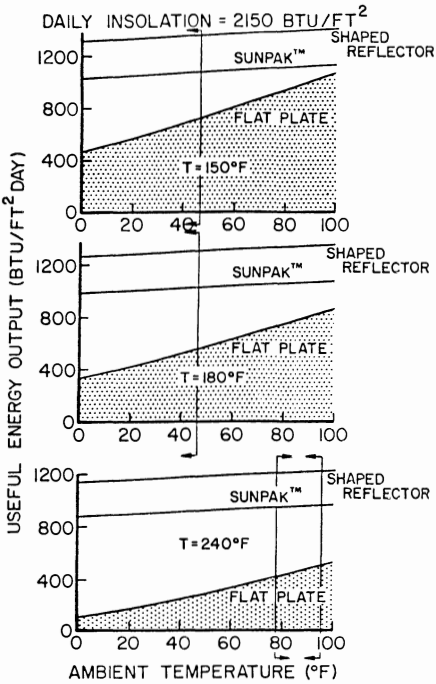

Figure 3

Daily Collector Output on a Clear Day

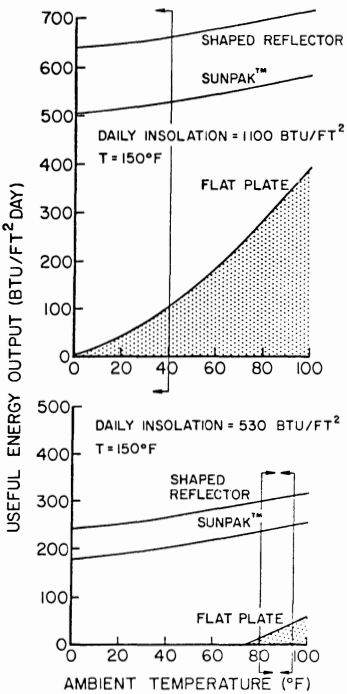

Figure 4

Daily Collector Output on an Overcast Day

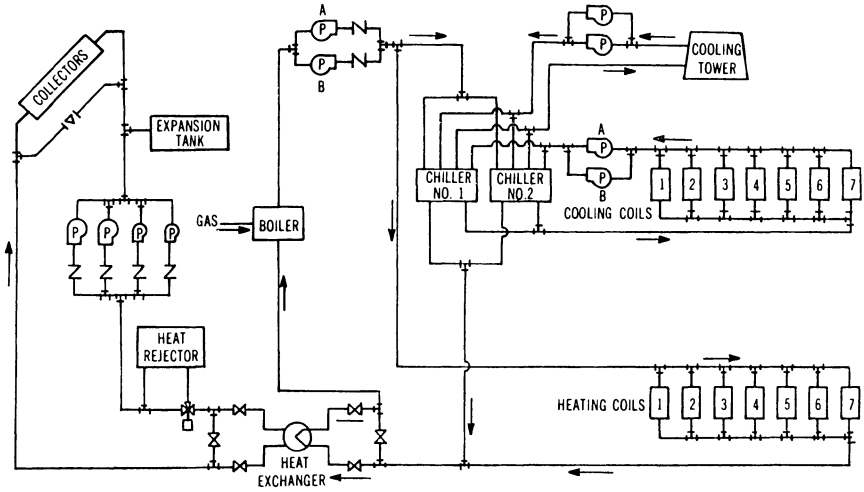

Figure 5

Irvine School Solar Energy System Schematic

HOW TO GIVE A GLASSBLOWING DEMONSTRATION

James Field Morris

Technological Institute
Northwestern University
Evanston, Illinois

A glassblowing demonstration should be interesting, entertaining, educational, and short; what to do and what is required equipment are most important. A good demonstration usually begins quietly, proceeds with a bang, gathers momentum, and finally moves even the most indifferent audience. But few of the onlookers ever realize how much work a glassblowing demonstration can become to be successful.

My demonstration usually consists of seven different parts showing various properties and uses of glass: (1) blowing and breaking a baloney sausage, (2) blowing and silvering a Christmas tree bulb, (3) stretching glass fiber, (4) explaining tempered glass, (5) spinning to form crown glass, (6) developing a vase, (7) and making a lung tester or whistling swan.

By carefully planning your equipment and materials, separating what is needed for each of the seven parts of the demonstration, you keep a consistent pattern of movement. The tools and materials should be as simple as possible and as few as necessary:

a torch

oxygen and regulator

air supply or nitrogen and regulator

city gas or propane with regulator

"C" clamps

glass knife

carbon taper

carbon flat

glass roller

silvering solutions and 3 breakers—1000ml, 100ml, 400ml

glass rod 8mm

glass tubing 8mm H.W., 12mm, 20mm, 25mm, 41mm

asbestos sheet to cover table

box of matches

didymium glasses

carborundum piece

rubber tubing

water – warm and regular

corks

cork with tube for vase holder

ink

white paper

fresh flower

Begin the demonstration making a baloney sausage, and end with the lung tester or whistling swan.

Baloney Sausage:

Use an 18" length of 8mm H.W. tubing. Heat 1" of end until glass is a solid rod on the end 1". Point tube downward and blow a long thin-walled tube 3' to 6'. Keeping pressure on the tube, hit it with a finger and it will explode with a loud bang and fine flakes will float to the floor. This wakes the audience and commands their attention.

Christmas tree bulb and silvering:

Use 41mm tubing 38mm long with pulled points at each end. Constrict one point at base of tubing to 10 to 12 mm. Close other end and then blow bulb 4 to 5 inches in diameter. Cool by floating bulb on stream of air or nitrogen. This impresses your audience.

Silver the bulb by wetting inside with dextrose solution, leaving one half to one cc of liquid in the bulb. Add silvering solution to the bulb and cork. Warm bulb under hot water removing cork momentarily to relieve pressure. Shake until the bulb is silvered.

Pulling glass fiber:

Heat 25mm tubing and have assistant run with one end to stretch glass into a fine tube. Show that the tube is hollow by putting a short piece into ink. Use white paper background to show that the tube was filled by capillary action. Blow into the tube to force the ink onto the paper.

Tempered glass:

Use a 12mm tube 8 inches long with pulled point on one end. Heat a section of glass leaving one half inch unaffected next to the point. Blow and pull to form a thin-walled section of tubing. Close pointed end leaving a 6mm thick bottom. While still molten, immerse the tube into water to the middle of the thin section. When cooled, remove from water. Show strength of outside by hitting the thick end being careful not to hit the tubing. Then drop a small piece of carborundum into the tube and the bottom will explode into small pieces.

Prince Rupert drops:

Heat the end of a 10 to 12mm soft glass rod with a gas oxygen hand torch. When it is molten, let it drip off into a large bucket of water. Prevent the drop of glass from touching the sides or bottom of the bucket. With practice, you will get some solid drops of glass. Burn off the tail two inches from the glass bulb. When this tail is broken, the piece turns into powdered glass.

Crown glass:

Use a section of 41mm tubing 38mm long with pulled point at each end. Close one end leaving a heavy bottom. Cold seal 15 inch length of 8 to 10mm rod to center of bottom. Hold by the rod and blow open the other end of the tube; reheat and flare. Then reheat the entire glass except the bottom where the rod is attached. When soft, put rod on roller and rotate rapidly letting centrifugal force throw out a flat piece of glass. Break rod at cold seal.

Flower vase:

Use a 6 inch length of 20mm tubing with a pulled point at each end. Constrict 1 inch from end to 15mm in diameter. Heat this 1 inch section and blow a small bulb; heat top, then bottom of bulb, pushing together to make a flattened bulb. This will be the bottom of the vase. Heat the center section leaving one inch of unaffected glass at the top end. Blow a 38mm bulb. Heat spots on outside diameter of bulb and indent at five places. Heat bottom half of this bulb in the large bushy flame holding one end firmly and rotating so glass will twist. Work toward bottom until a pleasing shape is formed. Open top end and flare. Heat sections and flute the top with graphite tool. Using a cork glass tube-holder in top, flatten the bottom. Place flower in the completed piece.

Lung tester or whistling swan:

Use a 6 inch length of 20mm tubing with a pulled point at each end. Pull one end down to 8mm tube three inches long. Constrict, slightly, a section one third distance from the other end. Blow a bulb in the center section to an egg shape, 30mm in diameter. Heat the remaining glass and stretch, bend, and blow to form an "S" shape. Form a beak and head by heating and pulling the glass end. Make a small hole at the back of the head. Bend the 8mm tubing up 45 degrees and flatten the bottom of the bulb. Cut beak at 8mm tubing to convenient length. Fill the completed swan with water and have someone try either to blow all the water out or to make a loud whistle by blowing into the end of the swan. The water blows into the face. This causes laughter, and it is a good end for the demonstration.

Comments concerning glass are given from an outline that is kept in view during the demonstration. An outline beginning with the history of glass and ending with information about the American Scientific Glassblowing Society is logical:

I. History of glass.
   A. 75,000 B.C. obsidian glass.
   B. 12,000 B.C. early glaze in pottery.
   C. 3,000 B.C. solid glass.
      1. First man made glass.
      2. Tut's tomb.
   D. 1,200 B.C. Egyptian glass pressing and molds.
   E. 300 B.C. invention of the blow-pipe.
   F. 200 B.C. Rome center of glassmaking.
   G. 1590
      1. Microscopes, telescopes, and thermometers developed.
      2. Professions of medicine and meterology developed.
   H. 1608 first industry in America, Jamestowne, Virginia. Countries leading in glass were leading in science.

II. Kinds of glass.
   A. Lime glass.
      1. 90 per cent of all glass today.
      2. Almost the same today as the first glass made.
   B. Boro-silicate glass.
      1. For research instruments.
      2. For cooking utensils.
   C. Lead glass.
   D. Fused silica.
   E. Formulas.
      1. One company, 100,000, 1963
      2. Same company melting 200 new each week.
III. Why glass?
   A. Inexpensive.
   B. Transparent.
   C. Inert.
   D. Easily worked.
   E. Ingredients plentiful.
IV. Manufacture of glass.
   A. Ribbon machine.
      1. 2000 light bulbs a minute.
      2. 3,000,000 a day on one machine.
   B. Pulling glass.
      1. Method of making small amounts of glass tubing.
      2. Marble pulled 93 miles.
      3. One pound – stretch around the world.
      4. Can be pulled one fifteenth of diameter of a human hair.
      5. Insulation glass fibers made by pulling with high pressure steam.
V. Tempered glass.
   A. Automobile windows.
   B. Glass doors.
   C. Chemcore – chemically tempered glass.
VI. Flat glass.
   A. History.
      1. Rolled.
      2. Poured and pulled.
      3. Spun.
         a. Crown or bull's eye.
         b. To 18th century in use.
      4. Cylinder glass.
      5. Modern up-draw and float.
VII. Comments about glassblowers.
   A. A.S.G.S.
   B. Attrition.

By proper choice of demonstrations, by knowledge of the subject, and by careful planning and preparation, your glassblowing demonstration will be a success.

Figure I
Baloney Sausage

Figure II
Christmas Tree Bulb and Silvering

Figure III
Pulling Glass Fiber

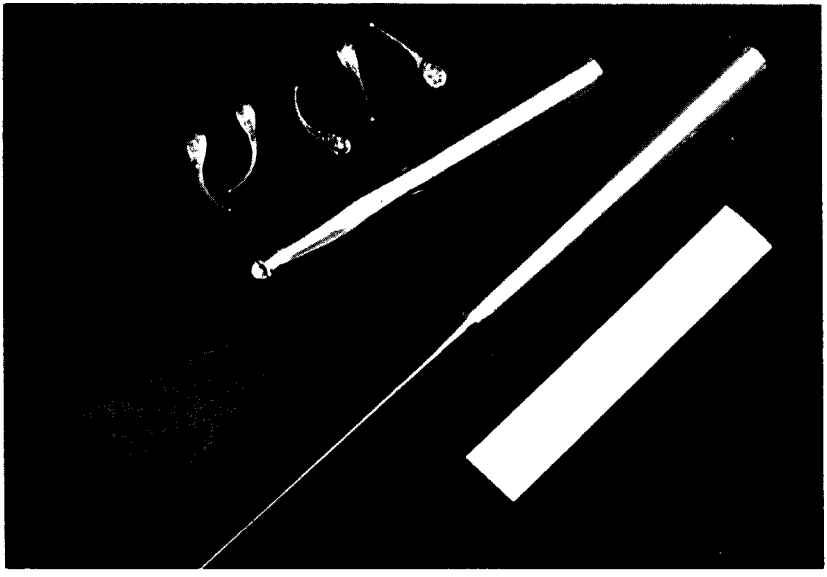

Figure IV
Tempered Glass and Prince Rupert Drops

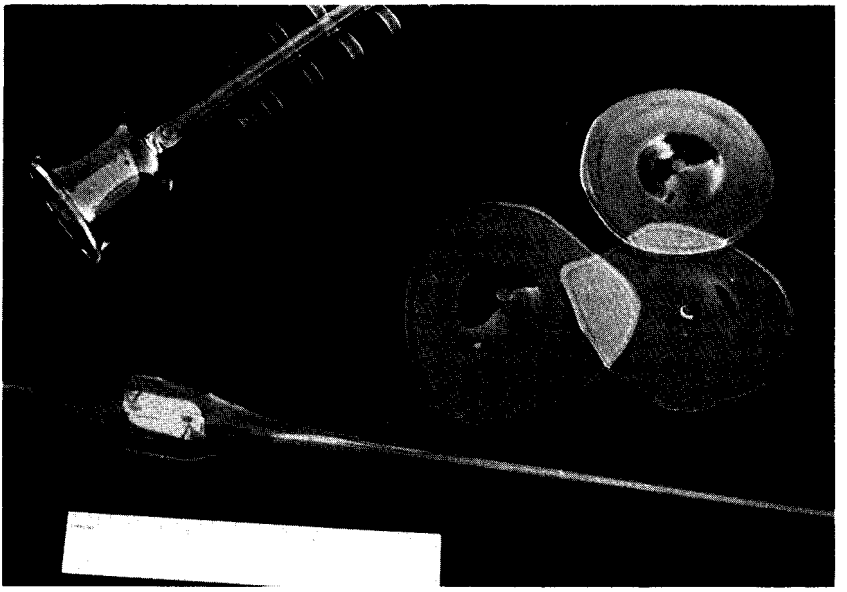

Figure V
Crown Glass

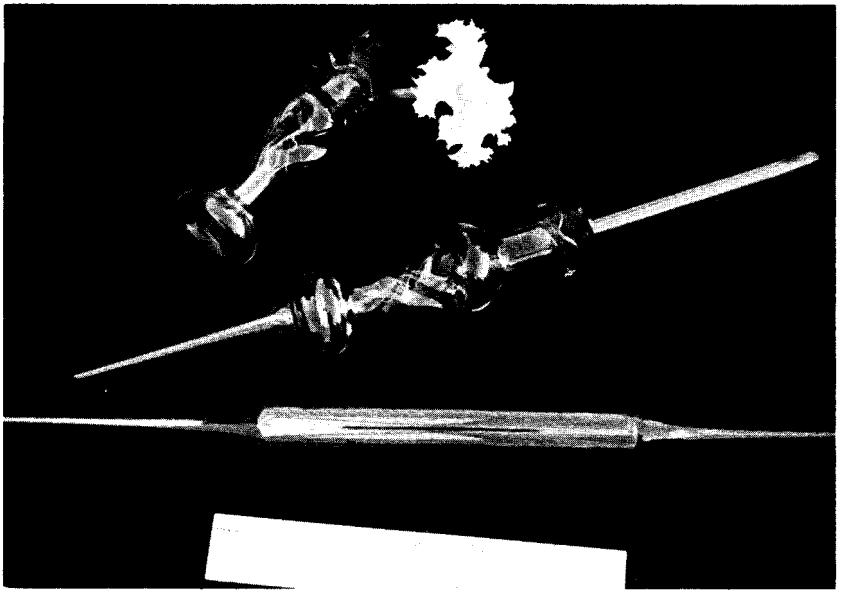

Figure VI
Flower Vase

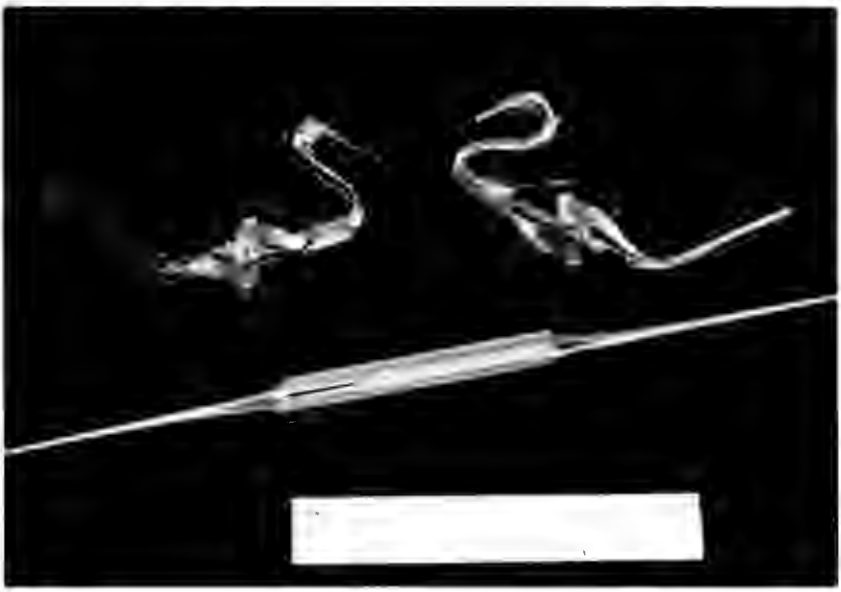

Figure VII
Lung Tester or Whistling Swan

WHEN STRIVING TO ACHIEVE ALMOST NOTHING CAN BE A CHALLENGE TO THE GLASSBLOWER

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Abstract

This paper deals with the vacuum rack, a responsibility that most glassblowers have encountered or will encounter during their employment as a scientific glassblower. The basic fundamentals of vacuum rack construction are known by most of us and the seals are straightforward but many of us work in laboratories where the chemist considers the glassblowers are expert in design, fabrication and leak detection.

The first part of the paper will touch on some basic fundamentals of vacuum rack design and the function of the components.

The second part will cover leak detection techniques and troubleshooting problems that occur when the rack is in operation.

The third part deals with safety tips and some devices which we have found beneficial in overcoming problems encountered in the lab.

Knowledge in vacuum technology can be a great asset to the glassblower when working with experienced research personnel, as well as in University laboratories, where the academic people have had little or no experience with vacuum systems. Hopefully there will be some information in this paper beneficial to the skilled glassblower as well as the apprentice, who might move into such an environment.

Basic Design and Functions

This paper will deal with what most of us in Chemistry labs consider high vacuum when the experimentalist hopes to attain an ultimate pressure of about 1×10^{-6} torr.

#1 These vacuum systems use a mechanical roughing pump capable of slightly better than 1×10^{-3} torr, liquid nitrogen cold traps, oil or mercury diffusion pumps, a manifold using high vacuum stopcocks and a pressure reading device. Selection of these components can be quite important depending upon the type of samples to be prepared. We are trying to get away from mercury pumps but there are occasions when the chemist objects to oil and it is also quite costly to convert the existing systems. Selection of stopcocks is also important where grease cannot be tolerated and in such cases either Teflon high vacuum stopcocks with "O" rings or metal needle valves may be preferred. When "O" rings are used, the "O" ring material must be compatible with the chemicals. Each of the pressure reading devices has its drawbacks, as well as its advantages, so the selection becomes a matter of economics or personal preference.

#2 The basic McLeod gauge is the most reliable but is inconvenient since the mercury must be raised and lowered each time you take a reading and must be trapped. It is useful down to about 1×10^{-6} torr.

#3 The cold cathode discharge gauge (Penning Gauge) is very convenient since it gives you a continuous reading from about 1×10^{-3} to below 1×10^{-6} but requires an undesirable outgassing period of time when initially installed or when cycling from a rough vacuum to high vacuum.

#4 An ionization gauge gives you a continuous reading and has a rapid outgassing feature but the cost of the power supply is prohibitive to many laboratories. The sensor using a filament which can burn out if the system goes to atmospheric pressure is a drawback.

Electronic pressure reading devices also have the disadvantage of malfunctioning and giving you an incorrect reading. My personal choice is a cold cathode discharge gauge plus a McLeod gauge which can be used to calibrate an electronic gauge that has malfunctioned or serviced. If both are to be used simultaneously, a trap must be installed to prevent mercury vapor from reaching the sensor; otherwise the reading will be the vapor pressure of mercury, which is about 1×10^{-3} torr.

Leak Detection and Troubleshooting

Once the vacuum rack is completed and there are no obvious leaks, the most common leak detection tool known to the glassblower or the experimentalist is the tesla coil. After starting the roughing pump and allowing some time to pump away the air and water vapor, the tesla coil should first be passed over each seal carefully keeping the probe close to the glass. If the spark appears to pass through the glass at any point, it indicates a leak and must be reworked. After determining that the system is leak tight, you may like to pass the coil over the seals again holding the probe far enough away from the glass to get the longest spark possible. You may puncture a few holes but if you don't the chemist will surely succeed once you are out of his sight. Try to explain to the research person that he punctured a hole rather than finding a leak. After being satisfied that the system is leak tight, the dewars can be filled and the diffusion pump started. Here is where patience must be advised especially when the inexperienced chemist expects the pressure to read about 1×10^{-6} in a couple of hours. We find that a new system requires nearly three days to reach this pressure as recorded on a cold cathode discharge gauge. If there is a rich pink discharge in the system when the tesla coil is applied at this time, you can suspect a leak but a bluish glow usually indicates water vapor.

Most problems occur after the rack has been in use or misuse in the lab. Many research people use coupling devices such as swagelocks, cajon fittings and "O" ring joints. Finding leaks at these points requires another method of leak detection. One technique involves using a tesla coil and a hypodermic syringe with acetone. If a pink discharge is present in the manifold, apply a small amount of acetone on each suspected area keeping the tesla coil a safe distance from the acetone or you may have a fire. Depending on the size of the leak, the pink glow will change greenish or disappear completely. This method is not suitable for grease or wax seals since they are soluble in acetone.

The last leak detection technique which I will describe is employed when you are using pressure gauges which depend upon ionizing the gases. These gauges are calibrated to read against the ionization potential of air so a reading would change if the sensor were exposed to gases of a different potential.

#5 By observing the slide it is easy to see why we should use helium gas. There is a significant difference between air and helium so, if we can flow helium gas past the leak, it should show up on our instrument as it replaces the air and reaches the sensor. This is exactly what occurs and the instrument indicates a lower pressure or better vacuum. Sometimes the response will be quite rapid and pronounced but with very small leaks it may be necessary to enclose the suspected area with a plastic bag to contain the helium.

The best method of leak detection is the helium leak detector but I will not devote any time regarding its use since you can consult the manufacturer's manual.

Troubleshooting vacuum rack problems is often tedious but can also be very rewarding if you keep a few notes and follow your self-styled attack. A very common experience for the glassblower is to have the research person inform him that he has a leak in his system when more times than not the problem is not in the glassware. Following are some of the problems encountered and the procedures we attempt to follow.

If the chemist uses an electronic vacuum gauge he suspects a leak when he gets a poor reading. If a quick check with the tesla coil shows no air glow, we can suspect a contaminated gauge sensor, a faulty instrument, low oil level in the roughing pump or contaminated oil in either the diffusion or roughing pump. It is very seldom the diffusion pump if a good quality of oil is selected. If an air glow is present, try to isolate the problem area by closing the stopcocks. A low oil level in the roughing pump, channeling of grease on ground glass fittings, deteriorated "O" rings or leaky ferrules on couplers may be the problem.

#6 Device for Safe Blowing Into Contaminated Systems

The device shown in the next slide we have found very useful in preventing moisture accumulation, keeping hazardous vapors from reaching the lungs and eliminating the possibility of air or oxygen combining with explosive vapors when repairing a rack. A nitrogen gas line is connected to the work piece and a blow hose connected to the tube exposed to the diaphragm. Adjust the gas flow so that there is a slight positive pressure exiting from the 3 mm by-pass hole when you blow against the diaphragm. Some final pressure adjustments are necessary when making the first seal but from then on you will find that intermittent puffing into the blow hose seems quite normal. This method is not as awkward as the "Y" tube method that you may be familiar with and I consider it safer.

Safety Blowout Ports

Since we had two explosions in our lab due to human error in shutting down a vacuum line, we have installed blowout ports at points where an excessive pressure might occur. In these two cases a fracture in the roughing line had occurred while the rack was unattended. When the broken line was found, a valve to the roughing pump was closed before removing the liquid nitrogen dewars from the traps. One theory was that liquid oxygen had accumulated in the traps and while warming, expanded; causing the explosion. Liquified gases can expand in excess of 600-1 when returning to the gas phase. The other theory was that there may have been a chemical reaction if the liquid oxygen had come in contact with the oil vapor from the roughing pump.

Liquified Gas Pressure Experiment

Using my annealing oven for an enclosure, I set up a simple system assimilating the apparatus and conditions leading to the accident. There is not enough time to go into the step-by-step procedure of the experiment but the experiment results are listed below.

#7 (System A)

1. No liquified gases formed in the trap while roughing pump was in operation even when system was open to the atmosphere (Stopcock "A" open).

2. With roughing pump off and Stopcock "A" open for 30 min about 25cc of liquid formed in trap.

3. When stopcocks were closed and dewar removed, pressure rose to 35 psi when ball joint released pressure.

4. Additional steps with the ball and socket sealed with wax resulted in the stopcock plugs blowing out.

5. After eliminating the stopcocks, the above step was repeated and the system sealed with a constriction before dropping the dewar. This resulted in a pressure rise to 275 psi when the glass system pulverized in an explosion.

(System B)

6. A simple 25 mm dia x 6" length vessel was tubulated with a 3/8" dia tube to replace the test piece and pressurized with a N_2 gas cylinder. The pressure rose to 490 psi when the vessel exploded.

General Conclusions

1. The ball and socket is a practical safety valve.

2. A slow pressure rise in glass apparatus will fracture it in the same manner as a chemical reaction.

As a result of our experience, we have printed a shutdown procedure for racks with cold traps and distributed one to each person with a rack.

A Protection Water Monitor

A cooperative effort between the glass and electronics shops produced a water flow monitor which we use on all water cooled apparatus such as diffusion pumps, distillation columns, etc. The device acts as a protection unit if there is an interruption of the laboratory water supply and also prevents flooding of a lab if a water line ruptures.

#8 The components required are a hydrostatic pressure switch used in washing machines, a solenoid valve placed in the water supply, an electrical box with receptacles and switches and a glass trap-like unit which serves as a sensor.

To put the system in operation, the feed line valve between the solenoid and the apparatus is opened and the water flow start by flipping a reset switch which opens the solenoid valve and allows the water to rise to the top of the overflow tube in the glass trap. The valve should be adjusted to avoid water waste yet enough flow to overcome fluctuations in line pressure. This head of water is enough to trip the diaphragm in the hydrostatic switch and when the reset switch is released, the water will flow until there is a problem. Should the water flow be interrupted the water level in the trip will fall, draining through the 3mm hole. This will trip the hydrostatic pressure switch which in turn closes the solenoid valve and breaks the circuit to the heating elements. A buzzer or bell can be incorporated in this unit which would warn the operator of a problem.

The water trap and hydrostatic pressure switch described above and shown on the right replaces the type shown on the left which used mercury contact with wires to complete a circuit. Frequently the wires became contaminated causing the unit to fail and mercury periodically spilled into the drain, collecting in the sink trap. These problems lead to the design of the present system.

#9 Detailed Scale Drawing of the Glass Trap

The size of the trap is important only to the extent that the head of water be sufficient to trip the diaphragm switch.

Devices to Prevent Mercury Spills and Vapors

There are still numerous pieces of apparatus used on vacuum systems requiring mercury such as diffusion pumps, toepler pumps, McLeod gauges, monometers, etc. We have taken several steps to prevent spills and reduce hazardous vapors.

#10 To lessen the danger of a ruptured boiler on a mercury diffusion pump, we have drilled a hole in the center of the heater pot which would allow the mercury to drain quickly as opposed to filling the hot heater cavity. A large coffee can with the bottom removed is used as a splash shield and a cake tin to contain a spill, attached with metal straps, leaving enough space for the electrical supply lead to pass between the can and the pan.

We enclose all other mercury vessels in coffee cans partially filled with plaster of paris.

Oil demisters should be installed on the roughing pumps to reduce the vapors being exhausted into the room.

Additional Safety Tips

Tape or screen enclose all flasks and dewars when possible.

Bake at annealing temperature all used glassware before reworking.

Always wear safety glasses around vacuum racks.

Whenever in doubt flush a used vacuum system with nitrogen gas before glassblowing.

Never trust the chemist who says that a system or used piece of glassware is safe to work on.

References for Vacuum Technology and Safety

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Safety in the Chemical Laboratory - Prepared by and published for the General Safety Committee of the Manufacturing Chemists Assoc., Inc., Wash., D.C.

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Author's Phone-Business 219-283-6443
Home 219-272-7867

Biographical Sketch of Author

Started glassblowing apprenticeship with General Electric Co., Schenectady, New York in 1946; left in 1952 to accept position at Oak Ridge Tennessee and accepted present position of glass shop supervisor at the University of Notre Dame in 1964.

Slides-10 35mm-Black and White

Aids-electric pointer

This paper has not been presented elsewhere nor has it been presented for publication elsewhere. As Mr. Blessing's employer I hereby certify its presentation.

Robert H. Schuler, Director

* The research described herein was supported by the Office of Basic Energy Sciences of the Department of Energy. This is Document No. NDRL-2009 from the Notre Dame Radiation Laboratory.

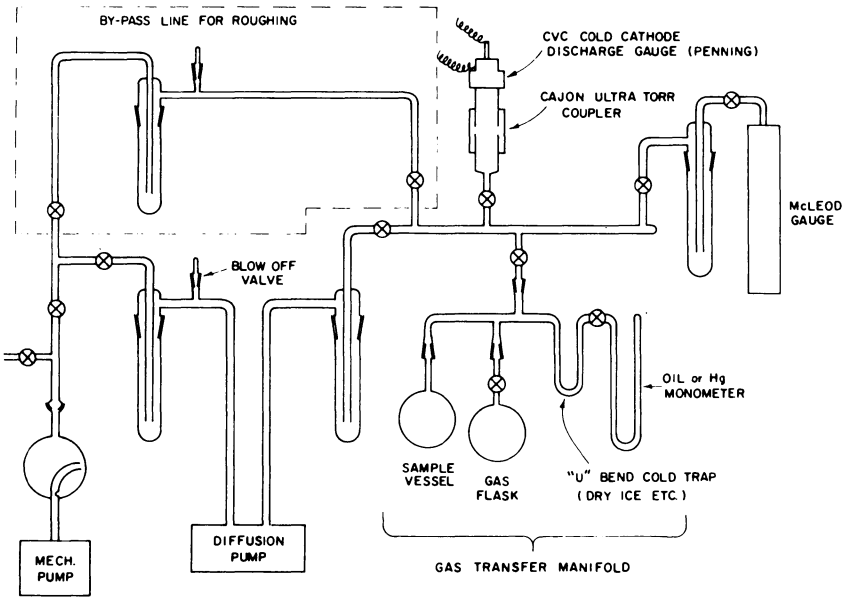
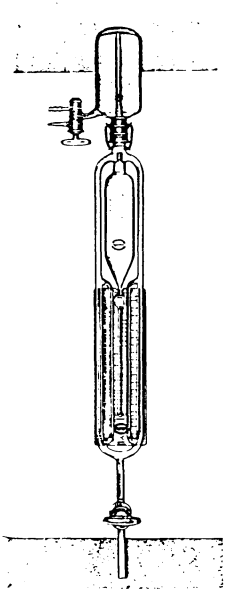


Figure 1



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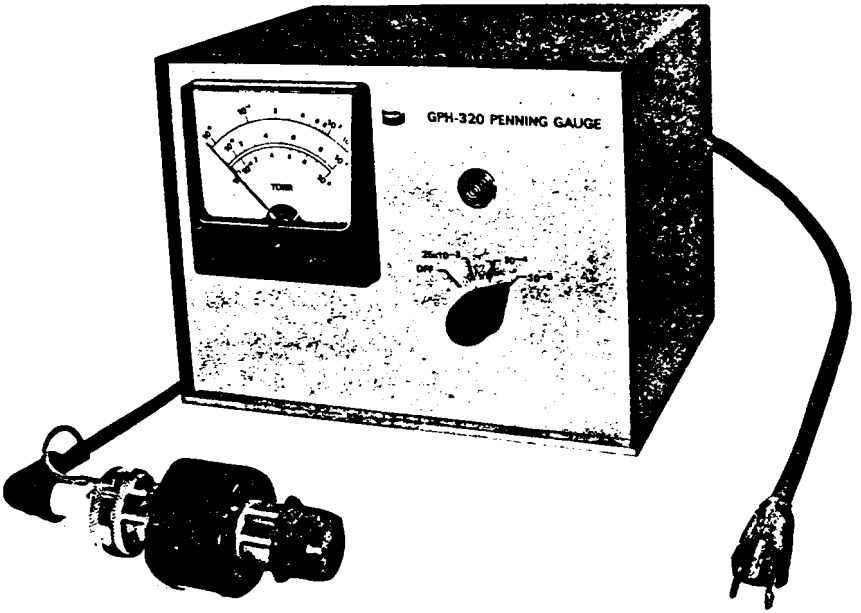

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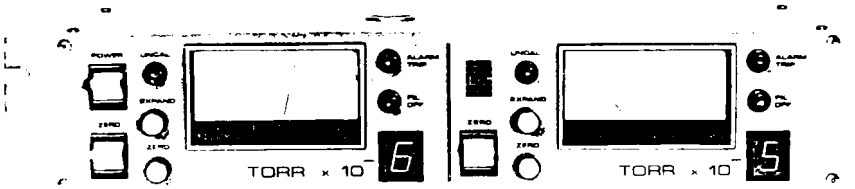


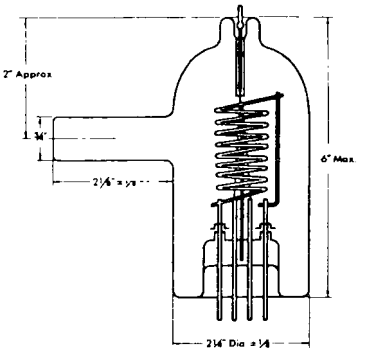


Figure 4

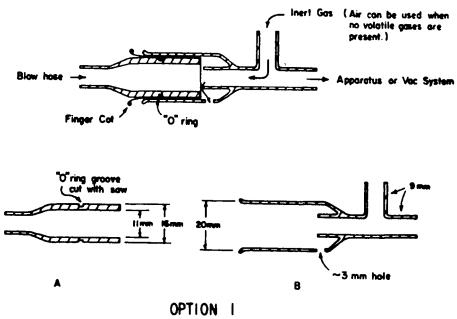
3.1 Sensitivity Of The Gauge For Common Gases

A Penning gauge calibrated for air will indicate a pressure for another gas in ratio f to the true pressure. The value of f is fairly constant for most gases over the greater part of the range in which the gauge is reliable.

Gas	f
Helium	0.21
Hydrogen	0.4
Carbon Monoxide	0.95
Nitrogen	1.0
Air (Dry)	1.0
Oxygen	1.23
Carbon Dioxide	1.23
Argon	1.39

To find the true pressure, the gauge reading is divided by f.

Figure 5



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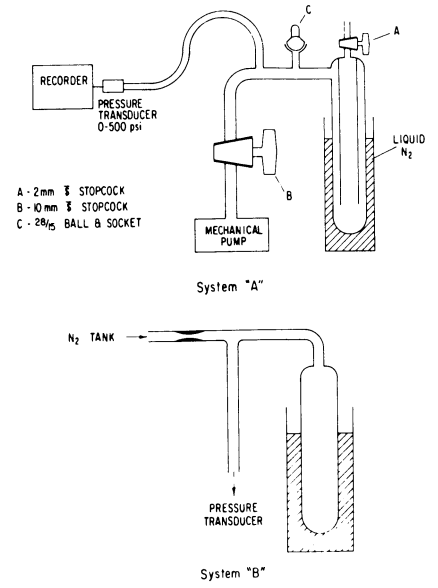


Figure 7

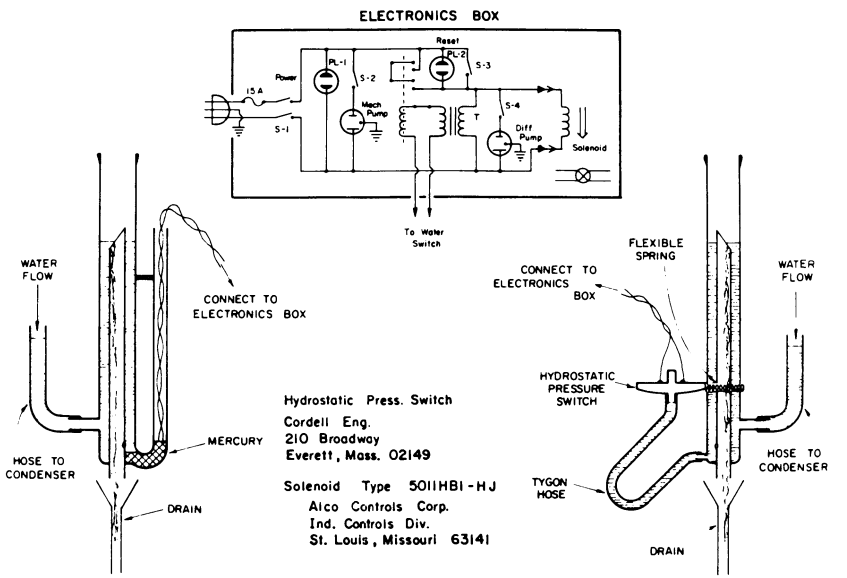


Figure 8

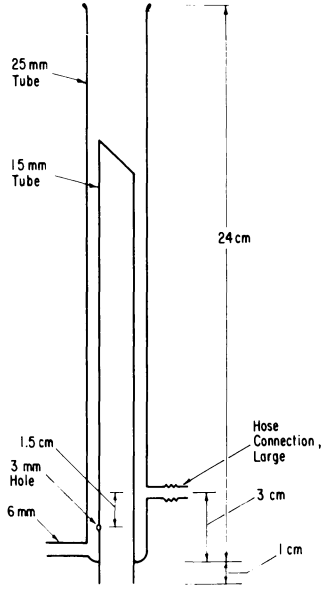


Figure 9

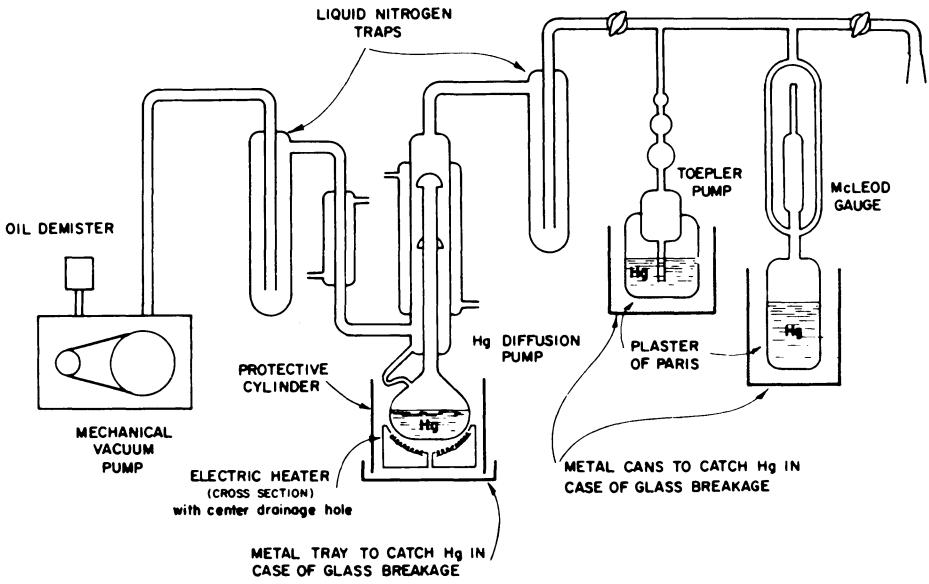


Figure 10

MANUFACTURE OF LARGE CHEMICAL STORAGE VESSEL

Joseph S. Gregar

In this presentation I will try to show, step by step, how to successfully achieve a completed storage vessel of extremely large size.

A Chemical Storage Vessel is a large cylindrical glass bottle with four flanged outlets, used for storing chemicals. #1 In the construction of a chemical vessel this large, the two difficulties of the glassblower are increased almost geometrically (in proportion to the size) of reaching and maintaining the heat needed to do the job, and trying to withstand that heat (on the body) when it is reached. This is the glassblower's main concern; taking for granted, of course, that he can accomplish the technical glassblowing details. The vessel must be hot enough at all times for successful completion.

To manufacture the vessel, the glass parts needed are: two-18" x 24" vacuum service bell jars and four - 2" conical pipe flanges. The other equipment will consist of: one - Litton Model K lathe with two planatary three jaw chunks, a variety of hand torches, a large vertical annealing oven with the internal capacity to anneal the completed vessel, and gas, hydrogen, and oxygen mixtures flowing through the torches.

The construction of the vessel was done in halves, taking one bell jar at a time; placing the various flanges on, and then splicing the jar halves together.

The first step is to put on a two inch conical flange on the rounded end of the first bell jar. To do this, an extremely large surface of the jar must be brought up to an overall temperature of at least 1600^{O}F. The temperature was measured with an electronic infrared thermometer. Once the correct temperature of the jar was achieved, the end where the hole was to be made was heated until the glass became softened. #2 A large glass rod was then used to pull the excess glass out, and the hole was pierced through. Piercing the hole was difficult because the bottom thickness of the jar was approximately 1/2" thick, making heat penetration difficult. After the hole was pierced, it was reamed up with a carbon rod to match the ID size of the flange. #3 At this point the bell jar had to be reheated because the overall temperature of the jar had dropped to 800^{O}F. When the jar was reheated to approximately 1600^{O}F, the jar hole and flange end were heated and then spliced together. To achieve a good splice, the two parts were heated until they reached a softened state and then joined together, concentrating the heat on the splice. #4 For this procedure, only gas and oxygen torches were used. When the two parts were joined, or fused, as one, it was a simple matter to then ream up the splice slightly with a long carbon rod. The splice is then completed. The jar must then be reheated to 1600^{O}F over a large area. #5 This flame will anneal the glass until it can be annealed in the oven.

The procedure just described, is then performed the exact way on the rounded bottom of the second bell jar, for attaching the flange. After completing the flange splice on the second jar, both jar halves are placed together in the annealing oven.

The next step is to attach the vertical flange which is six inches from the open end of the bell jar. This splice is more difficult than the previous flange splices, because the lathe cannot be used in aiding to make the vertical splice as it was used before. The first thing to do before starting this procedure for splicing, is to bring the jar up to proper temperature and then to #6 fire polish the end of the jar. The reason

for the fire polishing is because a sealed end is less likely to crack than a raw end. After fire polishing the end, the jar must again be brought up to 1600°F, so that a hole can begin to be pierced for the flange splice. A Litton hand torch with natural gas and oxygen was used at this point because it penetrated the wall thickness quickly. #7 When the area was hot enough, the hole was made by placing a solid glass rod on the spot for the flange and pulling out the glass. #8 With the wall of the jar pierced, the area was heated again and the hole was then formed with a carbon rod.

After the hole was made, the entire jar must again be reheated up to temperature in preparation of making the splice. The hole made in the jar must be perfectly matched to the ID size of the flange. Both glass parts were now heated at the same time. When the parts, jar and flange, were heated until a softened state, #9 they were then joined together working the torch around them. It is important to have a smooth interior surface on the splice to avoid having a cold seal. A Litton hand torch was used because it gave the best service for achieving a good splice.

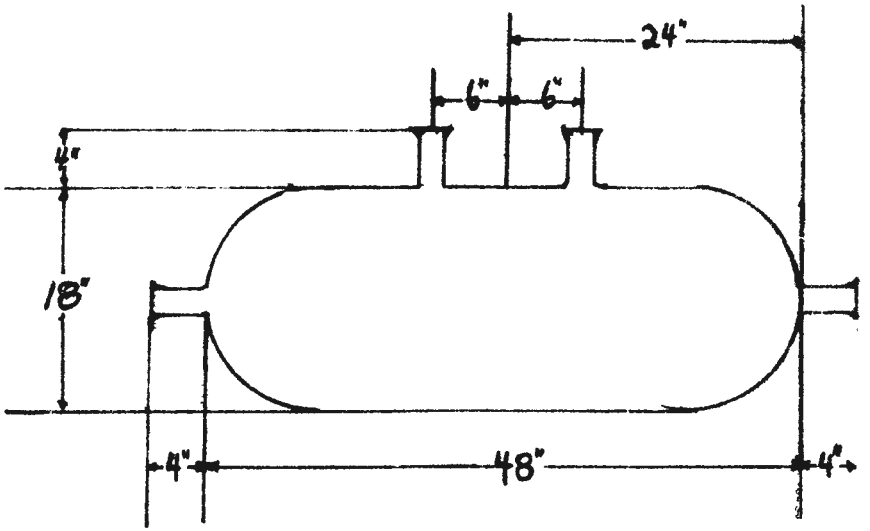
It is helpful at this point to use gravity by turning the jar down, #10 letting the glass flow in both directions. To insure that the ID of the splice is maintained, a carbon rod may be used to ream it out. The jar is again heated to 1600°F, and then slowly cooled to avoid breakage.

This process is duplicated on the open end of the second bell jar. When the second flange splice is completed, both jars are placed in the annealing oven.

The final step is to join the two jar halves together. This joining is aided by the use of the large capacity of the Litton K lathe, which held the two bell jars end to end. The gases used for this splice were hydrogen and oxygen through a Litton 12 tip - 7 jet ring burner. The two jars were brought up to temperature, at which point the lathe burners were turned on. #11 Both jars must be properly heated so that the heat has penetrated and reached the inside surface of the glass. When both ends of the jars are heated to a softened state, they are brought together and joined. The secret to this large splice is to have the lathe turning slowly, so that the centrifugal force does not bulge out the splice which will occur if the jars are turning too fast. #12 Continue to heat the joined area until both jars have blended into one and then use a carbon paddle to aid in making a clean, flat splice. #13 A large surface area of the jars is then reheated up to temperature to even out the strain patterns. Gradually the jar is cooled down over approximately 30 minutes.

#14 When the jar can be touched, it is carried to the annealing oven for it's final run. The Storage Vessel will then be completed.

To summarize, an extremely large Storage Vessel was created using only two-normal bell jars, four flanges, torches, and a lathe. I felt this procedure was not only interesting, but informative enough to be passed on to other glassblowers. It will let them know it can be done because it has been done, and may aid them if they should want to fabricate a similar vessel.



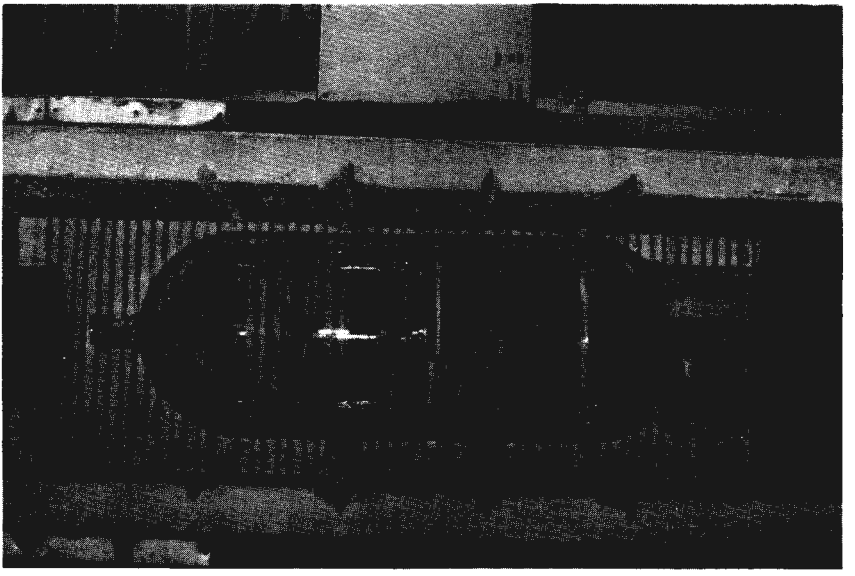


Figure 1

Figure 2

Figure 3

Figure 4

Figure 5

Figure 6

Figure 7

Figure 8

Figure 9

Figure 10

Figure 11

Figure 12

Figure 13

Figure 14

THE FORMING OF HIGHLY TOXIC FUMES IN THE AIR WHILE WORKING WITH GLASS

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V.P.I. & S.U.
Blacksburg, Virginia 24061

Everyone knows that a good ventilation system in the glass lab is essential because of the silicon oxide (SiO_2) and boron oxide (B_2O_3) developed when working with glass and especially quartz.

There are, however, other hazards to be considered when working glass and these hazards are too often overlooked.

The high temperature of the flame or other high heat sources can produce rather toxic gasses. High temperatures promote the unification of the nitrogen and oxygen present in the air and both gasses can form oxides of nitrogen. The most common combinations that are formed are:

Nitrogen Dioxide (NO_2)
Nitrous Oxide (N_2O) and
Nitric Oxide (NO)

Both the N_2O and the NO are colorless. The NO_2 is yellowish-orange and produces an odor very similar to the fumes of nitric acid. According to the following chemical reaction the nitrogen dioxide, when mixed with water, produces nitric acid.

$$4\ NO_2 = 2\ H_2O + O_2 \rightarrow 4\ HNO_2$$

This means that the NO_2 in the air, when inhaled, can mix with the moisture in the lungs to produce nitric acid. Practically any nitrogen-oxygen combination at high temperatures, together with the presence of water, could form nitric acid and therefore the NO_2 is not the only gas to be careful with in the glass lab. The American Council of Governmental Industrial Hygienists (ACGIH) publishes annually a list of so-called Threshold Limit Values (TLV's) and according to this information only 5 ppm (parts per million) of nitrogen dioxide is permissable in the air. The nitric-oxide level is set at 25 ppm and for the nitrous oxide or laughing gas, no limit is provided at this time.

The figures given by the ACGIH are based on the best information available and reviewed annually. The TLV's are considered as good-practice guidelines and have been accepted as official U.S. Government standards for industrial air control. OSHA or the Occupational Safety and Health Act follows the same regulations by law.

If a glass lab has insufficient ventilation, the 5 ppm of NO_2 will easily be surpassed.

Recently, a room of approximately 142 m^3 with practically no ventilation present was tested for this purpose by letting three benchburners of the type Carlisle (cc),* burn at approximately 3/4 of full capacity. Obviously, this test was somewhat exaggerated in comparison with actual working conditions.

A sample of the air was taken at 0 time and then every 5 minutes thereafter over a 15 minute period. The graph in Fig. I explains the results rather clearly, but also indicates that the ppm of NO_2 in the room just about doubled every 5 minutes. After 10 minutes a yellow haze was visible and after 15 minutes the room was filled with a yellow smog of highly toxid fumes that darkened as time went on.

A second test, that represented more normal circumstances, also showed above permissable amounts of NO_2 in the air (Fig. II). In this case, one torch was on at 3/4 of its full capacity, while the other two used the small middleflame only.

The nitrogen oxides formed this way are generally irritants, causing congestion of the throat and bronchi. Concentrations of 60 to 150 ppm causes almost immediate irritation of the nose and throat, with coughing. Some hours after exposure a sensation of tightness and burning in the chest can develop. Shortness of breath, sleeplessness and restlessness may also occur and finally loss of consciousness followed by death.

Techniques for Testing a Glass Lab for Oxides of Nitrogen

The ultimate way is to analyze an air sample by means of a mass-spectrometer. The mass-spectrometer method can detect traces of various gasses simultaneously in a relatively short period of time.

In principle, a one liter round bottom flask is provided with a greaseless stopcock and a standard tapered joint or other connection for adaption onto the mass-spectrometer apparatus. The flask is evacuated to approximately 10^{-4} torr, the stopcock closed and the testbulb removed from the vacuumline. (Fig. III)

The testbulb is brought into the room where the air needs to be analyzed and, by opening the stopcock, a sample of the air will be sucked into the evacuated bulb. The stopcock will be closed again in a few seconds.

Next, the bottom extention of the sample bulb is dipped into a low temperature mixture of dry-ice and actone while again on the vacuum line. This in-between manipulation will freeze the collected gases rapidly while most of the air in the bulb can be pumped off via the vacuum system. What's left over, after removing the cold mixture, is a purer gas that needs to be analyzed and consequently the final results of the test will be more accurate.

The readouts of the mass-spectrometer can be compared with a standard in order to calculate the final results of the air-sampling test.

Of course, not everyone has access to such expensive equipment and, therefore, other sufficiently accurate and less expensive methods are available. The next best and fastest way to determine the presence of toxic gases and in particular NO_2, is to utilize

*Product of the Carlisle Gas Burner Corporation

a Gastec Multi-Stroke Gas Sampling Pump* with Detector Tube No. 9L. The gastec detector exists out of a small section of glass tubing with inside of it a known prepared chemical that will discolor when it comes in contact with NO_2 containing air.

The glass sampling tube is connected onto a handpump and when the pump handle is pulled out to the end, a 100 cc air sample has passed through the chemical inside the glass. Since the sample tube has a logarithmic scale fired on the outside of the glass, we can immediately read the ppm of NO_2 because the color of the chemical changes progressively from top to bottom.

The difference in taints between the original material and the NO_2 absorbed material is clearly distinguishable inside the sample tube and a direct reading is possible.

CONCLUSION

It is obvious that the rather toxic NO_2 is easily produced and there is very little that one can do to prevent its development in the air under favorable circumstances. It's recommended to test your lab for the presence of toxic gasses developed by the heat of glassblowing torches or other high-heat sources under the various conditions that occur in your glassblowing operation. It would be impractical to make mathematical recommendations along this line, because the room size, ceiling height, number of heat sources, location of ventilation equipment and others differ from one glass lab to another. Therefore, adapt your ventilation to your own findings and needs until you reach a safe standard of air analysis in your workroom.

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*A product of the Bendix Corporation

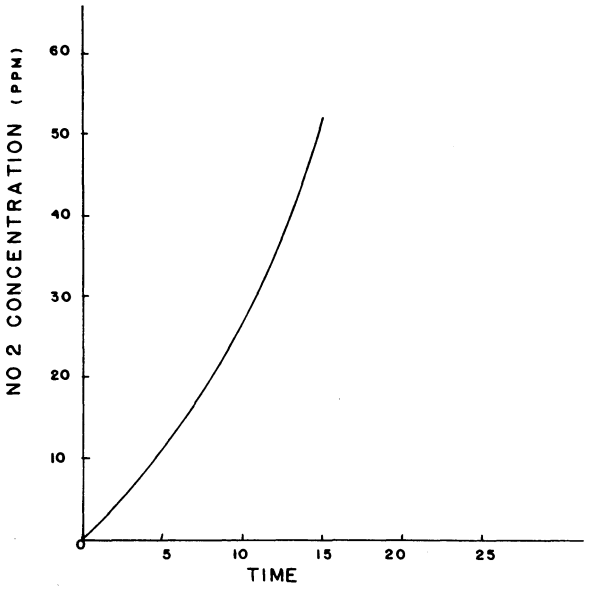

Figure 1

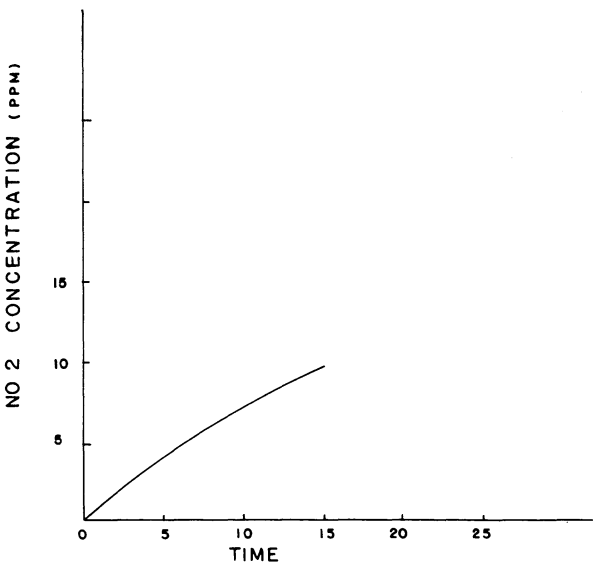

Figure 2

Figure 3

HOMAGE TO THE DISCHARGE TUBE

GEORGE BATLEY

The Lab-Crest Subsidiary of Fischer and Porter Company publishes a quarterly newsletter titled "Specialty Glass Items" and one of the regular features in this publication is a column, "From a Grain of Sand," with implication that all things have roots in humble beginnings.

Many of the complex products we fabricate from glass were at one time just a handful of sand if one stretches the imagination, and going a step further most of the sophisticated envelopes we make to house electrical, electronic or optoelectronic devices are in reality just modifications of the original "gas discharge tube."

In an early article for "Grain of Sand," we paid homage to the discharge tube as progenitor of some of man's more spectacular scientific achievements. At your Program Chairman's request, this paper is the full manuscript from which that column in "Items" was condensed, with some revision for this particular audience.

No one knows exactly who created the first discharge tube, for years just a laboratory curiosity, a scientific toy with electrodes sealed into the ends of the glass tube and with provision for evacuating the chamber. Faraday and Crookes did much of the pioneering work in attempts to explain the various optical and electrical effects produced at different stages of the evacuation process and certain phenomena still carry references to their names in tribute.

Goldstein is credited with coining the term "cathode ray" to describe the principal effect produced and this terminology persists today in limited usage, although it is now much more common to refer to these particles as beta rays or electron beams: they are all basically the same.

Several of you here may have had first experience with a discharge tube in Sign-shop practice for the Electric Sign industry was one of the first to exploit the principles on a commercial basis and many of our talented glassblowers of today learned their preliminary skills in these Shops. The electric sign is a discharge tube: a controlled electrical discharge through a confined rarefied gas atmosphere.

Thompson is credited with one of the first significant modifications of the discharge tube which he achieved by incorporating a small aperature in the center of the anode and a second chamber that permitted particles escaping through the aperature to be influenced by varying electrostatic or magnetic fields.

This modification permitted determination of the relationships between mass and charge, and ultimately to the discovery of the electron as a basic constituent of all matter. Coolidge made an important contribution in further modification of the Thompson tube by substituting a hot cathod system to produce electrons directly in a completely evacuated chamber. It is much more widely used today and we can say that hardly a home is without one for the Coolidge version is essentially the picture tube of our TV sets.

In experimenting with a discharge tube, Röntgen made a starting discovery, a different type of particle form exhibiting anti-cathode tendencies which he termed x-rays because of the then-unknown properties. Although the initial discovery was

entirely accidental, his follow-up is a classic in systematic scientific procedure and reporting and the speed with which his findings were put to commercial use is also unparalleled in scientific breakthrough. Within three months after the discovery was made known, x-rays were in general use in hospitals for diagnostic study of bone structure. Today Röntgen's x-ray is recognized as the equivalent of gamma rays emitted by naturally radioactive materials or by synthetically produced radioactive isotopes of almost any element.

Rutherford's name is associated with several important findings. In study of alpha rays emitted by naturally radioactive materials he is credited with discovery of the proton as another particle form produced in a discharge tube, a discovery that was instrumental in postulating theories connected with the nuclear makeup of the atom. His particle accelerator, a linear drift-tube, was just a modified version of the Thompson discharge tube, but forerunner of several ingenious means for particle acceleration and atom-smashing. With two assistants, Geiger and Marsden, in study of scintillations produced by ionic collisions within the nucleus of an atom they developed the first practical scintillation counter, another sophisticated version of the discharge tube. Rutherford's later studies of Soddy led to the transmutation theories of natural and induced radioactivity, the groundwork for subsequent nuclear fission and nuclear fusion developments with far-reaching changes in the fields of physics and chemistry.

Chadwick discovered the neutron, Yukawa the meson, both associated with revolutionary concepts of light. Kelvin's name is associated with the travelling-wave tube, a further sophistication of the drift tube and another accidental discovery developed from the explanation of peculiar behavior of a frightened horse. The same principles that allowed the horse to tow a loaded barge effortlessly down a canal could be used to permit an electron beam to piggyback on an electrical signal in much the same fashion that a surfer rides a comber to shore. Crookes' name crops up again in pioneering efforts leading to discovery of the photomultiplier tube, but the first practical model is attributed to Curran and Baker. The photomultiplier tube, the maser, and the gas laser are all complex versions of a discharge tube.

When we reflect upon the tremendous accomplishments developed from such a humble beginning as a discharge tube, a laboratory toy, we must also pay tribute to those unknown and unsung glassblowers whose skill and expertise made possible the prototype housings that permitted pioneering experimentation. Today as we try to harness solar energy, manipulate lased beams of light, combine electric, electronic, or optoelectronic signals with computer and closed-TV technology, the role of the glassblower has never been more important. Improvements in glassworking techniques, the marriage of glass, metal, ceramic, and even plastic components is opening doors for newer, better and more sophisticated developments. The future is indeed rosy.

In closing I would like to acknowledge the source of much of the material in this paper from "Physics for the Modern Mind" by Walter R. Fuchs and "A Layman's Guide to Atomic Physics" by J.M. Valentine.

About the Author. . . . . .

Mr. Batley has more than forty years industrial experience in the glass field, more than half spent in various technical positions with Corning Glass Works following education at Alfred University where he majored in Glass Technology. Other former employers were GTE-Sylvania and the Fredericks Company. He has specialized in development of new products and new processes, in which capacity he has assisted customer engineering and manufacturing staffs in more than three hundred industrial plants in the U.S., Canada and Mexico. He is currently employed at the Lab-Crest subsidiary of Fischer & Porter as Manager of Glass Research.

SUGGESTED FORMAT FOR A GLASSBLOWING DEMONSTRATION

Donald E. Lillie

Several times I have been surprised by glassblowers who have called me and have asked for assistance and suggestions for a glassblowing demonstration. Since we are such a select few we have a public and social responsibility to extend our knowledge and ability for public consumption. Fortunately, we have the most mystic and fascinating material known and has for centuries intrigued the average person. Whether it is civic, social, educational or fraternal, accept that invitation for yourself and your profession.

When addressing a group, make sure you are enthusiastic and sincere. Do not worry about being nervous since this has a tendency to make a more energetic speaker. Take command of the audience; make sure you are positioned so you can be seen and heard. Also attempt to eliminate any distractions if at all possible and be confident of yourself and your subject. I have chosen the technique of displaying one object at a time which is withdrawn individually from the case that I carry them. This circumvents the group being distracted by objects arranged previously on a table.

Today let's talk about the origins, history and applications of this glamorous, glistening material called glass. Nature manufactured it millions of years ago in the bowels of the earth in the form of obsidian and spewed it out of the volcanoes to solidify and become a material ancient man used for knives, arrowheads and even money. What do I mean by glass? I mean a substance that is a supercooled liquid; it does not melt at a specific point but gradually softens over a large temperature range. It is also noncrystalline which is demonstrated by its poor conductivity qualities. Besides a functional object, man ground and polished it into artistic items such as this obsidian idol.

Man's first endeavor to actually make glass was in the form of a glaze on small stones. The origin is probably attributed to the ancient copper smelting furnaces or tribal secrets such as beehive iron fusing techniques. Then for nearly three thousand years man ingeniously fabricated glass into many shapes before he discovered glass blowing. About 100 BC the Syrians, using a hollow metal pipe, began fashioning hollow vessels by simply blowing the moltened material into various forms. By the third century the Roman Empire had perfected all the known forms of decoration except that of using Hydrofloric Acid.

The epitome of glassblowing was achieved by the Venetians. In the 14th and 15 centuries they cleverly cornered the world glass market. They perfected the millifori technique. This vase has over ten different colors which must be thermally compatible.

The first industry in the New World was glassmaking, for in 1607, in Jamestown, Virginia, imported glassblowers were producing bottles such as this. Since labeling devices were unknown, the glassblower was required to imprint in a glob of glass on the side of the bottle an indicator. From this round configuration the bottle evolves to the familar early American flask. Our forefathers were the only society in the world that put presidential figures and political slogans on whiskey bottles. From China comes a selfclosing bottle. An imbedded marble is seated by internal pressure to close the container without the use of corks or caps. Another interesting bottle from China is this snuff bottle. It is blown, coated, and engraved before the artist paints this detailed picture on the inside of the bottle.

Glass was the material that man used to look into the heavens, for Galilee invented the telescope using glass lenses. Leeuwenhoek, the Dutch Scientist, employed a small spherical lens to make his first microscope. Imagine his amazement when this simple device magnified specimens one hundred times!! Man has used glass for centuries for humorous things. You are all familiar with the yard long ale glass or the glass boat. I have a replica of a German trick drinking glass. Once filled with ale, the poor victim would try many approaches before discovering a vent in the deer's nose and a hollow antler to act as a straw.

Man uses glass because it is transparent, rigid and chemical resistant. Here, I have a small tube partially filled with a liquid which when flipped makes a "clicking" sound. This is an example of the water hammer phenomenon. Without glass this simple reaction could not be truthfully presented.

The obvious question one asks is "What is the difference in the cost and composition of glass"? Quite simply, if a machine makes an object it usually is inexpensive and the glass is mass produced. However, if extreme care and selection is taken in the condition of the raw materials and the resultant piece is hand formed and hand finished, then the price will reflect these prerequisites. This goblet is hand formed so the thin bell, when struck, will give a clear, tonal ring. Also the personal polish given this piece when finished will command a $50 price tag and deserve the name "Stueben." At the turn of the century, a man by the name of Michael Owens invented a machine which could mass produce containers and simple items. So glass became man's servant and has benefited nearly everyone. In Corning, New York, a machine blows these light bulb blanks from a moving ribbon of hot glass at the rate of 2000 per minute. Flasks for laboratories are machine molded, such as this Erlenmeyer blank.

This whimsical material is said to be perfectly elastic. This blue glass coil will show that even though it is oscillated millions of times it will not fatigue or elongate. It can be made strong and durable. By controlled chilling, a glass hammer five times stronger than annealed glass can be made. Then a glass nail can be driven into this wood by use of this unusual implement. This same material in the hands of an artist can be sculptured into an expression of form such as this dogwood triple blossom.

In the future, glass will serve you in many new ways. A recent achievement is this Pyroceram dish which originally looked like this brown dish for ease of manufacture. Subsequent heating forms a space age glass ceramic.

As a highlight of your demonstration, you can actually make some small item which should not consume more than five minutes, such as a mouse, vase or traditional swan. Other things one might do is quench a quartz capsule to show thermal durability; temper a heavy walled tube to make a "baloney bottle"; draw a fiber or blow a thin bulb. The approaches are many and should be slanted to the interest of the audience. One should be careful to not field more than three questions from the floor. Remain after to answer any additional inquiries. Conclude your presentation with a forceful analogy relative to the group. Remember, being a dynamic speaker and presenting a memorable demonstration is a never ending undertaking.

A.S.G.S. 24th Annual Symposium

TECHNICAL PAPER ABSTRACT

by

John T. Balkwill

Manufacturing Techniques for Microchannel Plates and Their Application in Night Vision Image Intensifiers

A microchannel plate is a multichannel electron multiplier. The basic structure will be described as also will the operation of image intensifiers incorporating these devices.

A detailed description of the manufacturing procedure will be given showing the number of steps involved and stressing the importance of dimensional accuracy and cleanliness throughout the operation.

In conclusion, photographs taken at night with and without a night vision scope (image intensifier) will demonstrate the advantage of this device.

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MANUFACTURING TECHNIQUES FOR MICROCHANNEL PLATES AND THEIR APPLICATION IN NIGHT VISION IMAGE INTENSIFIERS

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INTRODUCTION

The microchannel plate is an array of continuous dynode electron multipliers. (Fig. 1) It consists of millions of microscopic hollow glass conducting channels fused into a thin disc. Since each microscopic channel represents a separate high gain electron multiplier, and perfect position registration exists between the input and output faces of the MCP for each channel, the MCP is ideally suited as an imaging electron multiplier. Four main sizes are manufactured: 18mm, 25mm and 40mm. The 18mm has 1,815,565 channels, the 25mm has 3,336,253, the 30mm has 4,428,029 and the 40mm has 7,956,805. The channel diameter is 11.5 microns; center-to-center spacing is 15 microns and length-to-diameter ratio is 45-1 (plates are approximatley .020" thick).

The main application of MCPs is in image intensifiers used in night vision devices. These devices enable the user to literally "see in the dark." Figure 2 shows the principle of operation of an image intensifier with a microchannel plate being used as an electron multiplier. Light from the object X falls on the photocathode which is a light sensitive layer on the inside of the fiber optics window. Photoelectrons, generated at the photocathode, impinge on the input side of the channeled array. The resulting secondary electrons are accelerated down the channels when voltage is applied between input and output electrodes of the MCP. These secondary electrons collide with the channel walls dislodging additional secondary electrons thereby producing electron multiplication or amplification. This phenomena is called electron "gain" and can be varied by adjusting the DC voltage to the MCP. Gains of several thousand are typical. Figure 3 shows a hand-held night vision scope. The scale shows that the device is about 4" long with conventional optics for viewing and focusing. Figure 4 is the image intensifier tube which forms the main body of the device. Figure 5 shows night vision goggles which use two image intensifier tubes with 18mm MCPs. Photographs illustrating the operation of these devices and taken through the hand-held scope will be seen at the end of the MCP fabrication and processing section of this presentation.

FABRICATION

Manufacture of microchannel plates can be divided into the following operations: Fabrication, Plate Preparation (Lapping & Polishing), Chemical Processing, Hydrogen Firing, Electroding, Final Test and Q.C. Inspection. All areas will be covered but only the fabrication will be dealt with in detail. The MCP is made from a high lead content glass, approximately 50% PbO_2. The main principle of the fabrication is the same as in fiber optics and (Fig. 6) shows the basic steps involved.

1. Single Fiber Draw
2. Multiple Fiber Draw
3. Boule Assembly, Press and Slice

4. Optical Finishing (Lapping & Polishing)
5. Electrical Processing (Etch, H_2 Fire, Electrode)
6. Electrical Test and Final Inspection

The starting material is lead glass tubing 1.350" in diameter with .150" wall. The core glass which is used to support the tubing during the fabrication is slightly less than 1.00" diameter and is a lanthanum optical glass, soluble in hydrochloric acid. The rod is held inside the tubing by a steel fixture which is also used to hold the assembly in the fiber drawing machine. The glass tube - rod assembly is preheated in the draw machine oven and slowly fed into the hot zone which is controlled at approximately 720 C. The rod and tube fuse together and the hot end drops off forming a fiber. This fiber is pulled by hand and fed into the hard rubber draw wheels. (Fig. 7) The draw speed is adjusted manually until the fiber is the right diameter (.030"). The machine is switched to automatic at this point and the fibers are cut to length, removed by the operator and gaged. One operator can run two machines quite comfortably. The single fibers are carefully cleaned before being packed into an adjustable carbon mold to form a hexagonal bundle which is heat treated in a nitrogen atmosphere until all the fibers (2437) are fused together. This assembly is called a tacked single.

The tacked single is hung in a fiber drawing machine in the same way as the single fiber rod and tube, and fibers are drawn in the same manner except that the temperature control is more critical in order to maintain the hexagonal geometry of the fiber. The fiber size is .0285" and is still a good hexagonal shape with the same number of single fibers in it (2437). Figure 8 shows the end of the tacked single being removed from the machine. Note the conical shape with the fiber still attached.

The multiple fibers are gaged on a machine which measures all three flats at one time over the whole length of the fiber. After gaging, the fibers are cut to length, cleaned very thoroughly and then packed into a twelve sided configuration in an adjustable fixture. Extra fibers are added as the assembled fibers are transferred to a precision bore lead glass tube which will eventually be the solid border of the MCP. This assembly is placed inside a lead glass envelope which has a metal tube sealed to one end. This envelope is sealed together on a vertical sealing machine (Fig. 9), and when cold the whole assembly is inserted in a metal pressure vessel with the metal tube attached to a vac-ion system. A furnace (Fig. 10) surrounds the pressure vessel and the temperature is raised slowly to the softening point of the lead glass. With a high vacuum inside (5×10^{-6} torr) and an external pressure of 150 psi, the fibers fuse together and are pressed into a solid glass "boule" in which the fibers still maintain their hexagonal cross section. Because of the vacuum and pressure, no voids or bubbles are included. This method is called "isostatic" pressing and is used because the glass can be pressed at a lower temperature. After annealing and cooling, the "boule" is removed from the pressure vessel and the ends which are somewhat distorted, are removed on a diamond saw ready for the next step which is grinding the boule to the correct diameter. After grinding the boule to size, it is waxed onto a lava or ceramic block, mounted in the slicing machine (Fig. 11) and cut into discs by the abrasive (400 grit silicon carbide) which is fed in slurry form onto the steel blades. The blades oscillate at a controlled rate and the boule is pushed against the blades by a vertically mounted air piston. The plates are sliced at an angle for electrical reasons and therefore they have to be rerounded, at the same time the edges are bevelled to remove the sharp corners. The machine used is a Bothner lens centering machine. The wheel is a metal bonded diamond wheel (Fig. 12).

Bevelled plates are loaded into a planetary lapping machine in carriers which allow the plates to be lapped on both sides at the same time. The grinding media is AlO_2 and the grit size is 5 microns. After lapping to the correct thickness the plates are removed, thoroughly cleaned and then polished in a similar machine which has polishing pads instead of a metal surface and the polishing media is cerium oxide.

The plates are now ready for the chemical processing, hydrogen firing, electroding and test. Etching is done in large containers of hydrochloric acid, diluted to suit the etch time. For example, 5% for an overnight etch. The plates are held in quartz racks giving maximum exposure of the plate surface to the acid. When all the core glass is removed, the plates are subjected to other etchants to prepare the channel walls for activation. After final rinsing and drying in a vacuum oven the plates are transferred to metal fixtures and placed in a hydrogen furnace. The hydrogen firing is critical and temperature and time can be varied to adjust the electrical parameters of the plates.

The plates are black when they are removed from the hydrogen furnace and the glass is conducting. In order to make electrical connection to the channels, and to connect them all in parallel, a thin metal film is evaporated onto both sides of the plates in a vacuum bell jar.

Electrical test is done in a vacuum chamber which simulates the geometry of the final image intensifier tube. All electrical measurements are recorded and a visual inspection of the output display is made for non-uniformity, spots, shading, etc.

The following pictures demonstrate the use of a night vision device attached to a normal camera.

A. Lab Technician	No light with N. V. D.
B. Front of House at Night	Thru N. V. D. - Night light in bathroom
C. Street Outside House	Thru N. V. D. - Street light - 2 blocks

ACKNOWLEDGMENT

I would like to thank Varian Associates for the opportunity to present this paper and my colleagues who helped with the preparation.

LIST OF ILLUSTRATIONS

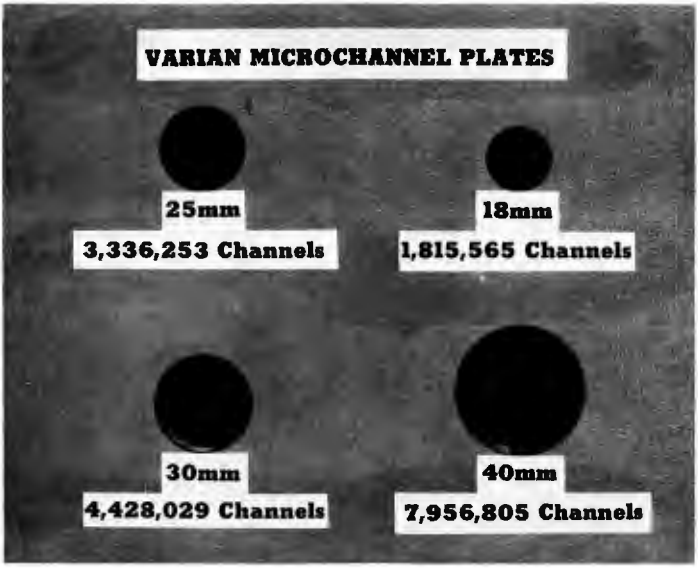

Figure 1
Microchannel Plates

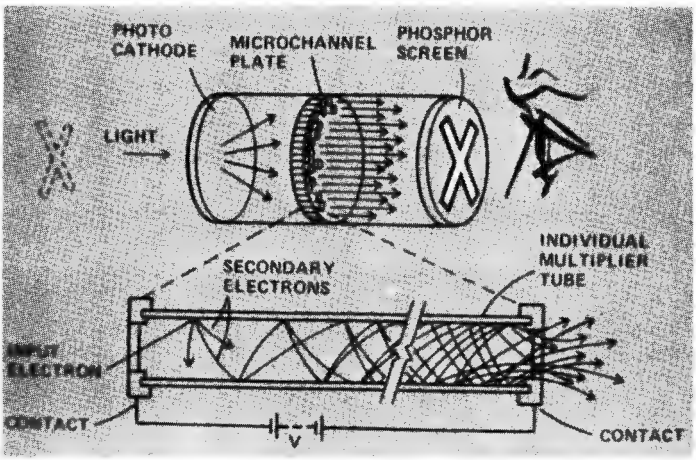

Figure 2
Image Intensifier Schematic

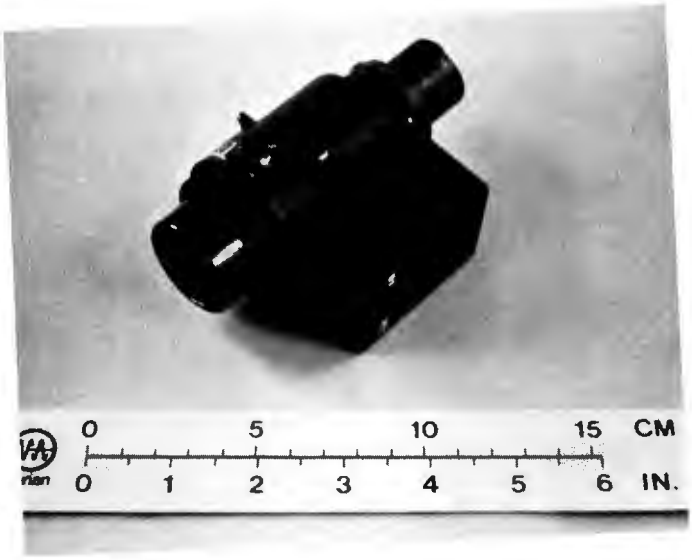

Figure 3
Hand-held N. V. Scope

Figure 4
Image Intensifier Tube

Figure 5
N. V. Goggles

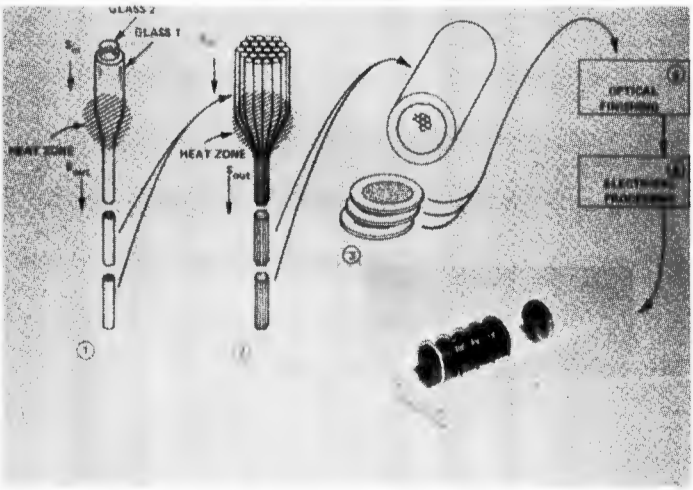

Figure 6
Fabrication Basic Steps

Figure 7
Fiber Drawing Mechanism

Figure 8
End of Multiple Draw

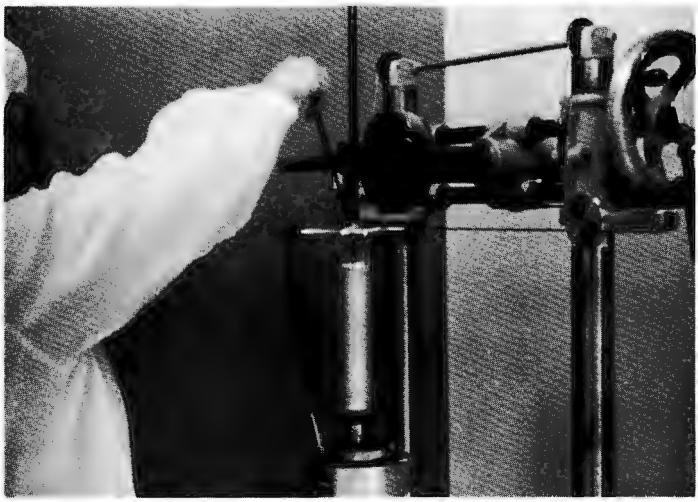

Figure 9
Vertical Sealing Operation

Figure 10
Boule Press

Figure 11
Boule Slicing

Figure 12
Re-rounding and Bevelling

A. Lab Technician — No light with N. V. D.

B. Front of House at Night — Thru N. V. D. - Night light in bathroom

C. Street Outside House

Thru N. V. D. - Street light - 2 blocks

The Application of Glass Technology to Novel Solar Energy Collectors*

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INTRODUCTION

In the last decade solar energy has enjoyed a tremendous increase in interest as a potential alternate energy resource. Unfortunately, solar energy though abundant, is quite diffuse. The peak solar flux at the earth's surface amounts to only 1000 watts per square meter, or about 300 BTU per square foot per hour. Even modest solar energy systems require large areas of collector to intercept sufficient solar radiation. Special attention must be paid to the collector, therefore, if the system is to be cost-effective. Due to the renewable nature of solar energy, the goal is to minimize the cost per unit of energy delivered, measured on a life-cycle basis.

For the solar thermal collector designer, this goal can be translated into four objectives. He must maximize the optical performance of the collector in order to absorb as much of the intercepted solar radiation as possible. He must minimize the thermal losses which occur once the radiation has been absorbed and converted into heat. He must minimize the cost of the collector so the initial investment is low, and he must maximize the durability of the collector so the cost can be amortized over a long time period. Clearly, these objectives are not particularly compatible. Traditional flat plate collector designs, for example, which, true to their name, contain a large flat metal plate absorber, can attain good optical performance and reasonable lifetime. Thermal losses tend to be high, however, particularly as operating temperatures approach 100^{o}C. Due to the massive metal absorber and the large amount of insulation required behind it, moreover, the cost of these collectors is difficult to reduce.

Optical concentration has long been used to minimize thermal losses in solar collectors by reducing the amount of hot absorber area necessary for a given entrance

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aperture area. Increased efficiencies and higher operating temperatures are the result. Focusing parabolic trough collectors, for example, may operate at temperatures up to 300°C. In order to properly image the sun onto the absorber, however, focusing collectors must accurately track the sun and must have high quality mirrors. These requirements increase the cost and impair the durability of the collector.

In an attempt to better meet all the design objectives, a novel class of nonimaging optical concentrators known as compound parabolic concentrators (CPC)[1,2] has been under investigation at Argonne National Laboratory (ANL) and at the University of Chicago. At intermediate operating temperatures, collectors employing compound parabolic concentrators have demonstrated improved thermal performance[3] over flat plate collectors while avoiding the tracking and mirror quality problems associated with focusing concentrators. As a bonus, the CPC-concept is easily coupled to present-day glass technology. After introducing the compound parabolic concentrator, this paper outlines ways in which glass has already been applied or can be applied, and points out the problems encountered.

NOVEL COLLECTORS

Two useful CPC-configurations, one with a flat absorber and the other with a cylindrical absorber, are shown in cross section in Figures 1-2. In both, properly shaped reflectors accept radiation incident on the entrance aperture and optically concentrate it onto the (smaller) absorber. The specific shapes of these reflectors and the theoretical principle for generating CPC reflectors for any convex absorber shape are discussed elsewhere.[1,2] It is noteworthy that these concentrators make effective use of the entire surface of a convex absorber--no part of the absorber is "shadowed."

As with all concentrators, the angular field-of-view of these designs is restricted to less than the full forward hemisphere. Nevertheless, nonimaging CPC's possess the maximum, field-of-view attainable for a given level of concentration, limited only by fundamental physical principles, whereas focusing concentrators fail this limit by a factor of three or more. Because of their wide field-of-view, it is possible to design CPC-trough collectors which concentrate sunlight by almost a factor of two and yet remain stationary like a flat plate collector. A fivefold concentration can be obtained in a CPC-trough collector requiring only monthly adjustments in tilt to follow the change in solar declinations. The nonimaging property of CPC's also lead to a marked insensitivity to reflector errors, either in shape or in specularity.

As one might reasonably expect, there are a few practical disadvantages to the use of compound parabolic concentrators in solar collectors. The theoretical reflector curves always touch the absorber surface, which would lead to an intolerable heat leak in the thermal collector. When the two elements are separated by a small gap to eliminate the thermal loss, then an optical loss arises. Since this gap is necessary, there is an ongoing effort to arrive at practical designs which minimize its effects.

Also, the CPS is best suited for low concentration applications. As the level of concentration increases, the height-to-width aspect of the CPC rapidly increases, so that the required amount of reflector material becomes excessive. Further, the optical performance becomes increasingly sensitive to the value of the hemispherical reflectance of the reflector material. It would appear that a five to tenfold concentration is the practical limit for CPC-trough collectors. Problems which require higher levels of concentration are probalby better solved with traditional focusing design.

APPLICATION OF GLASS TECHNOLOGY

Early prototypes of the CPC-designs shown in Figures 1-2 were built as all-metal collectors in analogy to flat plate collectors.[4] Flat or tubular metal absorbers were either painted black or given a selective (high solar absorptance, low-infrared emittance) black coating, and then supported in anodized, polished aluminum sheet reflectors bent to the appropriate shape. These proof-of-concept experiments demonstrated that compound parabolic concentrators did reduce the thermal loss in a collector while maintaining a wider angular field-of-view, as expected. At the same time, the experiments convincingly demonstrated that the all-metal approach did not properly address the other design objectives--maximizing optical performance, minimizing cost, and maximizing durability.

The first, obvious application of glass to compound parabolic concentrators was in the fabrication of the reflectors. Compared to the anodized, polished aluminum sheet used, second-surface silvered or aluminized glass mirrors could increase the optical performance, markedly improve the collector durability, and possibly reduce the cost. As an experiment, thin sheets of low-iron content glass were sagged against polished steel molds in an oven, and then aluminized. Optical tests revealed that the glass sheets had not conformed well enough to the mold. The work was abandoned before the process was adequately characterized because the resulting mirrors were heavy and because the fabrication process was too slow. However, the concept of glass mirrors needs to be reconsidered in light of the availability of micro-sheet glass. This material might first be aluminized in large sheets, then cut into individual reflector pieces which are finally mechanically deformed to the proper contour. If this is possible, then fabrication costs can be dramatically reduced. The appropriate experiments remain to be done.

The next application of glass to CPC-design was through the use of an evacuated receiver comprising a glass envelope around a flat metal absorber strip. This possibility, shown in Figure 3, was described in detail at the 21st ASGS Symposium.[5] Note there is necessarily a gap between the absorber and the reflectors to accommodate the envelope. Thermal loss from the absorber is minimized by the vacuum space, and durability is enhanced by the glass envelope. Corning Glass Works, Corning NY, fabricated a number of prototype receivers for ANL incorporating annealed copper absorbers (3.4cm x 122cm x .025cm) within a borosilicate glass envelope (3.8cm dia). The selective surface on the absorber was electrochemically deposited black chrome. To combat differential thermal expansion problems, the heat transfer fluid followed an attached hairpin loop of copper tubing, the inlet and outlet being brought through the same Housekeeper seal at one end of the receiver envelope. After evacuation, baking, and tip-off, a barium getter was fired in the vacuum space. The continuous service rating of these receivers was 230°C, and they have been tested to this temperature in a number of CPC-collectors. Although the prototypes were successful, much innovative work needs to be done to make glass-enclosed metal absorbers cost effective. For example, can this be done with a tubular absorber? Can the receiver be made in longer lengths--up to 2.5m--to reduce the parts count? Could the receiver be made in a once-through design with a glass-to-metal seal at each end? Questions also must be answered concerning long lifetime designs in the face of daily thermal cycling and possible helium permeation at elevated temperatures.

A similar application of glass technology was the incorporation of the all-glass vacuum receiver designs of General Electric, King of Prussia, PA (TC-100™ receiver), and of Owens-Illinois, Toledo, OH (Sunpak™ receiver). In both of these designs, a

smaller diameter glass tube with a selective coating on its outer surface is sealed within a larger diameter transparent glass tube in a gettered, Dewar bottle-like construction. The overall lengths are similar, 1.1m for OI versus 1.25m for GE. The OI receiver is fabricated from borosilicate glass (4.3cm and 5.1cm, od), and is intended to have the heat transfer fluid in direct contact with the inner glass surface. The GE receiver, on the other hand, is fabricated from soda-lime glass (T-14 and T-17), and requires a thin copper fin in contact with the inner glass surface to extract the heat and conduct it to the fluid. The selective coating in each case is a proprietary PVD coating. The CPC-collector design for these tubes is shown in Figure 4. Again the reflectors are anodized, polished aluminum sheets. As before, note there is necessarily a gap between the reflector and the absorber to accommodate the glass envelope. Tests of prototype collectors using each type of tube have been extremely encouraging, showing that equivalent or superior performance can be obtained with fewer tubes than in the original Sunpak or TC-100 collectors. Variations of this design are now in commercial production by several manufacturers. Nonetheless, important questions remain to be answered: Can the receivers be made in longer lengths to reduce the parts count? Can the selective coatings on glass be improved, since the absorptance is substantially below that of electrochemical coatings at present?

In the two previous examples, the evacuated glass envelopes enhance the receiver durability, but the conventional metal reflector remained exposed. To protect them, the collectors typically have an overall cover glass, which adds to the cost and weight, and which subtracts from the optical performance. The next two examples are yet-to-be-tried applications of glass technology which mimic the commercial lamp industry to bring the reflector into the vacuum space, producing a unitized collector and pointing the way to possible mass production.

The "floodlamp" collector concept, shown in Figure 5, has been proposed[6] as a way to mass produce one-piece conical CPC-collectors. Borosilicate glass envelopes blown to the correct contour and then aluminized internally would be attached to a selective coated metal stub absorber by means of a Kovar™ seal, then evacuated, baked, and tipped off. These collector lamps could then be laid out in large arrays, perhaps on heat pipe studs, for example. Crude daily tracking would be required since the collector is conical. At least one example of the "floodlamp" has been blown as a proof-of-concept, but most of the experimental work remains to be done.

The "flourescent lamp" collector concept, shown in Figure 6, has been proposed[7] as a way to mass produce one-piece CPC-trough collectors. Fabrication could begin with the redrawing of fluorescent lamp tubing to the correct contour. Although considerable theoretical effort has been invested in this concept, much innovative experimental and design work remains to be done. Ways must be found, for example, to incorporate the absorber tube, to aluminize or silver the reflector portion of the tube, and to apply a selective coating to the absorber. The question of workable vacuum seals remains open, as does the question of a once-through flow versus a return flow design.

CONCLUSIONS

The compound parabolic concentrator concept has demonstrated its usefulness in solar collector design. The ease with which present-day technology can be applied implies that dramatic inroads can be made concerning the cost and durability of the resulting collectors, while improving the optical and thermal performance. The effort is in its infancy, however, and much remains to be done by innovative workers.

ACKNOWLEDGMENTS

This paper is based on the collective labors of a score of colleagues at Argonne National Laboratory, at the University of Chicago, and elsewhere. Without their efforts the compound parabolic concentrator would still be an optical curiosity.

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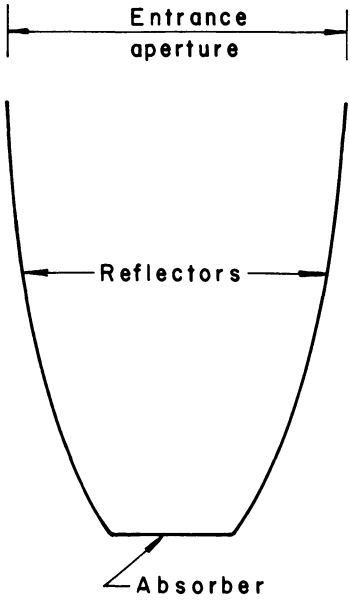

Figure 1
Theoretical CPC Design with a Flat Absorber

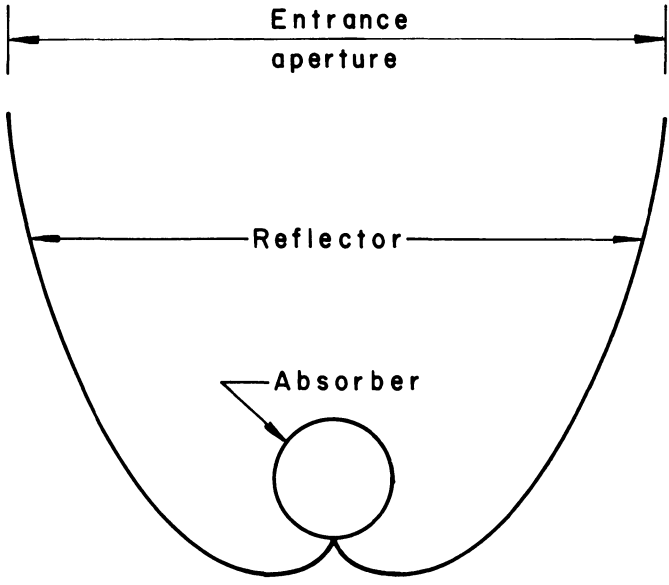

Figure 2
Theoretical CPC Design with a Cylindrical Absorber

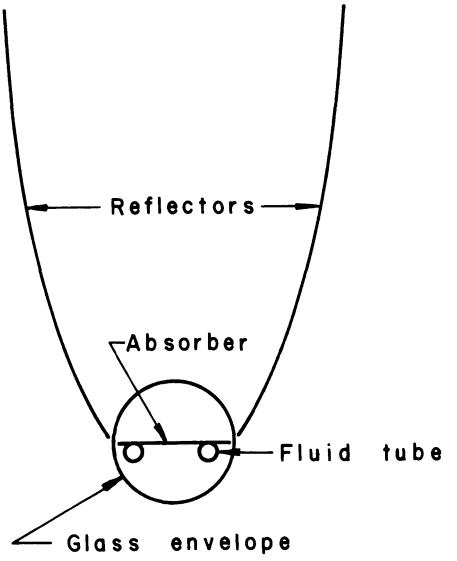

Figure 3
Practical CPC Design with an Evacuated Receiver

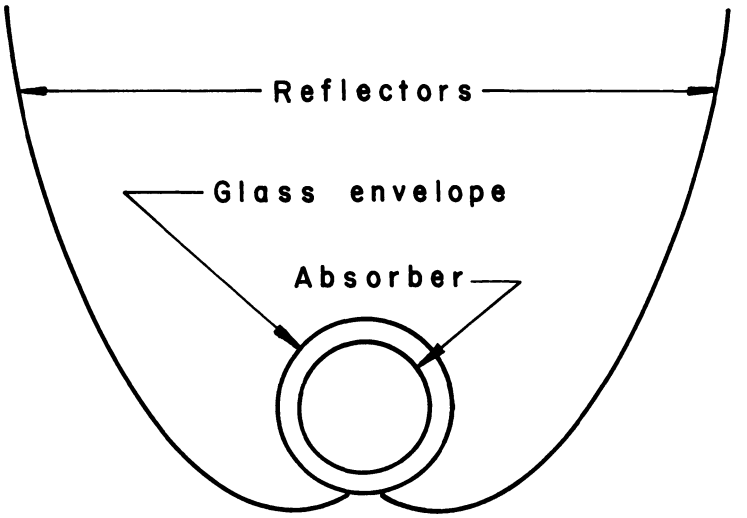

Figure 4
Practical CPC Design with an Evacuated Tubular Receiver

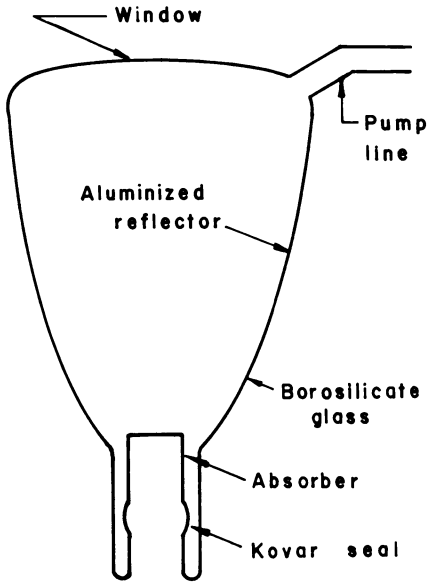

Figure 5
"Floodlamp" Collector Concept Proposed by D. Grimmer[6]

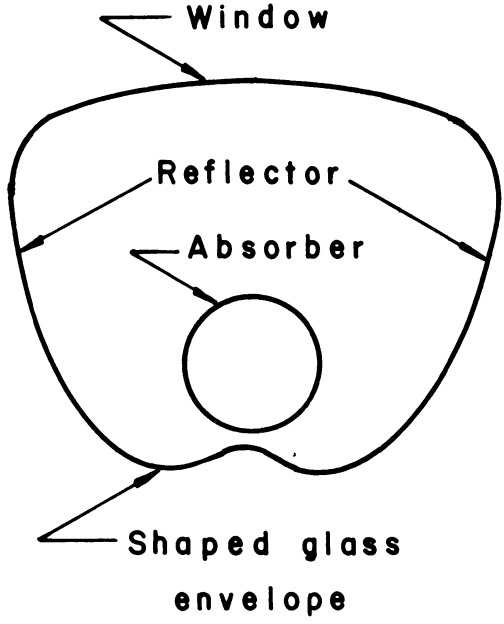

Figure 6
"Fluorescent Tube" Collector Concept Proposed by J. Garrison[7]

ELECTRICAL HEATER APPLIED DIRECTLY TO BOROSILICATE GLASSWARE

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INTRODUCTION

The need for an enclosure for a detector to analyze for alcohol in a human breath sample was requested to be made in my glass shop. This breath analyzer was to be portable, to be used in the field. To be an accurate and reproducible instrument, this enclosure must be heated to an elevated temperature. Glass was the ideal material for the detector housing since it is easily formed, chemically inert and an insulator. This housing had to have three tubulations, an inlet and outlet for the breath sample, plus a pressure switch tap and the end open to allow the detector header to be inserted. Also, since the housing was to be heated, two areas had to be flat to accept Klixon thermal switches to control the temperature. As you can see, we have created a very nonsymmetrical glass housing.

The advantages of applying the heater directly to the outside of the glass housing follow. An electrical heating element inside the housing would affect the result of the analysis. The temperature required could be controlled exactly, since the surface of the housing is in direct contact with the heater. Also, no glass-to-metal seals were required to power the heater.

METHOD OF APPLICATION

The material used as a heater element is actually a thick film ink. The ink is normally silk screened on a substrate and fired in a furnace to produce an electrical circuit. There are many inks blended: some for conductors, some for resistors, and some for capacitors. The material we used was the conductor ink. This is a mixture of glass, solvents and silver. When the ink is fired, the vehicle is burned out and a conductive film is left on the substrate.

Obviously, the substrate in my case is borosilicate glass, Pyrex brand #7740. Applying the ink to glass tubing is the most difficult problem. I used a 6 mm glass tube with a buret tip on one end and a funnel formed on the other end. The ink separates unless it is continuously mixed, so I inserted a fine wire down the tube (which I call a pen) to just clear the hole in the buret tip. That way, I could start and stop the flow of ink easily. A lot of technique was learned while doing the applying, and after a short while, it became very easy to "write" any pattern desired.

In the case of the breath analyzer, I chucked the detector housing in my glass lathe and turned the chuck with one hand while applying the ink with the other.

After applying the ink, I turned the lathe on and heated the part with a bunsen burner to pre-dry the ink. On cooling, an error can be corrected simply by wiping the dry unfired ink away and reapplying new ink to a cool glass part. The dry, unfired ink has a crusty, dull look and until it is fired, is very fragile. Care must be taken not to disturb the pattern to assure electrical continuity.

Parts can be dried in an oven at about 250°C but handling can obviously cause problems.

Satisfied the pattern is correct and the part is dry, an annealing burner was used to fire the ink. I used the Litton gas-air annealing burner. The gas-air flame is hot enough to fire the ink without distorting the glass part. On cooling, I measure the resistance with a bridge to assure continuity and also in this case to measure the resistance. This is to be sure it falls within our specifications. These housings were used in a portable unit and were powered with batteries so the resistance had to be close to an optimum value.

In most apparatus, the electrical source is easily adjusted so just about any conductor can serve as a heater. But when the electrical source is a fixed voltage, the resistance of the conductor pattern must be adjusted. To do this, the pattern can be made thin or thick, wide or narrow, long or short. This oversimplifies the problem, but with trial and error a great variance in resistance can be produced for an area to be heated.

Another technique can be used by blending resistor ink with conductor ink. A higher resistance can be produced. Again, trial and error was used to produce a specific resistance.

If the parts were dried and fired in a furnace, the resistance can be varied somewhat depending on a time and temperature cycle.

On producing a part with a heater, leads must be attached. A pad was made by applying a second coat of conductor ink and redrying, then refiring to thicken an area so a lead can be soldered. I used a soft solder with 1% silver, 36% tin, 73% lead, and a rosin flux. This solder wet the pad left for the lead easily with a small solder iron.

The part, when completed, was coated with a protective film since a metal temperature sensor was pressed against the heater pattern. This would obviously short the heater unless an insulator was used. A number of insulating materials were used. Silicone rubber was very effective, but epoxy resin also worked well. In other apparatus I have subsequently made, no protective coating was applied, and when the heater "burned out" I was able to repair the open area easily by reapplying ink and bridging the open gap.

I used a silver conductor ink supplied by the Methode Development Co., No. 80 CFN. The properties are:

Coefficient of Thermal Expansion: Compatible with borosilicate glass.

Chemistry: 86% silver, remainder glass and vehicle.

Solvent: Pine oil, butyl cellosolve acetate.

Resistance: 1.62 milliohms/square.

Firing Temperature: 3-5 minutes @ 780°C - 950°C

Solderability: 6 seconds @ 500°F - good
3 seconds @ 500°F - excellent

Cost: $17/troy oz.

The thick film industry has many ink manufacturers and I'm sure a number of different inks will work equally as well.

Some thick film ink suppliers are listed below:

Methode Development Company 7447 West Wilson Avenue Chicago, Illinois 60656	(312) 867-9600
E. I. DuPont de Nemours & Company Photo Products Department Electronics Products Division Wilmington, Delaware 19898	(800) 441-9475
Electro-Science Laboratories, Inc. 1601 Sherman Avenue Pennsauken, New Jersey 08110	(609) 663-7777
Plessey EMD 320 Long Island Expressway South Melville, New York 11746	(516) 694-7910

TECHNICAL ASPECTS OF BOROSILICATE GLASS PAPERWEIGHTS

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INTRODUCTION

In the last four years, I have spent some of my time exploring glass paperweights; at antique shows, in stores, in collections, and even at glass works in England and Scotland. While studying the classical weights and the techniques involved in making them, I became not only an admirer of these artifacts, but also an avid collector.

In history, we do not need to go back too far, as glass paperweight making only started in the 1840's, in continental Europe. The elements of glass weights have been known for centuries. Millefiori discs can be found on the Egyptian and Roman blown vases, and may even date back to the Syrians, 1700 B.C. About 1750, the Bohemian manufacturers tried to incrust glass ornaments. Many attempts failed, and only the French factories of Baccarat, St. Louis and Clichy are famed for having brought the art of incrustation to perfection.

The first paperweights appeared at the industrial Exhibitions in Vienna, 1845, and also later in London. Soon the art spread in Europe to Pietro Bigaglia of Venice, then to England, and at the beginning of this century, reached America's shores.

Close examination of some of the famous weights reveals a very large degree of technical knowledge and artistic talent. Where else, but in the making of paperweights can almost every technique developed in the history of glass working be found? Millefiori and lace cane making, lampwork, colour, design, incrustation, shaping, annealing and cutting: all are present in classic glass paperweight making.

However, this paper is restricted only to the discussion of the technical aspects of weight making using borosilicate glass.

For myself and for most of you present, there are a number of reasons for the preference for this glass. The scientific glassblower has the training, experience, knowledge and all the facilities to work with this medium. It is always available in many different shapes and forms, with uniform physical and thermal properties. Some weight makers chose this glass, but soon abandoned the idea and turned to soft glass, because of the numerous technical problems encountered in practically all phases of manufacture. Few prepared colours are readily available, and there are difficulties in producing coloured borosilicate glass. To me, all of these factors contribute to the challenge of perfecting a piece that can be considered a paperweight in the classical tradition.

DISCUSSION

Glass paperweights are usually blobs of solid clear glass with some artistic internal design. The design may vary, from lampworked ornaments, like flowers, insects, lizards or millefiori canes, to cameos made of glass, or even in some cases, ceramic.

Consider a simple clear paperweight and let it be built up to a fairly complex example. Let the blob of glass have a diameter of 2-1/2 inches, have a spherical dome and be flat on one side (Fig. 1(a) and 1(b)). The design is a single flower, made of coloured glass, with white petals, yellow center, green stem and leaves. To make it more complex and attractive, we could place a solid coloured base beneath the flower, e.g. dark blue (Corning No. 5420). On this contrasting blue glass, the above flower will come to life and appear more brilliant (Fig. 2).

To enhance the weight even further, we could add a thin coloured layer to the surface of the clear dome; this could be transparent, translucent or opaque, and is known as single overlay. To enable the beholder to view the incrusted ornament, windows or facets may be cut. For a weight of this size, the facets should be about 3/4 inch in diameter. Six facets are arranged symmetrically to form a circle around the dome, and a seventh is cut on the crown (Fig. 3(a) and 3(b)).

The appearance of the internal design depends on the type of facets, and is also influenced by the colour of the overlay. Through these facets, the flower will appear in its original colour and size, or smaller depending on whether the facets are level on concave. At the same time, through the dome the motif will be seen enlarged in its original colour, if there is no overlay, or with the colour modified by the transparent overlay. In the case of a translucent overlay, the ornament will be seen mainly through the facets. In the case of an opaque overlay, a second layer of different coloured glass can be placed on top of the first. In the case of this double overlay, the internal design can be viewed only through the facets. A second or third row of facets may also be arranged on the surface of the dome.

Colour and facet combinations, different motifs, plus the imagination of the glassblower, lead to an unlimited number of different glass paperweights, each with a different personality.

Let us discuss the different steps involved in the fabrication of a glass paperweight such as shown in the Fig. 3. These are:

a) Preparation of the ornament

b) Incrustation of the internal design

c) Shaping

d) Overlay

e) Cutting, lapping and polishing the facets

f) Annealing of inhomogeneous borosilicate glass

a) Ornaments

As long as good workable coloured glasses are available, no glassblower needs any introduction to glass ornaments or flowers of any kind. (See Bibliography No. 6.)

Perhaps millefiori and lace canes made of borosilicate glass deserve closer attention. These canes are solid glass rods, with different colours and designs in the cross section that run along the interior length of the rod, very much like a candy cane.

For example, consider a date (1979) for design in a millefiori cane (Fig. 4).

This rod is drawn down to about 6 mm in diameter, with blue numbers running inside the length of the white cane. To make this cane, use a blue glass rod (Corning 5420) about 6-7 mm in diameter and 75 mm long. Flatten and bend it lengthwise, so that when viewed from the end, it has the appearance of No. 7. Place it in a previously prepared borosilicate glass test tube, about 15 mm outside diameter and 150 mm long, so that the No. 7 fits exactly in the center. Pack with glass powder containing 3% SnO_2 (Ref. No. 6). A vibrograver aids in eliminating air pockets in the powder. Heat the tube slowly to melt and draw a rod of about 6 mm in diameter, by pulling it to about 20 - 24 inches long. If the tube is well packed with glass powder and dried in an oven, the result will be a white rod with No. 7 in blue in the cross section. Similarly, Nos. 1 and 9, or any other number, letter or design can be made with different colours. Once all the numbers are drawn, select the best, twist free 74 mm long sections and pack the different numbers together in proper arrangement ("1979") with glass powder in a larger test tube (28-30 mm outside diameter). Again heat to melt and draw it to suitable diameter (4-8 mm). The good section of this date cane, after annealing, may be cut into discs about 3 - 5 mm thick by different methods (abrasive or diamond impregnated cutoff wheels). Figure 5 shows cross sections of different arrangements of coloured glass rods to obtain lace canes. In Fig. 5(a), (w) white glass rods, 6 mm in diameter, alternate concentrically with clear ones of the same size, around a 10 mm diameter clear rod. In Fig. 5(b), different coloured glass rods (R, red, B, blue, Y, yellow, etc.) of the same diameters are alternating, also around a 10 mm diameter, clear rod. In Fig. 5(c), three different coloured rods are arranged in the center, in one plane, and packed concentrically in clear glass rods of the same diameter.

When the packages of rods, about 4 inches long (represented in Figs. a, b and c), are melted together and drawn to a smaller diameter, 5 - 6 mm, while constantly twisting, it will yield the lace cane. Complex canes are obtained, when a number of different canes, such as described above, are bunched together, melted and drawn again to a smaller diameter.

Silhouette canes are the glass rods where a simple outline of animals, flowers or even portraits is seen in the cross section. The minute details in the millefiori and silhouette canes are usually very characteristic of a particular factory or even an individual artist, very much like fingerprints. Close examinations at these minute details serve in many cases to trace the makers of antique paperweights.

b) Incrustation

Assume that we have an ornament to incrust, e.g. flower from Fig. 1, which is about 1-1/2 inches long and flat. Using the glassblowing lathe, make two pieces of clear glass from any convenient or available size rod (e.g. ~1 inch in diameter) as shown in Fig. 6(a) and (b). Place the above flower on a clean smooth graphite plate, heat the large glass blob (6 b) to red heat in the flame of a bench burner, and simply press it on the flower. For guide, use any suitable home made jig, or a piece of metal tube (2-1/2 inches inside diameter 1-1/2 to 2 inches long) to ensure that the design will be well centered. In this way, the flower will be pressed into the molten surface of the clear

glass and remain there free of air bubbles and cracks. Place it back on the lathe, but this time heat only the opposite part (6 a) on the facing surface, and while the lathe is turning at moderate speed, simply push them together. Note that the surface of the base part to be sealed (6 a) is slightly curved to eliminate the sandwiching of air bubbles with the design. Cut the stem on the right hand side with flame, and heat the sphere of glass to give the required shape. Use a graphite plate if necessary, but paddle marks are to be avoided. Finally, the stem on the left can be snapped off, leaving a "Pontil Mark." If desired, the pontil can be removed and smoothed with flame before annealing. (To perfect this procedure, some practice may be required.)

If the base of the paperweight is made of a suitable coloured glass, in this case blue, then we are at the shape shown in Fig. 2.

A motif holder, Fig. 7, may be useful, particularly if the design is a complex one. It is best made of brass, copper or steel. When this holder is lined with double-sided masking tape, the flower or complex pattern made of millefiori discs can be arranged on it and the whole assembly held by one of the chucks on the glassblowing lathe. In this way, the ornament will always be well located. However, the heat has to be kept away from it until the last second, because of the masking tape.

c) Shaping

The shape and clarity of the dome are important factors, since the appearance of the internal designs is dependent on them. Usually it is hemispherical, giving an enlarged view of the inside and, thus, the impression that the incrusted design fills the weight. The shape can be precisely controlled with a graphite tool by turning and heating the weight on the glassblowing lathe.

d) Overlay

When the shaping is complete, but the weight is still on the lathe, a thin layer of the surface of the dome can be coloured, before removal for annealing. The application of silvernitrate in a soft flame will give colours varying from transparent, through translucent, to opaque, depending on the quantity of silver diffused into the glass. This procedure has been demonstrated a number of times on different occasions at Glassblowers' meetings. It would be interesting to see what other chemicals could be used in similar fashion to achieve other colours on the surface of the glass. When a second overlay is required, usually over a first, white, opaque layer, the bulb technique is applicable. In this case, a thin wall bulb, slightly larger id diameter than the paperweight itself, is blown to the end of a coloured glass tube. Then this bulb is opened in the flame, so that it fits over the ready paperweight, Fig. 8(a) and (b).

The paperweight is positioned inside the prepared bulb on the glassblowing lathe and then it is collapsed onto the surface, carefully avoiding any air pockets being trapped in-between.

e) Faceting

If a lapping machine is not available, a silicon carbide cutoff wheel, or a steel or glass plate is used to grind the first facet on the crown of the weight. No. 150 grit silicon carbide slurry on the metal or glass plate is used, with a figure-eight motion until the size of the ground surface is satisfactory. Care must be taken so that this new surface will be parallel with the base. If a lapping machine or cutoff wheel is used, the

lapping is still brought to the stage of a matt flat surface using No. 300 and 400 silicon carbide slurry on the lapping plate. The directions of the side facets can be marked with a lead pencil on the circular lapped surface, to serve as guide. Fig. 9(b).

A piece of glass tube, about 16 mm in diameter and 15 mm long is glued to the center of the base, with an easily removable organic glue. Fig. 9(a). When lapping the facets on the side of the dome with the cutoff wheel, the weight is leaning on this tube, Fig. 10(b), therefore, its length will govern the angles and is to be adjusted before lapping begins. Once the sizes of the lapped surfaces are large enough to be able to maintain their angle, the lapping can be continued on the lapping plate, as above. The flat surface can be convered into concave, when it is gently rotated and held perpendicular against the edge of a small diameter, abrasive, cutoff wheel. The radius of the wheel will define the radius of the concave facet. Fig. 10(a). Concave facets can also be obtained using special tools of copper or steel machined to the desired radius and used with the appropriate silicon carbide slurry. A similar tool, with a diamond impregnated surface was also used with success.

Once the facets are complete with matt surfaces, the weight is washed in concentrated nitric acid, to remove all the abrasive particles, rinsed with water and dried. It is then placed in the annealing oven, and the temperature gradually brought up to 580^{o}C. The facets are fire polished while the weight is at this temperature, using the appropriate flame of a hand burner to polish one or two facets at a time, with the oven opened for a few minutes. After this, the weight is ready to receive its final, thorough annealing.

f) Annealing

When small amounts of metal oxides are mixed in borosilicate glass to obtain different colours, the thermal properties change slightly. This change may be enough to cause concern in annealing. Many weights made of blue glass, Corning 5420, sealed with clear 7740 and with incrusted design, failed. A series of annealing experiments was carried out in a specially designed furnace. Fig. 11.

1. Resistance heater element.
2. Thermal insulation.
3. Support for the weight.
4. Quartz discs.
5. Polarized plates.
6. Polariscope light and housing.
7. Thermocouples.
8. Digital thermometer.
9. Variable transformer.
10. Paperweight.

This is a tubular furnace, made of two semi-cylindrical resistance heater elements, 3 inches in diameter and 4 inches long, just large enough to accept one paperweight. At each end, a quartz disc (3 x 1/4 inches) is built in, one of them easily removable so that a weight can be placed inside, or removed when annealed. On the outside of each quartz window, at a safe distance from the heat, polarized plates are placed to create a

polariscope around the heated weight. For that, an appropriate light is provided in a housing at one end.

The temperature is controlled with a variable transformer, and monitored with a digital thermometer having two thermocouples, one above the paperweight and one below. In this furnace, the appearance and disappearance of the strain in the clear part of the weight, caused by the temperature, can be observed, while the temperature is simultaneously monitored at the two locations. In order to find the best way of annealing the inhomogeneous borosilicate glass, a number of weights of different coloured glasses and constructed in various ways were annealed, and the development of strain observed through the polariscope. This provided the opportunity to study the influence of the temperature, its rate of decrease, its gradient and time, during the annealing period. The strain found to be present in weights due to the effect of different colours, could be removed almost completely at a constant temperature. The time required to do that is, of course, inversely and approximately exponentially proportional to the difference between this constant temperature and the strain point of that glass which has the lowest value. The strain reappeared on lowering the temperature below this value and could not be completely removed. However, the purpose of the annealing was to control the amount of strain to a safe level, while the weight cooled to room temperature.

It was also observed that a temperature gradient across the weight had to be maintained during the cooling period, which means that control of cooling rate is important, and cannot be too slow, otherwise the gradient tends to vanish. It appears that the gradient is necessary to offset the effects of the different thermal expansion characteristics. Its sign and magnitude will therefore depend on the glass combination used.

The strain appears at a temperature just below the strain point at the interfaces of the different glasses, Fig. 12. After further cooling, the strain moves away from the interface, giving room for the next wave of strain. As the temperature lowers, the strain moves and disperses, and consequently weakens in strength. The intensity of the strain thus depends on how much the glasses differ. The degree of dispersion is influenced by the extent of fusion at the junction.

It seems that when fusion is not complete, i.e. the composition gradient is very steep, the strain developed at the interface due to slight difference in thermal expansion is not able to move away from the junction and spread, but remains close and intense, and may cause spontaneous cracking. This kind of junction occurs in many instances in scientific glassblowing, whenever slightly different glasses are joined together, e.g. in metal-to-glass seals, graded seals. In paperweights, it is critical, because a good fusion between glasses may result in a poor colour contrast, and fusing at slightly lower temperature may increase the danger of cracking.

Strain in weights made of Corning 7740 and 5420 could not be removed to a safe level, even with 500 hours of annealing at 485^{o}C, when the junction between the two glasses was made in a way different from described here, resulting in poor fusion at the interface. A well made junction between the above two glasses is best annealed for 48 hours at 485^{o}C and cooled to room temperature overnight, to withstand faceting and reheating to 580^{o}C to fire polish the facets.

Note the annealing temperature of these glasses: No. 7740 is 565^{o}C and No. 5420 is 547^{o}C, while the strain points are 504^{o}C for No. 5420 and 515^{o}C for No. 7740.

Discussion with prominent paperweight makers, who use ordinary or lead glasses, revealed that the problem does not exist to the same extent, because the different coloured glasses are much better matched. However, I have heard in collectors' circles that paperweights do spontaneously explode from time to time.

CONCLUSION

This may seem like a lengthy discourse, but is in actual fact only a mere précis of my findings. If I were to recount all my trials and tribulations, I would not conclude before the next symposium. When my failure rate was 100%, I would console myself by remembering that even the professionals have a failure rate of 50%. If you should decide that you still want to be part of the Paperweight Renaissance, do not be too ambitious at the outset. I have read that it took Charles Kaziun 4 years and $3000 to perfect the prototype of his now famous rose. (This figure would have to be multiplied by quite a high number to allow for inflation.) It is fortunate that Corning did not stage their exhibition until 1978, for had I seen, "Paperweights: Flowers which clothe the meadows" in my very early period, I am sure I would never have embarked on my perilous but exciting adventure into the world of weightmaking.

You and I may never produce a Clichy swirl, Melville rose, St. Louis marbrie or Baccarat bouquet, but we can explore, invent, create and find great satisfaction in paperweights. In the language of the great weights, je vous souhaite, "Bonne Chance."

ACKNOWLEDGMENTS

I am indebted to Dr. Raymond Bartnikas who offers me encouragement in all my endeavours, to Dr. Edna Dancy who contributed technical advice, and my wife, Maxine, who assisted me in compiling the information for this paper.

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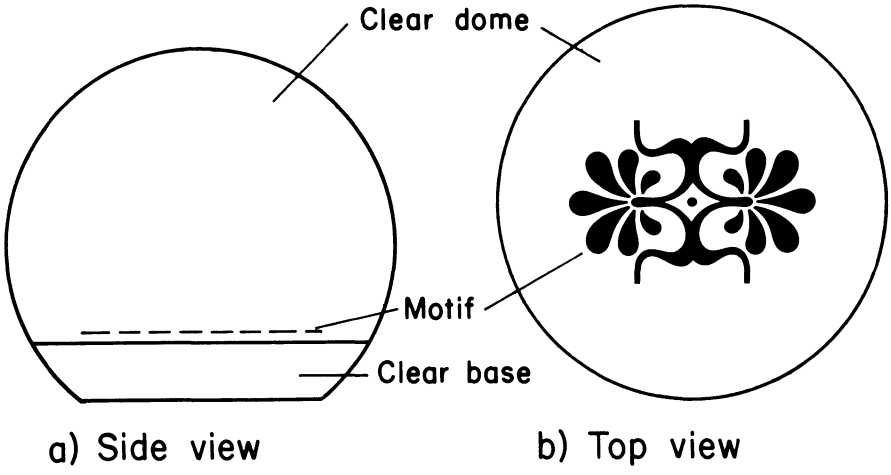

Figure 1

Paperweight, with Simple Flower Design

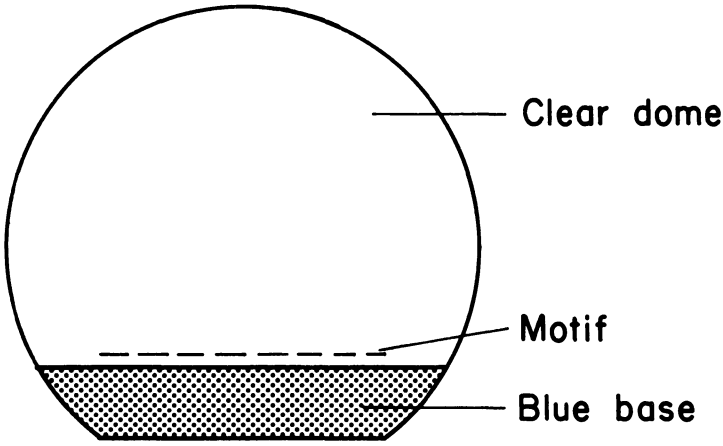

Figure 2

Paperweight, Clear Dome and Dark Blue Base

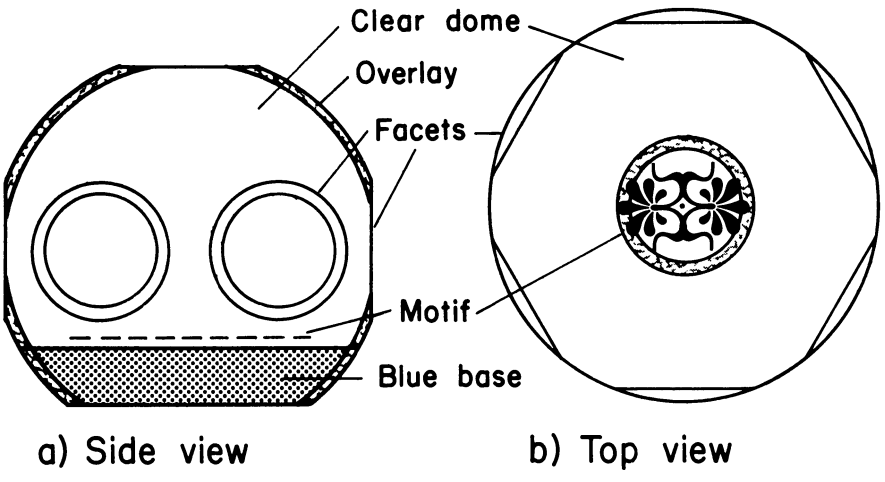

Figure 3

Paperweight, with Overlay and Facets

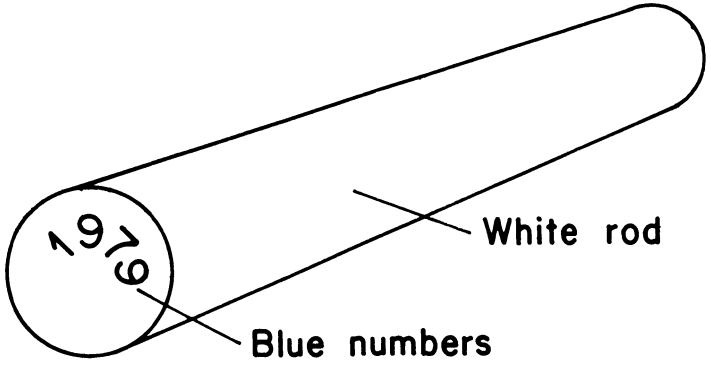

Figure 4

Date Cane

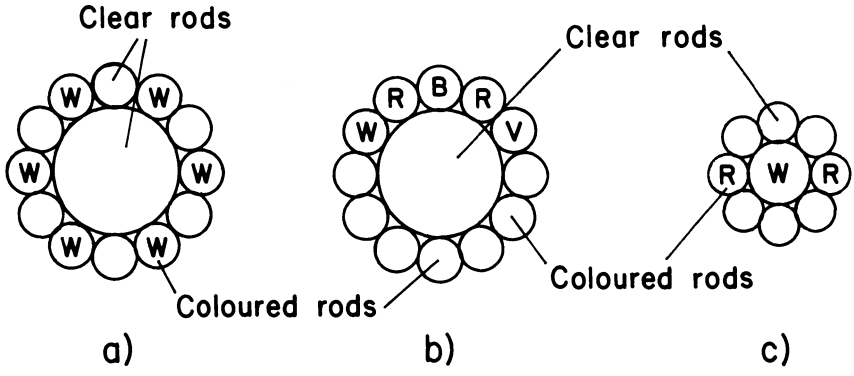

Figure 5

Bundled Different Coloured Glass Rods

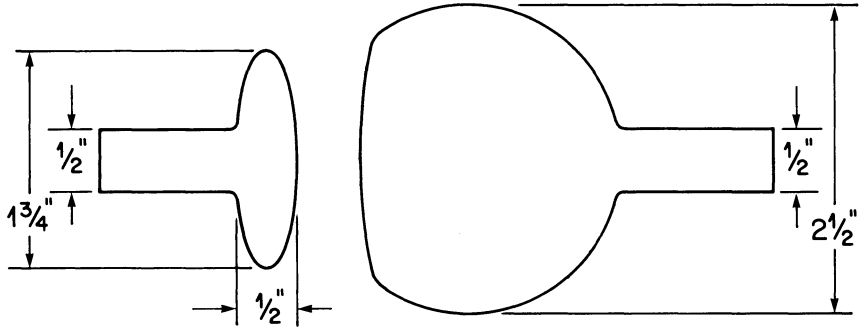

Figure 6

Prepared Clear Glass Blobs for Incrustation

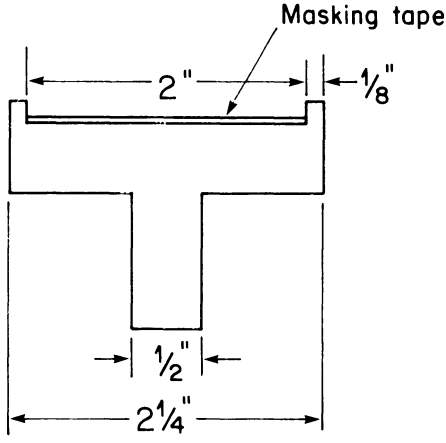

Figure 7

Motif Holder with Double Sided Masking Tape

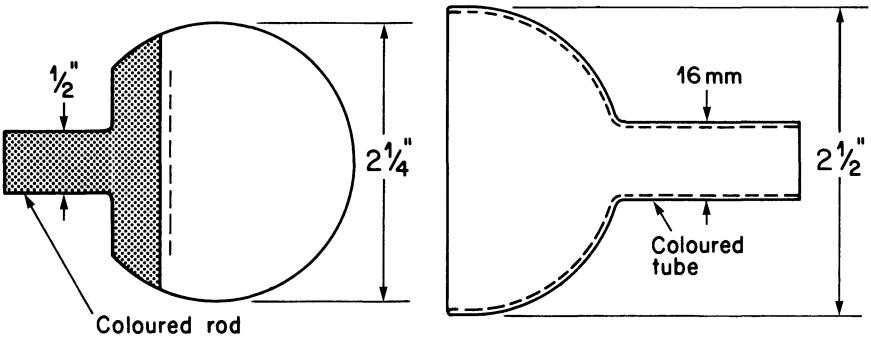

Figure 8

Prepared Overlay and Paperweight

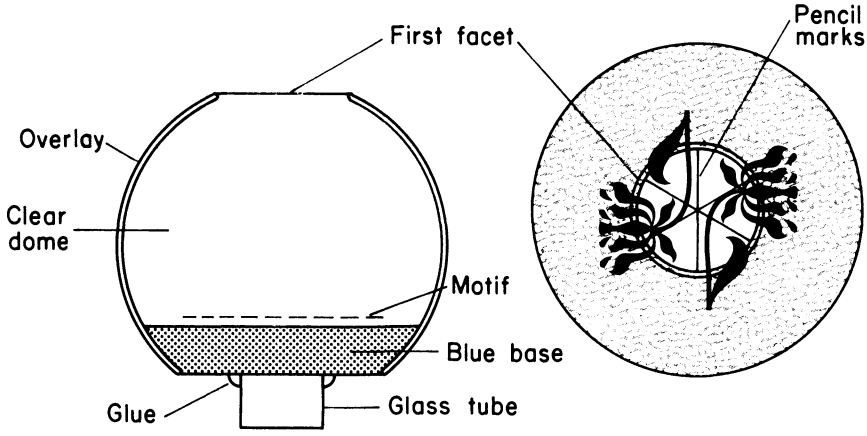

Figure 9

a) Glued on Guide Tube, b) Indication of Six Facets

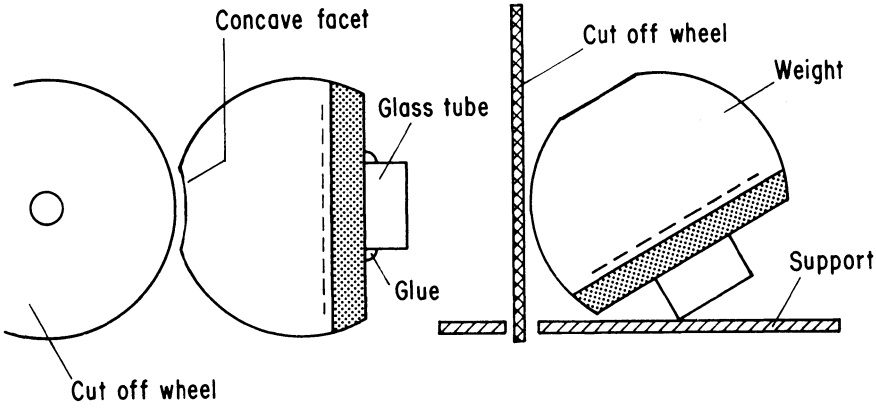

Figure 10

a) Concave and b) Flat Faceting with Silicon Carbide Cutoff Wheel

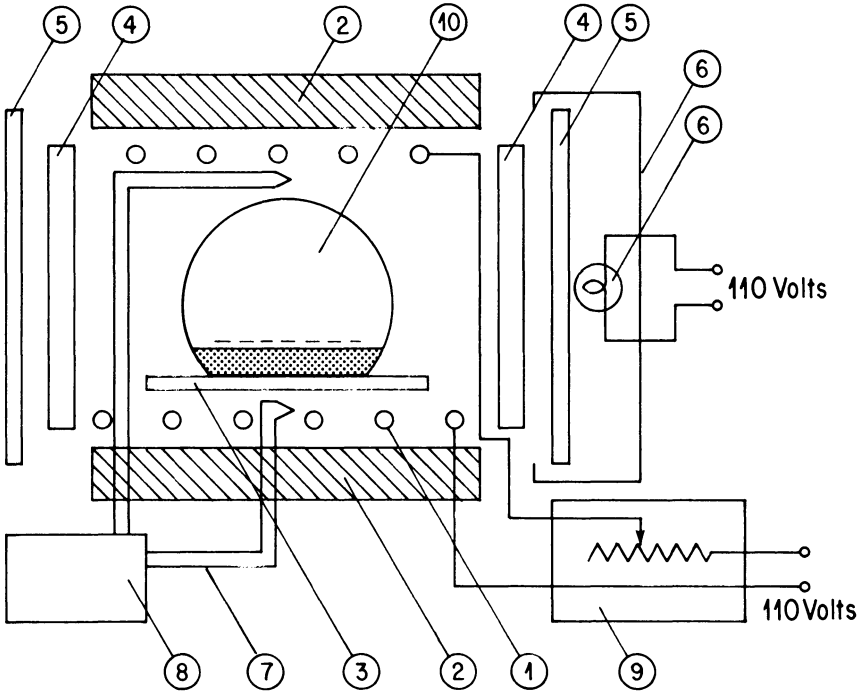

Figure 11

Annealing Furnace

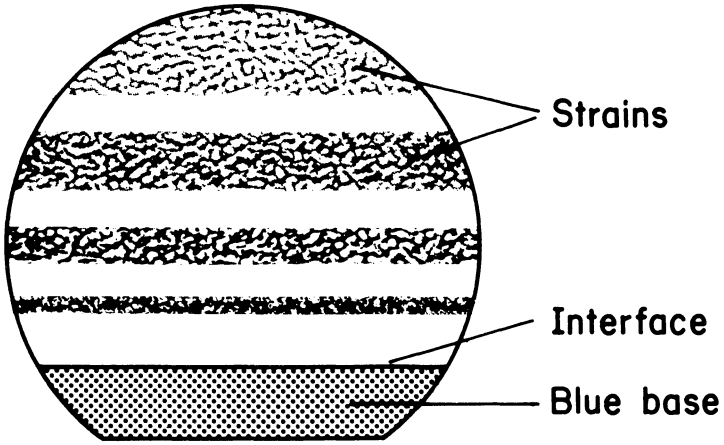

Figure 12
Waves of Strain in the Paperweight

U.S. DEPARTMENT OF ENERGY RECEIVES RECOMMENDATION TO FUND A.S.G.S. MEMBER's ENERGY-RELATED INVENTION

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Boise, Idaho 83705

ABSTRACT

In spite of the title this paper will present a few of the simple problems that the average glassblower may experience. The actual item or device of this paper does, of course, receive several comments. it is the finalized design of my improved low voltage fluorescent light bulb. My first report on this appeared in the 1976 Chicago Symposium Proceedings. That bulb was an all A.C. design. It still remains a viable device. The new improved design, however, although still using the line A.C. voltage - from any screw-type receptable - has an internal gas and mercury-vapor discharge that is direct current operated. Current control is still required. No ballast is used. Most technical matters have been fully resolved. In truth, the bulb is now at a point that a series of small batch runs (semi-production) will be carried forward. These runs will furnish the very necessary statistical data with respect to quality, life-expectancy, and luminous efficiency. Also, cost data will become availabe by this method. The transition from hand or custom made prototypes to semi-production units demands a most careful and well planned procedure. U.S. Letters Patent have been granted on the earlier low voltage fluorescent light bulb. A new patent application has recently been filed at the U.S. Patent and Trademark Office on the new D.C. bulb.

SLIDES: (To be used at Presentation of Technical Paper).

First: Let us glance at the few slides which I've brought along.

1. This grouping shows most of the components in their partially prepared state - but mostly prior to final glassblowing. There are two completed bulbs with regard to glasswork and processing.

2. This slide illustrates the type of stem we would now prefer to use. The two stems shown were given through the courtesy of EIMAC Corporation, a Division of VARIAN. EIMAC is at Salt Lake City. They are of Corning CODE 0772 (Nonex). They, I believe, are used on power tubes and certain radar vacuum tubes which EIMAC manufactures.

3. Our early preparatory attempt to make this type of stem. The bulb's envelope, lime glass, requires some glasswork for our purpose. The modification is a simple one.

4. This shows a four cathode and an eight cathode assembly.

5. The EIMAC stems approximate the size we would wish to develop.

6. This is a repeat view. All soft glass - excepting the two nonex stems from EIMAC.

7. This seemingly crude (even dangerous) arrangement is a means I have used for several years to enrich natural gas. Production, of course, would likely justify either oxygen or hydrogen. The fluid is high octane gasolene. A trance of a sulfur compound is added to the gasolene when this enriched gas is used for pressing or forming such items as the stems already illustrated.

8. This, unfortunately, is black and white. However, it does show the complete full flame that is attained using the gasolene. Color would give spectral evidence of the strong sulfur band produced. Prolonged use of this gasolene method is best avoided. Indeed, the accompanying unpleasant odor automatically limits ones use; e.g. Sulfur Dioxide! (I've checked this method for CO, and find that there is no appreciable generatoin of that vapor.)

I mentioned earlier of problems that the glassblower may face: What type of glass do we have on hand that requires immediate glasswork? What about its mass or weight? Its working characteristics temperature-wise? Softening, Annealing, and Strain Points. What is the proper flame source (RE: Fuel-mixture) that is advisable? These are only a very few of the important matters that must be determined. AND not surmised.

The bulk of my past three years of research on my present project has been mostly of pyrex prototypes. They were, for the greater part custom or handmade. On occasion I used my Litton, or my vertical lathe. However, during the past six months, I have slowly been converting to soft glasses. Soon, all bulbs made will be of the soft grades - excepting the quartz source tube columns - Clear fuzed quartz. (General Electric.)

Thus far, on the soft glasses, I have been using dumet, or its equivalent. I presently desire greater mechanical strength to my feedthrough leads, and plan to use the so-call SYLVANIA No. 4. (Driver Co. Newark, N.J., No. 426.)

One cardinal error I have continued to make is to use my oxygen-gas flame on the soft grades. With great care this is, of course, possible. But not as a production procedure, and certainly not for the unskilled glassworker. Hence, I soon will use the gas-air mixture.

One final error - my anneal. All hand. "Quick and dirty!" Literally so, since it includes the soot method of temperature indication - both on a heat-up procedure, and on the cool-down. It is quite successful, but slow and untidy.

Future possible test/development production will demand that I mend my ways.

THANK YOU.

Figure 1

From Idea-Theory · to Actual Practice

Figure 2

Waver head-pressed stems · Nonex
Courtesy of EIMAC A Division of VARIAN

Figure 3

Gasolene Used to Enrichen Natural Gas (CAUTION!)

Figure 4

In Color, Flame Gives Spectroscopic
Proof of Enriched Gas – Temperature-Wise and Combustion

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