Proceedings

THE NINETEENTH SYMPOSIUM
ON THE
ART OF GLASSBLOWING

1974

THE

AMERICAN SCIENTIFIC GLASSBLOWERS SOCIETY

Proceedings
THE NINETEENTH SYMPOSIUM
ON THE
ART OF GLASSBLOWING

Sponsored by

THE AMERICAN SCIENTIFIC
GLASSBLOWERS SOCIETY

HYATT REGENCY HOTEL
HOUSTON, TEXAS

June 18, 19, 20, 21, 1974

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THE USE OF DIAMOND BLADES AND SAWS FOR CUTTING AND MACHINING GLASS

J. P. LUNZER, *President*

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Before I proceed with my paper on Diamond Wheels, I would like to say a few words to bring you up to date on points I have learned since my last paper on the subject of small hole drilling.

Last year I stated that much depends on the individual operators' knowledge and skill in the techniques of drilling and that practice makes perfect. Well, in many cases with a new or unskilled operator the first 10 to 100 holes may be a fiasco. However, as I have said practice makes perfect and because there are no hard and fast rules that can be applied, I would like to make the following recommendations. These recommendations being based on factual data and experience accumulated over a period of time which covers many different types of glass, other related materials and a variety of hole sizes.

I would now like to give the following recommendations that we have found most helpful in the successful usage of our drills: the recommended speed for all drills, in the past, *was* a 3,000 to 12,000 RPM but through trial and error, we found that this did not apply to all drills. Although admirable for plated drills, which still remains constant today, impregnated drills were an entirely different story. To use impregnated drills *successfully* a much higher speed is required and we strongly recommend a drill press running up to 20 to 30,000 RPM. I repeat, this higher speed refers only to the impregnated drills.

We have encountered many cases where people have not been successful in drilling small holes or have had excessive chipping where the drill comes out of the holes, we have found that in most of these cases pressure has been used, excessive pressure on the drill head, which has harmed the drill and rendered it useless. So, we cannot stress enough that very low pressure on the drill will give much better holes especially in glass where for the last .004 to .010 of an inch of the hole being drilled the pressure should be cut down and if possible the speed of the drill increased.

Proper care and thorough experimentation will enable an operator to drill holes accurately and correctly. I stated last year, and I say it again, that many companies are using our standard drills successfully and getting as many as 1000 holes as small as .035, or getting 3 to 400 holes as small as .020 drilling through glass. But, of course, care must be used and patience is the word. It is our recommendation that if this is a production job, it should be given to a woman, as we find that women have far more patience for this sort of work than men, and patience is required in not forcing the drill through the glass, especially through the last portion where breakout often occurs.

My final recommendation regards the water pressure. When using water swivels or when the coolant is coming through in a continuous stream to the drill, we would recommend a much higher speed for small drills below 1/8 of an inch and a water pressure of at least 60 to 80 pounds per square inch, possibly as high as 200 PSI.

INTRODUCTION

Although diamonds have long been known as a very effective way to machine glass, they were not, until recently, used in abundance owing to their very high cost as a basic purchase.

Where an abrasive blade could be purchased for a few dollars, a user was hesitant to spend possibly hundreds of dollars in the purchase of a diamond blade. In the last few years, there has been considerable development in the improvement of diamond products and also, because the volume of manufacture has greatly expanded, users have found out that although the initial cost of the diamond blade may be high, the production, that is, the work produced, is far and away beyond that of an abrasive wheel. As a result, their usage has now become common to many users who would not have purchased them before.

* * *

There are basically three types of diamond blades depending on the bond being used. The first and most commonly used is the METAL BONDED BLADE, the second is the RESIN BONDED BLADE, and the third, is the PLATED BLADE. Nearly all diamond blades have a steel core which has to be used to give the blade its required stability. These blades must be correctly tempered and must run quite true so that the centers of these blades must be accurately made.

The first type of blade we will discuss is the METAL BONDED BLADE. The metal bonded blade consists of a steel core to which a diamond section has been attached consisting of diamond particles embedded in a metal bonded matrix. The diamonds used in metal bonded blades are either natural diamonds or synthetic diamonds. The grits can vary from as coarse as twenty mesh down to an eight to nine hundred mesh. The concentration, and here I shall give a general explanation of the word concentration, is the amount of diamonds, the caratage of diamonds in each cubic inch of wheel volume. The common denominator is 72 carats per cubic inch. This means that there is 72 carats of diamond material per cubic inch of total wheel volume.

Seventy-two carats per cubic inch is used to denote a hundred concentration. Fifty concentration would be 36 carats per cubic inch and twenty five concentration would be 18 carats per cubic inch. This formula is used by every American wheel and blade manufacturer.

The concentration, that is the amount of diamonds in the blade, does not necessarily determine the life of that blade. It could be assumed that as diamonds are the major cutting factor, the more diamonds there are in a blade, the better the blade will cut and the longer it will last but this is not the case. As a general rule, the coarser the mesh of the diamonds the larger the concentration required. Conversely, the finer the mesh, the smaller the concentration. This can be explained by the fact that the smaller the diamonds, the more diamond particles there are per cubic inch of volume. But, this also varies. In many cases, due to the type of material being used, the machinery and lastly, but most importantly, on the material being cut. A metal bonded blade will generally last longer than any other type of diamond blade but because of the hardness of the matrix it is not suitable where a very fine finish is required and will in many cases develop chipping.

The RESIN BONDED BLADE consists again, of a steel core to which is bonded the resin diamond section, consisting of diamonds set in a resin bond. These diamonds are usually synthetic diamonds, especially chosen for their purpose and normally cut very freely. These resin blades are the best to use when a good finish with little chipping is required. However, the resin bond on the blade is very fragile and can be broken easily. The resin bond also wears quickly and if used by an unskilled operator can wear out even more quickly. Therefore, when general purpose work is done on glass, it is recommended that a metal bonded wheel be used except where a very good finish is required. For this finishing, a resin bonded blade will do a far superior job.

The PLATED BLADE consists of the steel core to the edge of which diamonds have been electroplated, usually, in a nickle bath. These blades have all their diamonds on the surface. The plated blades will cut very freely and are excellent for short-run operations. Since, however, the diamonds are only plated to the outside of the blade, there is a comparatively small diamond area and will wear away quickly. To compensate for this, the cost of a plated blade is far lower than either a resin or metal bonded blade.

THE CUTTING OR GRINDING MACHINE

This part of the operation though normally taken very lightly is as crucial and important as the blade itself. The machine must be very stable, run perfectly true and have a motor capable of developing the speed required for effective cutting. Runout of the blade is a very important factor that should be looked into very closely.

When mounting the blade it should be seen that the flanges are large enough. When I say large enough, I mean as large as possible, since the larger the flanges, the less vibration and wobble can reach the cutting portion of the blade. These flanges should be perfectly clean and run perfectly true. The blade when mounted must be checked for runout as I am sure you will appreciate that every .001 of an inch of runout will be transmitted to the piece being run or cut.

COOLANT

Glass being one of the hardest materials to machine with diamond requires, except in special cases which I will mention later, a copious flood of coolant directed to the blade at point of cutting. The type of coolant is very important. There are many cases where only water and tap water, at that, is used and this is perfectly satisfactory as long as the blade is constantly kept cool and flooded. But an oil base coolant is far superior to even tap water to which an additive has been added. Mineral seal oil is good but the most preferred would be a mixture of ½ kerosene and ½ engine oil. There are some systems such as that used in the Pensar Combi-Cutter, which has a recirculating pump where the coolant is recirculated and this is an excellent way of cooling as long as the filter catches all the glass particles and they are not again applied to the blade in grinding.

When we first experimented with diamond blades on the cutting of glass, we generalized on the material and classified all glass as glass. We soon found out that there are various types of glass. Lead crystal glass machines fairly easily but quartz glass is very hard to machine with diamonds and needs a totally different type of diamond blade. The speed at which the blade passes the material at the cutting point is of course dependent on the diameter of the blade. As you well know an axial speed of say 5,000 RPM is quite different from the peripheral speed which will increase with the blade diameter. I will therefore refer to surface feet per minute and our recommendation is as follows: a speed of 4,000 to 6,000 S.F.M. is generally suitable but speeds do change according to the material and the size of the glass being cut. The feed, that is the feed at which the glass is fed into the blade is a most important thing. Never, I repeat, never force the blade into the material. Let the blade "feel" its own way into the glass. Although most cutting operations require a coolant there are some where the cutting must be done dry and this is possible by using the special bonds available for this purpose. Always inform your blade manufacturer that you are cutting dry otherwise your blade will heat up, load and consequently bind which in all probability will tear the matrix (bond) from the blade. I would recommend you avoid cutting dry whenever possible.

The diamond blade, once a luxury, is now a necessity and properly chosen and used is becoming a vital tool for glass cutting operations.

CORNING'S NEW MACHINABLE GLASS-CERAMIC

B. R. FEINGOLD

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Corning's Machinable Glass-Ceramic is a new material which can be machined to precise tolerances with conventional metalworking tools and equipment. Machining can be performed at feed rates and speeds typical for metals. Diamond tipped tools are not necessary. Machinable Glass-Ceramic does not have to be heat treated or fired after machining.

MGC is useful in situations where a special shape is needed but tooling for normal glass or ceramics would be uneconomical or too slow. MGC is also useful when tight tolerances or thin sections are needed since the expensive grinding operations that are required by glass are not necessary for MGC.

There have been other machinable materials before MGC, but there are none which have so many outstanding physical properties. In many cases MGC's unique combination of properties (such as high strength, vacuum integrity, thermal shock resistance, high dielectric strength, etc.) make it one of the only materials that can satisfy the application requirements. MGC has been used in situations where its machinability was incidental.

This paper will be divided into two parts; the first part will describe the machining characteristics of MGC and the second part will describe its physical characteristics.

MACHINABILITY

It is difficult to characterize the machinability of a material in a single index. However, it is useful to compare the ease of cutting in different materials in order to get a feel for machinability.

Machinable Glass-Ceramic cuts as easily as brass or aluminum (when cut with a hacksaw). Machinable Glass-Ceramic cuts 10 times more easily than pyroceram (when cut with a diamond saw). MGC sheet (.7" thick) has been cut with a diamond saw at a rate of 7" per minute.

All of the standard metalworking operations can be performed on MGC. Figure 1 shows a piece of MGC being milled. Figure 2 shows a piece that was turned and threaded and is in the process of being drilled.

Because of MGC's high strength it can be cut into very thin sections. Wafers with a 5 mil thickness have been cut with a diamond saw and 10 mil thick tubes have been turned from rod.

Because of MGC's very fine grain size, precision tolerances can be achieved. Parts have been machined with carbide tooling to tolerances of ± .002 mils. Figure 3 shows a case made from MGC; all dimensions in this piece are held within ± .002". The overall dimensions of the case are ½" x 1½" x 2.8" and the walls have been made as thin as .040".

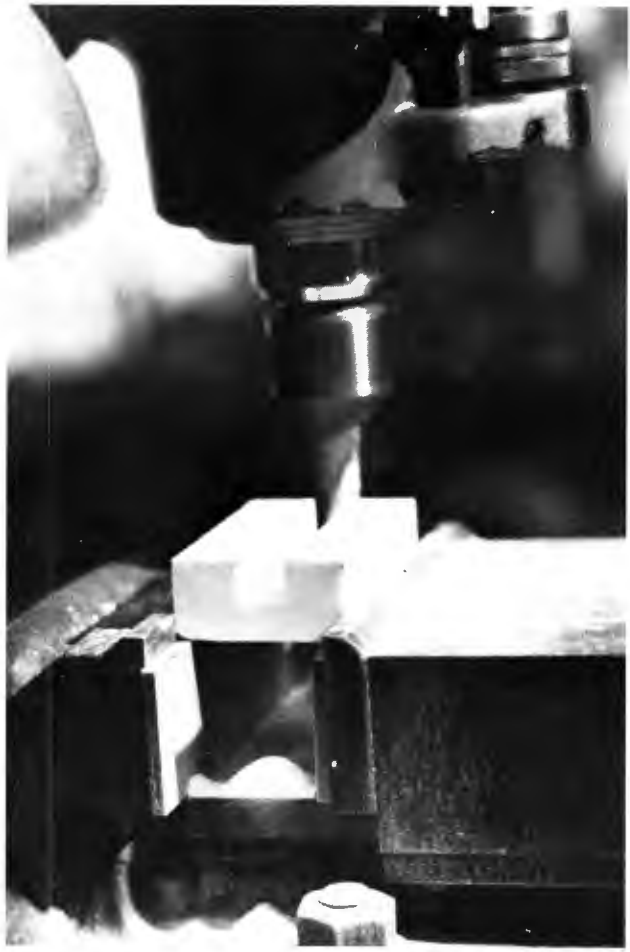

Figure 1

MGC can be polished to less than $\frac{1}{2}\mu$ in AA using cerium oxide on pitch. The surface finish as cut (with a diamond saw) has been as good as 4μ in AA.

MGC has been drilled and tapped for 2-56 screw threads.

GLASS-CERAMIC PROPERTIES

Machinable Glass-Ceramic is the latest material in the glass-ceramic family from Corning.

Glass-ceramics are materials that are melted and formed as glasses and are then converted by controlled nucleation and crystal growth into a

Figure 2

polycrystalline ceramic/glass system. In MGC's case, the polycrystalline phase is Fluorophlogopite; the microstructure consists of an interlocking lattice of Fluorophlogopite microcrystals surrounded by glass.

MGC's ease of machinability results from the fact that the fluorophlogopite crystals can be easily delaminated or separated, just like natural mica sheet. When a crack begins to propagate in MGC, it will propagate much more easily in the lamination plane. At the places where the microcrystals join, the propagating crack will change direction to stay in the lamination plane. The cracks will therefore have a tendency to bend back on themselves and chunk out the glass in the interstices. Since these changes in direction use up energy, the crack has a tendency to die

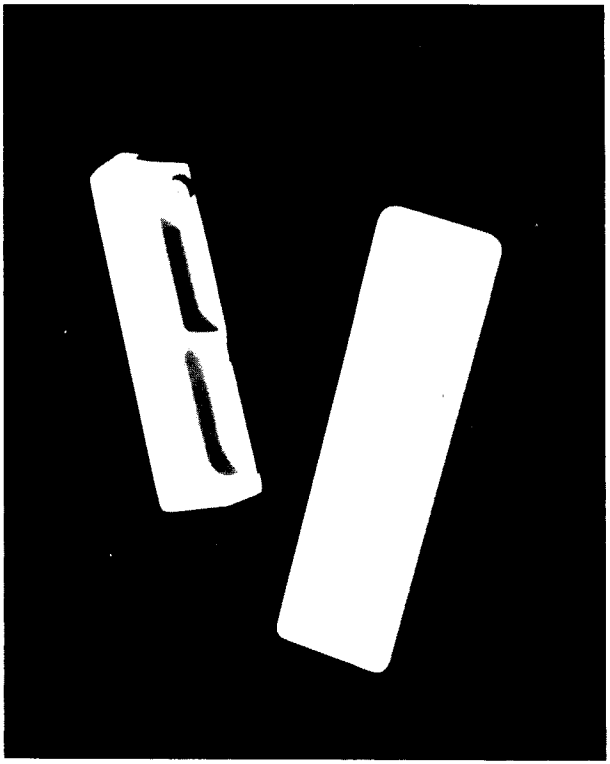

Figure 3

out before going far into the material. Therefore, the machining process is a microscopic, localized chunking out process.

This resistance to crack propagation explains MGC's combination of thermal shock resistance and high expansion coefficient. Although cracks may start from the thermal shock (or other reasons), they tend not to propagate through the piece.

The machining action described above indicates that MGC is machinable even though it is very strong and nearly as hard as ordinary glass. MGC's high tensile strength (MOR is 15,000 psi) is primarily due to the interlocking nature of the microcrystal lattice. The compressive strength (50,000 psi) is due to the glass phase of the glass-ceramic.

The glass-ceramic structure gives rise to physical properties that make MGC a very useful material. Figure 4 lists some of the material characteristics which contribute to its usefulness. Figure 5 lists the physical properties more quantitatively.

MGC's combination of properties suggests many applications, but there are two classes of applications that stand out.

Figure 4

Material Characteristics	Electrical	Thermal	Chemical
Code 9658	• High dielectric strength • High bulk resistivity • Low loss factor	• High maximum use temperature • Moderate thermal conductivity • High thermal expansion coefficient; can be sealed to common glass **Mechanical** • High strength • Insensitive to surface damage • Dimensionally stable and rigid • 50% greater impact resistance than Pyrex or Pyroceram • Isotropic and homogeneous	• Zero water absorption • Good resistance to chemicals • Not wetted by molten aluminum, magnesium or tin

	Thermal Shock Resistance	Surface Finish	Vacuum Performance
	• 800°C→ice water, without breakage • 200°C→ice water, without loss of strength	• Can be polished to finishes of 1 μ in AA or better **Metalizing** • Can be metalized using commercially available silver/glass pastes	• Zero porosity • Extremely low helium permeation rate • Does not outgas • Can be hermetically sealed to metals, ceramics and glass using solder glass

Figure 5

Physical Properties

Property	Test Conditions	Description of Test	Nominal Value	Units
General				
Density	Corrected to 4°C	10 g sample archimedies method	2.52 157	g/cm^3 lb/ft^3
Porosity	25°C	Hg intrusion	0	
Water Absorption		ASTM C-373, boiled in water and dried weight gain.	0	
Helium Permeation Rate	143°C		4.2×10^{-12}	$\frac{\text{cc stp—mm}}{\text{sec cm}^2\text{—cm Hg}}$
Electrical				
Volume Resistivity	500°C d.c.	ASTM C-657	10^7	ohm-cm
Loss Tangent	25°C, 10 kHz	ASTM D-150	0.003	
Dielectric Constant	25°C, 10 kHz	ASTM D-150	5.92	
Dielectric Strength	25°C sample thickness—10 mils, a.c.	ASTM D-149, 1 kV/sec. oil: Dow Corning-200	1000	volt/mil
	25°C sample thickness—10 mils, d.c.	ASTM D-149	3000	volt/mil

Thermal				
Thermal Expansion	RT to 400°C		94×10^{-7}	/°C
	RT to 600°C		110×10^{-7}	/°C
	RT to 800°C		123×10^{-7}	/°C
Thermal Conductivity	25°C	In-House Test Comparator	0.004	$\frac{\text{cal cm}}{\text{sec cm}^2\ ^\circ\text{C}}$
	77°F		11.68	$\frac{\text{BTU in}}{\text{hr. sq. ft }^\circ\text{F}}$
Maximum Use Temp.	Unstressed	Max. Temp. w/o change in crystal structure	1000 1800	°C °F
Mechanical				
Modulus of Rupture	25°C	ASTM C-158 modified for high temp.	15,000	psi
Compressive Strength	25°C, 10,000 lb. min.		50,000	psi
Modulus of Elasticity	25°C	Sonic Resonance Apparatus	9.3×10^6	psi
Shear Modulus	25°C	ASTM C-623	3.7×10^6	psi
Poisson's Ratio	25°C		0.26	
Knoop Hardness	25°C, 100 g	ASTM C-730	250	
Chemical Durability				
Resistance to Acid	5% HCl for 24 hrs. @ 95°C		87	mg/cm² wt loss
Resistance to Acid	5% HF for 24 hrs. @ 95°C		15	mg/cm² wt loss
Resistance to Base	N/50 Na_2CO_3 for 6 hrs. @ 85°C		0.12	mg/cm² wt loss
Resistance to Base	5% NaOH for 6 hrs. @ 95°C		8.5	mg/cm² wt loss

First, MGC is particularly useful when vacuums are involved. MGC does not outgas and is very vacuum tight (an order of magnitude better than Pyrex). Hermetic seals can be easily constructed between MGC and other glasses or ceramics using solder glass. MGC can be bonded directly to metal with epoxy or solder glass, or it can be metalized first and then brazed to metals. Since MGC is an outstanding electrical insulator it can be used for a variety of applications such as feedthroughs and standoffs as well as structural components.

The second class of applications is based around MGC's high temperature, high voltage electrical insulation capability. MGC has an extremely high dielectric strength and it is useful up to 1000°C. It can be used for substrates, for special insulators, for bushings, etc.

Machinable Glass-Ceramic is available in rod, bar and sheet form; the user can machine his part from the standard size pieces. Rod is available in 1", 2", 3⅝" and 6" diameters; sheet in ½", ¾" and 1" thicknesses, and bar in 1¾" x 5" cross sections. Since MGC is formed as a glass, it can be formed into "rough castings" to be finish machined later. Preforming shapes would be practical only for a large volume of pieces.

A LOW COST, SHORT LIFE, REUSABLE DIAMOND CORE DRILL

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All too often today we encounter jobs that require the drilling of straight, clean holes in glass components. For those of you who are fortunate enough to own a set of diamond core drills, this is no problem. For the glassblower whose department cannot afford the cost of a commercial set of core drills, the alternative approach, using carbide drills or a brass tube dipped in abrasive powder, is a slow and laborious method.

Those of you who were fortunate enough to attend the presentation by J. Peter Lunzer last year (a) might recall that during the film presentation, there was a short segment showing the construction of a homemade drill, but not much of a description was included. I have experimented with the idea and found that it solved many of my problems. I would like to share my findings with you.

THE DRILL

Basically, the drill is a soft iron tube with a 0.040″ wall thickness. For drills larger than 1″ diameter I have found that a 0.050″ wall thickness is preferable. Figure 1 is an example of the type of drill used in my shop. You will notice that each drill has a ½″ diameter shank. This allows it to be fitted to the adapter which will be described later. The working edge of the drill is notched with a sharp chisel, (Fig. 2-B). The notches should be about 1/32″ deep and as close together as possible (the closer the notches the greater the concentration of diamond particles at the working edge, thus a faster cutting speed and a longer working lifetime). Next, the notched edge is dipped into Duco Cement (b) and allowed to become tacky, after which it is dipped in 100 mesh diamond powder and allowed to dry. (Fig. 2-C). After the glue dries, peen the edge with a hammer to imbed the diamonds (Fig. 2-D). A close up sketch (Fig. 3) shows the end result. Be sure that the edge is mushroom-shaped as shown in the sketch. This gives the drill a working clearance and reduces drill hang-up.

The drill can be most easily dressed by drilling one or two holes in a glass plate. The drill is now ready for use.

When the drill loses its cutting efficiency, usually after about thirty to fifty ¼″ deep cuts, simply repeat the above procedure and you are ready to drill again. This may be repeated almost indefinitely as there is very little wear on the edge of the drill.

THE ADAPTER

Although you may drill simply by immersing the work piece in a coolant, there are times when this is not convenient due to its shape. In

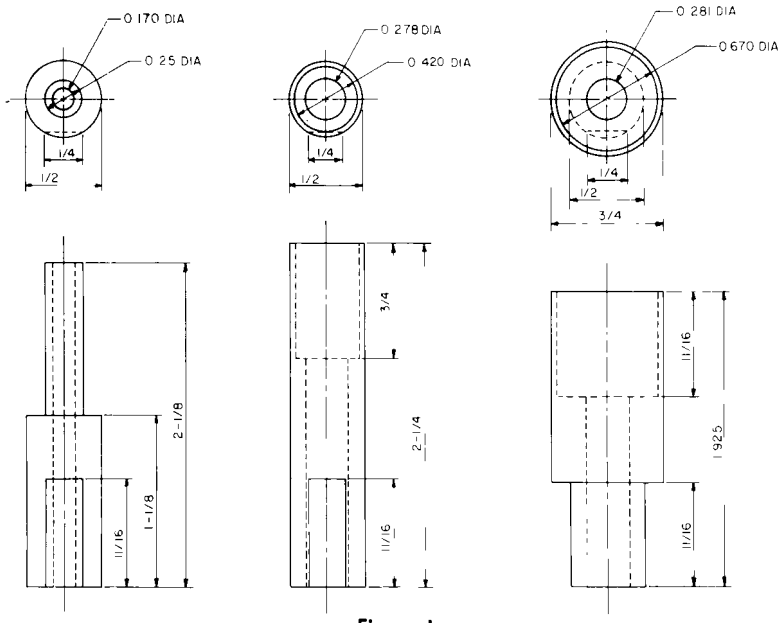

Figure 1

these cases a water swivel is almost a necessity. Also, applying the coolant through the center of the drill lubricates the drill much better and helps prevent core hang-up.

To fasten the drill to a standard water swivel requires that the drill be mounted in a collet which fits onto the swivel. If one were to permanently affix each drill to its own collet, this would require a considerable

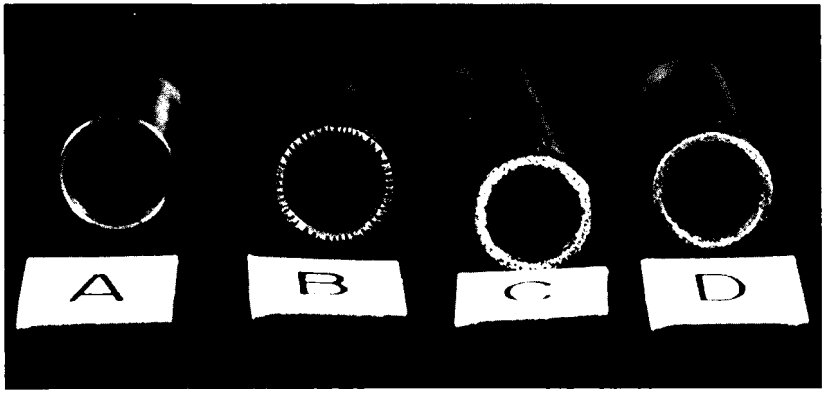

Figure 2

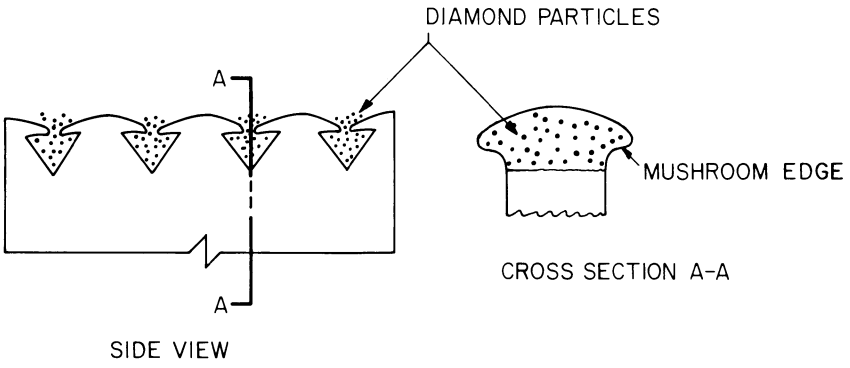

Figure 3

amount of machine shop time. Therefore, I constructed a collet which could be attached to any of my drills by using set screws. A detailed sketch is provided in Figure 4, and the complete set-up is shown in Figure

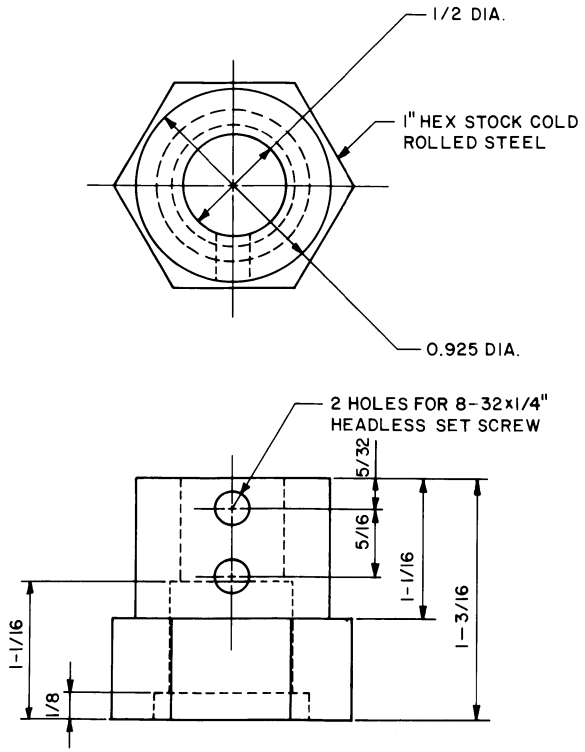

Figure 4

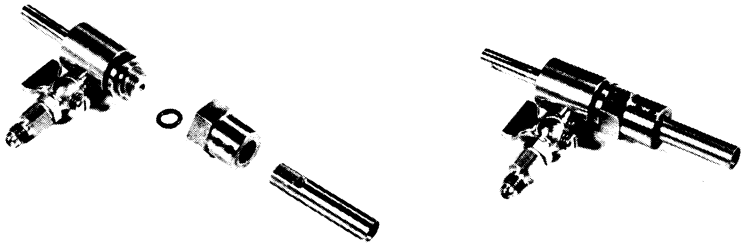

Figure 5

5. To minimize water leakage through the adapter, a rubber O-ring may be slipped inside the collet before screwing it onto the swivel.

One disadvantage of using the adapter is that it is difficult to control run-out at the working edge. This causes excessive tool wear and considerably shortens the drill life. However, if one considers the ease with which the edge can be reimpregnated, it is still a cheaper alternative than permanently affixing the drill to the collet.

CONCLUSIONS

Although I don't intend to imply that this drill is comparable to a commercial drill, when viewed in the proper perspective, it is a useful alternative, or a valuable supplement to a commercial set of drills. Its merits and faults are as follows:

1. Low Cost —
   It requires about $0.50 to $1.00 worth of diamond powder, depending on the drill size to impregnate the edge of the drill.
2. Re-usability —
   Although the drill requires some machine shop time to construct, this is negated by the fact that the drill may be reused almost indefinitely.
3. Expendability —
   One need not invest a lot of money in a drill needed for a one-shot or short run job.
4. Dimensional Accuracy —
   The drill is not as dimensionally accurate as a commercial drill. Due to the gradual wearing away of the mushroom edge, the drill will vary 10 to 30 thousandths of an inch in diameter during its lifetime.
5. Core Hang-up —
   Also, because of the mushroom edge you will experience more core hang-up than with a commercial drill.
6. Drill Speed —
   The drill does not have as great a concentration of diamonds at the working edge and, therefore, has a slower cutting speed.

ACKNOWLEDGMENTS

The author would like to express his thanks to Mr. J. Peter Lunzer, of Lunzer Industrial Diamonds Ind., New York, New York, the staff of the Physics Machine Shop and the Department of Chemistry at the University of Kentucky for their help and advice in preparing this paper.

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STRENGTH PROPERTIES OF GLASS

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INTRODUCTION

The strength of glass is an interesting and anomalous property. Since the breakage of glass can be its most frequent means of failure, such interest is more than academic. On the one hand the strength of glass is fairly constant ranging from about 8,000 PSIG to 11,000 PSIG for practically all commercial glasses. On the other hand, even within the same composition there can be a tremendous variation in strength depending on the glass surface. For example, a fine pristine glass fiber can have a strength of over 1,000,000 PSIG. The strength of glass is also affected by such conditions as environment, durations of loading, geometry, temperature, and state of annealing. Changes in the strength of glass under such conditions will be discussed and the practical implications of this for the scientific glassblower will be spelled out in terms of both the fabrication and designing of his glassware.

Glass in many respects is one of the most perfect engineering materials. It has no grain nor fibrous structure. When properly annealed it is highly isotropic and its physical properties like strength, hardness, thermal and electrical conductivity, refractivity, etc. are completely independent of orientation. Mechanically it obeys Hookes "law of elasticity" to the full letter of the law. Whereas metals and most other crystalline materials depart from the linear stress vs. strain relationship and show semiplastic flow at the upper regions of increasing stress, (Fig. 1), this is not so for glass.

From the very beginning of the application of stress to the very instant of breakage, glass exhibits strict linearity and concordance with Hookes Law. Coupled with this, the dimensional stability of glass, its excellent corrosion resistance, attractive economic cost, not to say anything of its transparency, should make glass a preferred engineering material. Unfortunately, the ready breakage of glass under certain conditions serves to severely limit its use.

It particularly behooves the glassblower and fabricator to understand the strength characteristics of glass and how they respond to the stresses of both his fabrication and of its subsequent use. With an informed approach this shortcoming in the otherwise attractive characteristics of glass can be compensated.

Pristine Glass

Interestingly enough the strength of glass under ideal conditions is tremendously high, exceeding even that of steel. Tests made on very fine

Figure 1
Stress-Strain Curve for Iron Wire

fibers of pristine or freshly drawn glass show a tensile strength of over one million P.S.I. Yet when strength tests are made on practical glass articles under typical conditions, values of only about 10,000 P.S.I. are obtained. Also familiarly enough, when a glass cutter moistens his scribed line on a glass plate, it can be broken more readily than when dry.

From the attempts to explain such curious behavior on the part of glass by such investigators as Griffith, Preston, Millikan, and others, a theory or rather set of theories has emerged regarding the strength of glass. While not completely satisfactory nor agreed to by all, they do present a fairly useful and acceptable picture of the mechanism of glass strength and breakage.

Surface Fissures and Stress Concentration.

One of the first explanations for the tremendous difference in the strength of glass under different conditions was presented by Griffith. In the first place, the strength of glass, based on computations of molecular forces should be very high, about 3,000,000 PSI, and this value in fact was approached by the pristine fibers.

However, the relatively low values found in practice with massive glass, Griffith inferred was due to stress concentration effects resulting from fine surface fissures. According to Griffith, the molecules in glass line up in groups that could result in decreased attractive force at certain locations and these could serve as incipient flaws. Rupture of glass concluded Griffith, entailed the formation of a new surface at the location of

the flaw, with a corresponding increase of surface energy. The breaking stress was mathematically expressed by Griffith as

$$R = \sqrt{\frac{2ET}{\pi \sigma C}}$$

Here, E is Young's modulus; T, surface tension; σ, Poissons ratio; and C, the half length of the crack.

Tests of this equation with data acquired from glass tubes and glass bulbs indicated that the maximum stress at the end of an advancing crack was 200 kilobars or approximately 3,000,000 P.S.I. This value is of the same order of magnitude as obtained from calculations of molecular cohesion and from experimental values obtained with fine pristine fibers.

Griffith's theory has been subject to criticism and modification by Murgatoyd, Jones, Charles, and others. Charles postulated a localized chemical reaction of the glass surface fissure with water vapor to explain the diminution in strength even of the fine fibers after exposure to the atmosphere for several hours. However, the basic concept of localized incipient flaws from molecular groupings or subsequent bruising of the glass, acting as severe stress concentrators has remained an effective means of explaining strength characteristics of glass.

Insofar as stress concentration resulting from flaws is the means of reducing the strength of glass, it would be well at this point to examine the nature of stress concentration. Let us assume that a flat rectangular bar has been found to have a certain tensile strength value. Now let another glass bar of the same dimensions and shape, but in which a small hole or notch has been made, be subjected to the same tensile test. Depending upon the notch geometry and size, it will be found that a much smaller tensile stress in relation to the removed material, will now suffice to rupture the second bar. In other words, the discontinuity acts as a stress concentrator, magnifying the stress by an amount known as the stress concentration factor.

This factor depends upon the geometry of the flaw. Ingles found that with elliptical discontinuities, stresses are increased by twice the eccentricity of the ellipse. Thus a narrow submicroscopic fissure as postulated by Griffith could have a hundreds fold stress concentration factor to account for the large degradation in strength for massive glass articles.

An interesting experiment in which it was illustrated that the strength of glass can be reversed from low to high was performed in 1846 by Brodman. By immersing massive glass specimens in hydrofluoric acid and tensile testing, he found that the strength of such specimens were increased several fold. Several hours later or after handling, the strength of the samples were found to have decreased to about their previous value. Etching the glass made the surface fissures rounder (Fig. 2). Subsequent handling permitted water vapor to contact them thus accelerating new microfissures.

Figure 2
Etching of Fissures

Strength Parameters

In light of the above, it will be helpful to examine strength values of glass under different conditions. In Fig. 3, we have a plot of typical breaking strengths of glass as a function of time. Curve A refers to the strength of annealed glass as tested in air. The sharp decrease in strength with time, particularly after the first few minutes as compared to the almost flat curve B which refers to annealed glass tested in vacuum, is explained well with the fissure-water vapor theory. By removing air and associated water vapor, the advance of microfissures and with it the static fatigue or decline in the strength of glass with time is markedly inhibited. This effect can also be achieved by immersing the glass in liquid nitrogen.

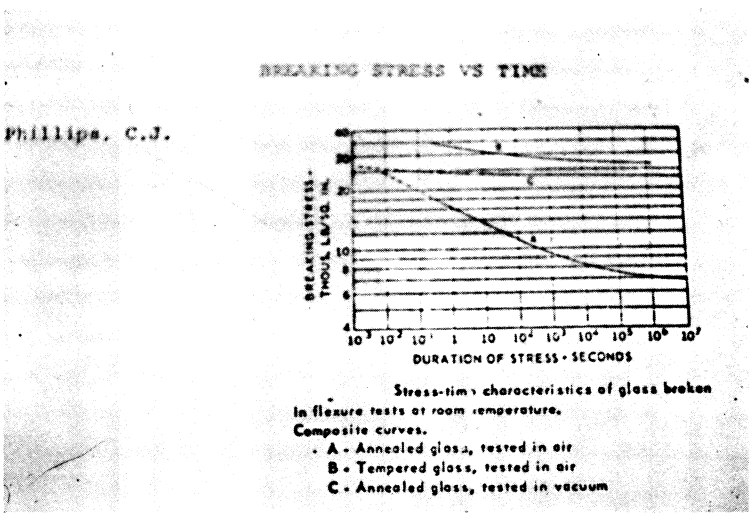

Figure 3
Strength of Glass as a Function of Time and Environment

In Fig. 4 we see the effect of temperature upon the strength of glass. At the minimum cryogenic temperature the maximum water removal effect is obtained and with it maximum strength. As ambient temperatures are approached the now present water vapor decreases the strength of the glass until it reaches a minimum in the region around 200°C. Because a thin molecular film of water is difficult to remove from the glass surface, the maximum strength on this portion of the curve does not occur until a little after 500°C.

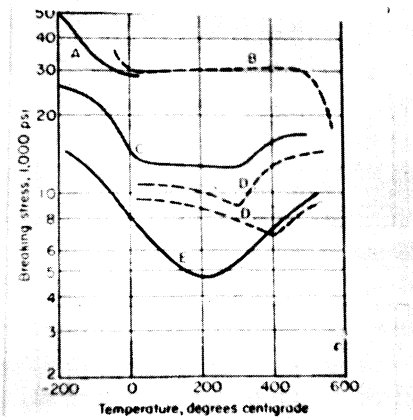

Figure 4
Strength of Glass as a Function of Time

Attempts have been made by Mould and Southwick and others to derive a universal fatigue curve. Glass specimens are generally pre-abraded in a uniform manner before testing.

Although this lowers the strength value, the glass samples are thus made more uniform in that they have comparable flow distributions. The adjusted strength condition is taken as σ/σ_n or the ratio of the strength in air to the strength in liquid nitrogen. This ratio is plotted against the log $(t/t_{0.5})$ or the log of reduced time to failure, where $t_{0.5}$ is the characteristic duration or the time to failure if a specimen is loaded to half its liquid nitrogen strength. Much remains to be done yet for a truly universal and accurate static fatigue curve and not the least of this is the accurate description of the state of the glass sample itself.

Strengthening By Design

In the light of such ever-present factors that can degrade the strength of glass, how can the glassblower or fabricator make his product more viable? There are two avenues. One approach is to design his product

and process to minimize surface tensile stresses. The second way is to neutralize tensile stresses by locking in a "permanent" compressive surface stress, namely tempering.

General and suggestive measures and cautions rather than specific and exhaustive ones to achieve the first approach are listed below.

1. Avoid excessive thermal shock and gradients. Glass is more vulnerable to thermal down-shock than up-shock because the former produces surface tensile stress while the latter is compressive.
2. Avoid scratches, bruises, and abrasion on glass since these increase and deepen surface flaws.
3. Hot glass in the temperature range below the strain point is more susceptible to breakage under thermal strain than when at room temperature. Not only is the strength of the glass lower, but in addition the coefficient of thermal expansion is greater.
4. Anneal glass as soon as possible after annealing since glass is weaker under long term strain than under short term strain.
5. Avoid glass to glass contact immediately after annealing or chemical cleaning because the coefficient of friction is at a maximum and the surface is more susceptible to abrading.
6. Avoid excessive "shape" stress concentration by avoiding sharp corners and reentrant radii. Use large radius of curvature.
7. Avoid excessive tensile stress intensity by providing adequate wall thickness for intended stress.
8. By coating glassware with lubricants, impacts tend to become glancing rather than bruising. Silicones, stearates and compounds of tin, titanium and others have been used for this purpose, but side effects on electrical and other properties have to be considered.

Strengthening By Tempering

With regard to tempering (Fig. 5) or locking in of compressive stresses on the glass surface, the most usual method has been thermal tempering. Here a compressive stress is locked in on both glass surfaces by first heating the glass to a temperature between its annealing and softening points and then quickly chilling it on all surfaces. The outer glass surface cools and becomes rigid first. As the inner portion cools it tends to shrink. Insofar as the outer layers are now "rigid", this contraction serves to put these layers into compression. It is this surface compression that has to be overcome by any potential breaking stress before fracture can occur. Thus if we take 1,000 P.S.I. as the practical working strength of the glass and then add 1,000 P.S.I. of compressive stress to it, the effective strength of the glass is doubled. The amount of tempering that can be imparted to a glass depends upon its thickness and coefficient of expansion. Thus more strengthening can be imparted to a soda lime glass than to a borosilicate one because of the lower thermal expansion of the latter. Modifications have been made on the above simple theory of tempering to bring it more in line with the "elastic" character of the so-called "rigid" condition as far as glass is concerned, and to reflect the fictive temperature equilibrium condition that is actually frozen in the glass.

Figure 5
Polariscopic View of Thermal Tempered Sight Glass

Recently, ion-exchange methods have been developed for chemically tempering glass. Here, a substitution is made of a larger ion for a smaller ion in the glass surface, like sodium for lithium, or potassium for sodium. This is done by immersing the glass, soda-lime, or alumino silicate, among others, for several hours in a molten bath of the appropriate salt. The effect of the larger ion in the smaller site of the smaller replaced ion is to cause a strong surface compression. Instead of being limited to increases in strength of 2 to 4 times as with thermal tempering, strength increases of even 10 can be obtained with chemical tempering. Other old and new methods of surface compression have also been employed, such as casing with a lower expansion glass, the utilization of devitrified crystals to produce compression, and dealkalization of the glass surface with the oxides of sulfur.

CONCLUSION

In summary, although the theoretical strength of glass is very high, this can only be realized under very special conditions. For glass articles of practical use, the effect of fissures, water vapor, and surface damage is to lower the strength to about 10,000 P.S.I. Add to this the effect of static fatigue and the necessity for a safety factor, and we are left with a typical design strength of 1,000 P.S.I. With all surface tensile stresses kept within respectable limits and with reasonable care, glass can safely provide an important material for engineering, science, and commerce. For higher stress requirements, the appropriately tempered glass can be used with an increased design strength, characteristic of the type of temper employed.

OBSERVATIONS IN A GLASS SHOP

R. J. BRUNFELDT

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Bartlesville, Oklahoma

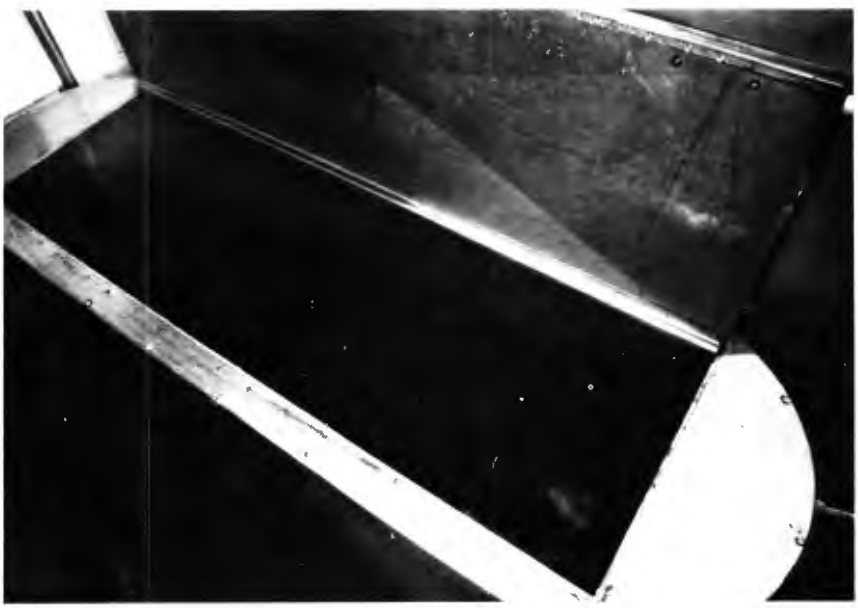
Science and technology have harnessed nuclear energy, developed antibiotics, produced color television and landed men on the moon. In each of these accomplishments the scientific glassblower has played some small but significant role. Yet it would be hard to find an occupation with as little consistency of training and background as the glassblower. Many times our job title doesn't really say what we do or indicate the necessary prerequisites.

We are a small group with specialized skills but overlapping many disciplines. Because of this our tools and techniques are also highly diverse. To the craftsman few events are more pleasurable and stimulating than visiting the workshop of his professional counterpart. This is especially true of the scientific glassblower, for by very reason of our disparity in background and sometimes considerable geographic distances between us, we find ourselves in occupation isolation. The sharing of our collective body of knowledge is the foundation upon which the American Scientific Glassblowers Society is based and the major force in dispelling the effects of this isolation. In this spirit, this presentation will take you for a brief visit to the glass shop of my employer, Phillips Petroleum Company. Because of the obvious limitations of this visit I will attempt to touch on only some of our tools and modifications to our equipment which may be unusual or whose purpose is to provide us with a maximum of functional flexibility and safety.

The shop is located at the Research Center (A), a campus environment set aside for our company research activities. We are located in a building designated for service to these activities. Proximity to the machine, welding, sheet metal and electrical shops is particularly convenient because of the integrated nature of our work. (B) the glass shop occupies two rooms. The fabricating room where our benches, lathes, ovens and stock are located, and the "back room" where our more messy operations are carried out — cutting, drilling, grinding, silvering, cleaning, etc. We are located on the ground floor with an outside entrance for receiving and shipping materials and the servicing of our hydrogen and oxygen manifolds. These gasses are piped in, we have no cylinders in the shop. There are three doors leading to the shop approximately evenly distributed for safety. Located near two of these doors is a switch leading to solenoid valves on our hydrogen, oxygen and gas lines. The valves are located out of the building near the source of these services. In the event of the need to evacuate the room, the switch at the door will interrupt the flow of these gases. The solenoids are normally closed so that a power failure will have the same effect. Exhaust hoods are located over each bench, glass lathe, sink, and cleaning tank.

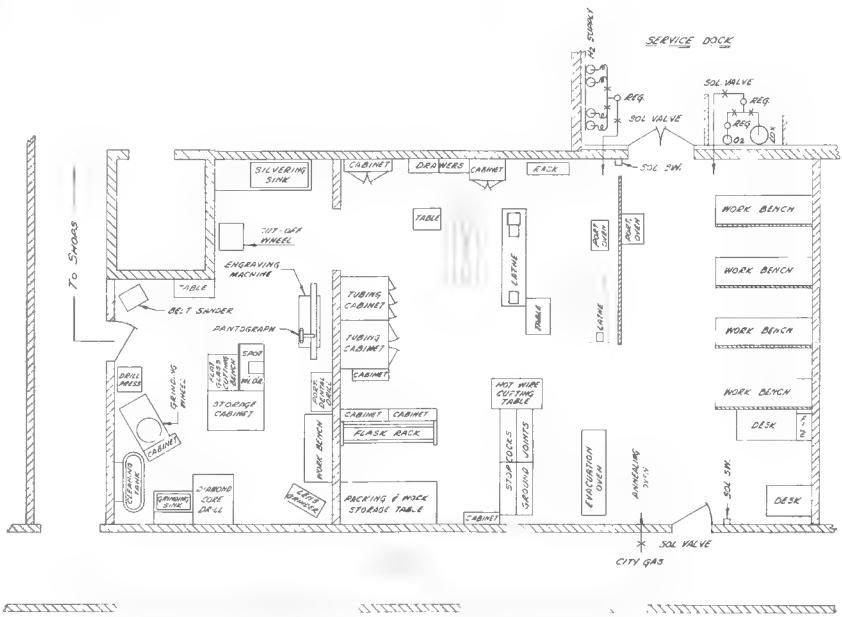
The cleaning tank (C) may be of interest to those of you who are called upon to repair equipment in which silicone grease has been used. You are, I am sure, well aware of the difficulties resulting from attempting to work on glassware which has not been thoroughly cleaned of this material. All too often a distillation head or receiver will arrive for repair, looking as though the operator thought silicone stopcock grease was peanut butter and the glass lines were home made bread. Stoddard solvent and alcoholic potassium hydroxide are the usual cleaners. This cleaning is usually a time consuming, tedious job. To resolve this problem we have a cleaning tank in our "back room" containing a hot, concentrated solution of a commercial rust stripper. This material, although made to remove rust from steel piping and tanks, does a beautiful job of cleaning glassware by simply soaking for about an hour, rinsing with water, and drying. Most important — the troublesome silicone is gone. The material is caustic, however, so that the use of rubber gloves, a covered tank, etc., are necessary safety precautions.

Most of us use one of the standard hand torches (D) in certain types of work. These are generally supplied with two or more changeable tips for necessary flame adjustments. We have found that some of the burner tips made for the electronics industry for use on their sealing machines and automated equipment are excellent all purpose tips for hand torches. These tips have a fine pilot ring around the center hole making the flame very stable. These allow us to pull the flame in to a very fine point or go out to quite a large flame without "blowing out". It will generally be necessary to modify the original torch neck by cutting the original threads



(C$_1$) Cleaning tank

(A) Phillips Research Center



(B) Sketch of general floor plan showing solenoid valves, switches and gas cylinders

(C₂) Cleaning tank

off close to the bend and attach a shortened ⅛" pipe coupling in this place. These tips may be used for torches burning natural gas, propane, mixed gas or hydrogen.

We have modified our glassblowing lathes in several ways to reduce set up time and give us increased flexibility. (E) A swivel support bracket is attached to each head stock. The swivels are of our own design to reduce flow resistance and "breathing". (F) A combination swivel support and hand torch mount is used on each tail stock. You will notice that we have two torches in this mount. We find this burner selection convenient.

(G) The spindle pressure control valve is used to divert the direction of blowing pressure with an outlet for attaching our blow hose at the front of the lathebed. Copper lines have been installed at the original spindle pressure control valve which replace the lines intended for foot control of air to the spindles. (H) This allows us to blow left, right, or both, with breath control, without separate hoses or tees.

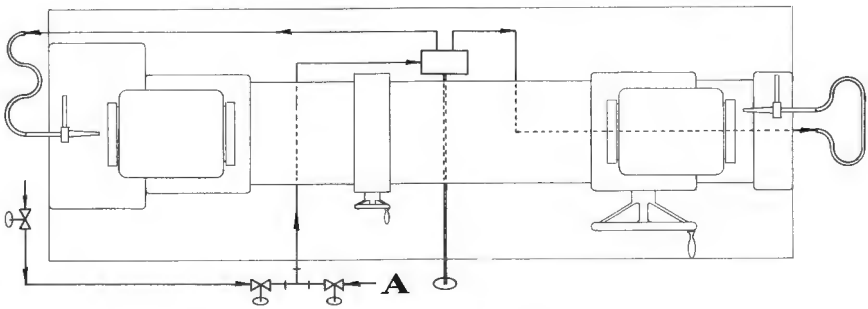
(D_1) Handtorch

(D_2) Handtorch

(E) Headstock swivel support

(F) Tailstock swivel support with torches in holder

(G) Front of lathe showing hose attachment

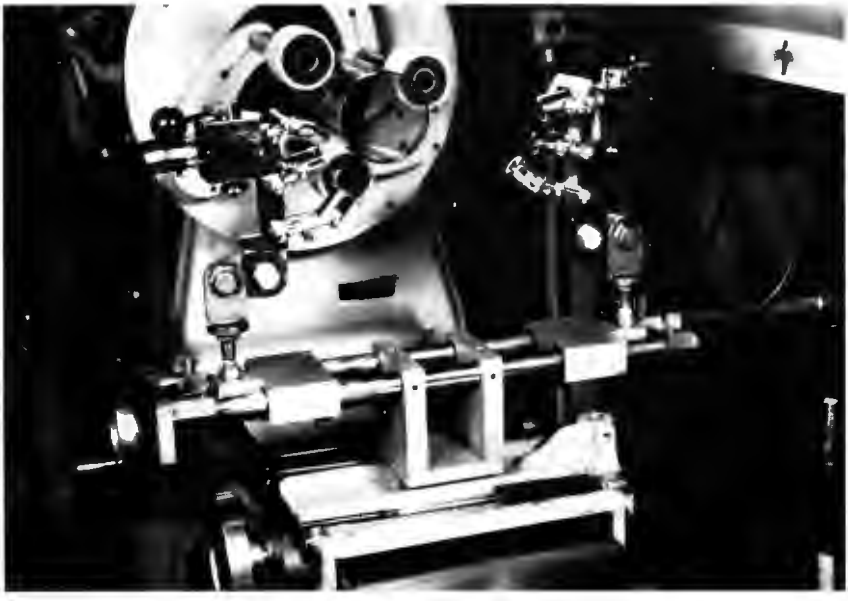


(H) Sketch of blow schematic of lathe

(I) Each lathe has been plumbed with a three-way solenoid valve between the hydrogen and gas lines feeding the bed burners and hand torches. Flipping the control switch allows us to select either of these fuels. The annealing burners, however, cannot receive hydrogen.

We have designed a burner assembly and mount for our large lathe. (J) This assembly is a cross-fire mount for six burner tips. With three tips on each side attached to a single lever, valve block; we can, by turning the top mounted control lever, select one or three tip function. (K) The block assembly is mounted on a vertical support which can, by crank adjustment, be moved in or away from the glass as size requirements demand. The vertical support can also be turned for moving in on a

(I) 3-way solenoid behind lathe



(J$_1$) Large lathe burner assembly (broad)

(J_2) Large lathe burner assembly (broad)

(K) Close-up of block burner valve

(L) Burners turned for shoulder seal

shoulder or away from a sealed tube which would bump the burner while rotating. (L) This unit is permanently mounted to the fire carriage and is used only on our large machine.

It is my hope that somewhere in this brief visit a thread has been added to the fabric of the glassblowing fraternity in the spirit of this Society. My sincere appreciation is extended to management of Phillips Petroleum Company not only for allowing me to bring this visit to you but for the recognition of the need and commitment to excellence in research.

A TEFLON AND GLASS SPINNING BAND COLUMN

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INTRODUCTION

The initial design phase incorporates drawings commensurate with function, utility, strictures, fabrication criteria and materials. That is another way of saying: first make a rough sketch showing the easiest way to make the apparatus so that it will work. Fig. 1.

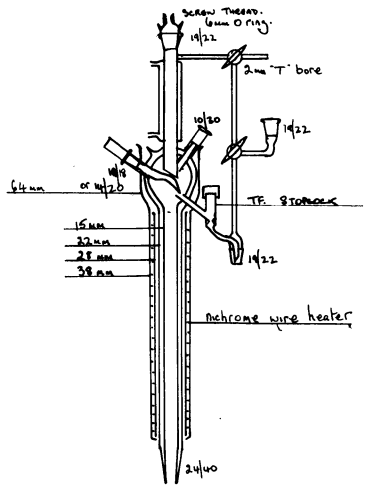


Figure 1

A spinning band column is essentially a one piece distilling column with a reflux ratio head, and where the band, which may be rotated by a stirrer motor, is analogous to column packing. The column under discussion was designed to give a sharp separation, between two high boiling temperature, chlorine compounds. The situation immediately suggested a spinning band column, with the band material to be a noble metal or teflon. The latter was chosen by fiscal necessity.

When designing distillation apparatus the five most important parameters to be considered are:

a efficiency, i.e. the total number of plates, H.E.T.P.,

b hold up, (defined by Carney),

c throughput, i.e. amount of material reaching the top of the column per unit time,

d pressure drop, i.e. differential pressure along the length of the column,

e inertness, i.e. non-reactive materials used, column, band, stopcocks, etc.

At least a cursory understanding of distillation techniques is necessary before the design can take shape. I am pleased to recommend glassblower Ed Wheeler's book to you, particularly the chapter on "Fractional Distillation", which has a wealth of references in the footnotes. Specific articles about spinning band columns are available in technical product bulletins and various journals.

CONSTRUCTION

The heart of the apparatus is the spinning band. Select a piece of ¼" x 18" heavy wall tubing with an i/d of ~2½ mm. Seal one end so that it may be attached to a 6 mm rod. Blow and shape a hole ~½" from the closed end so that it can accommodate the teflon rod, which should be 2' long and ⅛" diameter, with slightly tapered ends to ensure a snug fit into the ¼" tubing. Wrap the teflon around the tubing to about 15" along its length and make a similar hole in the side of the glass to accommodate the other end of the teflon. Fig. 2.

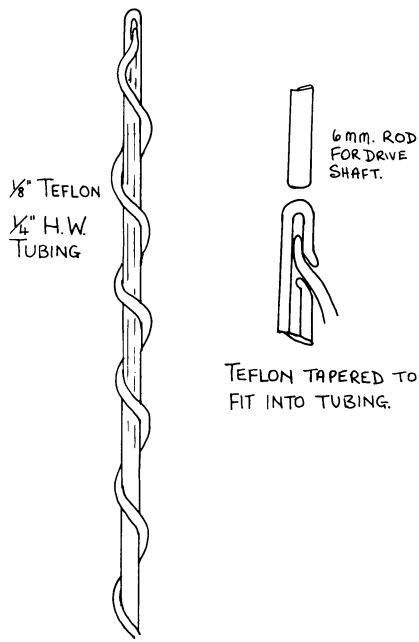


Figure 2

Select a length of 15 mm tubing into which the teflon spiral will fit and rotate contiguous with the glass wall. Shape one end to pass through a 24/40 inner joint, so that a dewar seal may be performed later. Coincident with the length of the band blow a prolate spheroid 45 mm x 55 mm

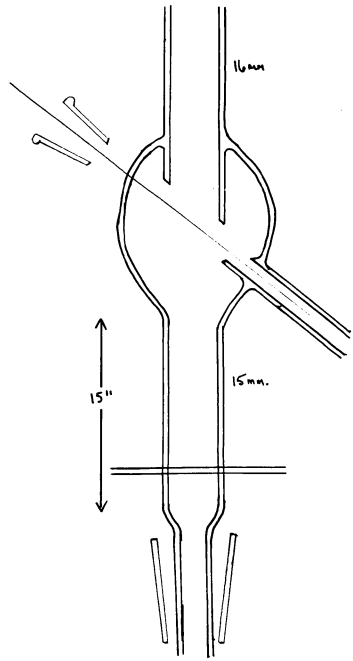


Figure 3

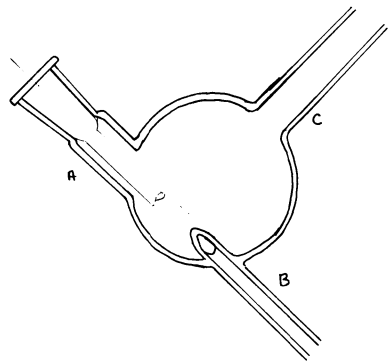


Figure 4

and through the rounded end seal concentrically a piece of 16 mm tubing about an inch long and ground to a drip tip, Fig. 3. While the seal is hot make joins and seal tubes A. B. and C., in a 120° radial configuration. For seal A attach a 14/20 outer joint, which may then be used for the alignment of seal B, which must also clear the 15 mm axis needed for the band to have complete freedom. Fig. 4.

To a 15″ length of 22 mm tubing attach a 24/40 inner joint, at the other end seal to a 6″ length of ~57 mm tubing into which the inner assembly will fit. Fig. 5. Seal the top and attach a prepared condenser coaxially and in a lathe. Complete seal A with the previously used 14/20 outer joint, seal B with a short length of glass the same size as the stop-

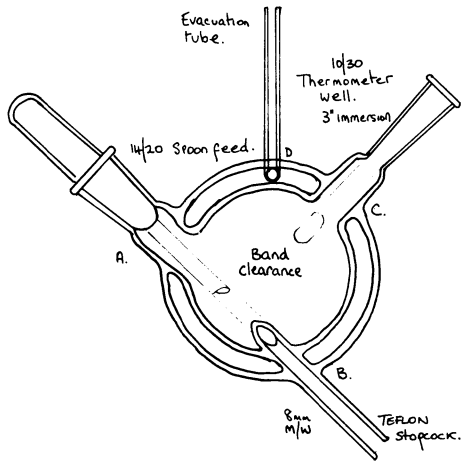
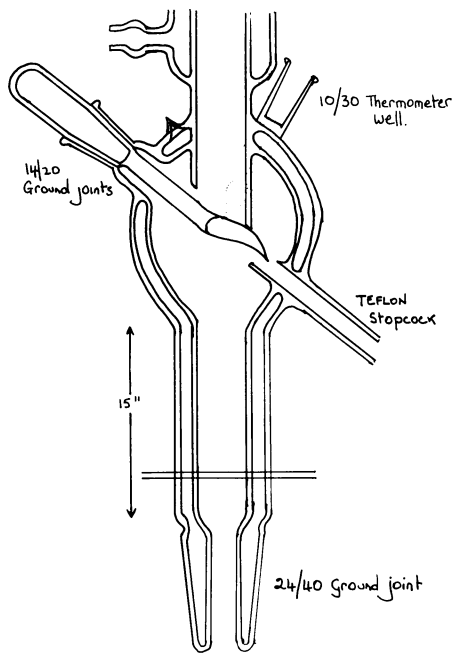


Figure 5



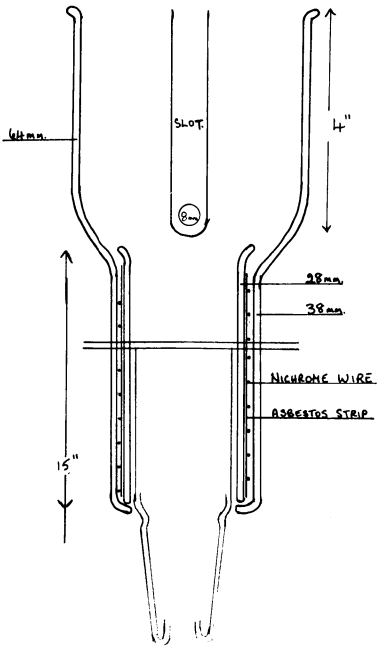


Figure 6

cock which will be added later. Seal a 10/30 outer joint to C to allow for a 3" thermometer immersion, join a short tube at D to facilitate the dewar seal at the 24/40 joint, and for evacuation and subsequent seal off after annealing. A heating jacket to compensate for thermal losses along the column is described in Fig. 6. The upper vacuum take off assembly is self evident. However care needs to be exercised in preparing the 14/20 inner joint spoon feed-through, where, by rotating the joint through $\sim$45°, the condensate may drip along the spoon to point B, or reflux. A judicious positioning may split the drip and afford (a not too precise) reflux ratio.

The whole apparatus took a full day to complete (less annealing and evacuation bake out time) with a minimally equipped glassblowing facility and low materials cost. Most importantly, it functions quite well with an efficiency of $\sim$100 plates low hold up, high throughput and minimal pressure drop.

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A MULTI-JACKETED GLASS APPARATUS FOR MEASURING THE LIMIT OF SUPERHEAT OF ETHANE AND ITS MIXTURES

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Mr. Chairman, Members of the Symposium Committee, Fellow Glassblowers, This paper deals with the construction of a Borosilicate Column used to determine the limit of superheat for ethane and its mixtures.

The first figure shows the completed column to scale with all dimensions in centimeters. In actual use, it is rotated 90° clockwise so that the 9 cm dia pot is on the bottom, the 2 mm O.B. pressure stopcock is on the left, and the open end of the 41 mm inner tube is at the top. In the figure on the screen or as shown on the drawing, the left thermal control jacket outlet tube actually comes up from the plane of the screen or drawing and the right upper inlet tube goes into the screen or drawing. The inlet and outlet tubes are shown in this "half section drawing" to be in the plane of the screen or drawing to show their relationship to the vacuum jacket. The 1 cm long 2.4 mm I.D. capillary tube, shown inside the 90 mm dia pot at the right end of the drawing, was sealed in without distortion by first laying rod around the tube to form a ring and then sealing it into the 41 mm inner tube as shown in Figure II.

This figure also shows the distances between the five major ring seals and their pre-seal diameters. All sketches are shown in the same orientation to show progression from one state to the other.

Figure III shows the lathe arms of the "HSJ" Model Litton with the headstock mounted EC Universal Planetary 3-jaw chuck with 2″ long 2″ diameter high purity graphite rings placed at the end of the 4½″ arms.

The blowhose is attached to a swivel connecting via rubber tubing to the 41 mm tube, from the headstock side, making possible the sealing of the first major ring seal between the 41 mm tube and the 90 mm pot tubing. The 90 mm tubing was sealed to the 41 mm tubing by blowing from the tailstock spindle hose nipple. Upon completion of this first major ring seal the hole was blown for the sealing of the temporary 8 mm sidearm. The sidearm is attached to the 90 mm pot tubing by blowing via rubber tubing threaded through the jaw pipe of the headstock chuck to the head spindle hose nipple. After this seal was made it was possible to form the flat bottom of the pot, 7 cm from the ring seal, by blowing through this temporary sidearm. Upon the completion of the flat bottom, the sidearm was bent to the right parallel to the center line of the column as shown in Figure IV.

This figure shows Litton's 6-jaw interdigital chuck holding the 41 mm tube and also the 52 mm thermal control jacket tubing for the sealing of the second major ring seal and the outlet tube.

*Work performed under the auspices of the United States Atomic Energy Commission

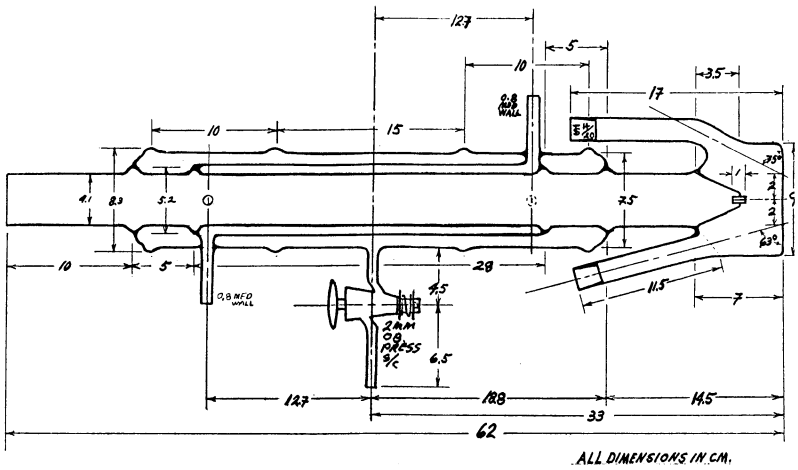

Figure I

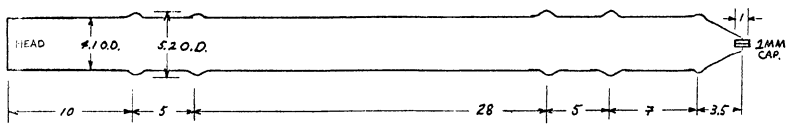

Figure II

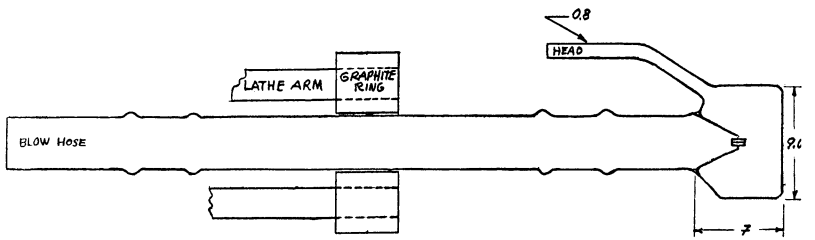

Figure III

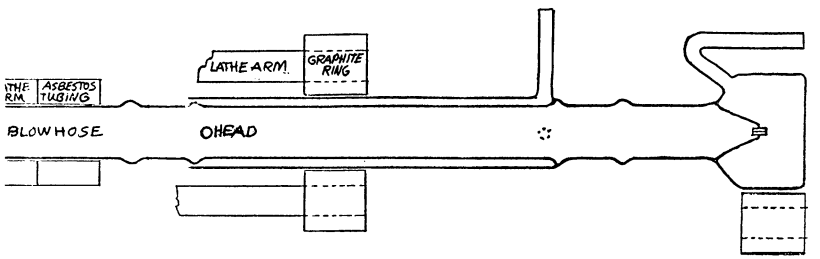

Figure IV

The outlet tube, having been presealed, was used for blowing, via the headstock spindle hose nipple, in making the second major ring seal and the sealing of the outlet tube.

Figure V shows the sealing of the third major ring seal. Note please that the graphite collars on the tailstock end are 4″ in diameter rather than 2″ as shown in the drawing.

Figure VI shows the sealing of the vacuum jacket tubing with the 2 mm O.D. pressure stopcock sealed in place so that it can be used for blowing, via the headstock. The thermal control jacket's inlet and outlet tubes were sealed off and blown out to fit inside the 75 mm tubing used for the vacuum jacket.

After the fourth major ring seal was made, with its expansion ring formed, the tubing of the vacuum jacket was heated and allowed to contact the outlet tube between the walls. A glass rod was used to pull out the glass in the center of the inlet tube and the 8 mm medium wall sidearm was sealed in place by blowing through the spindle hose nipple of the tailstock. The expansion ring between the inlet tube and the 6-jaw interdigital chuck was then formed.

Figure VII shows the sealing of the fifth major ring seal and the addition of the outlet tube with the expansion rings on both sides.

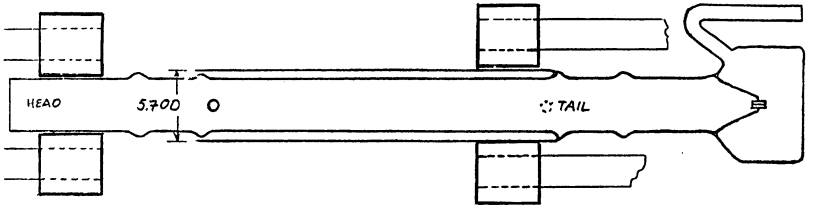

Figure V

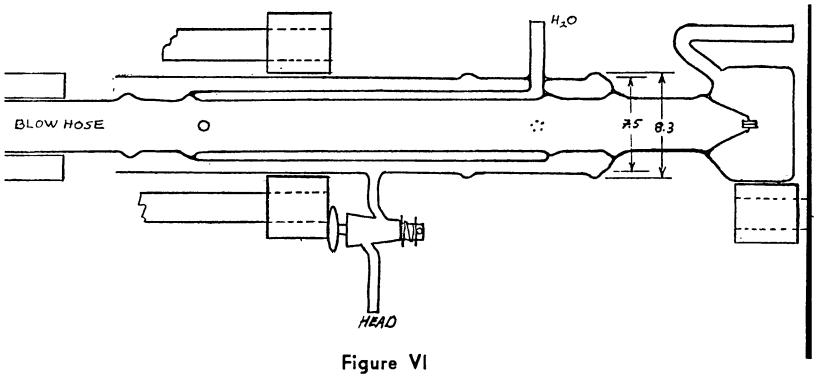

Figure VI

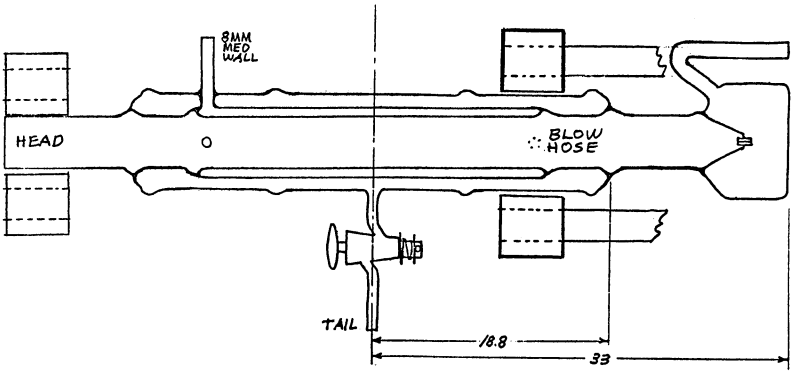

Figure VII

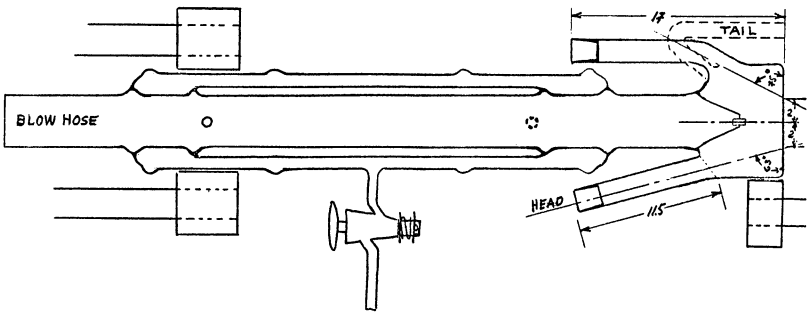

Figure VIII

Figure VIII shows the sealing of the straight 14/20 joint by blowing the hole in the pot via the tailstock spindle hose nipple, through the temporary dotted 8 mm sidearm. The joint was then sealed to the hole by blowing from the headstock spindle hose nipple via rubber tubing threaded through the headstock jaw pipe and the bore of the stopcock.

I took advantage of any anchor points through which the rubber tubing could be threaded to prevent shifting the position of the 14/20 joint during the annealing steps. Throughout the sealing of the vacuum jacket ring seals a polariscope was mounted behind the lathe allowing constant monitoring of stresss or strain in the glass.

The small, dotted line, temporary tube from the 90 mm pot was then pulled off and the hole blown for the bent 14/20 T outer tube to be sealed in place. After torch annealing, the column was removed from the lathe and annealed at 1040°F overnight.

CONCLUSION

In actual use by William Porteous, a thesis parts graduate student from Massachusett's Institute of Technology, the column, mounted in the vertical position, would be at room temperature and atmospheric pressure, at the top. The vacuum jacket would be evacuated before each experiment and the thermal control section would be controlled to a temperature between -30 to 10°C. The 90 mm pot was submersed in a slush bath at -95°C.

The liquid being tested is first emulsified with another liquid in the pot. A small amount of the emulsion is then forced by pressure via the bent 14/20 joint through the capillary into the 41 mm section of the column. A thermocouple is inserted in the straight 14/20 joint making possible pot temperature measurements during the experiment. The lighter drops of the test liquid soon begin to rise because of the warmer temperature of the upper part of the column. The drops continue to warm as they ascend in the column and eventually vaporize with a "pop" considerably above their normal boiling point. The temperature at which they vaporize is called their limit of superheat. (The limit of superheat is defined as the maximum temperature to which a liquid may be heated at atmospheric pressure.) This temperature is determined by positioning a thermocouple below the area, in the column's top, where the drops are vaporizing, and by placing a second thermocouple above the area. By the correct positioning of these two temperature sensing devices the area or zone temperature where the drops are vaporizing can be determined giving the temperature of superheat of the test drops.

This apparatus was successfully used for superheat determinations, withstanding all stresses, thermal and mechanical, without damage and is now on display in my office at the laboratory.

AN IMPROVED BURNER VALVE

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Texas Division
Freeport, Texas 77541

The versatility of the Corning style silent blast burner used for off-hand glassblowing has been severely handicapped by the incorporation of needle-type valves to control the mixing of the gas. A modified ball valve has been developed for this service. This valve allows the glassblower to switch from one type of flame to another with a minimum of manipulation. In addition, the valve will withstand the temperature requirements, apparently never needs lubrication and has a life expectancy in excess of three years.

The advent of the silent blast burner by GENERAL ELECTRIC[1] and its further development by CORNING GLASSWORKS[2] gave to the scientific glassblower a new tool with which they could best pursue that state of the art known as off-hand glassblowing.

Gone, but not forgotten, was the roar of the old cannon, pre-mixed gas burner. I can still hear the many explosive reports from this type of unit as the oxygen mixture became too rich. At times, certain gremlins always managed to change the gas mix when our patrons became irritable. As if by magic, the shop took on a peaceful atmosphere. The shouting was gone, jobs and techniques could be discussed, it even became a pleasure to answer the telephone. No longer was it necessary to wait for a happy hour to work on a delicate job. The versatility of this burner became quickly established and it was all too soon realized that its limitations were governed only by its operator.

A glassblower's expression of his full talents is best described by his ability to switch from job to job, whether small or large, simple or complex, with a minimum of effort. Isn't it great to see this artisan combine glass, metal, flasks, joints, stopcocks, and etc. with the aid of several hours of concentrated effort and perhaps sweat to produce an object of beauty.

The silent burner allows the glassblower to instantly switch from the smallest of pin point flames to those necessary to work four inch glass pipe. Its limitations were, to a big extent, governed by the cock type valve furnished as original equipment. At times, the valve's smooth operations was affected by the heat sink design of the burner and by the severe lubrication requirements caused by this heat. The valves controlling the oxygen were a constant source of trouble and required periodic overhaul. A good, safe reliable lubricant for oxygen was hard to find.

Soon the original valves were replaced with a needle-type valve. The heat and lubrication problem was now improved but the off-hand technique of glassblowing suffered. The quick change from one type of flame to another became a chore because a fair amount of manipulation was required to change the flame characteristics. As an alternate to flame changing, the glassblower now resorted to hand torches for flexibility.

Some three years ago we sought to develop a trouble free valve; one that required little or no lubrication maintenance. Also, one that could stand the temperature requirements. A stainless steel ball valve with a Teflon® seat was chosen; stainless steel because of its poor heat conductivity. The Corning burner's body can reach a temperature of 90°C. The Teflon, of course, was chosen for its lubrication characteristics. Following is a series of pictures to describe the valve, its modification, and flow pattern in comparison to other valves in use.

This is the Whitey[3] valve that was chosen for modification. It is a Number 43F2 from off the shelf stock. (Figure 1)

Figure 1

Figure 2 shows the radiator fins that were machined into the valve body to keep the valve cool. (Figure 2) Its temperature remains comfortable under all operating conditions. We also machined the bulk of

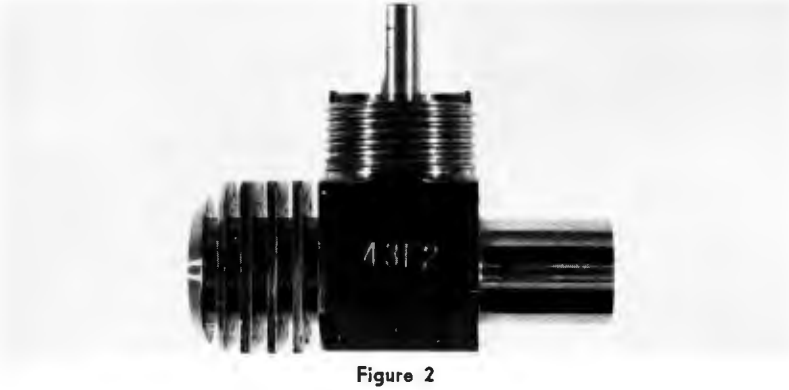

Figure 2

the metal off of the other end of the valve. This cuts the weight down and still gives plenty of body for the gas pressures that are involved. The inlet O.D. is about .525".

Figure 3 shows the valve disassembled. (Figure 3)

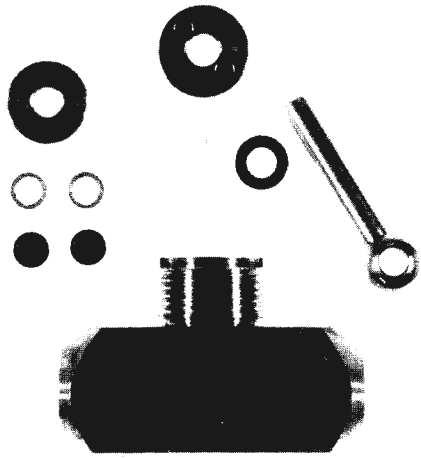

Figure 3

The restricting orifice was filed from 3/16" (.187") brass rod using extra slim taper files. The upper portion of the "V" slot is .140" wide and is about .137" deep. Using a jeweler's saw, a cut .017" wide x .025" is made in one end of the orifice. The slot gives the pin-point burner necessary for semi-micro work. The taper geometry can be varied at will.

Here is a sketch of the completed orifice. (Figure 4) The slug is no longer than .380" long. Note the position of the slot cut.

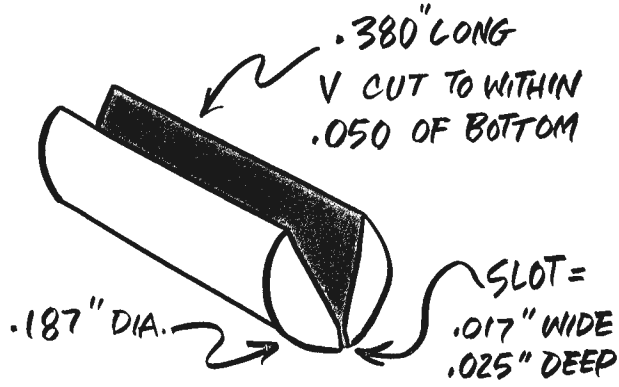

Figure 4

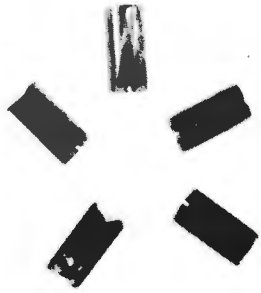

Figure 5

Figure 5 is a shot of several completed orifices.

The next figure shows the orifice soldered into the ball stem. (Figure 6) Stainless steel softer solder is used for this purpose. The orifice is first tinned, then slipped into the fluxed ball stem. A gentle amount of heat will fuse the orifice to the ball. Make sure that the orifice is orientated in the proper direction and that the slot end is pressed to the extreme end of the ball annulus.

Figure 6

The built-in stop located on the underneath side of the handle should be removed. The upper handle has had the stop removed. The valve can thus be used for right or left-hand service. Also, the plastic handle can be reshaped as desired.

A needle point flame is obtainable with this valve at 13 psi pressure. The maximum flow thru the orifice is greater than that of a standard $\frac{1}{8}$" valve. The torque necessary to move this valve can be set as low as 10 inch ounces. The torque is adjustable by tightening the packing gland located immediately below the valve handle. This adjustment can be made without removing the valve from the burner body. Just a flick of the fingertip can instantly change the flame's intensity. It may take some practice to get used to this very sensitive valve.

Figure 7 shows the flow curves of the cock, needle, and the ball valve. The flow patterns of the ball and cock valve are very similar. The needle valve handle has to be rotated $2\frac{3}{4}$ turns or 945° to achieve the same maximum flow of the ball or cock valve.

We are experimenting with other ball valves for burner service. Even inexpensive ball valves have proven satisfactory.

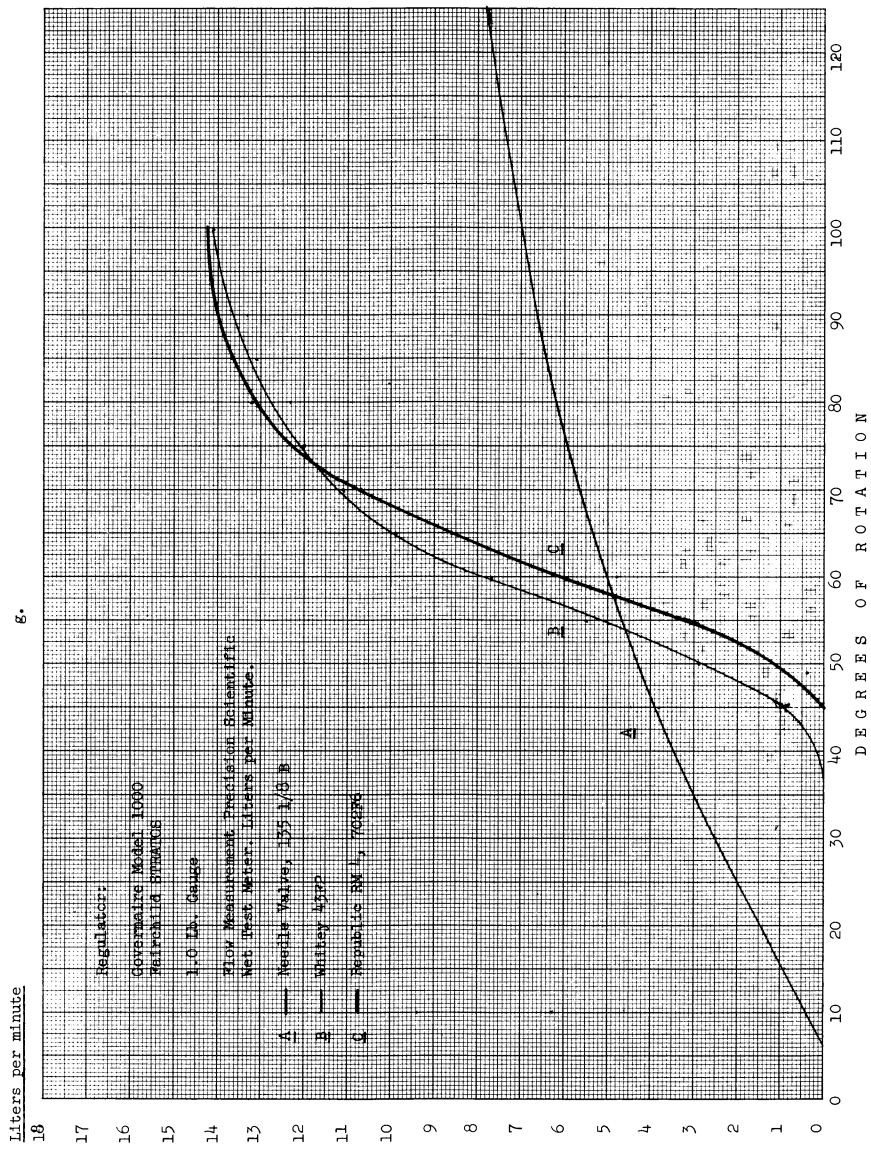

Figure 7

The redesign of the ball valve can be accomplished in 1 hour with a minimum of equipment.

1 General Electric Company.
2 Corning Glass Works, Corning, New York.
3 Whitey valve, manufactured by Whitey Company, 5679 Landregan Street, Oakland, California, 94662.

OFF-STANDARD SMALL DIAMETER BOROSILICATE GLASS TUBES

G. SARFI*
Hydro-Quebec, Institute of Research
Varennes, Quebec
Canada

INTRODUCTION:

A large variety of borosilicate glass tubes of various diameters are available commercially. But in many cases, glass apparatus manufacturers design particular instruments for certain purposes using off-standard inside or outside diameter tubes. These tubes are also available in large quantities from the manufacturers for the industry at a reasonable price. There is also expensive equipment on the market to convert diameters of glass tubes. For a great number of these specially designed glass instruments, it could well be profitable to use off-standard tubes. However, the availability of these tubes in small quantities does not permit production of such instruments. In some cases, even the idea of repair has to be rejected.

In this article, simple experiments are described to produce small diameter, off-standard borosilicate glass tubes of a reasonably good precision.

EXPERIMENTS:

A) To one side and both ends of a four feet long, 25 mm. outside and 22 mm. inside diameter quartz tube, a piece of quartz rod, 6 mm. in diameter and three inches long was fused. The quartz rod was bent to a "U" shape in order to fuse its other end to the opposite side of the same end of the quartz tube, as shown in Fig. 1.

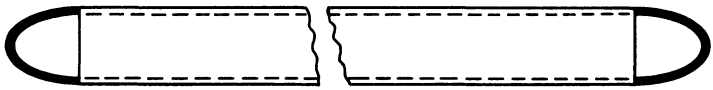

Figure 1
Quartz Tube

One end of a 90 cm. long, 7 mm. outside diameter, standard wall borosilicate glass tube was extended with the same type of glass rod, 8 mm. in diameter, 2 inches long. This rod was bent backwards to fuse its both ends together to form a loop. The other end of this tube was extended with a capillary tube of about the same dimensions as the glass rod at the previous end. This extension was then bent to about 180° to form a hook, in such a way that, this hook and the glass rod loop at the opposite end were in the same plane. This is shown in Fig. 2.

*Presented orally by Vincent C. DeMaria

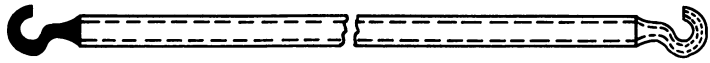

Figure 2
Borosilicate Glass Tube

The outside diameter of the above 90 cm. long tube was measured at different distances from one end of it, to an accuracy of ±0,001 of an inch (0,025 mm.). These readings were converted into milli-meters and recorded as shown in Table I. A vernier caliper, equipped with dial (reading to ±0,001 of an inch) was used. In most cases, about 0,003 of an inch out-of-roundness was observed. In these instances, the average readings were recorded.

A platinum wire, 0,020 of an inch thick, 15 inches long was tied to both ends of this borosilicate glass tube, and it was placed inside the center of the four feet long quartz tube (Fig. 1), in such a manner that at each end of the quartz tube, the free ends of the platinum wires were hanging out. The quartz tube containing the above tube with the platinum wires was placed in the annealing oven, and both ends of it were raised by using fire bricks to about a foot high. Then about 950 grs. of weights (ordinary pieces of steel having hooks welded to them) were tied to the free ends of both platinum wires. In this way, the two weights were continuously pulling the 90 cm. long tube, in the opposite direction. Then curved pieces of quartz, ½ inch long were placed on the top of the quartz tube to indicate the ends of the borosilicate glass tube. This is shown in Fig. 3.

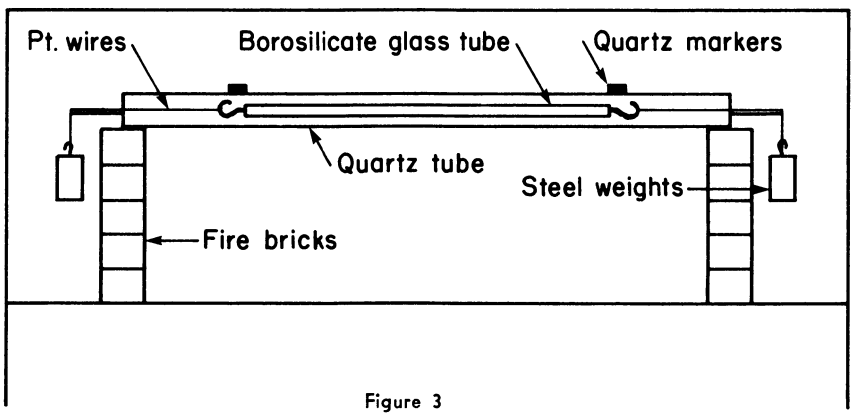

Figure 3
Annealing Oven

The annealing oven was closed and heated to 650°C. Next the temperature was raised to 750°C at the rate of 25°C at a time. With each temperature increase, the oven was opened for a few seconds to inspect if any elongation had taken place. At about 750°C, consider-

able stretching was noticed. At this stage, the oven was switched off and allowed to cool to room temperature. The borosilicate glass tube was then removed. It was found to be 111.8 cm. long, very straight, with no visible sign of any previous heat treatment. The diameter of this tube was measured at different distances from the same end as before. At each of these measured points, the tube was cut, and the inside diameter as well the out-of-roundness, was measured. Once again this data was converted into millimeters and recorded as shown in Table I.

TABLE I

90 cm. long, 7 mm. O.D. tube drawn to 111.8 cm. length							
Measurements Before Heat Treatment		Measurements After Heat Treatment				Calculated Data	
Distance from the End	Outside Diam.	Distance from the End	Outside Diam.	Inside Diam.	Wall Thickness	Outside Diam.	Wall Thickness
cm.	Milli-miters	cm.	Millimeters			Millimeters	
2	7.08	3	6,42	4,61	0,90	6,35	0,89
10	7.03	12	6,42	4,60	0,91	6,31	0,89
20	7.01	24	6,40	4,58	0,91	6,29	0,89
30	7.03	36	6,24	4,44	0,90	6,31	0,89
40	6,98	48	6,24	4,46	0,89	6,26	0,89
50	7.01	60	6,27	4.50	0,88	6,29	0,89
60	7.03	72	6,26	5.50	0,88	6,31	0,89
70	7.03	84	6,24	4.46	0,89	6,31	0,89
80	7.01	96	6,42	4.59	0,91	6,29	0,89
88	7.06	108	6,40	4.56	0,92	6,33	0,89
Tolerance:	7,03±0.05		6,33±0,09		0,90±0,02	6,305±0,045	

B) In the above experiment several difficulties were observed. The number of tubes to be stretched was limited to the number of quartz tubes. The platinum wire was not slipping at each end evenly. The following modifications were made to eliminate these practical difficulties. At one end of the inner wall of the annealing oven, stainless steel hooks were permanently mounted. To the opposite wall, at the same height, 1/64 of an inch diameter holes were drilled. On the outside of this latter wall, below the holes, one inch diameter pulleys were mounted. The borosilicate glass tubes prepared in the same manner as previously, were hooked at one end inside the oven to the stainless steel hooks. The other ends were hooked to the weights on the outside of the oven through the 1/64 of an inch diameter holes, using stainless steel wire, (0,020 at an inch thick) bent over the pulley. This is shown in Fig. 4.

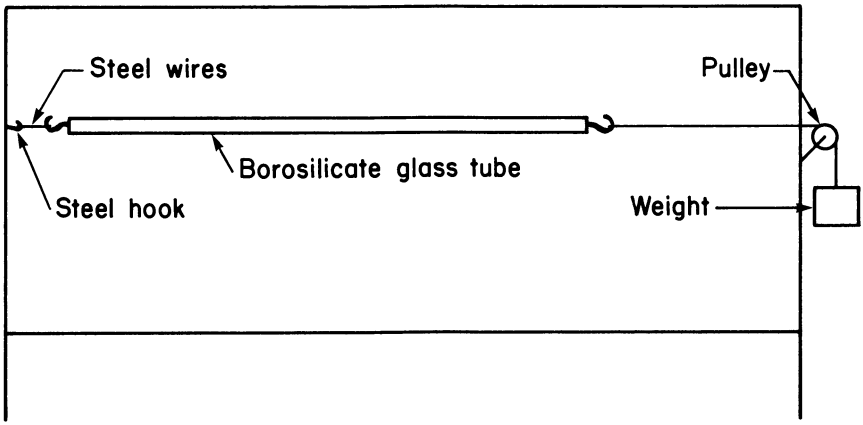

Figure 4
Annealing Oven

In this way, a number of tubes could be elongated at the same time. The elongation could be measured from the outside of the oven, without opening it, to a better degree of accuracy. In this fashion, a number of tubes had been elongated, in order to study the change in relation to their diameters and lengths. Some of the results are presented in Table II.

TABLE II

50 cm. long, 6 mm. O.D., Drawn to 86.5 cm. length.				98 cm. long, 5 mm. O.D., Drawn to 111.7 cm. length				98 cm. long, 4 mm. O.D., Drawn to 118 cm. length.			
Before Heat Treatment		After Heat Treatment		Before Heat Treatment		After Heat Treatment		Before Heat Treatment		After Heat Treatment	
Dist. from the end	O.D.	Dist. fr the end	O.D.	Dist. fr the end	O.D.	Dist. fr the end	O.D.	Dist. fr the end	O.D.	Dist. fr the end	O.D.
cm.	mm.	cm.	mm.	cm.	mm.	cm.	mm.	cm.	mm.	cm.	mm.
2	6,00	6	4,48	2	4,98	5	4,68	2	4,04	5	3,71
10	5,97	16	4,48	10	4,96	12	4,67	10	4,06	12	3,70
15	6,00	32	4,45	20	4,93	24	4,58	20	4,07	24	3,68
20	5,97	40	4,52	30	4,93	36	4,59	30	4,06	36	3,69
25	6,00	48	4,58	40	4,94	48	4,61	40	4,06	48	3,69
30	6,03	56	4,60	50	4,91	60	4,60	50	4,07	60	3,71
35	6,00	64	4,58	60	4,87	72	4,60	60	4,07	72	3,69
40	6,00	72	4,55	70	4,86	84	4.52	70	4,07	84	3,68
45	6,03	80	4,65	80	4,88	96	4.53	80	4,08	96	3,74
48	6,02	85	4,65	90	3,88	108	4,52	90	4,14	108	3,78
Tolerance: 6,00±0,03		4,50±0,10		4,92±0,06		4,60±0,08		4,10±0,04		3,74±0,05	

THEORETICAL EVALUATION:

Determine how much a borosilicate glass tube with diameters Do and do has to be elongated to obtain diameters D and d outside and inside respectively.

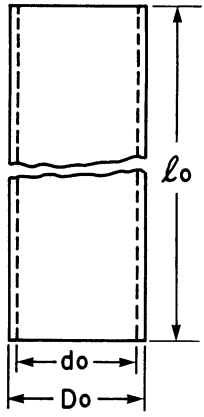

Figure 5
Borosilicate Glass Tube

The change in length, dl is the new length of the tube l, less the original length of it lo i.e.

$$dl = l - lo \qquad \text{I.}$$

The volume of the glass in the original tube, Vo from Fig. 5 given by

$$Vo = \frac{Do^2}{4} \pi\, lo - \frac{do^2}{4} \pi\, lo$$

And similarly, the volume of the glass in the same tube, V, after it has been elongated

$$V = \frac{D^2}{4} \pi\, l - \frac{d^2}{4} \pi\, l$$

According to the material conservation law, the volume of the glass in the tube does not change i. e. Vo = V

and
$$l = \frac{lo\ (D^2o - d^2o)}{D^2 - d^2} \qquad \text{II.}$$

Now, let lo be unity, 1 cm., then dl, change in length, will be cm/cm. In the Equation I and II replace the value of lo = 1 cm, also substituting the value of l from Equation II into Equation I we have,

$$dl = \frac{D^2o - d^2o}{D^2 - d^2} - 1 \qquad \text{III.}$$

From the experiments, concluding that the ratio of the diameters of the tube will remain constant during the process i.e.

$$\frac{Do}{do} = \frac{D}{d}$$

and

$$d = \frac{Ddo}{Do} \qquad \text{IV.}$$

Replace this value of d into Equation III.

We have $$dl = \frac{Do^2}{D^2} - 1 \qquad \text{V.}$$

And the wall thickness of the new tube t, in terms of its diameters, given by the Equation

$$t = \frac{D-d}{2} \qquad \text{VI.}$$

The same for the original tube, to

$$to = \frac{Do - do}{2} \qquad \text{VII.}$$

Replacing the value of d from Equation IV into Equation VI.

$$t = \frac{D}{Do}\left(\frac{Do - do}{2}\right)$$

But according to Equation VII.

$$t = \frac{D}{D}\,to \qquad \text{VIII.}$$

NUMERAL EXAMPLE:

A) To evaluate the validity of the above Equations, use data from Table I. The 90 cm. long tube was stretched to a total length of 111.8, therefore the elongation, dl in cm/cm.

$$dl = \frac{111.8 - 90}{90} = 0.2422 \text{ cm/cm}$$

$$0.2422 = \frac{7.01^2}{D^2} - 1$$

$$D = 6.29$$

And the wall thickness:

$$t = \frac{toD}{Do} = \frac{1 \times 6.29}{7.01} = 0{,}89 \text{ mm.}$$

Theoretical values obtained in this way, using data from Table I, are tabulated in the same table as "Calculated Data" to permit comparison of theoretical and experimental values.

B) Given a standard wall 90 cm. long 7 mm. outside diameter (1 mm. wall thickness) borosilicate glass tube, whose outside diameter is to be reduced to 6.36 mm. (¼ of an inch.). To determine to what length it must be stretched, use Equation V.

$$dl = \frac{D^2o}{D^2} - 1 = \frac{7^2}{6{,}36^2} - 1 = 0{,}211 \text{ cm.}$$

dl = 0,211 means each centimeter of the above tube has to be stretched by 0,211 cm. i.e. 90 × 0,211 = 18,99 cm. The total length will be 90 + 18,99 = 108,99 cm. to yield an outside diameter of 6,36 mm. The wall thickness of this resulting tube is given by Equation VIII.

$$t = \frac{toD}{Do} = \frac{1 \times 7}{6{,}36} = 0{,}90 \text{ mm.}$$

The values of dl's and t's were calculated for some possible useful tubes and the obtained data is tabulated in Table III.

TABLE III

Original Tube				Resulting Tube			
Outside Diam.	Inside Diam.	Wall Thickness		Outside Diam.	Inside Diam.	Wall Thickness	dℓ
Millimeters							cm/cm
4	2.4	0.80		3.50	2.10	0.70	0,306
5	3.4	0.80		3.50	2.38	0.56	1,040
6	4.0	1.00		3.50	2.34	0.58	1,938
8	6.0	2.00		3.50	1.76	0.87	4,224
6	4.0	1.00		4.50	3.00	0.75	0,777
7	5.0	1.00		4.50	3.22	0.64	1,419
8	6.0	2.00		4.50	2.26	1.12	2,160
7	5.0	1.00		6.36	4.56	0.90	0,211
8	6.0	1.00		6.36	4.78	0.79	0,581
8	5.0	1.50		6.36	3.98	1.19	0,581

CONCLUSION:

From the above results of the experiments and the theoretical evaluation, it seems that with this technique any previously specified inside or outside diameter off-standard borosilicate glass tube can be produced in small quantities. The evenness of the resulting tube depends on the precision of the original tube, as well the uniformity of the temperature inside the oven.

In Table I, the diameter of the initial tube varies between 7,08 mm. and 6,98 mm., that is 7,03 ± 0,05 mm. The same tube, after being redrawn has a diameter of 6,33 ± 0,09 mm. Theoretically the latter measurement should have read 6,305 ± 0,045 mm., which is practically the same tolerance as originally. In this range of diameters, the value given by some manufacturers is ± 0,30 mm. The above data is well within this specified value, before and after heat treatment. In Table II, similar interpretation can be obtained.

In Table I, the measurements indicate a variation in wall thickness of 0,90 ± 0,02 mm., which is in agreement with the theoretical value of 0,89 mm. and the specification of 0,10 mm. given by manufacturers of glass tube.

AN APPARATUS USING GLASS BATHS FOR PARALLEL DRUG STUDIES WITH SMOOTH MUSCLE TISSUE

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Various experimental techniques have been used to study the effects of drugs on the contractibility of smooth muscle tissue. Glass muscle baths from macro to micro size have been used for many years. In using these baths, there is the problem of having well controlled parallel washing cycles and of maintaining a constant volume of the salt solutions surrounding the muscle preparation.

In this apparatus, four muscle baths are operated simultaneously, maintaining isolated muscle tissue at body temperature in a balanced salt solution. Viability of the tissue is maintained by this physiological salt solution and a constant flow of oxygen through the glass baths. A drug is added to each bath and smooth muscle contractions are measured with Grass force displacement transducers supported over each bath and they are recorded by a polygraph.

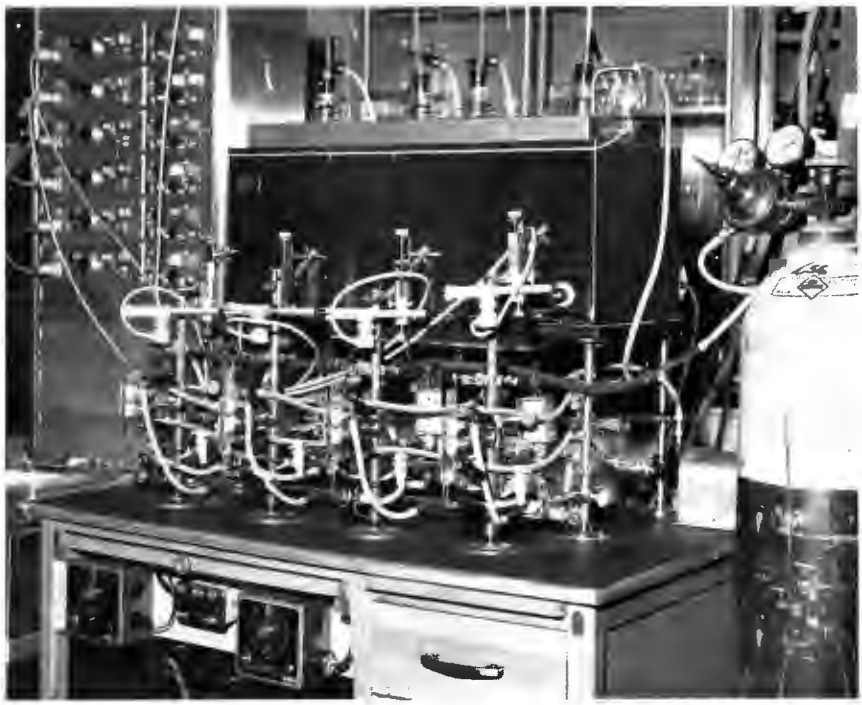

Figure 1

A description of the apparatus follows:

The complete set-up was mounted on a desk (Fig. 1). Drawers are used for spare parts and casters allow the apparatus to be easily moved. A Thelco constant temperature bath was mounted on a platform at the back of the desk. Four one gallon bottles to hold the wash solution are held in place in the bath by a cover constructed by our machine shop. Rubber stoppers are fitted in each bottle top to hold a glass tube line and a breather tube. The glass tube line extends down the rear of the bath and connects to a Teflon® stopcock manifold under the platform. The manifold is constructed so that each muscle bath can be supplied by any of the four bottles.

From the stopcock manifold, glass lines are connected to glass solenoid ball valves. These are connected to the muscle baths at the front of the desk. Ball and socket joints are used in certain points to allow for easy dismantling of parts for cleaning.

An oxygen glass manifold is mounted in back of the baths and carries the gas to each bath. Flow is regulated by Teflon capillary needle valves. An overflow glass manifold is mounted in the same location to carry away overflow from the muscle wash cycle.

A vacuum glass manifold was mounted on the rods supporting the platform under the Thelco bath. This manifold is connected to an adjustable vacuum tube inserted in each bath, to remove excess solution from the muscle baths to the proper level. Two more glass manifolds are mounted in the same area to carry water from the Thelco bath to the heated jackets of each muscle bath and return via a small electric pump.

A more detailed description of the glass parts follows:

The smooth muscle baths are made with a water jacket. The inner chamber is 1.2 cm. outside diameter and 8 cm. long (Figures 2 and 3). The jacket holds a constant temperature from the water from the Thelco bath. Above the top ring seal, an overflow tube is sealed to a short extension of the inner chamber. A detachable muscle tissue holder is inserted in each bath. A length of stainless needle tubing is bent in a sharp U to serve as a holding clamp at the top of a bath. A V hook of nichrome wire is soldered near the bottom of the needle tubing. The section of tissue is hooked at this point. The other end of the tissue is connected to the Grass transducer which is clamped over the bath.

At the left of the bath is another length of needle tubing connected to the vacuum. By adjusting the section inside the bath to the proper height, the desired liquid level is maintained.

At the bottom of the bath, a ¼ inch ball valve is constructed to hold the liquid level. A short length of capillary tubing is ring sealed inside the inner tube just above the valve. A glass luer joint is sealed to the capillary tube. An easy disconnect is made between this joint and a similar joint on the oxygen manifold using metal outer joints and capillary bore plastic tubing. The short length of 8 mm. tubing below the valve is bent to a right angle for connection to the solenoid valve. 12/5 socket joints are sealed to the back of the water jacket for connecting

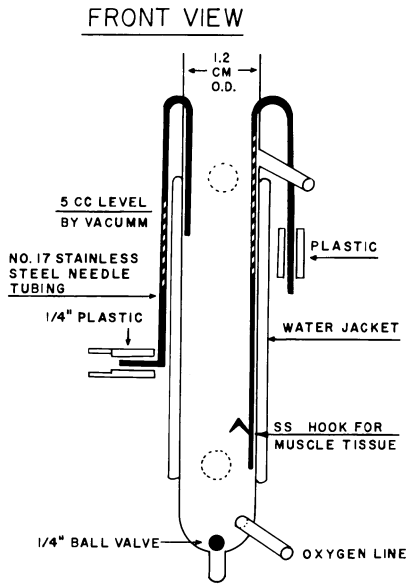

Figure 2

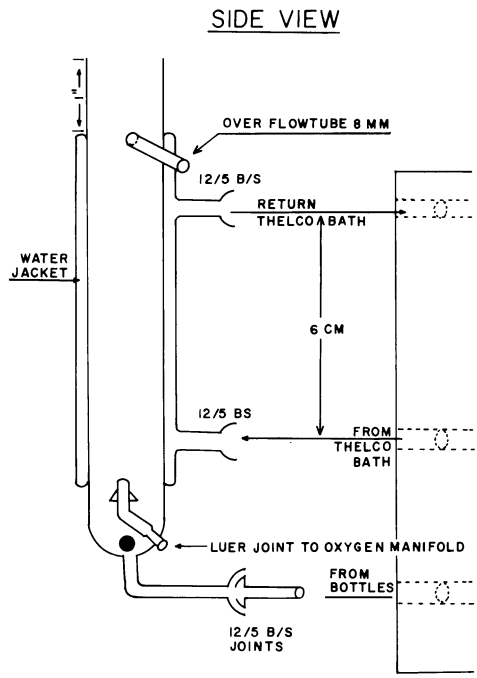

Figure 3

lines to and from the Thelco water heater. This maintains the muscle bath at the proper temperature.

The solenoid glass check valve (Fig. 4) is made using 14 mm. tubing for the outside chamber. The $\frac{3}{8}$ inch ball is sealed to the end of a length

GLASS BALL SOLENOID FLOW VALVE

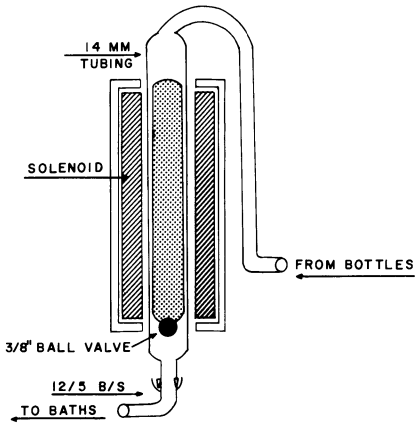

Figure 4

of 11 mm. tubing for the inner core. Sections of welding rods were used for the metal core. A ball and socket joint is used at the bottom for easy cleaining, etc. The solenoid metal frame is removed. The metal inner tube is removed and 5.8 inch hole is drilled through that end. The unit is reassembled and the upper glass part of the valve is inserted through the solenoid.

The oxygen manifold has four stopcock units, one connecting to each bath. The Teflon needle valve regulates the gas flow to the muscle bath. The two glass luer joints are connected by the metal joints and plastic tubing.

Electric timers, at the front of the desk, regulate the wash and fill cycle and the vacuum level cycle by push button controls. Once the proper salt solution level is established by vacuum, drugs are injected into the bath and a permanent record of muscle activity is made via the force transducers and the polygraph.

Acknowledgements are given to our machine shop and Joseph Giaquinto for the metal and electrical work for this unit, to Ellen Miller for the sketches I have used, and to Zigurd Mielens for his contribution in preparing the apparatus and this paper.

A GLASSBLOWING COURSE FOR GRADUATE STUDENTS

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Almost every glassblower at some time has been asked to teach glassblowing, especially those associated with educational institutions. Yielding to constant requests, I began a course in basic glassblowing employing the usual free-hand approach. After ten weeks, the results were far from gratifying. Prior to the next quarter, I thought long and hard about the direction and results desired from such an endeavor. Obviously, we are not attempting to train professional glassblowers but to induce another skill that a scientist can utilize; a skill that can be re-employed even after considerable time intervals. Many of our graduates are subsequently employed by small companies or remote colleges where they must perform many of their own glassblowing requirements. Others unite with large companies or Universities with professional glassblowers so their basic glassblowing skills are seldom used.

I therefore concluded that an effective course should include:

1. The history, development, artistic and esthetic aspects of glass.
2. Physical and chemical properties of glass.
3. The ability to effectively communicate with the glassblower through acceptable drawings and specifications.
4. Because of the large diversity of skills of the students, to move and manipulate the hand torch instead of the hot glass.
5. To instill confidence, responsibility and correct technique in the student.
6. To offer the course on a voluntary, non-credit basis to attract only the interested and sincere student.

The present Elementary GlassBlowing course consists of 8 weekly sessions of one and one-half hours each. Initially the course starts with a 45 minute lecture on the history of glass with visual aids to emphasize various periods. Samples of obsidian or volcanic glass are shown to define it. Egyptian beads as man's first attempt at glassmaking, a Syrian vase to illustrate the invention of glassblowing. Most objects are reproductions but help to depict the ideas put forth. Examples of more complicated apparatus emphasize the application of basic techniques. Finally, examples of tempered glass, annealed and special glass i.e. Photochromic are shown and discussed. The remainder of the session covers types of stopcocks and joints, their correct designation, explanation of their sizes and various special components a glass shop may use. Safety and caution are firmly stressed with relation to the torch and material.

Subsequent sessions require:

(a) cutting and cracking tubes of various diameters
(b) sealing two tubes of equal diameters
(c) sealing two tubes of different diameters

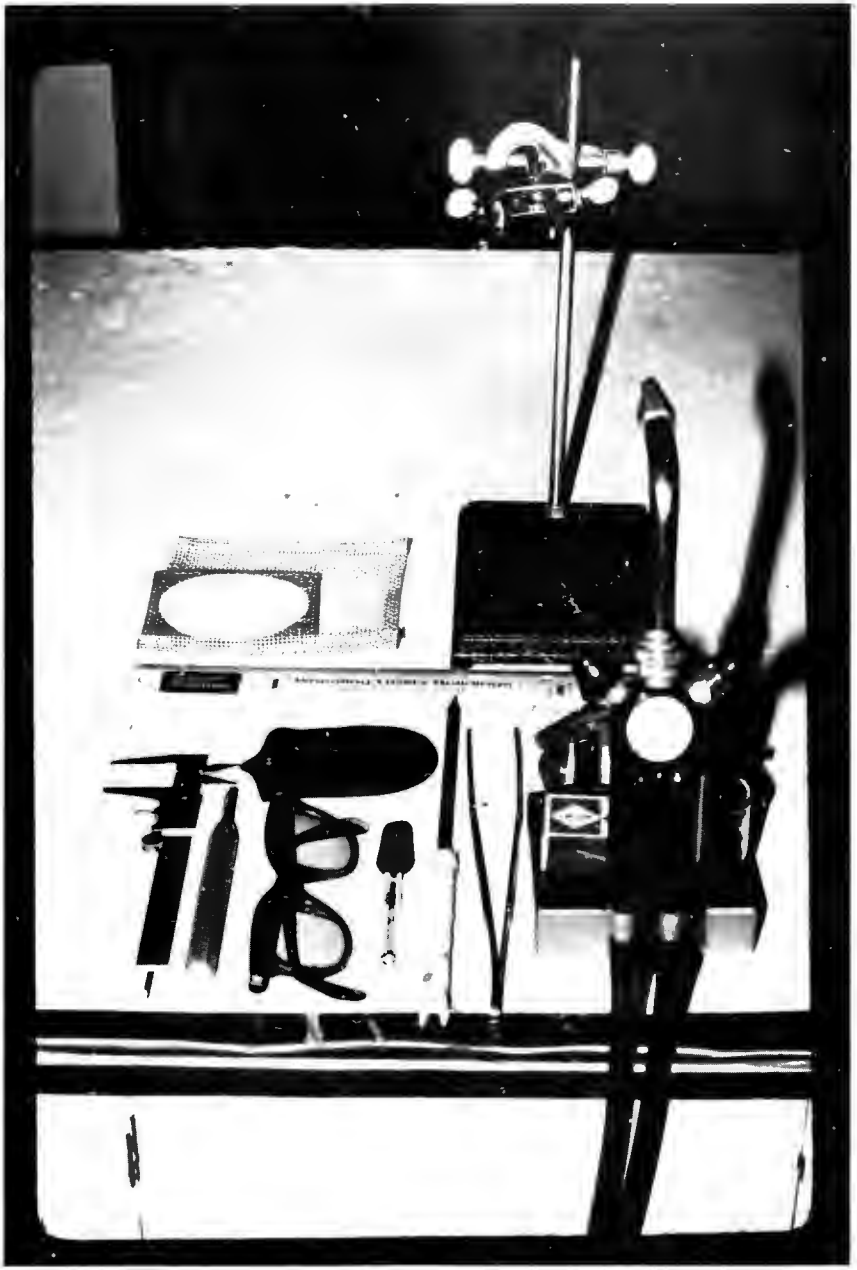

Figure 1
Student Glassblowing Equipment

(d) simple bends without blowing
(e) simple T seals
(f) small basic trap; then a small condenser
(g) combine all techniques to fabricate a vacuum manifold which must be vacuum tight and dimensionally correct.
(h) the last session covers laboratory techniques i.e. freeing frozen stopcocks and joints, grinding stopcocks for a better fit, sealing a tube under vacuum and if time permits basic artistic techniques.

Each station is supplied with the following

(a) National band torch #1, 3, 5 tips
(b) Laboratory Supply torch stand
(c) carbide Knife
(d) glasses, carbons, blowhose with swivel, corks, metal stand, etc.

Each station was equipped for less than $75 and the student bench was assembled from surplus equipment for approximately $50. Economy is

Figure 2
Various Seals Required in Course

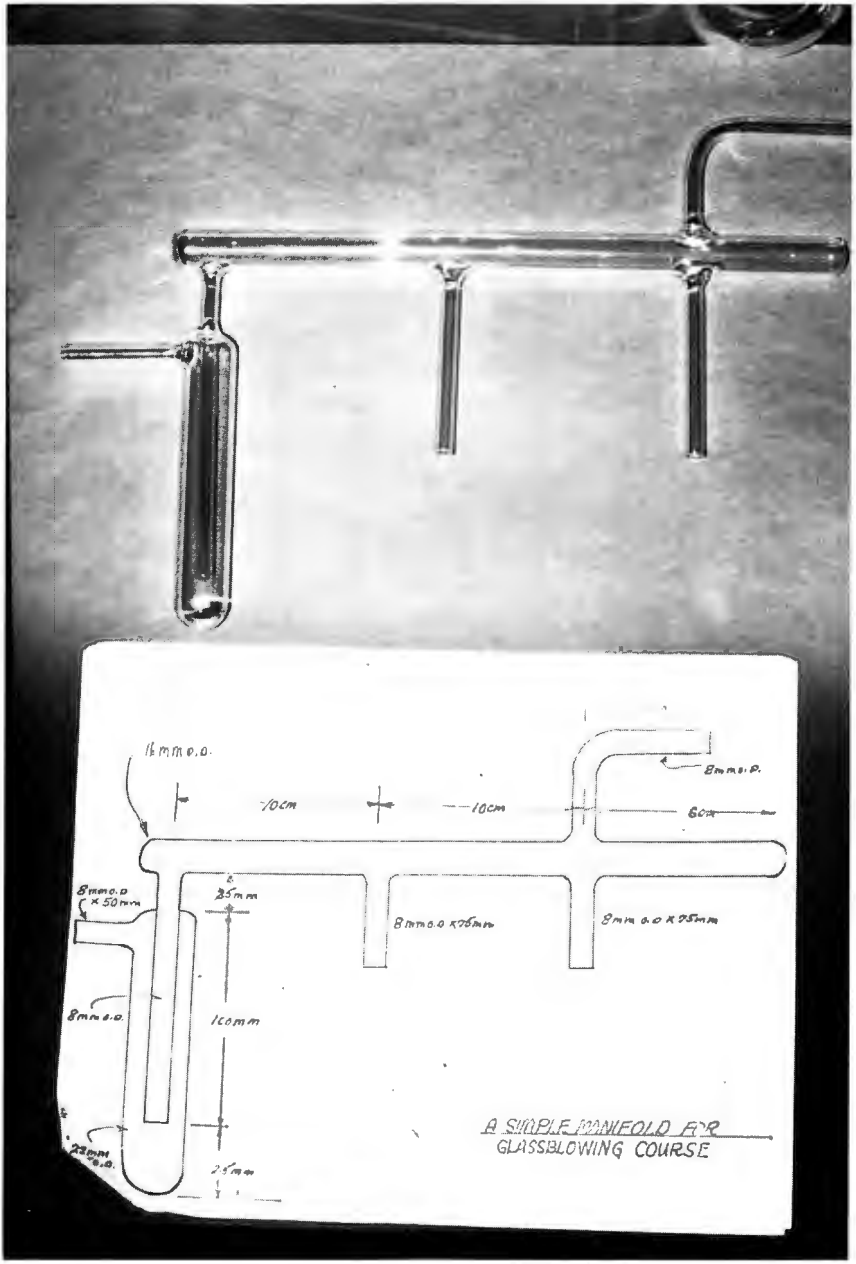

Figure 3
Manifold and Drawing

important when associated with a state institution. The tubing required for the course was simply 8 mm O.D. Standard wall and 19 mm O.D. Standard wall; 3 mm and 6 mm rod were used for filling and pull off operations.

After an initial demonstration of the required fabrication, the student is closely supervised especially the first two or three sessions. Confidence is emphasized and torch technique stressed. Cleanliness and organization are also required.

Subsequent questionnaires and personal conversations have proved that much general information about glass and useful techniques on glassblowing have been acquired by the graduate student to help him supplement his research and professional future endeavors. I am expressly indebted to my assistant, Jerry Cloninger, for his technical assistance and suggestions.

A VESSEL TO PERFORM PULSE RADIOLYSIS OF LIQUID AMMONIA IN A HIGH PRESSURE VESSEL

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The complete title of this paper should be, "A quartz vessel to perform pulse radiolysis[1] in a high pressure vessel[2] of liquids which have a vapor pressure of more than one atmosphere at room temperature or liquids which cannot be contained with a ground glass seal in the absence of grease."

Although the title sounds very technical, I will break it down in a way that means something to us as glassblowers. The experiment may mean little to us but the requirement for a rather unique piece of apparatus presented a challenge which most research glassblowers would welcome.

The basic requirements involve a vessel which will contain a liquid, has a movable part to allow compression of the liquid when subjected to pressures ranging from 1 atmosphere to 100,000 psi, and has optical windows suitable for good transmission of ultraviolet light. These requirements were met and found successful for ultra-pure water with a design that came about through the combined efforts of the glass shop and Dr. Farhataziz[3] who conducted the research.

Figure 1 shows a cross section view of the high pressure vessel and the water syringe.

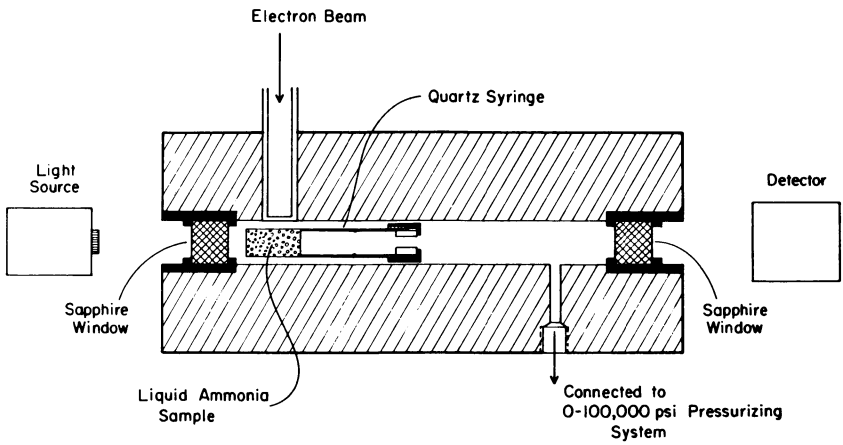


Figure 1

*The Radiation Laboratory is operated by the University of Notre Dame under contract with the U. S. Atomic Energy Commission.

The materials used in these syringe type vessels are precision bore tubes .597″ ID x 1 mm wall, precision ground OD quartz tubes to fit the precision bore tubes with .0005″ clearance, 1 cm^2 Suprasil[4] tubing and Suprasil windows of 1/16″ thickness. These parts are assembled as shown in the figure.

This design posed no problems when used in the pressure vessel since the water acted as a sealant between the plunger and barrel.

It is very important to note at this point that there is no differential of pressure between the inside and outside of the syringe as the pressure is slowly increased from atmospheric to 100,000 psi. As the pressure increases, the plunger slowly moves into the barrel. Many of us have heard the saying that gases can be compressed but not liquids. It might be of interest that at these pressures, the water is compressed about 20%.

As the old saying goes "one thing leads to another" and there became an interest in doing the same type of research with liquid ammonia. To avoid any confusion at this point, I must mention that we are dealing with pure ammonia gas NH_3 which is a gas at room temperature and atmospheric pressure and is not to be confused with ammonia as many think of it in the home which is ammonium hydroxide NH_4OH and is a liquid. It is now evident that we have a new situation which leads us into the main topic of this paper and the design of a modified syringe.

A suitable sample of liquid ammonia can be collected in a syringe barrel by submerging the closed end in dry ice and flowing NH_3 into the open end. (The gas liquifies at $\sim$ —40°C.) If we can now insert the syringe plunger and lock it in place, the sample, upon returning to room temperature, will remain as a liquid if the syringe is gas tight and will withstand 150 psi. (The 150 psi is the internal pressure exerted when the liquid tries to return to a gas at room temperature.)

We are now confronted with 3 new problems as opposed to the water syringe.

1. The syringe must be gas tight.
2. The syringe must withstand pressures in excess of 150 psi.
3. We need a locking device to hold the plunger.

We proceeded with the theory that an "O" ring between the plunger and barrel would give us a gas tight seal which we knew would be vacuum tight but needed final testing for 150 psi. Since we have no glass machining devices in our shop, the Pistorius glass saw seemed to be our only choice for cutting a groove.

Figure 2 is an exploded view and an assembled view of the fixture which I designed.

This fixture is used to hold the plunger and allow it to be rotated against the saw blade. Although it was made specifically for this application, the design lends itself to modifications which may be useful to some of you. For example, the base plate could be made any length with the tailstock movable to accommodate any length of tube, the chucks machined with a taper on the inside to accommodate different diameters, or chucks of other sizes could be machined.

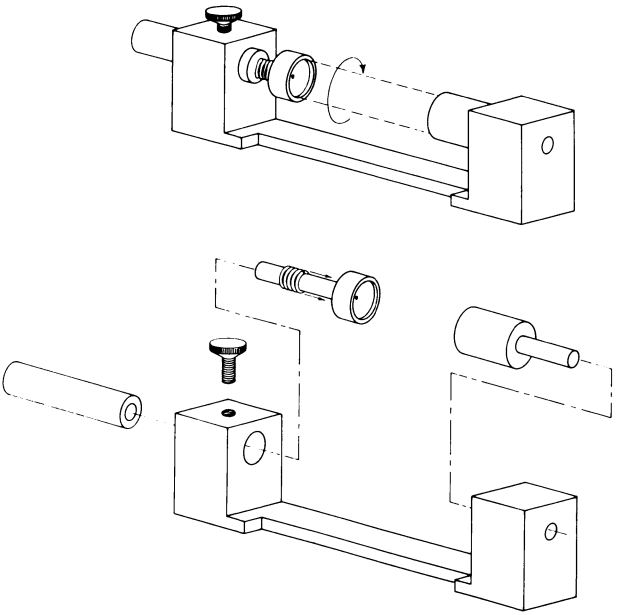


Figure 2

Because of the close fit between the plunger and barrel, the depth and accuracy of the groove became such an important factor that an adjustable stop for the saw table was imperative.

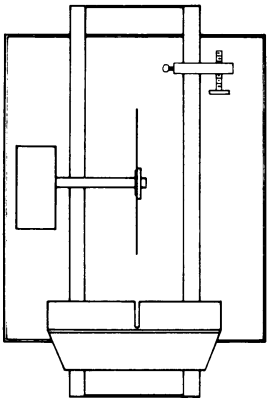
Figure 3 is a simplified top view of our Pistorius glass saw with the adjustable stop installed, a view of the factory table stop, and a detailed view of my design for the adjustable stop.

We are able to control the depth of our cut to within .001". A trimmed new blade is quite essential and the width of the blade is ideal for the "O" ring used. There are likely many cutting or grinding operations where this adjustable stop could be beneficial in other shops.

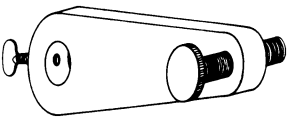
Figure 4 shows an exploded and an assembled view of the ammonia syringe.

Our next problem entailed determining whether our syringe could withstand an internal pressure of 150 psi. There was no concern with the plunger which needed to be at least 2 mm wall thickness to accommodate the "O" ring groove. The strength of its window seal was also enhanced by the direction of the pressure. The major concern was the barrel and its window seal. We made up several barrels with a window seal at one end and a reduced section at the other that could be attached to a compressed gas tank for testing.

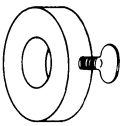
Before showing the results of the tests, I would like to describe the window sealing technique referred to as molecular bonding. I have been



Top view of Pistorius Glass Saw



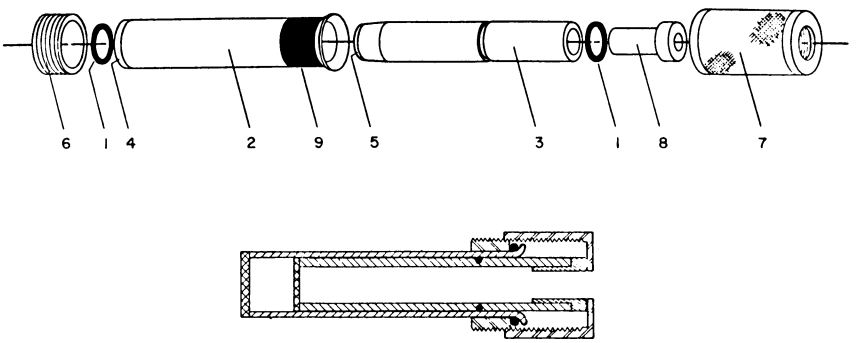
Custom piece to replace factory stop



Factory stop for saw table

Figure 3

unable to find this method spelled out in detail although it has been mentioned in previous "Proceedings" by Liebson[5], Campbell[6], and Schulze[7].



1 ● - "O" Rings
2 - Precision Bore Quartz Tube .597" ID x 1.5 mm Wall
3 - Precision Ground OD Quartz Tube To Fit Precision Bore with .0005" Clearance x 2.5 mm Wall
4 - Quartz Optical Flat $^{3}/_{32}$" Thick
5 - Quartz Optical Flat $^{1}/_{16}$" Thick
6 - Stainless Steel Sleeve Threaded on Outside
7 - Stainless Steel Cap Threaded on Inside To Match Sleeve
8 - Teflon Stopper To Serve As Cushion
9 - Teflon Tape

Figure 4

We have chosen this procedure the past 6 or 7 years for all quartz optical flat sealing operations such as window dewars, cylindrical optical cells, rectangular or square tubing cells where one or more sides need to be replaced with an optical flat window or any device when it is necessary to seal an optical window of minimal distortion. By comparing different techniques mentioned in other papers, we find molecular bonding no more time consuming and it leaves little or no divitrification and little or no distortion depending upon how well the matching surfaces have been prepared.

Using No. 600 grit silicon carbide slurry on a metal lapping plate[8] (Figure 5) we hand grind the end of the tube for the seal by grasping it with our fingers and with a figure-eight pattern motion grind the end until we attain a flat 600 grit matt surface (5-10 min.). The lapping plate is then covered with a nylon cloth[9] and a slurry of jewelers rouge is used to polish the ground surface until it becomes transparent or glossy (5-10 min.). This step is necessary so that the sealing operation can be observed as will be shown in a later figure. The flatness of the quartz plate or disc as supplied by the vendor is adequate for our seal. We now cut the disc to the exact outside diameter of the tube and prepare to make the seal.

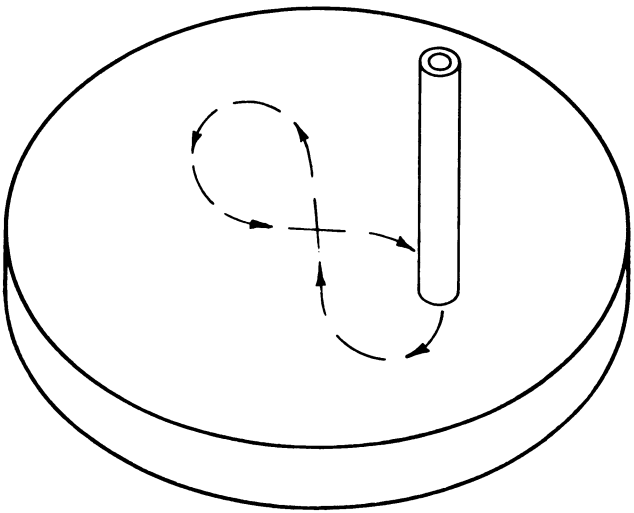


Figure 5

Many tools seem to have more than one use and this happened to be the case with the "O" ring groove cutting fixture which can be used as the window sealing fixture (Figure 6).

Although it is possible to make these seals with the window merely resting on the tube end, the seal takes place with much more ease when pressure is applied. Here we are using the tension in the spring to supply

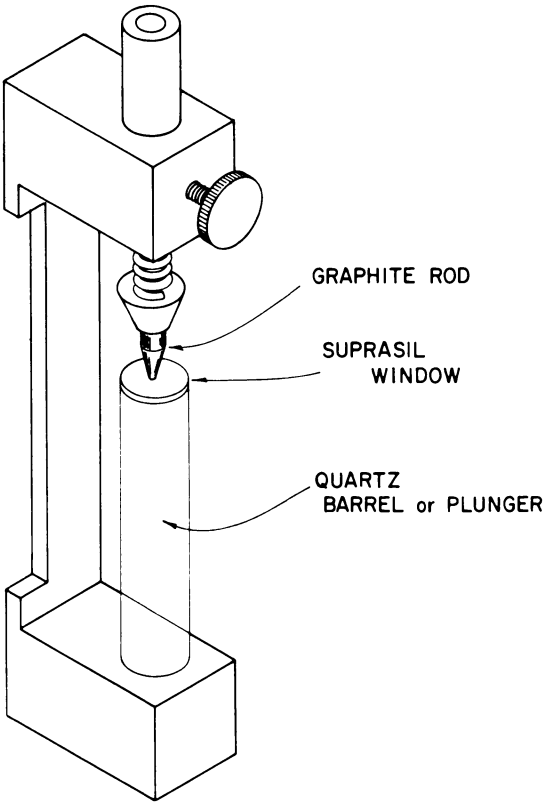


Figure 6

the force, but in many cases we use a vacuum pump. When using the vacuum pump, we have a bleeder valve in the suction line to regulate the pressure according to preference. I favor the first method since the vacuum pump exerts a pulsating force.

When the end of the tube is prepared properly and the window set in place, interference fringes can be observed at the interface between the window and the end of the tube (Figure 7).

To see these, the observer must position himself properly above his work and develop a well trained eye. They appear more clearly to me through didymium lens than clear lens and more easily seen under a red lamp. These interference fringes (dark and light spaces) move and change shape as various pressures are applied. The type of geometry they assume is directly related to the flatness of the matching parts and when they show up as concentric rings, the fusion occurs with lesser heat and more uniformity.

The sealing operation should be preceded with a thorough warming of the window and adjacent area followed by local heating with a back

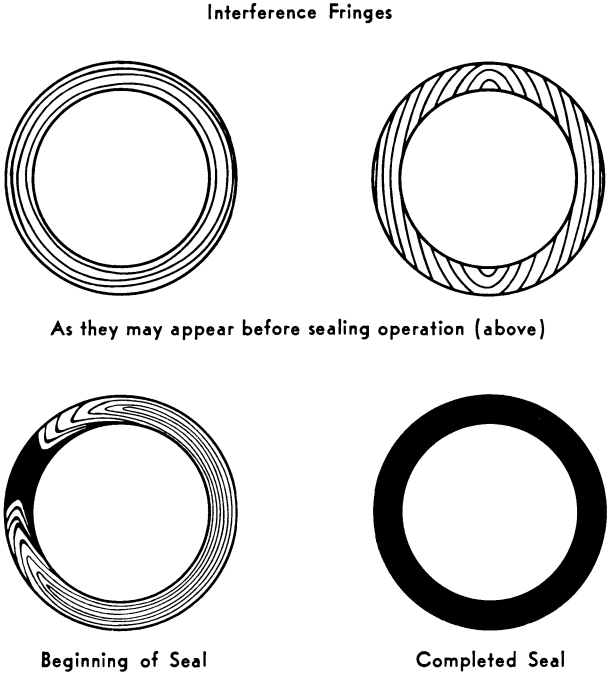


Figure 7

and forth motion of the torch. Favor the outside of the seal area avoiding unnecessary heat on the window. A No. 1 torch tip on a national torch supplies sufficient heat and the flame required to cause fusion is not bright enough to require quartz working glasses. Initial sealing is observed as a dark line appears where the inner wall of the tube meets the window and progresses across the full wall thickness of the interface. By moving around the perimeter of the window, the seal is completed as you would chase a crack in repairing a piece of apparatus.

For additional strength we thoroughly seal the outside junction of the window and the tube. The barrel is then oven annealed.

The question may arise concerning the strength of this type seal since it appears to be cemented rather than sealed. The following testing results (Figure 8) should answer some of your questions.

	Barrel Wall Thickness	Barrel Window Thickness	Fracture (psi
Seal described	1 mm	1/16" Suprasil	∿ 125
Seal described + seam seal	1 mm	1/16" Suprasil	∿ 180
Seal described + seam seal	1.5 mm	3/32" Suprasil	∿ 260

Figure 8

The final problem of holding the plunger against 150 psi was solved with my design for a coupling device which can be seen in Figure 4.

The open end of the barrel is flared slightly against which an "O" ring is placed to act as a cushion. A stainless steel sleeve threaded on the outside was made up to slide over the barrel and held in place against the "O" ring by using a few wrappings of Teflon tape between it and the barrel. The plunger was fitted with a Teflon piece machined as shown to serve as a cushion for the cap portion of the coupler. The cap portion was machined and threaded to match the sleeve as shown.

In conclusion it might be said that the device in its entirety has limited applications, but a number of other samples can be processed in the syringe. Some of the common ones are chlorine gas, sulfur dioxide and hydrogen iodide.

The main object of this paper is not the apparatus itself but the presentation of steps involved in constructing the apparatus with the hope that you may apply some of the techniques in solving some future problems in your own shop.

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5. ASGS Proceedings 1964—Low Temperature Glass Seals—Liebson.
6. ASGS Proceedings 1969—Glass Techniques for Laser Fabrication—W. W. Campbell.
7. ASGS Procedings 1973—Construction Techniques used for Micro High Purity Silica Cells—W. J. Schulze.
8. 8″ wheel with cloth clamp made by Buehler Ltd.
9. AB nylon cloth for 8″ wheel made by Buehler Ltd.

IN ATTENDANCE

The following are on record as having attended the Nineteenth Symposium on the Art of Glassblowing held at the Hyatt Regency Hotel, Houston, Texas, June 18-21, 1974. As a fully paid registered participant, these persons are entitled to a copy of the Proceedings.

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