PROCEEDINGS

2nd International
and
26th National
Symposium
and
Exhibition
on the
Art of Glassblowing

1981

THE AMERICAN SCIENTIFIC GLASSBLOWERS SOCIETY

Proceedings

2nd International and 26th National

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Art of Glassblowing

Sponsored by
The American Scientific Glassblowers Society

Dunfey Atlanta Hotel
Atlanta, Georgia
June 22-26, 1981

THE AMERICAN SCIENTIFIC GLASSBLOWERS SOCIETY
Toledo, Ohio

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Library of Congress #58-3756

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CONTENTS

COLORED LOW EXPANSION GLASS

Vincent C. DeMaria
Vitro Dynamics Inc.
Rockaway, N.J.

The first colored glasses resulted from impurities in the sand and minerals used in early glassmaking. The history of glass before 1500 BC shows the additions of copper for green, cobalt for blue, antimony for yellow and iron for a variety of colors used in Egypt, Mesopotamia and China. The controlled addition of colorants to produce predictable glass colors occurred later, after the metals were isolated during the middle 1700's. This resulted in a slow change from guarded trial and error secrets to scientifically controlled chemical formulations. This progress has contributed to the present development of colored low expansion glass.

As you know, until now most colored glasses have been produced in soda lime and lead glass. The original colorants favored the lime and lead to produce brilliant colors. In addition, the lower melting and rework temperatures of these soft glasses did not destroy the colors produced from copper, iron and antimony.

Now, more than sixty years after the invention of Pyrex® glass, hundreds of attractive, brilliant and fiery colors have been produced in low expansion borosilicate glass with the aid of modern materials and technology.

Producing this broad spectrum of colors, in less than tonnage quantities, required a new approach differing from tank or pot melting. First we start with cullet to eliminate the fusing and fining of the high silica ingredients which can destroy many colors. Then we subsitute refractory colorants for many of the well known soft glass coloring pigments. However, in doing this some interesting problems develop. For example, a high temperature pink, purple and maroon can be obtained with exacting proportions of tin oxide and chrome oxide. In this case, the pink is produced when a thin layer of chrome is deposited on the insoluble tin oxide by thermochemical reaction. The reaction occurs during fusion or in a separate crucible. However, this will occur only in an absolute oxidizing atmosphere and in the absence of reducing impurities such as metals, carbon and certain metal oxides. The problem is further compounded because the pink color prefers the presence of calcium to develop. Additions of lime can alter the coefficient of thermal expansion and must be added in cautious amounts.

An excellent high temperature yellow can be obtained with uranium oxide. Unfortunately, since the atomic bomb, uranium chemicals cannot be purchased in this country. A high temperature subsitute was found which produces our bright yellow from a combination of praseodymium and vanadium. This brilliant yellow has excellent flame rework properties.

In search of refractory colorants we are sometimes limited by the state of the art. The best fiery reds and oranges have been and still are produced from mixtures of cadmium and selenium. Most of the soft glass reds, oranges and yellows produced today use these materials. The colors develop only under reducing conditions and the ratio of cadmium to selenium determines the glass color. At melting temperatures, cadmium and selenium vaporize and their retention for color control becomes difficult. At best, these colorants produce borderline high temperature, low expansion, reds and oranges. However, with a little experience the rework temperatures and techniques can be mastered to make cadmium selenium colors an important addition to a complete palette of low expansion colors.

Gold and copper will produce reds by striking. When the initial yellowish glass is reheated the colloidal suspension of these metals develops the familiar ruby red. The reaction prefers a lead containing glass. However, for low expansion glass we would find copper is not a suitable refractory colorant for rework temperatures and today's gold price market would make gold prohibitive to use.

Glass is colored by one or more of four methods:

1. Ionically; here the metal oxide dissolves into the glass solution producing transparent colors such as cobalt blue.

2. Atomically; in this case the colorant does not dissolve but remains in suspension giving opaque colors such as tin oxide which gives opaque white.

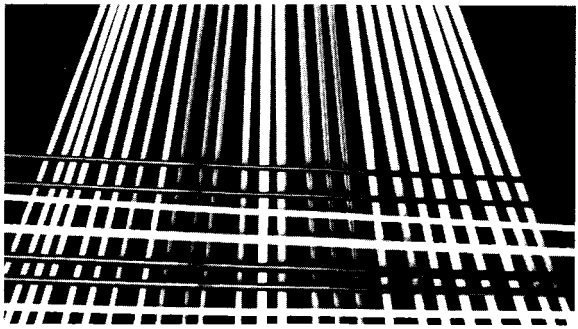
3. Colloids; in which the colorant remains in colloidal suspension such as copper and gold as just described in the production of ruby red.

4. Radiation; which can be by x-rays, gamma rays or visible light. The popular sun glasses with silver ions producing a reversible color effect by light radiation is an example.

Most colors develop best in the presence of other elements which further enhance their brilliance. While these elements determine color, their addition to low expansion glasses are not always possible due to resultant changes in the desired physical properties. For example, in a cobalt-chrome, blue green glass the color goes from blue in a high zinc glass to green in a glass with no zinc. Violet colors develop best in no zinc but high

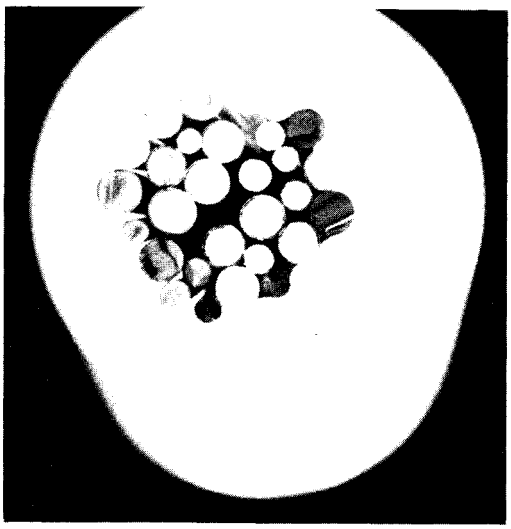
calcium, while a synthetic ruby pink is produced with high zinc and low calcium.

In spite of this and other problems, attractive colors can be produced in low expansion, high temperature glass. Sixteen of the more popular colors were selected for marketing under the name COLREX.®



Here you see a rainbow of colors produced in four and six millimeter rod that will satisfy the palette of most glass artisans. The coefficient of thermal expansion of Colrex®allows all colors to be directly sealed to Pyrex®7740, Kimax® KG33 and non-domestic glasses such as Duran 50, Hysil, Nife, Thermisil, Phoenix and Razotherm.

Colrex was developed specifically for reworking in gas-air-oxygen or gas-oxygen flames. The colored rods are characterized by a thin clear glass sheath of Pyrex 7740 or Kimax KG33. The purpose of the clear glass cladding is 1 - To help buffer excessive heat, protecting the more heat sensitive colors, 2 -To provide a uniform surface gloss to all colors including some matt producing high refrac-

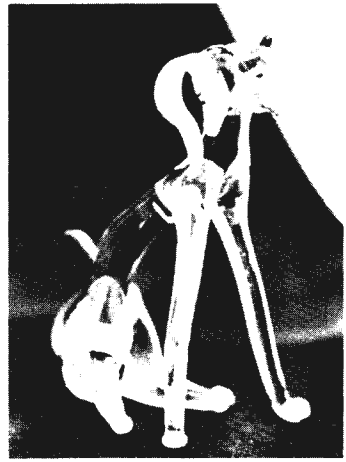


tory colorants, 3 - To prevent color changes in colors sensitive to reducing flames other than lead or antimony which are not used in Colrex, 4 - To allow the use of colored glass rods for food or medical use where Pyrex glass has been approved. (For some applications the pin hole free cladding may require the cut ends to be capped with a dab of clear rod.) and 5 - To provide quality control from manufacture to the ultimate user. Stock rod will develop circumfrential cracks with minute changes in expansion between the clear glass sheath and colored glass rod. This visual inspection assures the sealability of Colrex rods.

Flame working with colored borosilicate glass will be a new and often challenging experience for many of you. Manipulation and fire setting techniques require some modifications to obtain professional looking projects. For most colors you will have to cut back on working temperatures by staying away from the flame's inner blue cone. Getting to know which colors can take intense heat will frequently determine color choice for a particular job. Clear rods are usually heated above their working temperatures to reduce viscosity and obtain smooth flowing joints. To obtain this effect with some colored rods requires pushing the softened glass with graphite rods and paddles. The use of shears for cutting softened glass will eliminate color loss from high temperature burn-off. In general, allow time for heat to penetrate into the rod rather than increasing temperature and remove the glass from the flame immediately on completion.

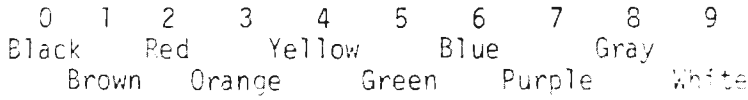
Exceptions to the rule are for knitting or weaving glass. In this operation any color can be worked in the hottest part of the flame. This is possible because as fast as the glass is softened it is formed and removed from the flame. A little color can highlight the beauty of clear crystal spun glass or blown ware. Simple

shapes such as this bent rod dog become meaningful when the characteristic features are emphasized with colored glass. A little colored glass can be made to go a long way. Use natural colors or select complimentary colors for eye pleasing effects. Non-fading colored glass eliminates painting and opens new areas in giftware, costume jewlery, glass flowers and paperweights made for pleasure or profit.



Numerous technical applications exist for colored glass and new uses are sure to appear. Color coding for permanent indentification of flange position, stopcock open direction and numbering are accomplished with

COLREX



colored glasses. Here the universal numbering system used for color coding electrical resistors is shown. These colors surpass mil specs for permanent non-fading colors in that they are also high temperature and chemical resistant. All of these colors except gray are in the standard Colrex assortment of sixteen colors. Gray is available on request from special stock colors. Short ends of colored rod sliced into thin discs and stored in partitioned plastic boxes make a ready source of material for glass shop color coding.

About thirty-five years ago I spent an afternoon with a Mr. Muller, glassblower at the Museum of Natural History in New York City. Working with colored soft glass he created biological specimens such as the ameba and protazoa. Medical models in glass of the human brain and body organs were reproduced from textbooks and used for demonstration at medical schools. Glass parts were first made then assembled with extreme care in preheating and annealing. Models took from a week to several months to produce.

This slide shows a scanning electron microscope view taken at UCLA of a monkey's brain magnified four thousand times. The newspaper article titled, "How Brain Cells Talk to One Another", showed a black and white picture which had numbers inserted to identify parts. This reproduced glass model, in which the film has not duplicated all colors, shows small blood vessels in red which are serving nearby nerve cell fibers in yellow. Lavender strands from the nerve cells carry drop-like pockets of nerve-signaling chemicals. Tan fibers are from a neuron-aiding glial cell. This model is not to scale and represents about an hour-and-a-half's work. While some

preheating and annealing was required, it did not have the frustrations associated with soft glass model making. Those of you inclined with patience may find biological model making an enjoyable hobby for fame or fortune.

After two years of Colrex development work, we are now committed to supplying the glass fabrication industry with the low expansion colored glasses. In the very near future you can look for transparent, opaque and striped colored tubing up to 50mm O.D. Additional rod colors will be added in tones and shades for the natural flower makers. Those of you interested in making paperweights and costume jewelry can look forward to a unique modern version of millifiore rods for obtaining new effects. Satisfying your requests for assistance or specials is our committment.

MANUFACTURE OF SEVEN BARREL MICRO PIPETTE BLANKS

Victor G. Plumbo
Friedrich & Dimmock Inc.
Millville, N.J.

The manufacture of seven barrel borosilicate glass micro-pipette blanks 1.00mm O.D. x .58mm I.D. using Kimax or Pyrex glass tubing, for physiological studies utilizing a Scotch Tape technique.

The developement of a **simple way** to make economical uniform multibarrel glass electrode tubing with good tolerances of each barrel and well maintained structural shape of the complete pipette blank has helped to accelerate the developement of advanced instrumentation for greatly improving the physiologist techniques in the field of Micro-Iontopherisis -"The study of living cells and their behavior when stimulated by electrical impulses and or direct electrical ejection into cells of drugs and other solutions by minute electrical charges." Physiologists are routinely pulling micro-pipette electrode blanks to tip diameters of .1 micron (.000039") and finer with open lumens. This presentation will deal with the making of a suitable seven barrel glass micro-pipette blank with each barrel having an approximate size of 1.0mm O.D. with an I.D. of .55mm, and with each tube in the array having an integral glass fiber drawn into its lumen. The purpose of glass fiber is to enhance the filling of the micro-pipette blank, after it has been pulled down by the physiologist, who uses a special micro-pipette blank puller for filling and implantation with the micro manipulator (under a microscope unto the living cell to be studied). This is a simple method of building a micro-pipette blank as it does not require the use of a glass lathe, but a relatively simple apparatus employing an electric resistance heater, and glass tubing redrawing techniques.

The following materials are required:

1. A supply of 9mm O.D. by 2mm wall Kimax or Pyrex Glass Tubing. Kimax or Pyrex glass rod drawn down to .036" by 4 feet long.
2. A roll of 3M Scotch Brand Clear Tape Highland No. 5910, ¼" wide.
3. The apparatus for holding, heating, feeding and subsequently drawing of the glass tubing into convenient lengths for recutting into 4 or 6" lengths for use by the physiologist.

Step No. 1. Set furnace controller to 1610 degrees F.

2. Select each piece of glass tubing to be redrawn down for uniformity and straightness as much as possible. Blow out I.D. with air and clean the

O.D. with alcohol or other suitable solvent.

3. Arrange tubing in a cluster bundle of seven with the center tube extending out from the rest about 2″ as per sketch.
4. Use the Scotch tape and tape rear end of the glass cluster with the one glass tube extended in a circular manner exerting as much pressure on the tape as possible to hold cluster together without snapping the tape. (About five turns of the tape superimposed is sufficient.) Move to the other end of the glass tubing while holding the tubes secure in one hand to keep them in line and wrap this end with the Scotch tape about 3″ in from this end. You should now have the tubes in line so you can complete the taping. Divide the tubing distance so you can space the tape about three or four superimposed turns every 4″ apart. You will now have all the barrels securely held together. It should look like this sketch.
5. Place one length of .036″ O.D. glass rod × 4 feet long into each one of the seven barrels. Put a dab of rubber cement or Crazy glue into each lumen of the blank (chucking end) to secure the glass rod to each individual tube. Let dry a few minutes.
6. Chuck up redraw blank to the chuck by means of the extended portion of glass tubing. No turning should be necessary.
7. Feed the first six inches of the 7 barrel blank into the hot furnace. You will notice the Scotch tape in the furnace area immediately burns off causing some smoking. Don't be concerned. This is normal. The whole configuration will be held in place by the Scotch tape outside the heat zone; thus the whole glass structure is maintained as the individual glass tubes are securely held together with the Scotch tape band pressure every 4″.
8. After the tubing bundle in the furnace reaches a sag attitude about the softening point of the glass, place the feeder in the go position and grab the tubing in the furnace with a pair of tongs or other suitable grabbing device. Give the hot glass a twist to take advantage of adhesion, attenuate glass allowing it to cool and attempt to insert the tubing between the pulling rollers. The tubes may not adhere to each other at first, but don't be discouraged, they will.
9. Draw off the tubing and measure to get the outside tubing diameter to measure about 3mm across. This should be about 1mm O.D. for each of the glass tubes in the cluster. After you get on size check with a jewelers loupe to see if the individual glass tubes in the cluster have the glass fiber adhered to each of the inner walls. Your yield will be from 200 to

250 feet depending on how quickly you get on size or less if time is lost due to some other inconvenience.

THE BOLOGNA BOTTLE AT 6000 FRAMES PER SECOND

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INTRODUCTION

Tempered, or case hardened glass, demonstrates strength of forces in equilibrium. The bologna bottle is an example of tempered tubing; it provides an excellent example of tempered glass, in that it affords a close look at forces in equilibrium.

DISCUSSION

Bologna Phials, or Philosopher's Flasks, are perhaps better known among glassworkers as color proofs. They are used to determine the color and quality of the glass batch. The glassblower gathers a small amount of glass on his blowpipe and distends it by blowing. The bubble is then elongated by swinging the pipe to and fro keeping the end fairly heavy. This action cools the exterior quickly, while the interior cools at a slower rate thus tempering the phial. The piece is then cracked off the end of the pipe which leaves the piece in a phial or bottle shape with the one end rounded and heavy and the other end with a fairly wide opening (1).

When glass is tempered by cold immersion as in the bench version of the bologna bottle, the same phenomenon occurs. This difference in cooling rates leaves the outer surface in a state of compression and the inner surface in a state of tension. The density of the outer surface is greatly increased, enabling the surface to withstand great mechanical shocks. The key to this phenomenon is the uniformity of the stress which can be seen readily under polarized light and again when the fragments from the bologna bottle are examined after explosion.

The inner surface is left in delicate tension because it cools at a much slower rate, thus allowing us to explode the bottle with the slightest surface rupture.

This is an event I have witnessed many times at normal speed in countless demonstrations and explanations of the forces and stresses involved. As the title indicates, this paper and accompanying film gives us a look at the bologna bottle from a high speed perspective.

We are now able, with the use of a high speed camera, to see these forces change from stability to disintegration (2). The film provides a look at the uniformity of the explosion and the delicate balance between compression and tension. It enables us to see the action of the tungsten carbide chip as it makes contact with the inner surface. The slightest contact with the inner surface by the carbide chip causes the bottle to explode with great force. The chip appears to go through the wall of the bottle as if it were passing through a drop of water. The disintegration of the bottle is shown to be extremely uniform and rapid at first contact, it then appears to travel more randomly and slowly to final destruction (see Figures 1-5).

Although weight and size of the particle used to rupture the inner surface is not a factor, in this instance the carbide chip used was rectangular and approximately 1 mm $\times$ 1.5 mm $\times$ 3 mm and weighed .0276 grams. What is important to the destruction factor is that the particle has hard, sharp edges in order to fracture the delicate inner surface of the bottle.

When one considers the time element involved, approximately 0.0001 of a second, for complete destruction, one can better appreciate the tremendous tension involved. Yet, because of the denseness caused by outer surface compression, the bottle cannot be destroyed by repeated hammer blows.

At this writing no information has been found by this author as to the exact date or origin of the development of the bologna bottle.

I can only theorize that the bologna bottle predates the Rupert drops based on the fact that it is known they originated as color proofs. Let us also surmise the origin of the name "bologna phials" as being derived from the Italian city of Bologna, which has a history of glassblowing dating back to the 13th century.

CONCLUSION

Whatever its origin, one cannot help but believe this glass curiosity or "frigger" as it was once called, made a very important contribution to the development and use of tempered or safety glass available today. It also provides, of course, an interesting and exciting example of the mysteries of glass.

REFERENCE

1. "Prince Rupert Drops and the Bologna Phial" by T. Taylor Seago — reprinted from "The Pottery Gazette and Glass Trade Review", April, 1943.
2. Wollensak FASTEX Camera — Category II, WF14 (400 ft. capacity).

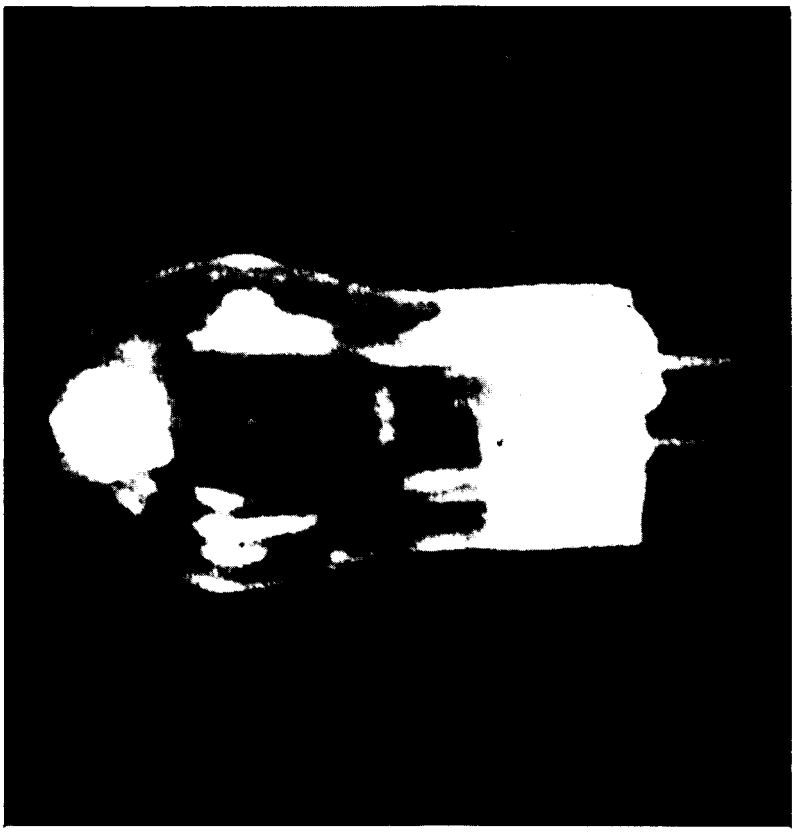

Figure 1. Before impact

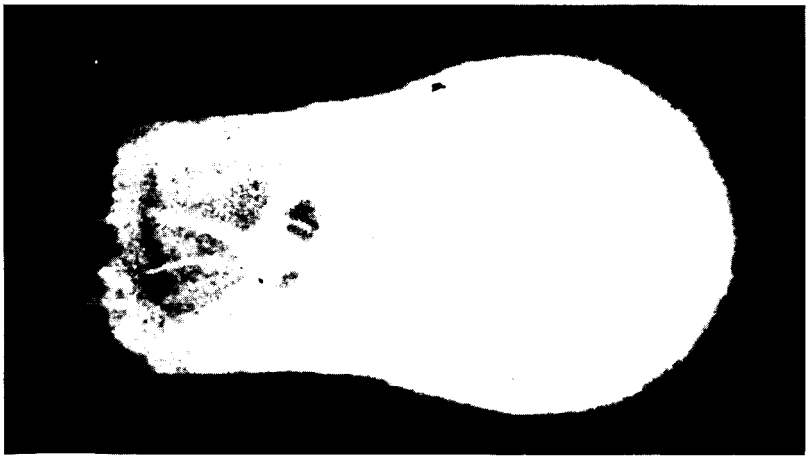

Figure 2. Time zero (moment of impact).

Figure 3. 2.2 milliseconds (14 frames after initial impact).

Figure 4. 7.8 milliseconds (47 frames after impact).

Figure 5. 13.3 milliseconds (80 frames after impact).

TWO ELECTRONIC PROJECTS

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When I was approached by the Hazardous Wastes Committee concerning the waste mercury that was generated by the University of Virginia I suggested reclaiming as much of it as possible. The triple distillation apparatus in E. L. Wheeler's book *Scientific Glassblowing*[1] seemed ideal for this purpose. Having fashioned a model of his device I encountered problems locating heaters of the correct dimensions and power. Also, the variable transformers which were to be used as controllers were quite expensive. I decided to make my own heaters and controllers. I constructed three cartridge heaters from materials available in my glass shop. The three controllers were made from inexpensive materials readily available from a local electronics supply store.

CARTRIDGE HEATERS

The materials used to fashion the cartridge heaters consisted of fused silica tubing, nichrome wire, unburned asbestos paper and aqueous furnace cement. To determine the right amount of wire to use in the heaters I used the chart (Figure 1), which combines Joule's Law, watts = ohms x amps^2, and Ohm' Law, volts = amps x ohms.[2] This chart shows all the algebraic combinations of these two laws. Wheeler had called for 115 volt heaters rated at 300 watts. I wanted to construct heaters of even greater power to provide a significant margin for error because the dimensions of the boilers on the drawings were not given. I decided to make heaters that would have power of 400 watts when 75 volts was being used. According to Figure 1:

$$R = \frac{E^2}{P} = \frac{(75 \text{ volts})^2}{400 \text{ watts}} = 14.06 \text{ ohms}$$

The resulting 14 ohms of resistance needed to be correlated to the nichrome wire itself. Since the 24 gauge nicrome has a resistance of 1.671 ohms per foot[3]:

$$\frac{14.06 \text{ ohms}}{1.671 \text{ ohms foot}^{-1}} = 8.41 \text{ feet} = 8 \text{ feet } 5 \text{ inches.}$$

This length of wire is used to wrap a tight coil along a 5mm mandrel. The coil is slightly stretched so the adjacent helices will not short by touching.

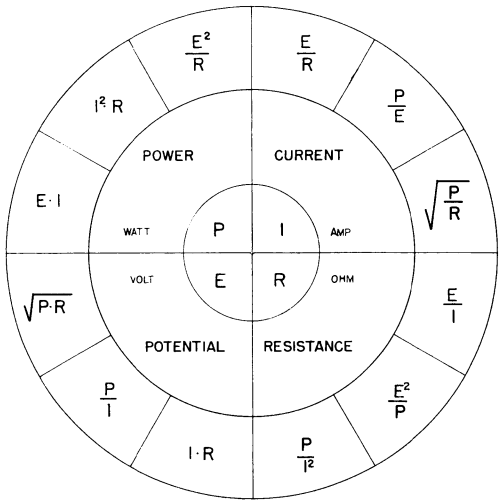

FIGURE 1

CARTRIDGE HEATER

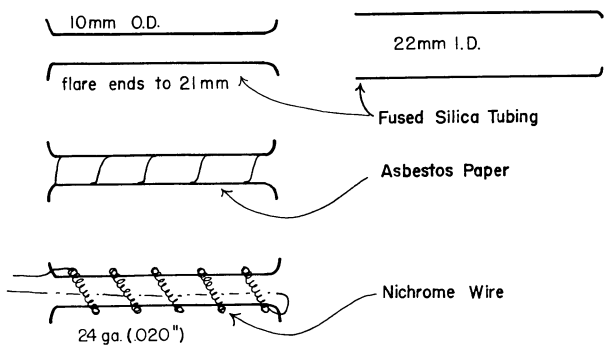

FIGURE 2

Figure 2 shows the heater assembly. The inner tube of 10mm outside diameter fused silica is flared at both ends so it will just slide into the 22mm inside diameter outer piece. Wet unburned asbestos paper is wrapped around this core. While the asbestos paper is still wet the nichrome coil is wound around this core. The flared ends of the core are slotted and the ends of the nichrome coil are threaded through these slots to keep the coil positioned. Water soluble furnace cement is used to paint the whole assembly both to keep the coil positioned and to contain the asbestos fibers. After the cement has dried the core assembly is slid into the outer tube. The outer fused silica tube is spot heated and dimpled to keep the inner tube assembly positioned. The outer fused silica tube is used to protect the borosilicate boiler from the hot nichrome wire which could sag, bite into the borosilicate, and break it. This could result in a major mercury contamination. Asbestos clad copper wire leads are attached via silver soldered lugs to the ends of the nichrome heater and from there to the controller.

VARIABLE VOLTAGE CONTROLLER

FIGURE 3

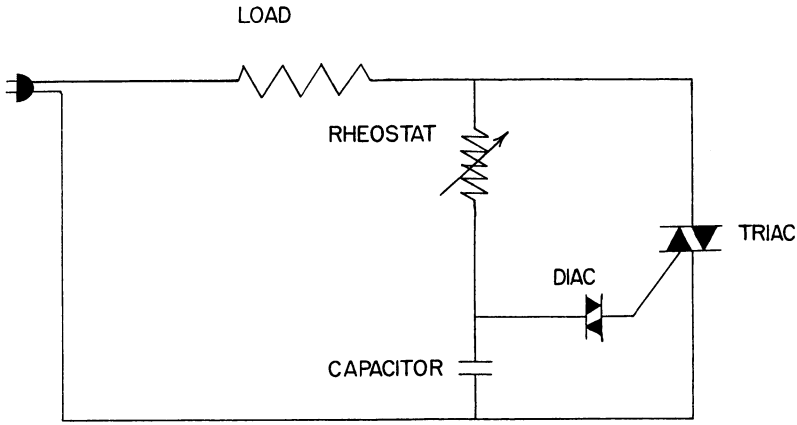

Figure 3 shows the schematic for a variable voltage controller. With alternating current the voltage moves from zero to plus through zero to minus and back to zero for the beginning of the next cycle. Since the voltage passes through zero twice each cycle the heater is being turned off twice each cycle, or 120 times each second. The function of this controller is to lengthen these times that the heater spends off.

The load in Figure 3 is the cartridge heater described above. A semiconductor is an electronic device which is sometimes a conductor and somestimes not depending on the circumstances. A diac is a semiconductor. It will not conduct until the voltage, or potential across it reaches its triggering value of 28 to 34 volts. Once it triggers it will remain conducting, even if the potential drops below its triggering value, until the current falls below 200 mA[4]. This part is an ST-2 diac.

A triac is also a semiconductor. It will conduct if there is a potential at its gate lead. The gate lead of the triac in Figure 3 is the one connected to the diac. The triac controls the heavy current of the circuit. The diac controls the triac. Thus the control of the time-base of the diac means the control of the entire circuit. This triac is an SC146B[5]. It is suitable for peak voltage of 200 volts and can handle 10 amps of current. Other triacs are available which can handle much higher potentials and currents.

The remaining two parts of the variable voltage controller are a variable resistor or a rheostat and a capacitor. They provide the time-base for the diac. A capacitor is an electronic device which stores a charge on its parallel plates. This one is rated at .1 microfarad. A resistor is an electronic device which limits the voltage across it. A rheostat is a variable resistor. This rheostat is rated at 0 - 200,000 ohms. The rheostat controls the amount of charge on the capacitor plates by varying the voltage. When the resistance of the rheostat is low then the charge on the capacitor plates is high. As a result, the charge on the capacitor will keep the diac conducting. The conducting diac will in turn keep the triac conducting. But, if the resistance of the rheostat is high then the charge on the capacitor plates is low and quickly depleted by the diac. If the resistance of the rheostat is high enough the diac will not reach its triggering value and the circuit will remain off.

The components in the periphery of Figure 3, namely the load and the triac, are the components through which the main current flows. The interior components are there to control the triac. The resistor-capacitor pair controls the amount of time that the diac triggers the triac.

The recycling of the hazardous mercury waste has been cost effective from the first use of the triple distillation apparatus. In addition to the savings the problem of finding appropriate dump sites for this chemical was virtually eliminated.

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SCIENTIFIC GLASSBLOWING TRAINING PROGRAM AND CERTIFICATION IN FRANCE

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It has been 50 years since an illustrated poster invited "parents who are justly concerned with the future of their sons to write to the Director of the ECOLE DORIAN, 74, Avenue Philippe Auguste, PARIS XI, to obtain free information about a program of scientific, industrial, medical, and artistic glassblowing. The section was created by the initiative of the Syndicate of makers of glass blown using a torch." The opening was in October 1931; the shop was installed by Mr. Paul Boudrie, head of the glassblowing shop at Rhone-Poulenc (a large chemical firm), who didn't stop his magnificent teaching mission until 1960, when he retired. He has trained 300 students, most of whom are scientific glassblowers today.

In 1947 another school named ECOLE des FEUILLANTINES opened in Paris. In 1968 it merged with the LYCEE TECHNIQUE DORIAN, (previously named ECOLE DORIAN) today the only school of that kind in France.

Before 1968 the L.T. DORIAN offered only one type of program for both the C.A.P. (Certificat d'Aptitude Professionelle)[1] manually oriented program and the more academic B.E.I. (Brevet d'Enseignement Industriel)[2] which was later replaced by the B.T. (Brevet de Technicien)[3]. It is only in recent years after the ECOLE des FEUILLANTINES merged with the L.T. DORIAN, that a new program was tailored to meet the needs of the job market. Now, rather than every student having the same curriculum, some will receive rigorous manual training and earn a C.A.P. and others will earn a B.T. indicating preparation to work in research.

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[1]Certificate of Professional Aptitude
[2]Diploma of Industrial Arts
[3]Technical Diploma in Scientific Glassblowing

Entrance to the School:

To give a better idea of the general instruction level for entering students you should know that the school system in France is based on 13 years before entering the university.

To be admitted to the program leading to the degree of B.T. the student has to have accomplished 10 years of schooling, and the average age is 16 years old. Since the number of places in the class is limited, only the best qualified students are admitted.

To get into the program leading to the C.A.P. the student has to have accomplished 9 years of schooling. The average age will be 15 years old.

Both of the programs are 3 years long. There is a possible specialized year if the student wishes to learn how to make neon signs. The B.T. program emphasizes sciences and technology in order to produce a technician with a solid base should the student wish to further his education. This paper will concentrate mostly on the B.T. program.

Aptitude:

Being a glassblower demands an analytical spirit, decisiveness and problem solving ability, in addition to manual dexterity, a sense of aesthetics, imagination, and good space perception.

Sixteen students started the B.T. program three years ago, only nine will finish. The quality of the average class is such that seven of this final nine might be expected to receive a diploma.

Instructional Staff:

There is a total of 6 glassblowing teachers. For the B.T. program they are Mr. H. Luneau, the head lecturer and coordinator of the glassblowing department of the school, Mr. Peyrussie and Mr. Garnier (specialized in lathe work). For the C.A.P. program there are Mr. Gachet, Mr. Baehr and Mr. Beaujard (specialized in neon signs). All these men are professional dedicated teachers who have been selected from among the best of their peers.

The rest of the curriculum is taught by other faculty of the LYCEE TECHNIQUE DORIAN, as the glassblowing program is one of several programs offered by the school.

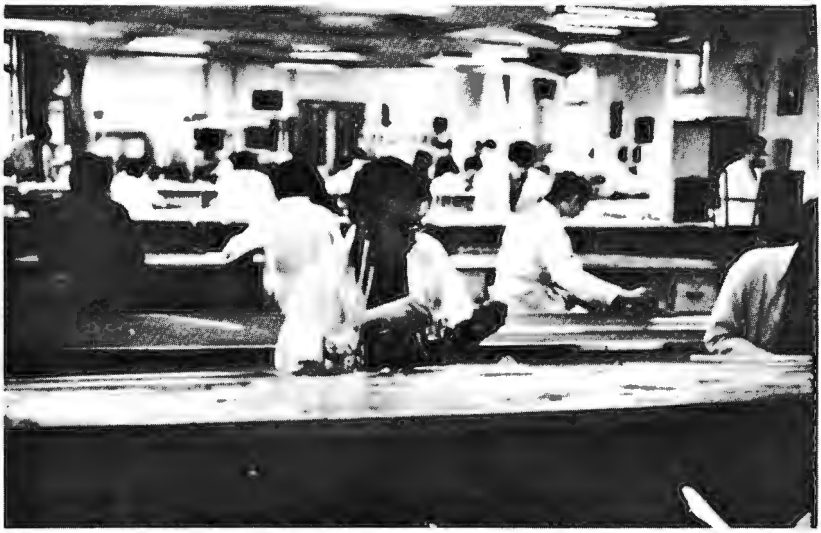
Facilities:

The glassblowing section is part of the LYCEE TECHNIQUE DORIAN, a technical school which prepares students for various specialities with a curriculum in engineering. There are around 800 students, about 70 of them enrolled in the two glassblowing programs of three years length.

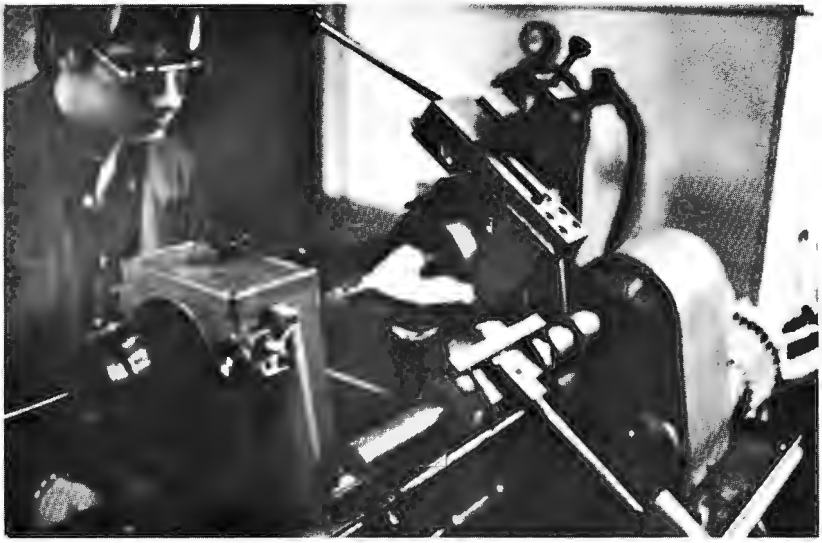
The workshop area consists of one large room which can accommodate 40 students at once. It is very well lighted and equipped with a good ventilation system. In this room can be found four large benches, glassracks, wet cutting machines, drilling machines, ovens, polariscopes, lapping machines, and 7 small lathes. (Pictures I and II).

The burner used by the students is of French design. It is a sideways on turret type. (Picture III) You can move it away from the table.

Another room is specially equipped with five large lathes, (Picture IV), with canopy hoods installed above for ventilation, with an oven, a cutting wheel, and a polariscope. In a third room neon sign work is done, in addition to vacuum techniques. For general studies, the classrooms and labs are shared by all the students of the school.



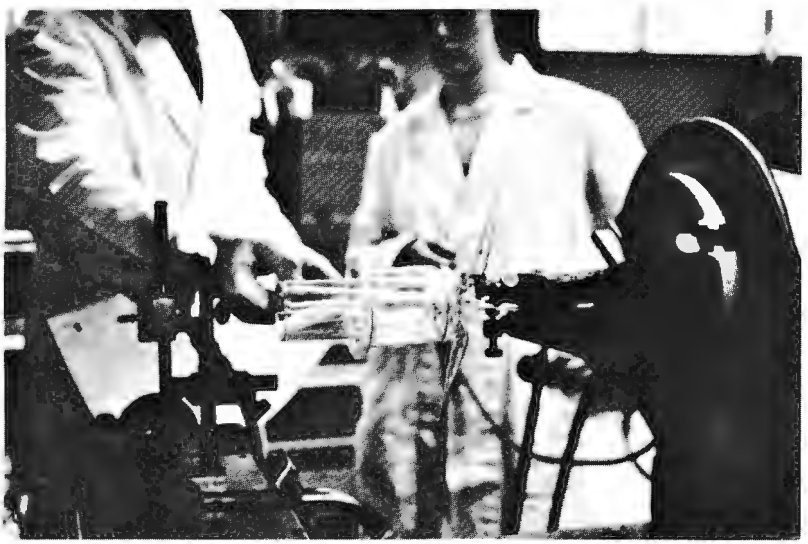
Picture I



Picture II

Picture III

Picture IV



CURRICULUM

As I mentioned earlier, this paper will only emphasize the three year program leading towards the B.T.

In Figure 1 you will find an outline of the curriculum. At the end of the first and second year the faculty examines the grades of each student to determine if he is qualified to proceed to the next year in the program. Figure 2 is a sample of a weekly schedule, and Figure 3 is a diagram which defines the number of hours the students are exposed to scientific glass-blowing.

I shall now describe the various courses in the curriculum.

FIG. 1

CURRICULUM

SUBJECTS	HOURS PER WEEK 1st Year	2nd	3rd
A) General Studies			
1) Liberal Art			
French	4	3	3
History and Geography	2	-	-
Economics	-	-	1
Foreign Languages	3	2	2
TOTAL	9	5	6
2) Sciences			
Mathematics	5	3	2
Physical Sciences: Physics	2	2	2
Chemistry	2	1	2
Labs	1	-	-
Mechanics	-	1	-
Electricity	-	2	2
TOTAL	10	9	9
3) Art	1	1	1
B) Physical Education	2	2	2
C) Professional Studies:			
1) Technology of Construction: Drafting	5	5	6
2) Glass Technology	1	1	1
3) Glassblowing Lab	11	16	15
TOTAL	17	22	22
GRAND TOTAL	39	39	40

FIG. 2

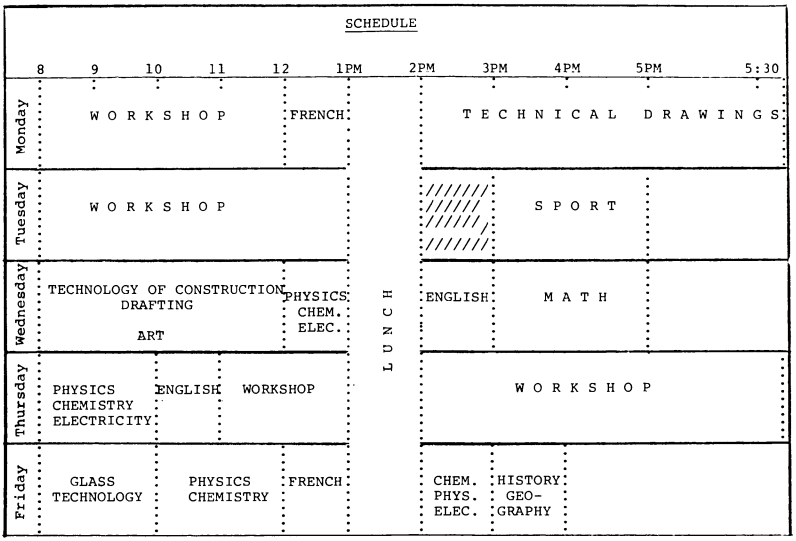


FIG. 3

NUMBER OF HOURS OF GLASSBLOWING
UNDER THE B.T. PROGRAM

1st YEAR	S.I.[1]	2nd YEAR	S.I.[1]	3rd YEAR	TOTAL
11 hrs x 38W[2] = 418 hrs.		16 hrs x 38W[2] = 608 hrs.		15 hrs x 38W[2] = 570 hrs.	In School 1596
	40 hrs x 6W = 240 hrs.		40 hrs x 8W = 320 hrs.		S.I.[1] 560 hrs
658 hrs		928 hrs		570 hrs	TOTAL 2155 hrs

[1] S.I. (Summer Internship) in Industries or Research labs from 6 to 8 weeks.

[2] 38 W. (the student is attending the school about 38 weeks a year).

TECHNOLOGY OF FABRICATION
GLASS TECHNOLOGY

1st Year

1. General principles of setting up a glass shop: Fuel distribution, control and security, ventilation
2. Equipment necessary: machines, torches, tools, glassblowing lathes including their installation, maintenance and security.
3. Metrology: (science of measurement)

2nd Year

Glass properties

Chemical: classification of principle components of glass, quartz, different allotropic varieties

Physical: fusion, solidification, supercooling, vitrification, crystallization

Mechanical: coefficient of expansion, curves, transformation points, studies with dilatometers, coloration, discoloration, opalization

3rd Year

1. Studies of various processes of fabrication and transformation of glass
2. Various melting techniques, old and modern
3. Fabrication of flat glass
4. Thermal treatment of glass: tempering, annealing procedures, measurement of strain and birefringence with polarized light
5. Complement to physics course on apparatuses utilized in vacuum techniques — pumps, manometers, gauges
6. Complement to technology of construction course: machines utilized for glass working

WORKSHOP

1st Year Introduction to glassblowing:

Uniform rotation of glass tubing, various techniques of cutting glass, pulling points, straight seals (same diameter, tapered), lateral seals, round bottoms, flat bottoms, blowing bulbs, bends, flaring, elementary work on capillaries and rods.

Practicing assembly and synthesis of techniques:

After studying the function of simple apparatuses utilized in chemistry experiments the student reproduces the blown glass object using his acquired knowledge.

DIAGRAM 1

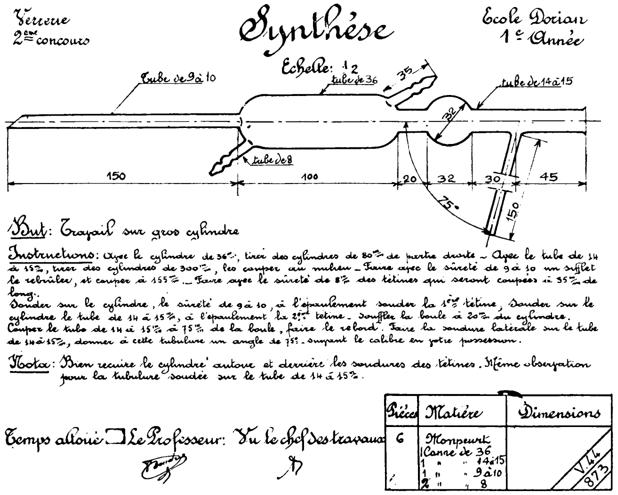
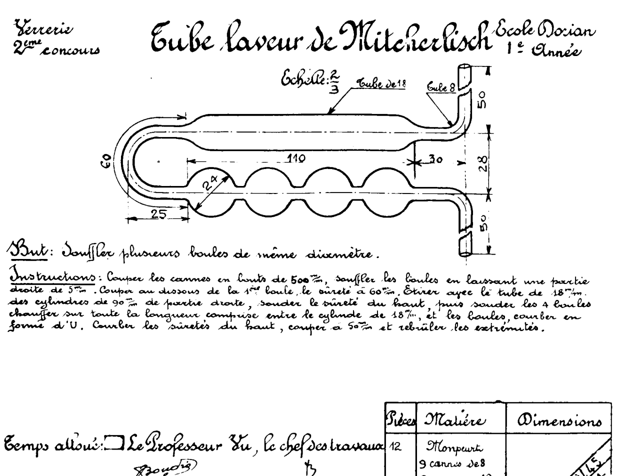


DIAGRAM 2



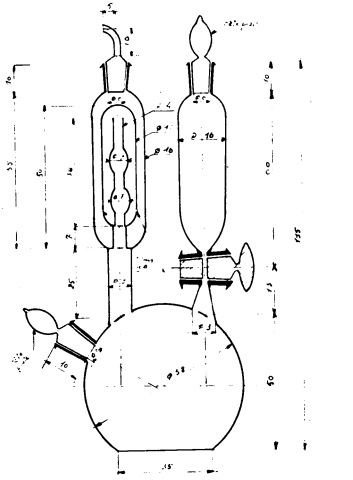
Metrology: Use of special measuring tools to control dimensions.

2nd Year

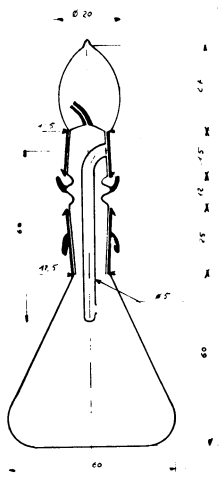
Perfecting blowing bulbs and lateral seals, learning to make coaxial seals (ring seals), side ring seals, fabrication of various types of condensers, transformation of tubes using molds, making stopcocks and standard taper joints.

DIAGRAM 3

APPAREIL DE SCHROEDTER.



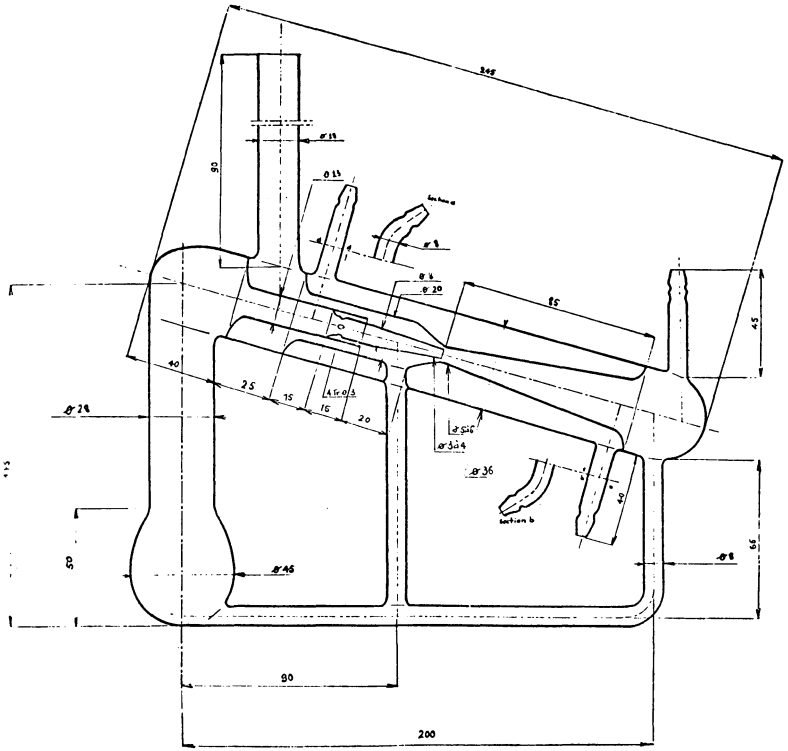
FIOLE COMPTE-GOUTTES



Metrology: Verification of dimensions: parallelism, angles, tolerances, thicknesses; also the effect of annealing with a flame compared to an oven, and qualitative studies with the polariscope.

3rd Year

This year is dedicated to the application of knowledge acquired during the two preceeding years in order to fabricate apparatuses like diffusion pumps, and various types of distillation apparatuses (Diagrams on pages 35 and 36).



TROMPE A VAPEUR
DE MERCURE
DUNOYER

échelle : 1

DIAGRAM 4

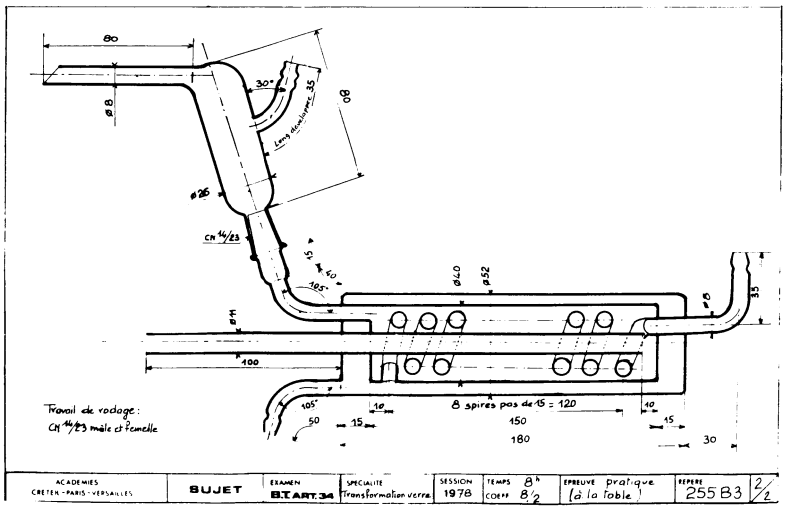


DIAGRAM 5

The student is introduced to special glass including quartz, and glass to metal seals (tungsten, platinum, molybdenum, copper-clad, Dilver P, Kovar). The use of the hand torch is taught and applied to vacuum line construction. Small and large lathes are introduced as is drilling and grinding glass.

Metrology: testing of stopcocks, joints, testing of tightness under vacuum of glass to glass and glass to metal seals.

Fabrication Studies:

Commencement of work, record keeping, establishment of ranges and corresponding detailed analytical files. Studies of fabrication tools, timing excercises, introduction to machining.

DRAFTING AND TECHNOLOGY OF CONSTRUCTION

A group of courses in design and construction are intended to teach five basic priciples:

1. To visualize objects in space

2. To read a design
3. To learn how to analyze a technical problem, to recognize technical functions and put them together logically
4. To design pieces and create forms
5. To establish a functional quotation

Industrial Design:

In the first year the student learns how to work neatly, use design instruments correctly, define in detail the forms of an element, and understand the function and utilization of a single ensemble. This involves learning to choose views properly, draw to scale and in perspective, etc.

In the second year the student learns how to analyze a technical problem and draw a flow chart of the solution. He should be able to draw designs of one piece and of groupings freehand and using various instruments.

In the last year the student begins to choose forms for their functionality, calculate quotations and verify them, and solve numerous problem exercises.

Descriptive Geometry:

Descriptive geometry is a one year course taught in the second year of the program. The usual concepts of geometry are taught:

1. Points, lines and planes
2. Shapes: prisms, pyramids, cylinders, cones, spheres, toruses
3. Tangents
4. Helices

Technology of Construction:

In the first year, students concentrate on two subjects. The first is various joints and all the difficulties which may be encountered in making them. The second is valving and how to choose and execute the appropriate system.

In the second year calibration is taught, as are the various types

of condensers and extractors.

In the last year the topics covered are distillation apparatuses, barometers, glass to metal seals, and optical polishing.

CHEMISTRY

First Year

The introductory course presents the theory of atoms and molecules and descriptive chemistry essential to the practicing glassblower.

Second Year

Inorganic chemistry for glassblowers is taught in the second year of chemistry. The chemistry as well as the mechanics and physics of metals are taught. Metal oxides and mixed metal oxides are used to introduce the descriptive chemistry of glasses. Phase diagrams are used to explain a number of properties including crystallinity, viscosity, and coefficients of expansion.

Third Year

The last year of chemistry is concerned entirely with organic chemistry. The special ability of carbon to catenate helps explain similarities among compounds, and functional groups such as alcohol and carboxylic acid help explain the differences among compounds. The most important synthetic techniques are carried out in the laboratory by the students.

MECHANICS

Last Year

A number of important mechanical principles and the applications essential to the modern glassblower are taught in the last year of mechanics: pulleys, friction, and gears. The effect of mechanical forces on materials is covered. The principles of operation of simple machines, such as levers, pulleys, and inclined planes are explained in terms of the equality of work done by the motor and of the resistance.

ELECTRICITY

Last Year

The properties of alternating currents having been covered in previous years, the last year goes on to electric machines and electromagnets. The principles and operations of alternators, transformers, and motors are taught, as are several machines which operate on direct current. Elementary electronics is also taught in this course. The principles are developed and applied to vacuum tubes and semiconductors.

MATH

First Year

Algebra and geometry are taught during the first year. Algebra is primarily review, but exponents, polynomials, and functions are introduced in this part of the course. The students are taught to use numerical tables, including log tables, and to use slide rules. Geometry is divided into five sections: plane geometry, space geometry, vectors, trigonometric functions, and graphing. Plane geometry treats circles in depth. Space geometry deals with the intersection in space of two lines, two planes, or a plane and a line. Projections, important to glassblowers in reading blueprints, are also taught. The vector segment of the course covers all aspects of that subject: properties, algebraic operations, translation, and rotation. Trigonometric functions and the use of tables are taught. Graphing in cartisian and general rectilinear coordinations is used in the final section of the course to illustrate functions, vectors, and other ideas covered previously.

Second Year

Introductory calculus, trigonometry, and advanced geometry are taught in the second year. Derivatives are covered after a review of the concepts of limits and functions. Examples are given. In the trigonometry section students are taught to manipulate algebraically the trig functions to which they were introduced during their first year. In the final portion of the course the students are taught the formulas for calculating areas and volumes of all the important geometric shapes.

Final Year

In the final year, more applications of derivatives are taught. Graphical representation of differentials is used to introduce definite integrals. A number of definite integrals are taught and the students are shown how integration can be used to calculate areas, volumes, and moments of inertia. The last section of the course deals with logarithms and exponentials in base 10 and base e. Their properties are taught, as is their relation to roots of numbers.

PHYSICS

Last Year

A number of principles directly applicable to glassblowing are learned by the students: viscosity, capillarity, solution theory, the law of partial pressures of gases, spectroscopy, and the wave nature of light. Vacuum techniques with distillation applications are also covered extensively in this course.

FRENCH LITERATURE

1st Year

French Composition: One week out of two an hour of French composition will be dedicated to the correction of a written exercise done outside of class and discussions of its thoughts and maxims. The other week is to be reserved for group exercises of composition and editing. If necessary there will be a brief review of grammar with excises.

Recitation: Once a week a short poem or prose, studied and discussed in class will be memorized and recited with emphasis on elocution and diction.

Study of authors: From the sixteenth century to today

2nd Year

One week out of three, students will review a literary work written outside the class. The other two weeks they will do oral exercises.

Authors: Cultural implications of work by ancient and modern authors, the situation of France today, problems of civilization, and elementary principles of economics will be studied and developed.

3rd Year

Students continue the study of literature and advanced composition. They write themes on human problems at work. Modern methods of psycho-sociology investigation, and moral ethics on the job will be developed.

HISTORY

The course will cover the aftermath of the second French Empire to the eve of World War II.

GEOGRAPHY

Physical and human geography will be covered, one half a year for each. Under the first part, the student will study the surface elements of the Earth's environment, the relationship among these elements, as well as some notions of oceanography. The climatic phenomena and their application to the human factor will be the subject of the second part.

FOREIGN LANGUAGE

1st Year

Today in France, in general, the study of foreign languages starts in the sixth grade. When the students enter the L.T. DORIAN they already have four years of a foreign language. At the L.T. DORIAN the students have a choice between English and German. In the program of scientific glassblowing English is the only language taught.

At the beginning there is a general review and afterwards, a program to enrich vocabulary and grammar with an introduction to business communications.

2nd and 3rd Year

Students learn to utilize audio-visual methods and they practice professional communication. They are introduced to technical vocabulary (tools, machinery, and fabrication terminology). They study the technical revolution of yesterday and today and the continuous nature of this phenomenon.

ART

The artistic education concentrates mostly on cultural aspects. It is less important for the students to draw than to be conscious of all the plastic forms and problems that the world today encounters.

With help of courses directed in this manner the students will acquire the means to build a more harmonious life environment later in life, to be able to judge art objects, and generally everything which has an art application.

Some of the topics are: Graphic art, stained glass, glassblowing, painting, sculpture, architecture, the relationship between aesthetics and ma-

CAP. VERRERIE SCIENTIFIQUE

CONTENT OF EXAMINATION

Each test is graded from 0 to 20 and a weight of the test is given by the coefficient. A 10 average or above is required for graduation. A grade of less than 12 in the practical test will eliminate the candidate.

AREA OF TESTING	Duration in hours	Coeff.
I. Practical test:		
Scientific glassblowing[1] --	up to 16	10
II. Written test:		
French composition pertinent to the profession ----------	1-1/2	2
Math ----------	1-1/2	2
Technical Drawing ---------	2	2
III. Oral testing:		
Glass technology ----------	1/2[2]	3
Legislation, working regulations and laws accident prevention, hygiene ----------	1/2[2]	1

[1] The practical test is divided in two parts, one based on quality using soda-lime glass and the other part being a speed test. The quality test has a coefficient and a duration double of the one based on speed.

[2] The candidate has 1/2 hr. prior to the exam to prepare the given subject.

FIG. 4

chines, and man in his environment. Many field trips are taken by students to various museums to illustrate this course.

At the conclusion of the third year towards the end of June, the examinations for diplomas are taken by the students of the school and qualifying independent candidates (persons who have been trained by other means but desire to obtain certification).

In Figure 4 you will find the content of the examination for the C.A.P. and in Figure 5, the content of the examination for the B.T. along with an example of the practical test of the year 1973. Diag. 6 (at the bench). Diag. 7 (at the lathe).

B.T. VERRERIE SCIENTIFIQUE

CONTENT OF EXAMINATION

Each test is graded from 0 to 20 and a weight of the test is given by the coefficient. A 10 average or above is required for graduation. A grade of less than 10 in the practical test will eliminate candidate.

AREA OF TESTING	Duration in hours	Coeff.
General Studies:		
Written Tests		
French and Economics	3	5
Mathematics	3	5
Oral Tests		
Foreign Language	1/2*	2
Electricity and Mechanics	1/2*	3
Professional Studies:		
Written and graphic tests		
Physics and chemistry	3	6
Technical Drawing Technology of fabrication	6	6
Practical Test		
Scientific glassblowing	12**	8
Oral Test		
Glass technology Technology of fabrication	1/2*	4

*The candidate has 1/2 hr. prior to the exam to prepare the given subject.
**Consists of bench and lathe work.

FIG. 5

DIAGRAM 6

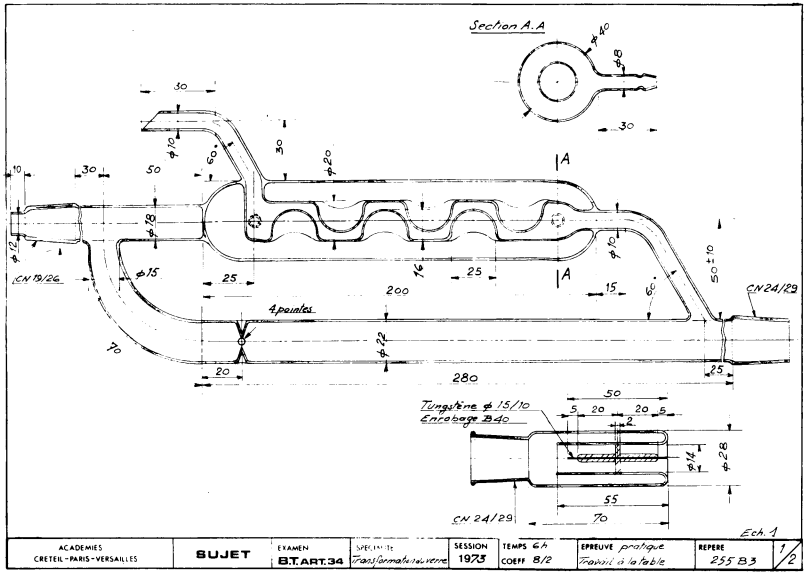
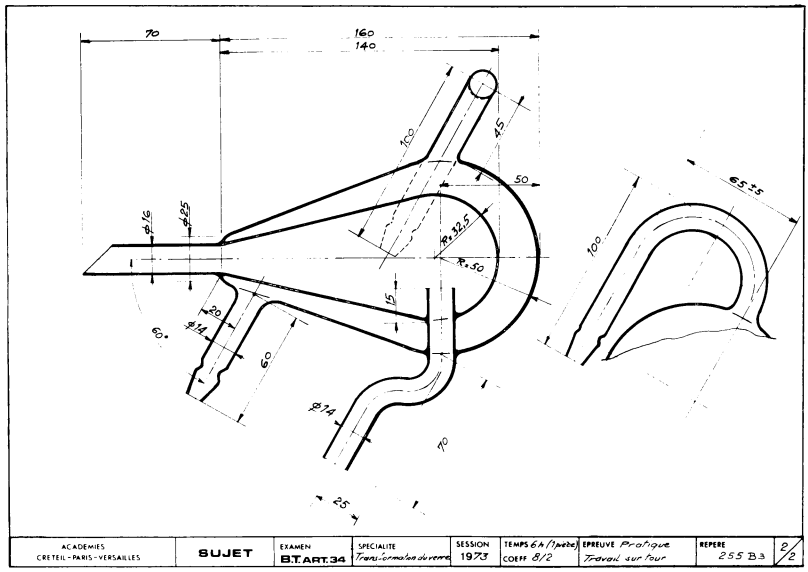


DIAGRAM 7



CONCLUSION

Certification is a form of peer recognition of an individual's professional ability. The assumption is often made that a person who is certified is more qualified than one who is not. This assumption, of course, is not always true.

In the USA in simplest terms, certification as a word is ordinarily used as a voluntary process carried out by a non-governmental organization. Registration and licensing on the other hand are procedures performed by governmental agencies.

In France, the diploma given to candidates who merit, called B.T. or C.A.P., are in short a certification, a registration and licensing all together since the training program is under care of the education branch of the government. The diploma is dispensed by that branch and the government has required, for the last 20 years, young potential employees to be in possession of one of these diplomas in order to qualify for a government job. In the private sector the situation is similar, and in general, except for older established glassblowers, promotion and wages are influenced by these diplomas. Moreover, if you want to open a privately-owned shop you will have to be certified.

There are three main ways to learn glassblowing: through a school, under a commercial enterprise program, or under a Master. A fourth one is the empirical method and is not recommended today.

For French people who are serious about learning glassblowing it is strongly recommended that a youngster who wants to enter the profession, unless issue of a parent himself an artisan glassblower, attend the L.T. DORIAN.

In France most agree on the principle that a school is more prepared and has a better aspiration for dispensing fundamental knowledge to a student, as opposed to a commercial organization which has a tendency to prepare their trainees to serve their own interest. To a certain extent it is also true, too, for the Masters. Their weakness is that although they excel in their field, they may not be such good teachers. To support this statement the percentage of success in the exam for the diploma is greater for those who have attended school than for the independent candidates. Although every individual Master has his limitation, the L.T. DORIAN provides students with six teachers and each of them has a specialty to offer! Having tenure allows them unfettered pursuit of academic innovation, professional dedication, and to train in the best interest of the profession and the nation.

After viewing the curriculum, the content of the examination, and listening to my presentation, most of you realize that the L.T. DORIAN has no pretention to deliver to the professional community minimaster glassblowers. The relatively short workshop program and the fact that the learning of scientific glassblowing is a lifetime project, does not permit this.

The L.T. DORIAN rather gives a well-rounded glass technologist. He will be perceptive and prepared to further develop his academic and manual knowledge when he is in contact with elderly peers, researchers, and engineers in our dimensionless future.

In France, I can say, the L.T. DORIAN has been and is today the cornerstone of scientific glassblowing.

ACKNOWLEDGMENTS

The author wishes to express his appreciation to Mr. H. Luneau, head lecturer in scientific glassblowing and the faculty of the L.T. DORIAN whose fine teaching has gotten many students, myself included, off to an excellent start in their professional career.

The author also wishes to thank Dr. Hulen B. Williams, Dean of the College of Chemistry and Physics, Louisiana State University, Baton Rouge Louisiana, for sponsoring the preparation of this paper including the clerical assistance of Ms. Susan Scott. Special thanks to my dear wife, Anne, who will never let me present another paper again!

Addendum: Starting in September 1981 the selection of students will be changed. All students entering the L.T. Dorian will follow the same program the first year in order to make a more rational choice of their specialty whether it be glassblowing, electronics, surveying or fine mechanics. Because the first year will be more general a fourth year of instruction will be necessary in order to teach all the techniques of glassblowing.

For the future the possibility of a fifth year leading to an advanced diploma is being discussed.

Interested parties are invited to contact Mr. H. Luneau at the Lycee Technique Dorian, 74 Avenue Philppe-Auguste, PARIS 75011, FRANCE.

LIGHTGUIDE FIBER PRODUCTION & A UNIQUE TORCH DESIGN FOR QUARTZ PREFORMS

Brian Lynch
Product Engineering Control Center
Western Electric Company
(Atlanta Works)

INTRODUCTION

At Western Electric we have for many years manufactured copper wire cables for the express purpose of telephone communications and data links. In recent years the Bell System has been researching and developing an alternative media for transmitting messages by pulsing a laser beam signal over a hair thin optical fiber that has to be produced to very stringent specifications using ultra pure chemicals and clean air environment.

We gave it a fancier name, that of lightguide. Within the last year we have progressed from the development lab into the production shop, although a tremendous amount of on-going development still continues.

The manufacture of lightguide cables consists of five basic steps:

1. Make a preform.
2. Draw that preform into a fiber.
3. Mount 12 fibers in a ribbon form.
4. Cable up to 12 of these ribbons in a protective covering.
5. Connectorize fiber ends for installation.

In the first part of this paper we will cover only the manufacture of the preform and fiber in some detail, and touch very lightly on the ribboning and cabling. The second part of this paper will deal with torch characteristics and design considerations that lead to a novel torch design used in the manufacture of preforms.

PREFORM MANUFACTURE

In preform manufacture, there are three well known techniques **(Fig. 1).**

1. V.A.D. or vapor axial deposition.
2. OVPO or outside vapor phase oxidation.
3. MCVD or modified chemical vapor deposition.

All three systems produce silica oxide ($Si0_2$) by reacting $SiCl_4$ vapor with 0_2 in a heated zone. Controlled amounts of $GeCl_4$ and $POCl_3$ vapors

are also added to alter the refractive index of the deposition layer and to reduce the sintering temperature respectively.

We will confine our discussion to the MCVD process as it happens to be the one we employ at Western Electric.

To produce a preform with the MCVD process, we must first start with a substrate tube of high silica quartz — dimensions of the tube being 25 mm O/D × 19 mm I/D × 1.3 m LG. After carefully measuring and characterizing the tube using a laser micrometer and microprocessor, those tubes that are within specification are cleaned by acid etching to remove any contamination that would be detrimental to finished product.

Figure 2 shows schematically the equipment and method required to produce a preform. The tube is placed in a glass lathe between two synchronous chucks with one end inserted into a rotary connector, while the other end is flared and welded to a 50 mm diameter exhaust tube.

Each lathe has its own mini-computer and control unit that will track and automatically hold programmed set points, such as 0_2 and H_2 flow to torch by means of two flow controllers; torch velocity along the lathe bed; surface temperature of tube via a pyrometer feedback system; and chemical vapors in correct proportions from the MCVD unit to the lathe.

The operator can manually override and alter the set points in the computer should the need arise. Chuck speed and certain heat controls are manually set. In operation the torch will travel from right to left at a slow speed during its working pass and return at a fast speed. The length of torch travel up and down the lathe being controlled by limit switches mounted to the lathe bed.

The first few passes are known as polish passes. They are performed at a high temperature (≈1700 °C), and condition the tube for the even deposition and temperature control to come later.

After the polish passes, a phosphoborosilicate vapor mixture is introduced into the tube where it is reacted by the torch as it travels along the length of the tube. The resulting layers that are deposited provide a barrier that will prevent any contaminents left in the starting tube from penetrating or diffusing into the subsequent deposition layers.

In the MCVD unit, the 0_2 passes through flow controllers at a rate set by the computer and bubbles through each chemical to produce vapor. All the vapors flow into a common feed line where they are mixed and directed into the center of the rotating tube. By traversing the torch slowly down the length of the tube, the oxide particles formed in the reaction zone are driven to the tube wall by thermophoretic forces and fused into a fine layer

of pure glass on the inside wall of the tube approximately 15 to 18 μm thick. This process will continue until about 50 separate layers have been deposited and fused in - each layer, because of an incremental increase of $GeCl_4$ will have a slightly higher refractive index.

After all the deposition passes have been completed, the tube is collapsed into a solid glass rod, or preform as it now becomes known. At the end of the collapse cycle, preform is separated from the exhaust tube and removed from the lathe.

It is very important that the tube remains perfectly circular and straight during the collapse phase. This is accomplished by balancing the flame stagnation pressure with a positive internal tube pressure and reducing the torch speed to a very low velocity **(Fig. 3)**. As the heat softens the tube, so the surface tension in the glass shrinks it.

The following table gives some preform manufacturing data:

FUNCTION	AVERAGE TIME (HRS.)
Positive pressure polish) Barrier layers) Deposition layers)	5 - 8
Collapse	3 - 4
Avg. total time for manuf. one preform	8 - 12 Hours

Surface temp. of tube 1500 to 1900°) Depending on the phase
Torch speed – 4 to 150 cm/min.) of preform manufacture

Figure 4 shows a typical preform making station — the mini-computer and main control unit are in the foreground, the lathe is located inside a vented compartment with a slide down front door. The MCVD unit, although not seen, is located at the extreme end of the lathe compartment.

FIBER DRAW

A fiber of 125 μm diameter that is to be drawn from the preform will be an exact duplication in all respects except that it will be some 18,500 times smaller in area (or 136 times smaller in diameter). We define a fiber as having a "core" consisting of the deposited layers, and a "clad" — this being the original starting tube. The core layers being deposited in increasing refractive index gives the required parabolic profile as seen in **Figure 5** — this is called a graded index fiber. The shape of the index profile influences the pattern of the laser beam passing down the fiber such that all

the light pulses will travel at the same rate and arrive at the other end of the fiber at the same time. If the index profile were "stepped" or of a single index value, some of the light would travel directly down the center of the fiber, while other light beams would be reflected back and forth across the fiber. Thus, the light pulses down the center would reach the other end of the fiber before those being bounced back and forth. It will be seen that the interface between the "clad" and the "core" acts as the internal reflecting surface because of the lower refractive index of the clad.

We mentioned before that the fiber drawn from a preform will inherit the same characteristics — this includes any imperfections such as bubbles, airlines and abrasions. As you can imagine, these imperfections will become tremendously magnified in relation to the final fiber size leading to possible breaks or high transmission losses in the fiber. Great care is taken not to abuse the outside diameter of the preform once completed — grease and moisture from finger prints, for example, can have a severe detrimental effect on the final fiber strength.

Referring to the schematic in **Figure 6,** it is seen that drawing the preform into a fiber is done in the vertical position, with one end of the preform being gripped in a chuck and slowly lowered into a furnace (or hot zone). Either graphite elements that are resistively heated or zirconia rings that are inductively heated are commonly used to heat the preform by radiation.

The furnace produces sufficient heat to soften the glass from which the fiber can now be drawn. In order to protect the surface integrity of the fiber during subsequent manufacturing and installation steps, a coating is applied after the fiber leaves the furnace.

The use of laser measuring devices are used to monitor the diameter of the fiber and the concentricity of the coating around the fiber. Although not shown here, the fiber passes through an optional in-line proof tester that will continually test the strength quality of the fiber. The strength of a perfect fiber at this time is greater than that of steel having a comparable diameter.

In order to minimize microbending stresses being induced into the fiber during take-up, minimum winding tension must be accomplished at the take-up spool. This is achieved by forming a catenary loop between the capstan and the take-up. A CCTV camera is employed to detect changes in the height of the catenary loop, and through an electrical control feedback regulates the take-up motor speed to within $\pm 1\%$.

The speed (V_p) with which the preform is lowered into the furnace, based on volumetric equilibrium conditions, is:

$$V_p = \left(\frac{D_f}{D_p}\right)^2 V_f \text{ - mm/sec}$$

Where: D_f = Diameter of fiber
D_p = Diameter of preform
V_f = Velocity of fiber

Fig. 7 shows the progression from the starting tube, to the preform to the spool of fiber.

RIBBONING AND CABLING

Once the fibers have been produced, a ribbon will be formed by positioning 12 of the fibers side-by-side and sandwiching them between two layers of plastic tape (mylar or similar). The lightguide cable is finally produced by enclosing up to 12 ribbons (144 fibers) within a series of extruded plastic jackets and metal strength members.

The finished cable is taken up on a large reel **(Fig. 8)** ready for connectorization and shipment after being thoroughly tested for quality and function.

During installation, the cable can be buried in the ground, pulled through existing duct work under the streets, or suspended as an aerial cable.

To conclude this portion of the paper, I think it is interesting to review some facts to give you some idea of why the fiber optic field is a fast growing and highly competitive industry, especially in the telephone communications market.

The following table shows the comparison between a cable made with copper wire to one made with optical fiber.

	Copper Cable	**Lightguide Cable**
Design	900 pr. screened pulp, 22 Ga.	144 fibers
Size	2.85 inch diameter	≃0.5 inch diameter
Weight	4000 lb/1000 ft.	80 lb/1000 ft.
Transmission Rate	1.5 megabits/sec	44.7 megabits/sec
Voice Channels	10,800	48,384
Repeater Spacing	1.14 miles	≃4 miles or greater

Fig. 9 gives some idea of the overall cable diameter when compared to the ear piece of a telephone handset — there are 144 fibers contained in the center square.

TORCH DESIGN

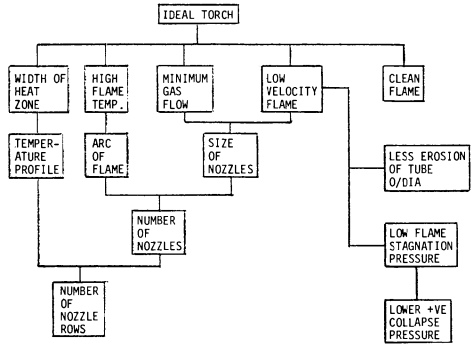
Let us now discuss in more detail the evolution of the torch design that we are presently using in our preform manufacture.

In the MCVD process, both the heterogeneous and homogeneous reactions take place in the hot zone created by the torch. From the heterogeneous reaction, a thin glassy film is deposited on the inside wall of the substrate tube, while the product of the homogeneous reaction — the doped silica particles — are deposited on the tube wall downstream of the hot zone.

The moving hot zone must be maintained at a temperature high enough to sinter these particles into a clear vitreous layer down the length of the tube. Of extreme importance is the temperature profile as seen by the tube surface. This profile must be smooth as shown in **Fig. 10** — any dips in the profile peak would lead to erratic deposition because of the hot zone temperature variation.

Rotating the tube in a lathe ensures that the localized heated zone is uniform all round the tube and that the deposition is evenly distributed on the tube wall. As the deposition temperature is normally above the softening point of the tube, rotation is also very important to prevent sagging and distortion within the heated zone. Thus deposition can be carried out at temperature as high as 1800 °C if required. These factors become critical when we recall that the deposition phase lasts for up to eight hours.

The following block diagram gives the basic design considerations that we looked for in an ideal torch for our particular production process:



The correct width of the heat zone is important to control optimum fining of the doped silica particles, and greatly influences the shape of the temperature profile. Too wide a zone tends to make the flame turbulent, resulting in two distinct heat zones, thereby giving a temperature profile that has a double peak. Under these conditions there is a greater possibility of the preform tube crank-shafting or going elliptical. The heat zone, therefore, should be kept relatively narrow.

A high flame temperature, as we have previously discussed, is essential for the fining operation. Constant and uniform temperature around that part of the tube being heated is a function of the arc of flame that partially surrounds and impinges on the tube.

For obvious economic reasons, minimum gas flows are desirable. The torch must have a flame velocity that is as low as possible, yet will still perform its primary function. A low velocity flame, apart from being quieter in operation, will minimize the erosion, or wearing away, of the starting tube outer diameter. Another benefit of the low velocity is that the flame stagnation pressure would also be proportionally lower, thus enabling the positive pressure used during polish and collapse to be as low as possible. The flame must be free of all contaminates such as oxidized flame nozzle material and air-borne particles, etc. that could be easily impinged onto the tube and fused in by the flame.

In reviewing the basic nature of a torch design, we know that a good flame is one that remains anchored to the nozzle of the torch **(Fig. 11)**. This is achieved by a velocity balance wherein the flame velocity, tending towards the nozzle, slightly exceeds the physical exit velocity of the gases based on the dimensions and configuration of the nozzle. Should the velocity balance be reversed, the flame would lift away from the nozzle and possibly extinguish itself.

The maximum temperature normally occurs at the tip of the inner cone, a fact that is critical to know when holding the relative distance between the torch nozzle and part being heated constant for any length of time. This ensures optimum usage of gases in achieving the desired temperature. There are two basic styles of torch designs that we considered for our process:

The first is a "pre-mix" style where two or more gases are mixed in a common chamber before being introduced to the torch nozzle(s) where the flame is formed **(Fig. 12).**

The disadvantages of this style of torch are:

1. Because of the high gas velocities necessary to achieve the required temperature, the flame becomes very turbulent and produces what we term a "hard flame" leading to unwanted erosion of the outer diameter of the substrate tube.
2. The possibility of "flash back" is high because if the flow of 0_2 should somehow be interrupted or reduced, the flame could very quickly chase its ignition source, the H_2, back into the mixing chamber and possibly back down the supply line to the main tank.

The main advantage of this style is the low manufacturing cost.

The other style is the "surface-mix" in which the gases are fed through the torch body in their own separate passage ways and mixed at the exit of the torch nozzle(s) where the flame is formed **(Fig. 13).**

The advantages of this style of torch are:

1. The chances of flash back are virtually eliminated.
2. A resulting low velocity flame for the temperature requirements — what we call a "soft" flame.
3. A low flame stagnation pressure resulting from the low flame velocity.

The disadvantage is a relatively high manufacturing cost.

In the early days of our development program we used both the premix and surface-mix style of torches that were, and still are, available on the commercial market.

Two to four of these torches were arranged around the tube as shown in **Fig. 14.** As you would expect, this arrangement had several disadvantages:

1. A large heat loss due to the lack of flame containment and inefficiency because of the greater amount of combustible gases required to achieve the desired temperature.
2. The temperature around the tube could not be held constant. More often than not this temperature variation led to tube sagging (or crankshafting). The tendency for the tube to go flat or oval in collapse instead of remaining circular was also a problem.
3. Because of the close proximity of the torches, they become extremely hot after many hours in operation. As a consequence, some of the flame nozzle material was oxidized and carried by the flame to impinge on the outer surface of the tube. Such a flame is commonly referred to as a "dirty flame" and is to be avoided in order to retain the strength integrity of the final fiber product.

By constructing a torch body that was semi-circular in shape containing a continuous array of flame nozzles, it was possible to produce a confined and continuous arcular flame to the tube being heated (**Fig. 15**). Because of the confinement, the flame and the tube temperature remain very constant for any set of gas flows. As the characteristics of the surface-mix torch were more in line with our concept of an ideal torch, our design was based on that style.

The original idea was to use two of these semi-circular torches to completely encompass the rotating tube. Each half to be adjustable, away from and towards the tube, to compensate for the necessary temperature variations during the process and the diameter difference as the tube is finally shrunk into the solid rod form. However, using just one-half of the torch, positioned on the underside of the tube, worked so well that we have retained this method, thereby saving on gas consumption and additional torch costs.

A further important and economic consideration is to obtain the optimum length of finished preform from the original starting tube. It follows that the bulkier the torch body design, the more limiting would be the overall length of the torch travel on the lathe. As an example, a reduction of a ½ " of torch travel at the end of each pass would mean a potential loss of nearly ½ km of fiber from the finished preform. It was, therefore, essential to design the torch body and its mounting to the lathe carriage as narrow and compact as possible.

Looking at the cross-section of the torch in **Fig. 16,** we see that it consists of four basic parts (Ref. U.S. Patent No. 4,231,777, Lynch, etal):

Two side plates, through which the gases are directed into two separate chambers created by an outer support member and another member into which the ends of the flame nozzles are oven brazed. The volume of each chamber ensures that the gases are distributed evenly to the nozzles throughout the arcular shape of the torch. In addition, each side plate contains a passage way close to the nozzle tips through which cooling water continuously flows. The partial view to the right is looking directly into the end of the flame nozzles. In order to further control the distribution balance and the stoichometric mixing of the gases, the flame nozzles themselves are constructed from circular tubes confined in a tight array by the two side plates and by the outer support member on the extreme ends. In this configuration, the 0_2 flows through the center of the tube; while the H_2 gas flows through the passage ways created between the tube outside diameters.

The gas flow through and around the nozzles assist the water in maintaining both the nozzles and torch body at a sufficiently cool temperature to prevent oxidization of the metal. To further reduce the chance of material oxidation, the entire body and nozzles are made from stainless steel — a metal that oxidizes at relatively high temperatures. The components for this torch have to be accurately machined and fitted together to prevent internal mixing of gases within the torch body and leakage to the outside through the interface joints.

In **Figure 17** the assembled torch is shown mounted to the lathe carriage heating the preform tube.

SUMMARY

I hope that this presentation has given you at least a brief insight into our approach to the production of optical fibers in this relatively new field of telecommunication technology. Because of the limited time, we did not discuss the many tight specifications and conditions that have to be met in order to produce an acceptable product. As we are in the leading edge of this highly competitive and expanding technology, future specifications can be expected to be much tighter and production environments to be far more critical.

The torch design that we have discussed is but a small improved step in the evolution of optical fiber production and although not yet completely optimized, this design has fulfilled most of our requirements for an ideal torch.

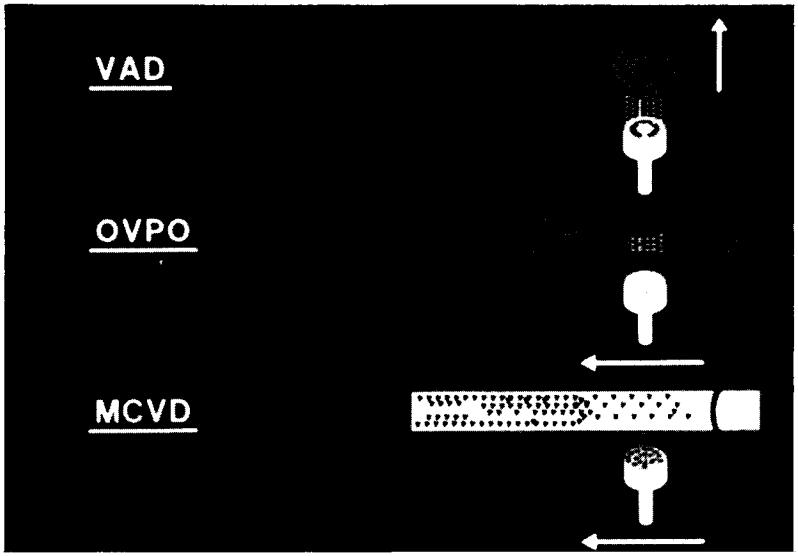


Figure 1 Methods of Preform Manufacture

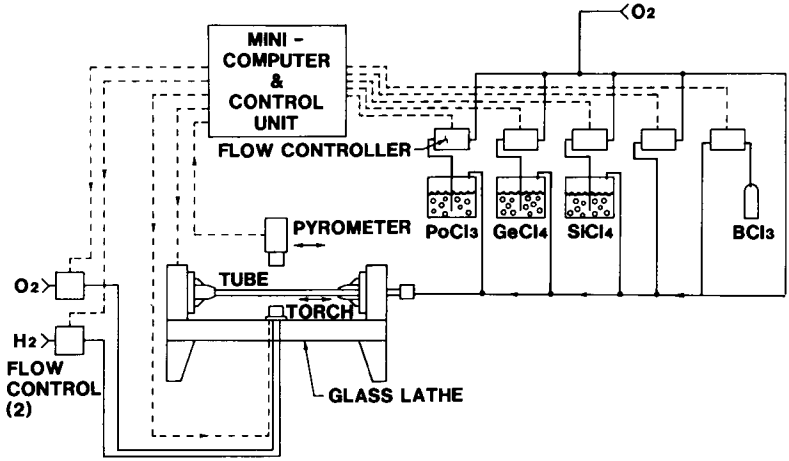


Figure 2 Schematic of Preform Manufacturing Equipment

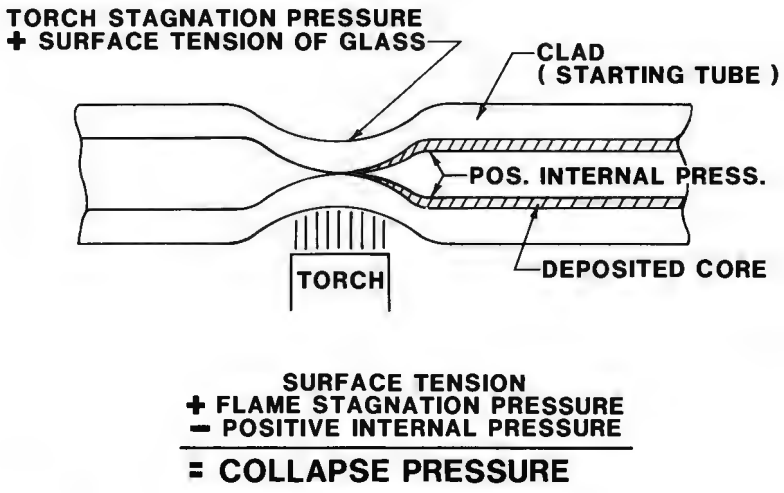


Figure 3 Balance of Forces During Collapse

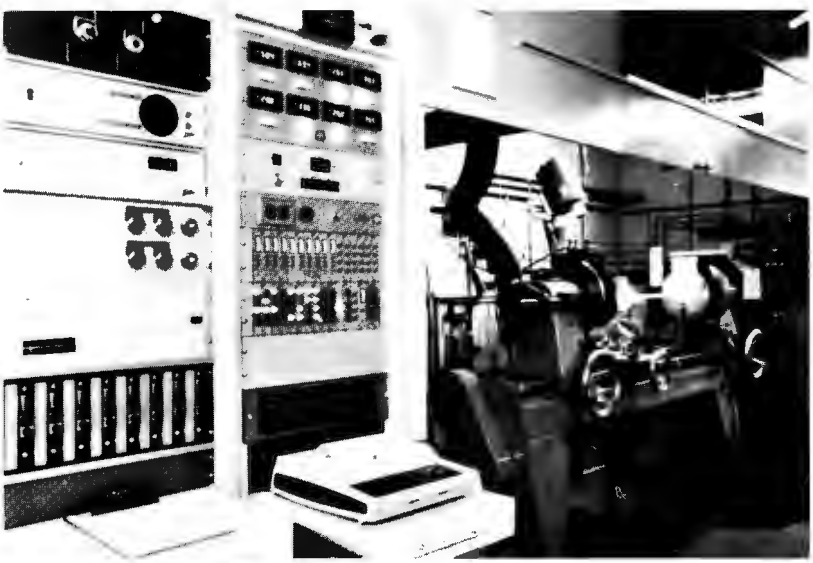


Figure 4 Typical Preform Making Station

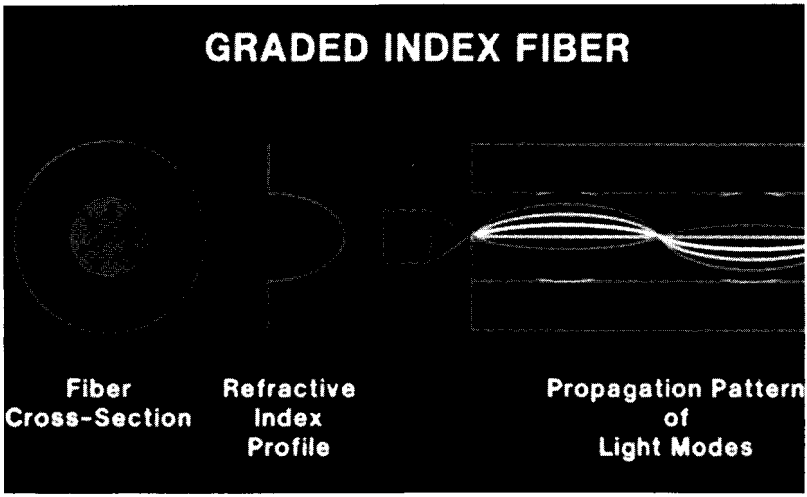


Figure 5 Graded Index Fiber

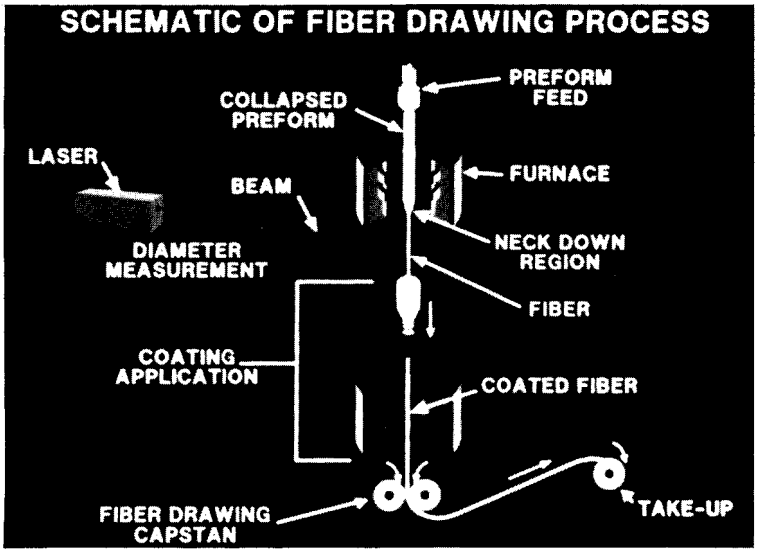


Figure 6 Schematic of Fiber Drawing Process

Figure 7 Starting Tube, Preform & Reel of Fiber

Figure 8 Lightguide Cable Being Taken-Up

Figure 9 Comparison of Lightguide Cable to a Telephone Earpiece

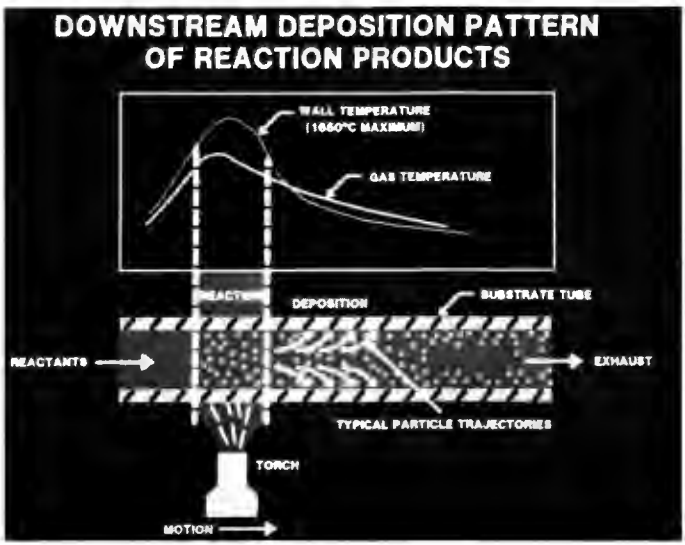
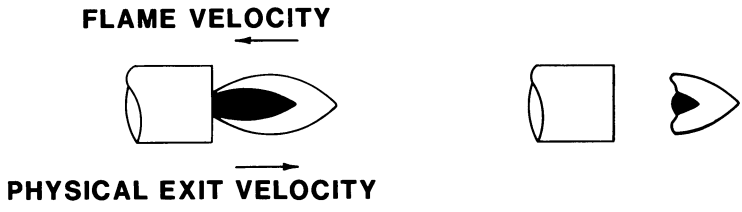


Figure 10 M.C.V.D. Process with Temperature Profile



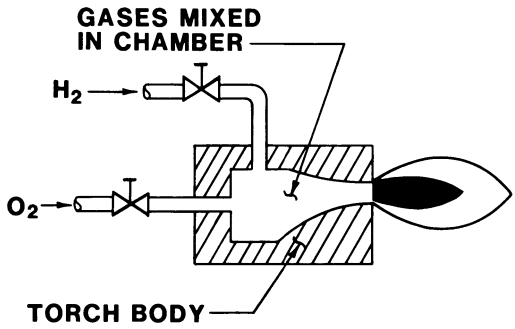
ANCHORED FLAME

- FLAME VEL. SLIGHTLY EXCEEDING EXIT VEL.

FLAME LIFT

- EXIT VEL. EXCEEDING FLAME VEL.

Figure 11 Torch Velocity Balance



DISADVANTAGES

- CRITICAL GAS VELOCITIES
- POSSIBLE FLASH BACK
- "HARD" FLAME

ADVANTAGES

- LOW MANUFACTURING COST

Figure 12 Pre-Mix Torch

Figure 13 Surface Mix Torch

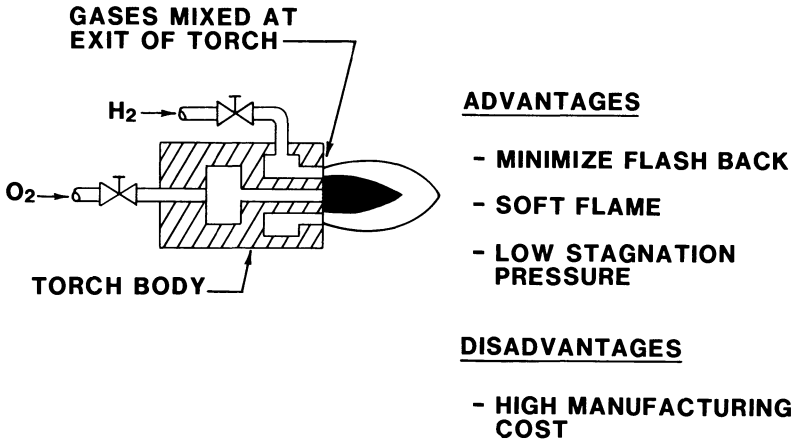


Figure 14 Arrangement of Single Torches (Commercial Type)

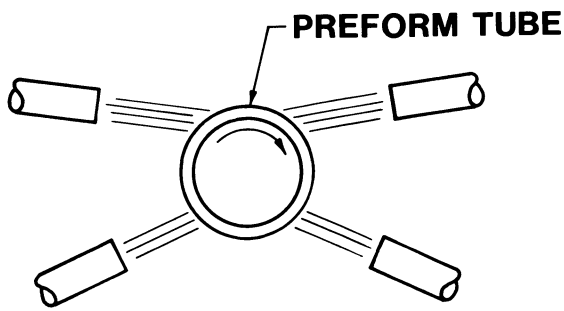
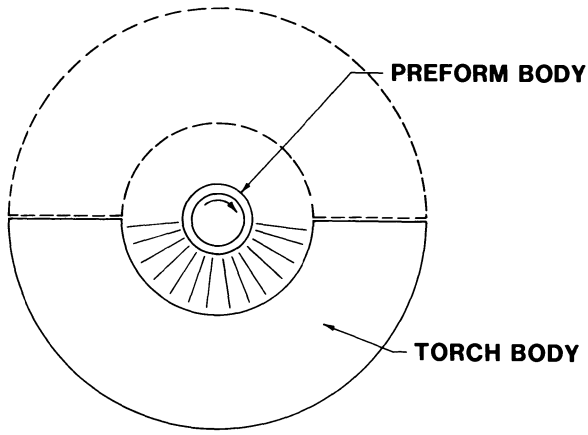


Figure 15 Western Electric Design Torch



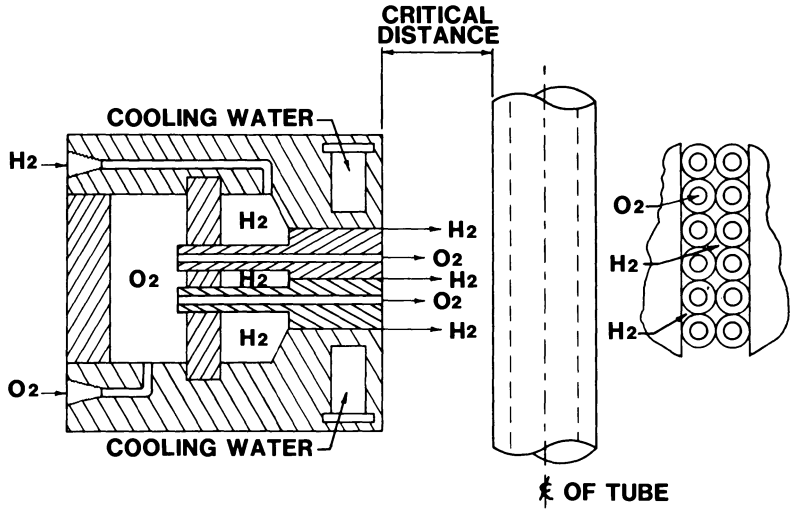


Figure 16 Cross-Section of W.E. Torch

Figure 17

W.E. Torch Heating the Substrate Tube

THE CONSTRUCTION OF A SMALL VOLUME CHEMOSTAT

Alex Stuart

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Introduction

The chemostat that I will be speaking about is designed for the continuous growth of small volumes or bacteria.

One of the earlier design of chemostats had a cooling coil between the inner body and the outer jacket, so it was impossible to pack or use a holder, Figure 1. By using the flange method that I will describe later, these difficulties were overcome. Like quite a few glassblowing projects, I started with a chemostat with an inner body of 40 m/m diameter then came the usual requests: "Can you make it twice as big?" So this steadily increased up to 100 m/m inner diameter, but no matter what the inner dimensions were the method remained the same.

Method of Construction

Figure 2 shows the complete chemostat. This was achieved like so many other pieces of scientific glassware, in stages. The first stage is the inner body and this is shown in the following Figures 3 and 4. You will notice the method of sealing sintered discs in using a hand torch. I find this technique simple and effective up to 50 m/m diameter. After the sintered disc is sealed into the inner body, the various side arms are made using the technique of blowing through solid rod, this insures no pin holes. With the inner body finished the next stage is the making of the side condenser. The coil for this is wound on a mandrel by hand and then sealed into its outer jacket, Figures 5, 6, 7. The construction of the main body is achieved by flanging up the shank of the joint that will be used at the top of the chemostat. Figure 8.

The flange marked "A" is to be the same diameter as the main outer body "C". Next the inner body "B" is sealed to the flange and well run in by hand torch, Figures 9, 10, 11, 12. Now the outer body "C" is sealed to the edge of the flange, Figures 13, 14. The various side arms and outlets are made working from the seal end downwards, thus pre-heating the sintered disc and ensuring it will not crack, Figure 15.

The next stage is the top side ring seal through to the inner body, when this is completed the top hose connection is made, Figure 16. The third stage is to seal on the side condenser and with the strengthening rod sealed in place this makes for easier working, Figures 17, 18, 19. With the various side arms and outlets completed and working downwards towards the sintered disc the next step is to join the bottom hose connection on Figure 20 and lastly the side ring seal next to the sintered disc is completed. At this stage I put the chemostat into a hot annealing oven and anneal Figure 21.

After annealing, the last stage is completed and this is, a long capillary tube is sealed through at the top of the chemostat the length of the capillary tube extends down to about 10 m/m from the sintered disc. Figure 22. Using this method it is possible to construct chemostats with 100 m/m diameter sintered discs in complete safety as compared to the conventional method of packing which has to be removed out from the bottom and then the bottom closed off.

Although the techniques described are used in the fabrication of chemostats they could be applied to make ring seal types of glassware which cannot be packed or held from the inside, for example large liquid nitrogen traps, various types of diffusion pumps, skirted neck dewars Figure 23.

Acknowledgments

I would like to thank the University of Aston in Birmingham for the opportunity of presenting this paper and Dr. P. Gilbert now of Manchester University and Professor M. R. W. Brown of the Pharmacy Department, University of Aston in Birmingham for their help.

Figure 1

Figure 2

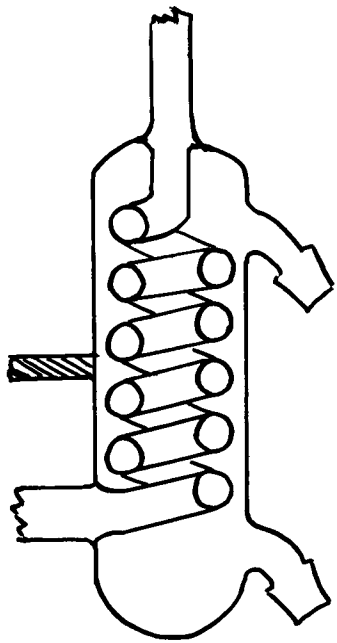
Figure 3

Figure 4

Figure 5

Figure 6

Figure 7



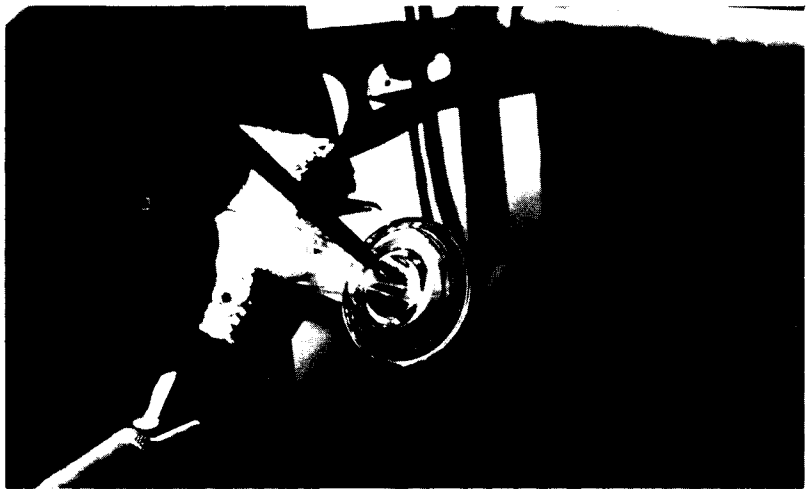
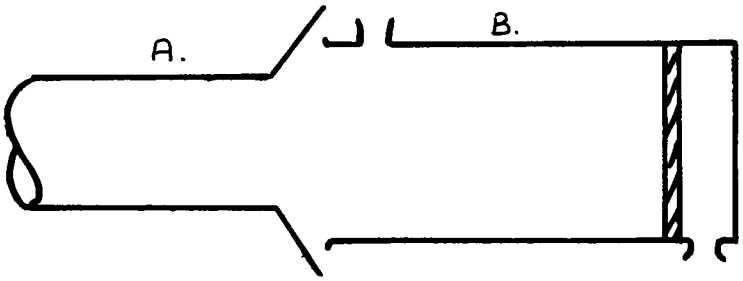


Figure 8

Figure 9



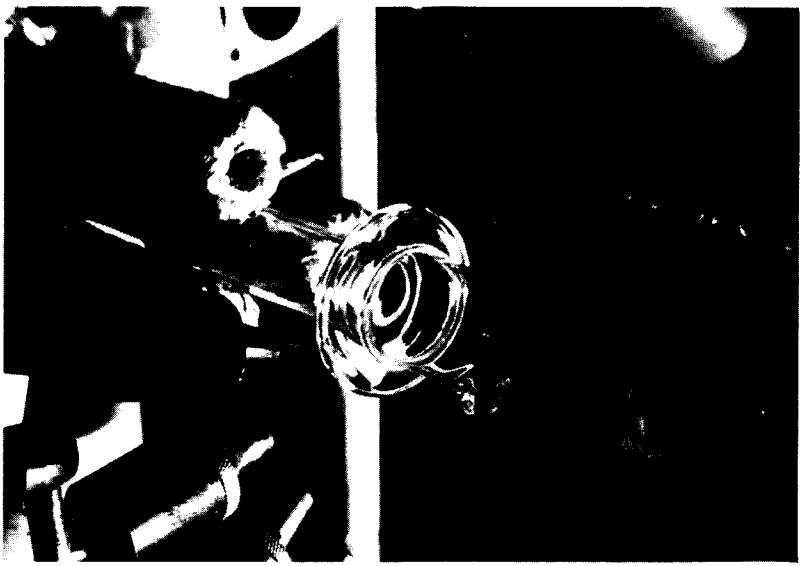


Figure 10

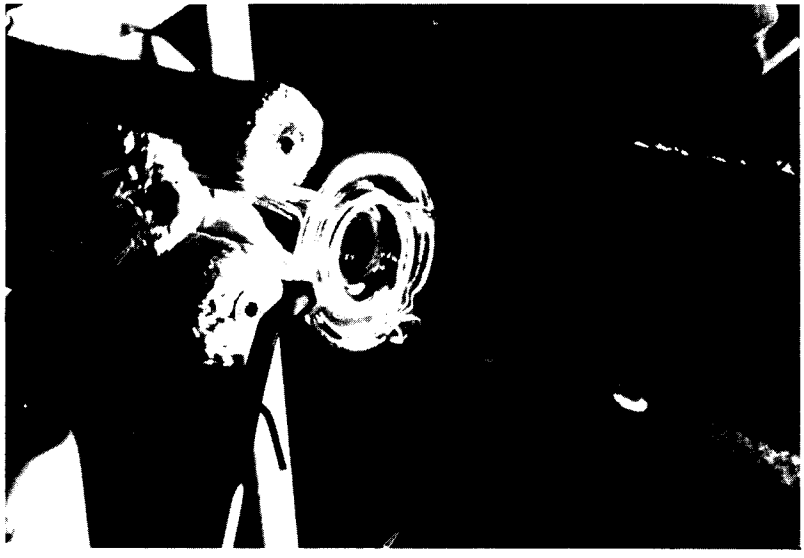


Figure 11

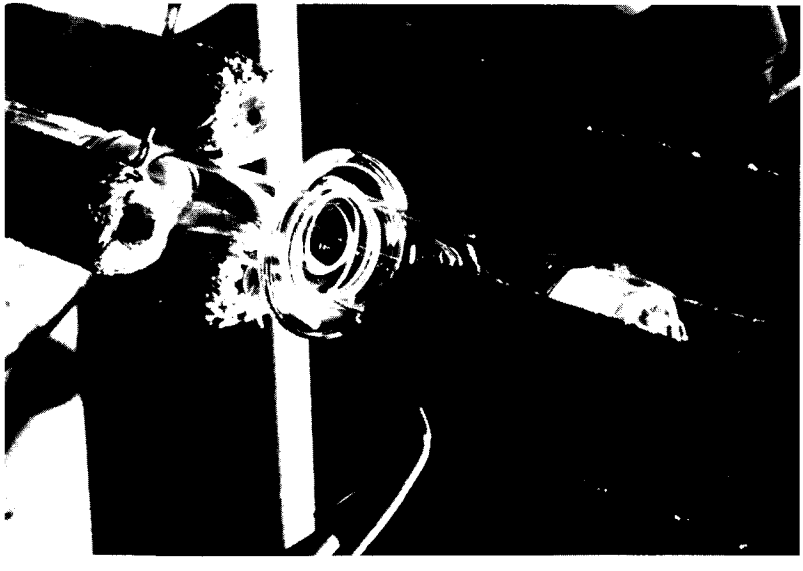


Figure 12

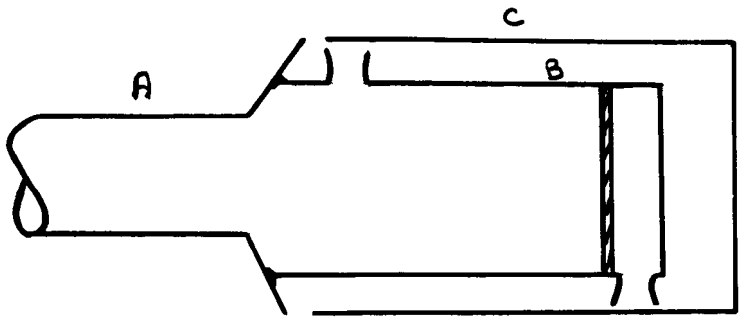


Figure 13

Figure 14

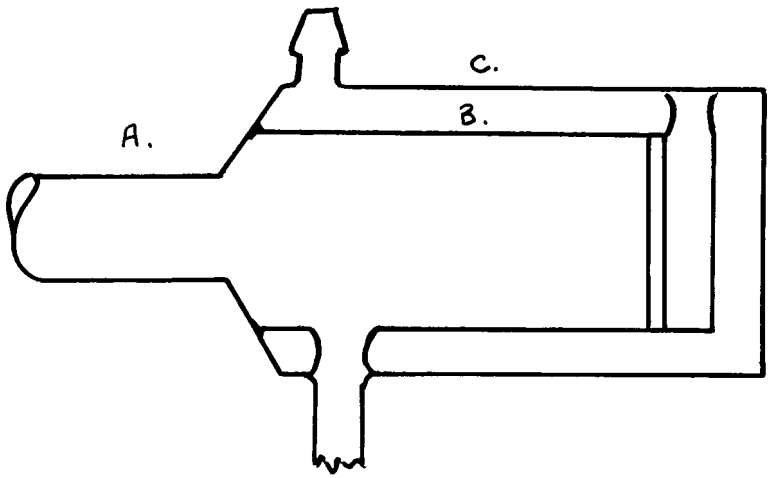


Figure 15

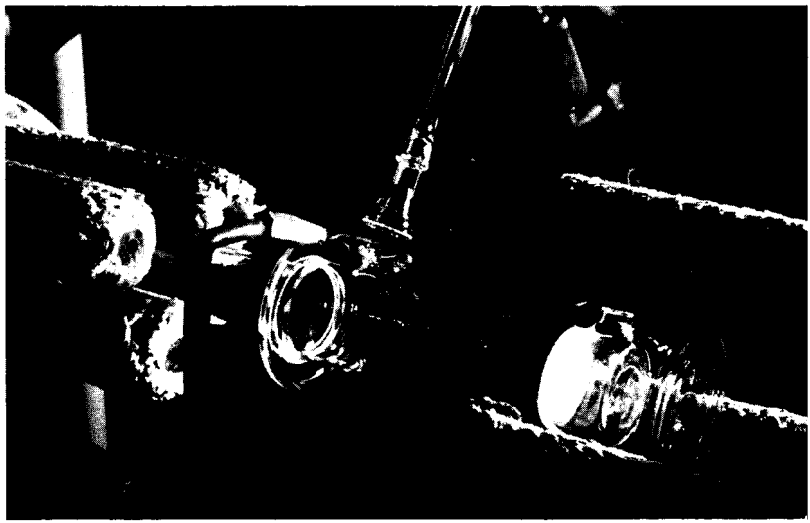


Figure 16

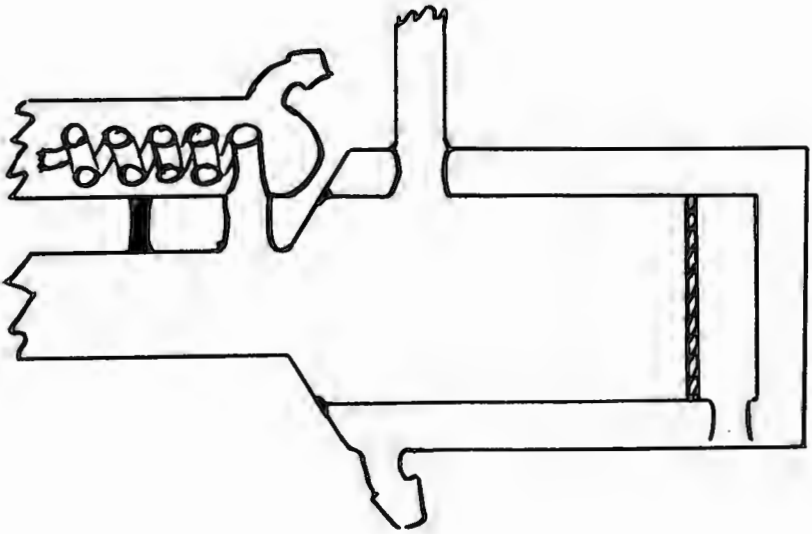


Figure 18

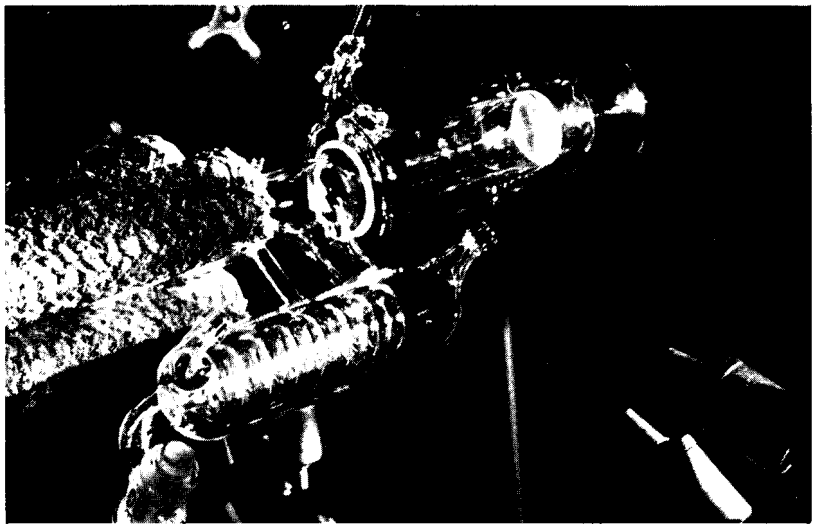


Figure 19

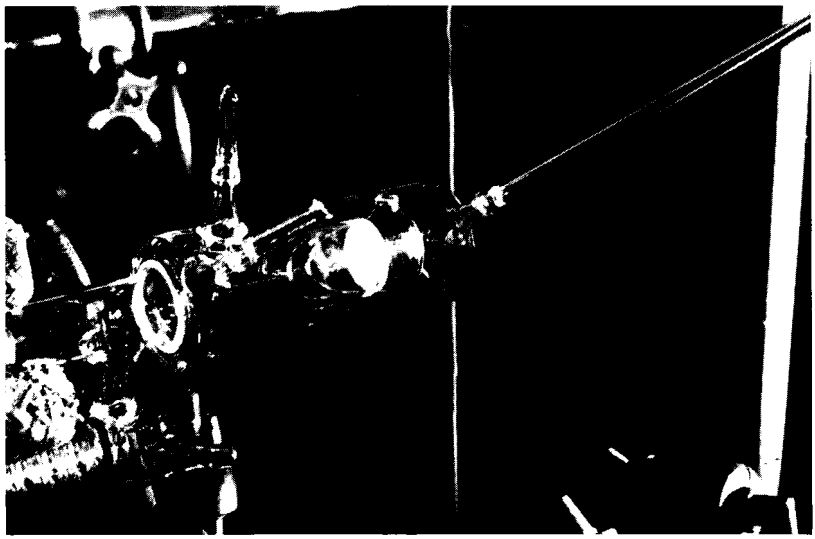


Figure 20

Figure 21

Figure 22

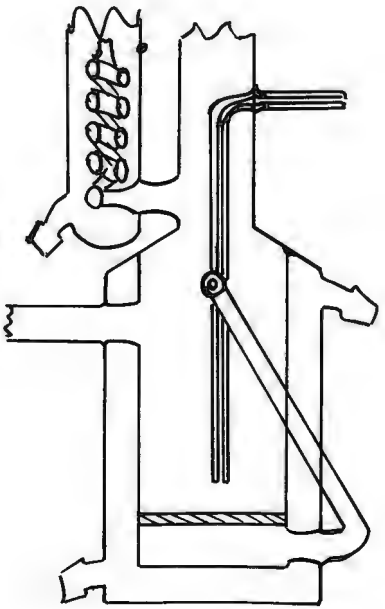


Figure 23

PRESSURE TESTS ON GLASS FLOWRATOR* TUBES

George Batley
Manager of Glass Research
Lab-Crest Scientific Glass Company

Introduction:

As the name implies a Flowrator* tube has something to do with the flow of fluids. It is used in measurement of the **rate** of flow, and it is - in a broad sense of the word - of tubular shape, Flow-Rator-Tube!

This particular tube I am holding, and which is the subject of this paper, is typical of many Flowrator* tubes. It has been reformed from glass tubing to a precision internal geometry that incorporates a taper. The taper is an essential property for intended use. The tube also embodies flutes to provide guiding of a float, another essential component. Guiding allows free vertical travel of the float while limiting lateral movement that could introduce errors of measurement. There are transition areas at each end of the tube to permit assembly into a fixture and to accomodate necessary gasketing and hardware, which also retains the float within the metering cavity. And then there are permanent markings that include the metering scale, indentification logos and nomenclature, and a white backing that accentuates the scale and float position in use. The **complete** assembly is a flowmeter, in a form that allows fitting into a desired piping system.

The Flowrator* tube is a **precision** glass component of somewhat complex tubular configuration. In the process of fabrication it has undergone several manufacturing operations that include cutting, reforming, grinding, beveling, annealing, fire-polishing, calibration, decalling, partial tempering, and also various chemical treatments such as glass-prep, washing and degreasing. The final inspection involves a complete check-out of dimensional and calibration tolerances and accuracy, as well as a critical examination for visual flaws; but this is not enough.

Several industrial applications for glass flowmeters require the transport of fluid at pressures and temperatures considerably higher than atmospheric and/or ambient conditions. Like fittings and other components of a glass process pipeline, the flowmeter also is rated for

* *T.M. - Fischer & Porter Company*

intended usage. This particular tube carries a rating for operation up to 200 psig pressure and 200°F temperature for almost any fluid exclusive of four major categories; hydrogen fluoride (gaseous or aqueous), hot phosphoric acid, hot alkali of high pH concentration, and super-heated steam. Good chemical durability is realized through use of a borosilicate glass composition such as Corning's #7740, Kimble's KG-33, or Schott's "Duran". But to assure compliance with published ratings, some **physical** tests are requisite.

Routine physical tests are performed on each and every production lot of tubes. One such test involves internal hydrostatic pressurizing at room temperature for a short-time interval at double the tubes' rated pressure. This is performed on a statistical sampling plan basis in accordance with accepted industry standard procedures. On occasion some rather elaborate tests are performed under laboratory conditions, usually for special-purpose evaluation. It is one of these special programs that I wish to comment on at this time.

Special Test Programs:

From associated work being conducted in the Fischer & Porter Metallurgical Labs on brittle materials, the question arose as to whether recent fracture mechanics literature, on predictions for time-to-failure of brittle materials under load, could also be applied to Flowrater* tubes. In addition, increasing industrial requirements for applications at elevated temperatures had raised a question of whether rated temperature ranges could be extended without incorporating a derating factor, and if necessary, to what degree and in what form. And, thirdly, from product liability angles, how would any change, if proposed, affect current risk factors associated with product liability.

Since the fracture mechanics study was the principal consideration, and since any fracture analysis requires fractured samples, it was proposed that test specimens be incrementally loaded to burst pressure under controlled conditions of time, and of temperature. Since it was also presumed that relationships would be curvilinear rather than linear, it would be necessary to incorporate **at least three** different levels of temperature and of time. Sample size for each group was set at five to be consistent with routine test procedure but each sample of five was replicated at least three times for statistical evaluation purposes; all in all a minimum of fifteen samples each for nine major test

groups, or 135 total tubes. Some spin-off tests were anticipated along the way so it was felt that total tubes required would approach the 200 quantity, and this automatically determined the particular item to be used; there were sufficient quantities of this tube in shelf stock to meet the requirement.

Two of the time increments were more or less predetermined; our version of the instantaneous test with time-to-failure under load being in the 10-20 sec. range, and the 5-minute hold period, or 300 sec. range, used for regular tests.

Repeat of these two increments would permit comparison with other tube sizes and types, for which we had an excellent background history. The third increment was arbitrarily chosen to exceed the 1000 sec. range usually considered a dividing line between short-time and long-time loading; we chose 1200 sec. or 20 minutes. These three test increments would permit a plot over three of the eleven decades published for static fatigue relationships with time under load for borosilicate glass. It would also be in the more sensitive portion of the range.

Two of the temperature increments were also pretty much predetermined; ambient temperatures and the 200°F upper limit for the current tube ratings. The third temperature level was somewhat arbitrarily chosen, but it was determined as best compromise of some limitations imposed by other physical conditions. We could use actual burst pressure data for comparison with previous experimental history, but for the proposed fracture mechanics study, all data had to be converted to stresses being imposed at the time of failure. To preclude any significant variations of thermal stress, it was desired that test samples be at 95% thermal equilibrium throughout during test. We also wanted to use fluids consistent with past tests for comparison purposes. To obtain fracture fragments in a form that would permit the fracture mechanics study, this meant eliminating any generation of gas at the temperatures and times involved, to prevent associated secondary fragmentation that such gas formation would produce upon failure. We had wanted to use 350°F as our third tempereature increment but compromised at 300°F to meet all other conditions desired.

All testing was performed manually using a hand-pump system similar to that employed in automotive garages, but modified to provide some additional desirable features. For the instantaneous tests,

the sample was pumped until failure occurred. This was a form of **disrupted** continuous loading in that additional load was applied only during the down-stroke of the pumping motion and check-valved to provide a pressure hold during up-stroke. Pumping was moderately fast and deliberate to prevent a bouncing motion of the gauge needle, yet sufficient to reach burst pressure in the 10-20 sec. desired interval. Lazy-hand gauges were used to give a direct reading at failure.

For time tests the procedure was modified. In these cases the sample was pressurized at a specific gauge pressure and held for the allotted time, then depressurized and retested at another increment 50 psig higher, this procedure being repeated until failure occured. In contrast to the disrupted continuous loading used for instantaneous testing, time tests were a step-increment loading procedure. Although unnecessary for such tests, the same lazy-hand gauges were used to minimize gauge factors, and gauges were checked for calibration at start and finish of each test segment.

The test fluids were various mixtures of ethylene glycol, in water. Such mixtures are used in routine testing to provide lubrication of the pump's internal working mechanisms and also to permit easy cleaning of test survivors. For ambient temperature tests, performed on a bench with a convenient sink at hand, the test sample was filled with water, coupled to the piping system and bled off to minimize air pockets. Fluid in contact with the test sample was essentially 100% water. For oven tests a different set of conditions existed and fluids were modified accordingly. To allow for volume expansion during heat-ups, the system had to be left open so that the additional volume could be run back into a reservoir and not add load pressure until ready for actual test. At 200°F temperatures the fluid in contact with test sample was a 50% mixture of glycol in water, but at 300°F temperatures it was 100% glycol. The test fixture was a modified standard flowmeter housing with provision for a bleed-off by-pass and for quick-connect and quick-disconnect to the pump system. Normal gasketing was used with exception of high-temperature tests in which cases a special o-ring had to be substituted because of temperature limitations of the standard part. Oven tests included a dummy set-up with thermocouples to provide a complete temperature history of each test case, monitored by a multi-point recording potentiometer. To permit safe handling in dissembly of the broken tubes, each test sample was given a spiral winding of clear "Scotch" tape prior to test. This

winding also facilitated fracture analysis studies immeasurably.

Test Results:

This particular test program extended over an eighteen-month period with two months in preliminary planning and preparation stages, fourteen months for collecting actual test data, and then two months for evaluation wrap-up and reporting phases.

Over 200 tubes were tested, although formal parts of the program included only about 160. Spin-off tests included evaluation effects of such details as the spiral wrapping, the three different test fluids and the special o-ring. Some tests were also run at 350°F and some with a cooking oil as test fluid. Plans for artificially abrading some test groups were discarded for reasons that will be discussed later. It was even found necessary to determine the location in the system to install the pressure gauge for minimal extraneous effects.

So, when it was all over, what had we learned? Before trying to answer this question, I would like to refer to the classic definition of a camel; or of an elephant, if you prefer. It has been applied to either, "A camel is a horse that was put together by a committee". We had a lot of interesting data, and a lot of interested groups, and so we got a lot of interesting interpretations. If your committee is large enough, you can even get a version of a horse that's a cross between an aardvark and a unicorn. And, this is as it should be. We are not a non-profitmaking organization; at least not willingly or willfully. Our research is more of the applied type rather than pure research, and various groups are usually more interested in their own particular areas and applications in these areas than they are toward the complete program.

We had a large block of test data of a nature that did not allow analytical evaluations. The "Scotch-tape" windings preserved fragmentation patterns in a one-piece form that was almost like a complete reconstruction. This permitted thorough fracture analysis studies with both halves of the primary break origin easily obtainable and in a form that was ideal for the principal fracture mechanics study. Break origins for the most part were various types of superficial surface damage inherent to manufacture of a "tooled" product. Of course, everyone has their own interpretation of "superficial". We are no exception, but our version is quite stringent in this respect.

From an engineering viewpoint, test data indicated that the pressure rating which had been established for this tube was an excellent and accurate representation of tube performance. A little on the conservative side, perhaps, but from a product liability viewpoint this is a desirable feature.

From Manufacturing Control and Quality Assurance perspective, data indicated that procedures were adequate and under reasonable control. There's always room for improvement and such areas were pin-pointed, however. On the basis of burst pressure data, there was no indication of temperature degradation over the range under study; if anything, a slight improvement with increased temperature. But there were some anomolies, as is usually the case when dealing with glass.

Original planning stages had allowed for some test groups to be artificially treated by abrasion. This is a ploy often used to obtain data with less scatter, or dispersion, and it does simulate a degree of service usage in a manner of speaking. The original plans had been predicated upon opinions built up over the years that tubes "always break from outside surface origin". This was a decidedly false premise; for the complete program we actually had more breakage from inside surface origin than for outside surface, a ratio of 56:44. Since there was no simple way to abrade the inside surfaces uniformly, this phase of the program was dropped. Even so, the data dispersion, although wider than we would have liked, was within expected parameters for an off-the-shelf condition of a manufactured product such as this one.

Discussion:

Any evaluation for **static fatigue** relationships with **time under load** was almost exactly as predicted from published literature covering this subject. These factors were unquestionable and undebatable. There was even a suggestion that some of the anomolies connected with **temperature** relationships included a time factor also, as a sort of correlated interaction instead of two completely independent variables.

To reach the 95% thermal equilibrium desired for oven tests required a 3-hr. heat-up period, during which the inside surface of the tube was in contact with test fluid and also for which, as measured by the lag in heating rate between inside vs outside surface temperatures,

tensile strains as great as 250 psi could have been introduced. It had been presumed that this would produce minimal and insignificant effect, but such was probably not the case.

One of the most interesting and most perplexing results observed was a tendency for break origins to shift from a perponderance of external surface to a preponderance of interval surface origin with both increasing temperature, but also with increasing time in contact with test fluid without the temperature variation. This may have been due to a reduced effect on outside surface from the higher temperature conditions, at least in part, but it also appeared to be correlated with weakening effects on the inside surface, an actual degradation of sorts.

The fracture mechanics analysis was very enlightening in this respect. Early calculations showed good correlation with published litterature, particularly that for work performed at the Nat'l Bureau of Standards. Then the correlation seemed to go haywire, until failures were separated into two completely differing classifications, inside surface and outside surface origins. And then the real breakthrough. Mirror patterns for failures originating in the transition area of the tube showed different detail than that originating anywhere outside this zone. At the transition area, diameter decreases about 10% in a short ½'' space at the same time wall increases at 50%. We had a configuration factor, which although slight was quite apparent. By further classifying for break origin within and outside the transition zone, all data correlated very well with published literature. Four different sets of data, four different sets of curves, but each set in good correlation with the expected results as established in published literature.

The fracture mechanics study indicated a degrading of strength with respect to temperature, but for unquestionable results would have necessitated extending the program into the 4th and probably the 5th time decade. We had been able to dovetail 20 minute hold increments into a normal daily routine, but there was no way that this could be performed for 3-hr. and 30-hr. test increments.

Further work appeared unwarranted at this time since the tests had also shown one other very enlightening condition. For over 200 tubes broken in the program, not a single tube had failed from inside surfact origin in the tempered portions of the sample. Tempering for the outlet end of the tube had originally been incorporated to

strengthen this section from "water-hammer" effects produced by sudden shut-off of appreciable flow. The compressive strains produced at the surfaces also seemed to inhibit whatever mechanics were involved in both the time and temperature relationships.

From a practical point of view, any desired result, either from less risk in product liability perspective or from tube derating as an extension of operating temperature ranges, appeared entirely feasible by the simple easy and inexpensive expedient of tempering both ends of the tube instead of only one.

Our first excursion into the realm of fracture mechanics had been very interesting and very educational. From early pioneering work in the 1920's by Griffiths, Wallner and Preston, to name a few of the countless thousands who have contributed since, to the real scientific approach of the 1970's, the study of the fracture process has steadily advanced until it is now a valuable industrial tool. The various factors that conspire to trigger off a chain reaction that we call "catastropic failure" leave unmistakable indentity behind on the fractured surfaces. As we learn more and more on how to interpret these details, we come closer and closer to realizing what glass really is.

We are quite confident that in future A.S.G.S. Symposia you are going to hear more and more about this relatively new science, "Fracture Mechanics".

PRINTING ON GLASS WITH PRECIOUS METALS

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Introduction

In your experience as glassblowers, you have probably come across times when you would like to make a permanent imprint of a design on glass. This may be in the form of a decorative design, or for a strictly utilitarian purpose, where a standard decal is not readily available. While there are many ways by which such printing can be done, most are not suitable to the average glassblower with limited facilities, or are only suited for mass production techniques. One such technique utilizing the silk screen process was described in *Fusion* in 1969.[1] The purpose of this paper is to detail for you a relatively simple photographic method by which these prints can be made.

While a photographic darkroom can be an aid, if you have access to one, it is not necessarily essential. This puts the method within the capabilities of everyone here. If a darkroom is available, it will make it possible for you to be more creative to your approach to using this method. However, in the interest of simplicity, I have prepared the paper keeping only to the basics for those of you who will not have ready access to a darkroom.

I became interested in a better method of printing on glass ten years ago, when I needed to designate that a piece of art work, which I had made, was to commemorate our 25th wedding anniversary. I did it, but I knew that there had to be an easier way to print on glass. Over the intervening years, through trial and error, and with the help of a Kodak publication,[2] I evolved the method, which is presented here in ten easy steps.

PHOTOPRINTING ON GLASS

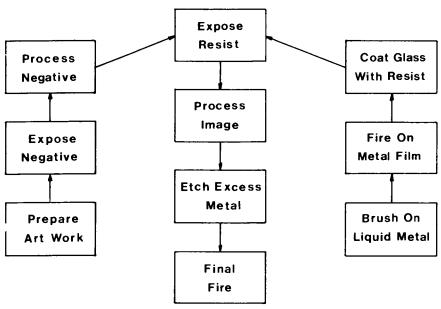


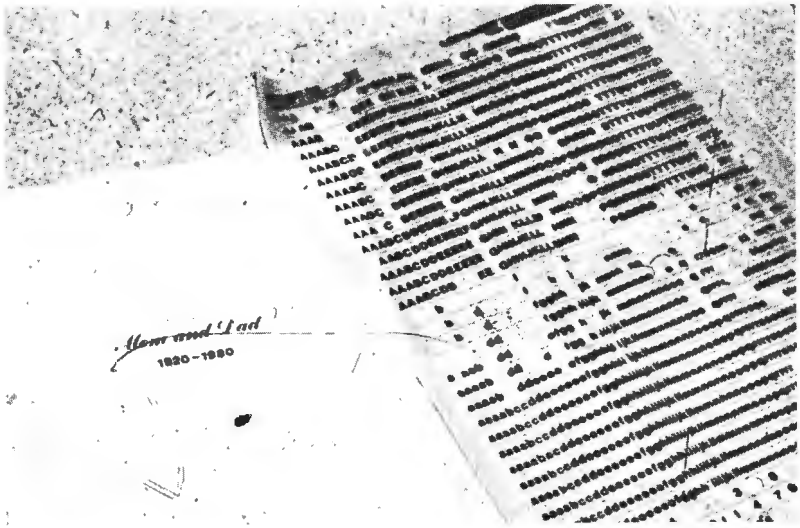
FIG 1

1. Prepare Art Work

The simplest method for making up the art work for your project is to use rub-off type dry transfer lettering. This is availble from art stores in many sizes and types of lettering and many decorative designs are also available. Since the easiest way of making a negative is to make a contact print directly from the art work, choose the same size of lettering that you want in the final application. The rub-off letters are applied to a transparent film, which has been taped to a ruled background to give proper alignment. If several lines are to be made, with each one centered over the next, the lines can be made separately, cut apart and taped to a glass plate, centering each as you go. This eliminates the chore of figuring the center of each line.

Alternatively, the art work can be made larger than needed and then reduced to the appropriate size by means of a copy camera, or an enlarger used backwards as a camera, if you have one available. The advantage of this method is that one size of lettering can be used for all your art work, reducing it as necessary, instead of buying many sizes of lettering sheets.

FIG 2



2. Expose Negative

A good high contrast negative has to be prepared and a high con-

trast film is needed to give sharp blacks and whites. I use Kodalith sheet film, cut to the appropriate size as needed. This film has a distinct advantage in that it can be handled much the same as photographic paper, that is, under a safelight in a darkened room, so that you can see what you are doing throughout the whole process.

If you are making a contact print, cut a piece of film to fit your art work from the sheet, place it emulsion side up, place the art work upside down on the film and weight it down with a glass plate. If a contact copy frame is available, it can also be used instead.

The negative is now exposed. I use my electronic flash unit from my 35mm camera to expose it, using one flash at six feet. If you don't have an electronic flash, you can use the flip-flash on a 110 or 126 camera. One flash at 12 feet will do the job.

3. Process Negative

I have found that Kodak D-8 developer produces an intense contrast negative, although it is not usually recommended for this film. A one quart stock solution is made up according to instructions and diluted one part to four parts water for use. Mix only enough for immediate use, as the mixture does not keep.

Development time will depend on the exposure, but its progress can be watched. For the exposure example I gave, the time will be about 45 seconds. When the reverse side of the film shows the image in clear black and white, the film is placed in a conventional stop bath for a few seconds and then is fixed in a standard fixer solution for the time recommended by the manufacturer, usually about five minutes. All these supplies mentioned are obtained from your local photographic supplier. The negative is then washed for at least ten minutes in running water and air dried in a vertical position to avoid streaking.

As you can see, this process is very short and if a good clear, high contrast negative is not achieved, it can be easily repeated.

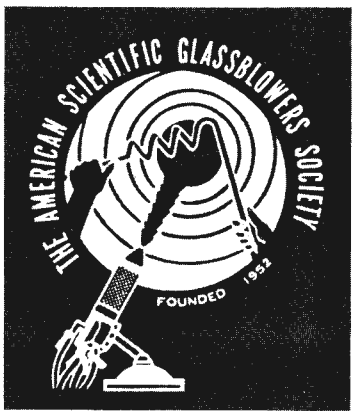


FIG 3

4. Brush on Liquid Metal

There are a number of precious metal formulations available, depending on methods of application and substrate material used. I have found that for printing with platinum, Hanovia Liquid Bright Platinum #05 is the best choice and for gold, Hanovia Liquid Bright Gold #4813 is good.[3] Both of these are composed of the precious metals salts dissolved in an organic essence made with oil of lavender. In addition, certain base metals are added to provide proper fluxing to bond to the glass. The fluxes are not identical for the platinum and the gold, and consequently a slight difference in adhesion is apparent. If abrasion will be a problem, choose the platinum, as it bonds slightly more securely to the glass.

The glass should be clean and dry. Mark off the area to be printed. I use a Sharpie pen for this purpose, as it burns off clean in the firing later. Using a small camel's hair brush, coat the entire area marked off with the liquid gold or platinum. Do not apply too heavy a coat, as in firing it may leave a hazy rather than a bright film. A proper film will be a light amber color, with just enough applied to allow the coating to level out evenly.

FIG 4

It might seem a bit wasteful to use so much excess film, when only a small part will apear in the final print. However, when you take into account the fact that it takes about two grams of the liquid to cover

one square foot, the cost breaks down to about five cents per square inch at today's precious metal prices.

The film may be air dried for about an hour, or dried more rapidly at about 65°C (150°F).

5. Firing on the Metal Film

Until you are familiar with the process, it might be wise to consider double firing of the ware--that is, first firing to a low temperature, about 250C (800°F) and then continuing with the photoprinting process. This permits easy correction after the print has been entirely completed, as the film has not been tightly bonded and the areas needing touch-up can be scraped off mechanically. A second firing to mature the image then finishes the process.

Alternatively, the film may be fired to fix it permanently and any correction necessary can still be done, as will be pointed out later.

In the firing, the ware is placed in a cold oven and brought up to temperature in about one hour. Sufficient ventilation should be provided in the oven up to 400°C (750°F) to allow for complete burnout of the organic binder in the film. Unless the volume of the oven is large, it may be necessary to leave the oven door open slightly. When the temperature reaches 400°C, the door can be closed and the firing completed. The table below gives the firing temperatures recommended for various glasses.

Maturing Temperature Table

	°C	°F
Soda Lime	600	1100
Borosilicate	675	1250
Fused Silica	725	1350

It is important that the ware be soaked at the proper temperature for one half hour to assure proper bonding to the glass.

6. Coat Glass with Resist

FIG 5

The metal film is now coated with a photo resistant material. The resist used is KTFR, Kodak Thin Film Resist. This material is specially formulated for certain metals and glass and is available in one quart sizes from Kodak Graphic Art suppliers. Your local camera supply store can probably point you to the nearest supplier. As supplied, the resist is quite thick, and should be thinned with KMER thinner in a 50-50 proportion. Since the solution settles on standing, it should be shaken to mix thoroughly before application. Allow the bubbles to dissipate, as they will form pin holes on the film.

The resist is flow coated over the metal film by pouring slowly from the container so as to cover the entire metal film. This should be done in a dimly lit place. I work in a darkened room, with only the light from the hallway to illuminate the piece as I coat it. The light from a yellow anti-bug light can also be used, if it is at a distance from your work place. A tray or beaker to catch the excess should be placed under the piece and this excess material can be returned to the original container. When it is fully coated, the piece should be allowed to drain in a vertical position.

It is desirable to allow the piece to dry long enough for all solvent to evaporate, preferably over night. Drying can be hastened by allowing the resist to dry to the touch, about ten minutes and then oven drying at a temperature not to exceed 65°C (150°F) for about twenty minutes. If oven drying is used, the piece should be allowed to

cool to room temperature before exposure of the resist.

7. Expose the Resist

The next step is to expose the resist. If the piece to be printed is cylindrical in shape, the negative can be positioned in place over the metal film in a dimly lit room and held securely in place by taping it to the glass with electrician's black vinyl tape. It can be lifted easily to reposition, if necessary, and it masks off areas beyond the negative, where metal film will have to be removed. If the piece is flat, the negative can be held down to the piece with a quartz plate, which will pass the ultraviolet light used to expose the resist.

Since a cylindrical form will not print well around the curved surface, it will have to be masked off in small sections of about ¾ inch, again using the electrician's tape, and exposed a section at a time.

Exposure is made using a Sylvania Sun Lamp bulb, with the exposure time being one minute and the lamp face eight inches from the surface of the glass piece being printed. The lamp must be warmed up for at least one minute before beginning the exposure, since it does not produce much UV at first. You will probably wish to put the lamp in

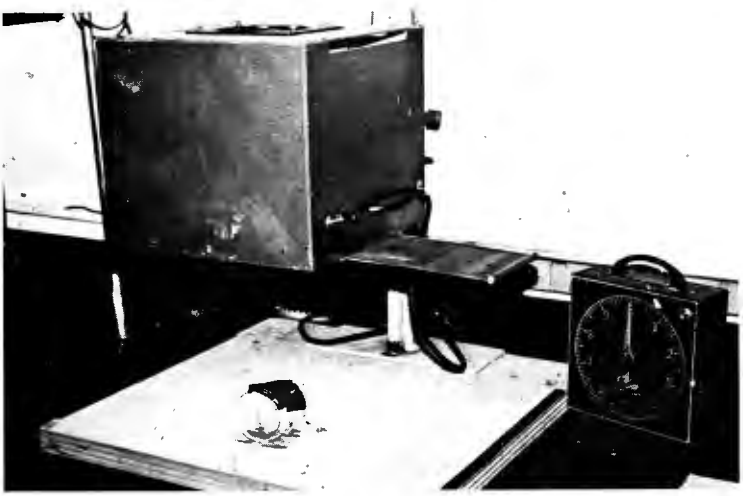


FIG 6

some kind of enclosure to shield the piece and your eyes from the light. A slide shutter will help when making the multiple exposures needed on a cylindrical piece. Do not shut off the lamp between these exposures, as it has to cool down to room temperature before relighting it.

8. Process the Resist

After removing the negative, the resist is developed in a dimly lit room by immersing it in Stoddard's solvent for two minutes, without agitation. This solvent is widely used by the dry cleaning industry, so you should be able to get it from a local dry cleaner. After removal from the solvent, the image should be sprayed gently with xylene from a wash bottle over the entire surface. This serves to clean the residual resist film from the image. Be gentle! Too strong a spray can loosen the exposed image as well. The developed piece is now washed with a gentle flow of water, as from an aerated faucet, to remove excess solvent. The piece is dried in air and the image can now be inspected.

FIG 7

Any pin holes or scratches on the negative will be apparent, as the resist will remain in these areas. It can be scraped off when dry to expose the bare metal film for subsequent removal. Any design that did not come out as clean as you would like can also be cleaned up at this time.

9. Etch Excess Metal

All the metal film which is not protected by the resist can now be removed with aqua regia. A sufficient amount of the acid is prepared, using one part nitric acid to three parts hydrochloric acid, to cover the metal film in a tray or beaker. The acid takes several minutes to begin

a strong reaction. Allow it to stand until it has a strong golden color, or even begins to bubble gently. Immerse the piece and the unprotected metal film should disappear almost at once. Rinse the piece with running water and distilled water to avoid water spotting. Air dry.

10. Finish Fire

If you have elected to use the double-firing method described earlier, you now have a second chance at final cleanup of the image. I usually use a sharp pointed scalpel to scrape away any excess metal, which is now very visible. The image is now fired to maturing temperature, held there for thirty minutes and allowed to cool in the oven. This bonds the metal firmly and also burns off the remaining resist.

If you fired the film completely the first time, the resist will still remain on the film after the acid etch. While it can be scrubbed off with solvent, it is preferable to refire the piece to the annealling temperature to burn off the remaining resist.

FIG 8

This completes the entire procedure. I have spelled it out in considerable detail, keeping in mind the many pitfalls which I encountered. If all has gone well for you to this point, you are ready to give the piece your final inspection and sit back and admire your handiwork. Good Luck!

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3. Available in 100 gram bottles from Hanovia Liquid Gold, Engelhard Minerals and Chemicals Corp., 1 West Central Ave., East Newark, NJ, 07029

pH ELECTRODE MANUFACTURE

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Introduction

History

Observed Phenomena
Early Investigators
Early pH Electrodes

Glass Formulations

Properties of Glasses for Ph
Improvements in Glass Formulations
Mechanism of Action
Temperature Dependance

Applications

Medical Field
Food Industry
Chemical Process Industry
Research
Environmental Field

Fabrication of Glass Electrodes

The glass electrode for measurement of pH has its early origins in other naturally occuring materials that were found to have an affect on the pH of water. Materials such as common clays and some fibers, which occur in nature, show ion exchange capacity, i.e., they are able to absorb ions of one species (from aqueous solution) in exchange for other ions contained in their matrics. Zeolites and wormwood were used as far back as Biblical times to purify water. In that way, they were precursors of our modern ion exchange columns - 'water softeners' -that are used today to treat and cleanse our water.

The electrochemical properties of glass were first studied by Lord Kelvin in the 1870's. In 1906 Cremer measured the electrical signal produced by a glass bulb when one side was exposed to an acidic solution. Haber and Klemsiewicz, 3 years later, made a careful study of the electrical potentials developed on glass surfaces and related them to change in pH. Haber concluded that the glass electrode was a "perfect hydrogen electrode".

Further investigations were made by scientists in the first half of the twentieth century into the electrode functions of different glass compositions. Some glasses containing sodium or zinc, acted partially as sodium and zinc electrodes respectively. All, however, showed a response to hydrogen ions. In other words, they were able to register a **pH value.**

Those who set out to determine the optimum glass composition for response to hydrogen ions came up with a soda-lime glass of a low resistance. The glass having the composition SiO_2 72%, Na_2O, 22%, and CaO 6% was chosen.

The composition of the soda-lime glass has been modified in many ways to improve performance of the pH glass membrane in many applications. The original soda-lime pH glass formulation has been adapted to overcome limitations encountered and to extend the applications of the pH glass membrane. One of the very first modifications to be made was the substitution of lithium oxide for the sodium oxide (soda) in the original pH glass formulation. The objectives of the modifications in the glass composition were to make the pH membrane more selective for hydrogen ions in the presence of other ions. Casium, Barium, and Lanthanum oxides have also been used for this purpose.

The mechanism by which the pH glass membrane responds to $H+$ ions to produce an electrical signal (potential) that can be read on

a meter, is the key to the formulation of pH glasses with particularly desirable properties. In particular applications it may be desirable to have a pH glass formulation that performs well at extremes of temperatures, extremes of pH, or in chemically or physically hostile environments.

The understanding of the mechanism of action of the pH glass membrane is at this time incomplete but several things are known. Experimentation has shown that an ion exchange takes place at the glass membrane surface, where the glass is in contact with an aqueous solution. In fact, each surface of the glass membrane, both inner and outer surfaces, participate in ion exchange. In the bulk of the glass membrane, the signal is transmitted by the mobility of charge carriers. That is, ions of positive charge migrate through the glass matrix in response to the difference in ion exchange activity at the two surfaces. Thus the pH glass membrane can be looked at as a sandwich of functional layers. The two outer layers function by ion exchange; the inner bulk layer functions by charge movement through the glass matrix.

In addition to the glass composition of the pH membrane, the size, shape, and design of pH electrodes are variable over wide limits to suit the probe optimally to the desired application. pH Electrodes are used in a wide variety of applications in the medical field, food industry, chemical process industry, in research, and in environmental studies.

pH Electrodes (and $pC0_2$ electrodes made from pH modules) are important to modern medical care. They measure the acid-base balance and $C0_2$ tension of the blood providing important information to the physician charged with maintaining optimum respiratory balance in his patient. pH Electrodes are used by food processors to monitor the acidity of foods. In most cases, a narrow range of pH assures quality of the product and provides protection against spoilage. In the fermentation and pharmaceutical industries control over pH is essential to the efficient operation of the fermentation process. In the chemical process industry, control over pH is very often the most important point for control of the desired chemical reactions. In environmental studies, pH is realized to be an important variable to monitor whether it be the measurement of pH of process effluents or the determination of pH of acid rain. pH Electrodes have been made to measure the acid-base balance in a great diversity of applications, and the size, shape, and design of pH probes have taken on that same diversity.

In fabricating pH electrodes to suit each application, the glassblower fabricating a pH probe must have a knowledge of all the materials available to him or her as well as knowledge of all the skills of working those

materials to optimum designs.

The early design of the pH glass electrode may well have had its origins in the R&D laboratories. They were rather bulky and fixed with a spherical membrane which often had a diameter in excess of one inch and were extremely fragile. The modern day trend is to down size the electrodes, since this will allow the user to make a pH determination with a small sample volume.

We will now discuss some techniques for the manufacture of pH glass electrodes. First, however, we have to determine the glass to be used for the stem or main body of the pH glass electrode. Since most pH membrane glasses presently available can be classified as being soft glass and having a coefficient of expansion range from 90×10^{-7} to 111×10^{-7}, we selected a Potash-Soda-Lead glass with a coefficient of expansion of 89.5×10^{-7} for the stem or body.

Basically, there are two types of pH glass electrodes: (A) the single pH glass electrode and (B) the combination pH glass electrode. The single pH glass electrode will always be used in conjunction with a reference electrode, the latter incorporates a reference electrode comprising a liquid junction which makes contact with the test solution through a porous junction **(see Figure 1).**

The single pH glass electrode is of a simple design, generally consisting of a glass stem with a membrane of pH glass, blown on one end. It is filled with an electrolyte and a silver half-cell, which communicates with the electrolyte and is sealed in at the opposite end.

Two of the methods used to put a glass membrane onto the stem or body of a glass pH electrode are application (1) direct from a melt of pH glass (2) from an ingot of pH glass which is softened by heating in the conventional way with a gas-oxygen flame.

The first method, direct from the melt is a simple one. Pieces of pH membrane glass are put into a crucible made from pure platinum, heated until the melt is formed; the temperature is kept at a constant level. The glassblower then has to insure that the air bubble activity in the melt has ceased. Several lengths of stem glass tubing have been prepared and the glassblower can now proceed with the next step. He or she brings one end of the stem tube very close and in a vertical position to the surface of the pH glass melt **(see Figure 2).** Keeping the stem glass in this position for a couple of seconds to allow the rim to be warmed up sufficiently before lowering it onto the surface of the melt to form an intimate contact with it. Care has to be taken to avoid an overlap of the membrane glass on the stem

tubing. Removing the stem tubing with the blob of pH membrane glass at the end and apply a pressure inside the tube to form the chosen membrane configuration (**see Figure 3**). However, this method has its limits, since one can only make two configurations of membranes, the spherical and domed.

The second method follows the conventional way of heating an ingot of pH membrane glass by means of an oxygen-gas flame. The scientific glassblower who will be asked to manufacture pH glass electrodes in this fashion has to acquaint him or herself to these rather soft glasses.

Furthermore, it is a necessity for the scientific glassblower to get the "feel" for the correct heating temperature and the length of time in which this heat is applied to the pH membrane glass. It has been demonstrated during practice runs that most of the pH membrane glasses are very sensitive to overheating and/or extended heating time cycles. However, once this "feel" is mastered, the manufacture of pH glass membranes in the easiest configuration, the spherical one, is a straight forward job.

Other configurations and the use of different types of pH membrane glasses pose other problems and difficulties for the scientific glassblower, which can only be mastered by practice.

It is my opinion that the order of difficulty in manufacturing the various configurations are as follows; the easiest being **spherical** and progressively more difficult are **domed, cylindrical, conical, needle, flat,** and **capillary.**

So far, I have been talking about single pH glass electrodes and earlier on, I mentioned that this type will always have to be used in conjunction with a reference electrode (**see Figure 1**). We will now discuss some techniques in manufacturing a combination electrode. The combination electrode is actually a single pH glass and a reference electrode built into one body. The liquid junction of the combination electrode is located in the outer body, close to the pH glass membrane, yet separated from it (**see Figure 4**).

The following is a graphic display of the different phases in the manufacture of a combination electrode (**see Figure 5**).

The manufacture of the capillary configuration in pH membrane glass is a rather delicate operation, due to its very small size. This is dictated by the need to measure very small quantities of a test solution. The capillary tubing is hand drawn to an outside diameter of 0.9mm and a wall thickness not to exceed 0.1mm and cut to the desired length. Capillary tubing in lead glass are made in the same fashion and with the same diameter. A given length of the lead glass capillary is then sealed-on to both ends of the pH

membrane glass capillary. This in turn is then built into the main body of what is to be an ultra-micro pH glass electrode.

Figure 6 is a diagram of a supporting tool used in the manufacturing of the ultra-micro pH glass capillary.

Conclusion:

This paper is intended to attempt to unravel some of the mystics surrounding the pH phenomena. It highlights the role in which the scientific glassblower takes part in the design and the manufacture of pH glass electrodes in general and also explores his or her ingenuity to engineer the supporting tools.

I realize this paper touches only a small aspect in the wide field of pH electrode manufacture.

I would like to take this opportunity to express my gratitude to my co-worker, Dr. Wayne Crowe, Manager and Director of our Research and Development facilities, who has given me so much guidance and support in putting this paper together. I also wish to thank the General Manager, Mr. Ihsan A. Haddad of Ingold Electrodes (USA) and Dr. W. Ingold of Switzerland for giving me the opportunity to devote the time to work on this paper.

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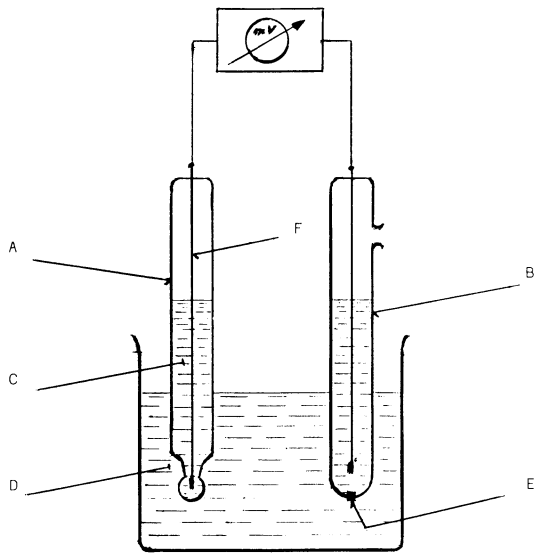


A - Single Glass pH Electrode

B - Reference Electrode

C - Internal Electrolyte

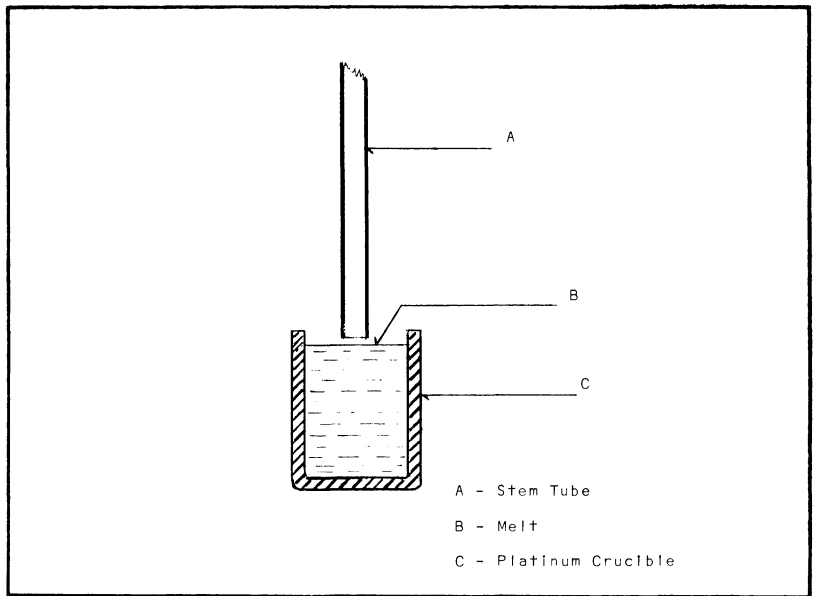
D - Test Sample Solution

E - Liquid Junction

F - Silver Half-Cell

FIGURE 1

FIGURE 2



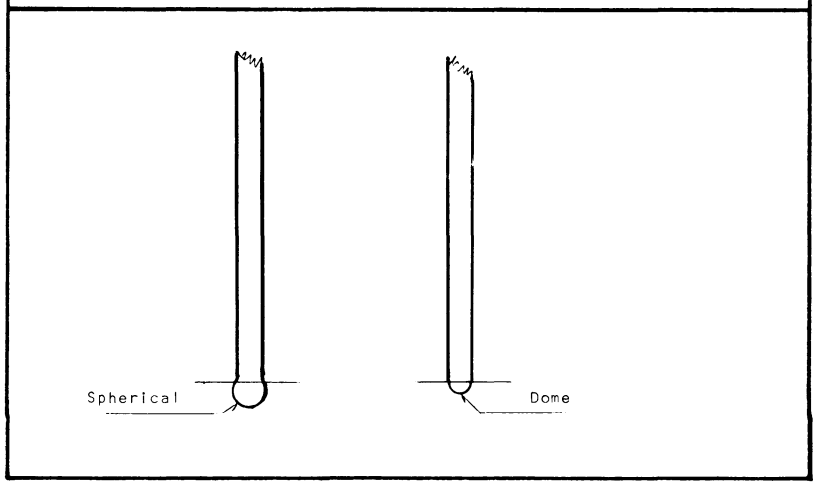
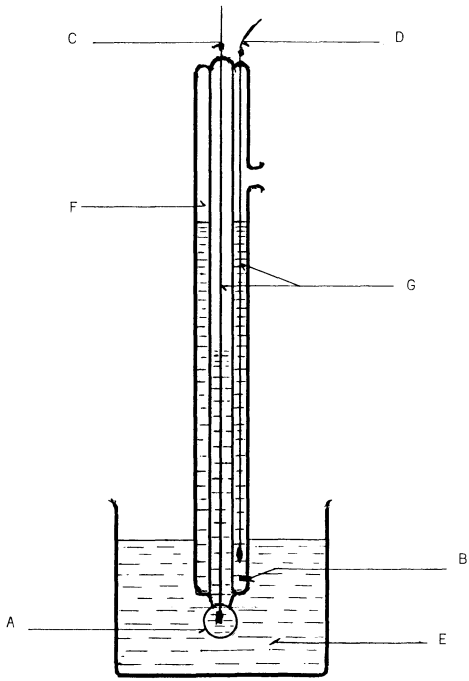


FIGURE 3



A - pH Glass Membrane

B - Liquid Junction

C - Connection to Half-Cell - glass Membrane electrolyte

D - Connection to Half-Cell - reference electrolyte

E - Test Solution

F - Reference Chamber

G - Silver Half-Cell

FIGURE 4

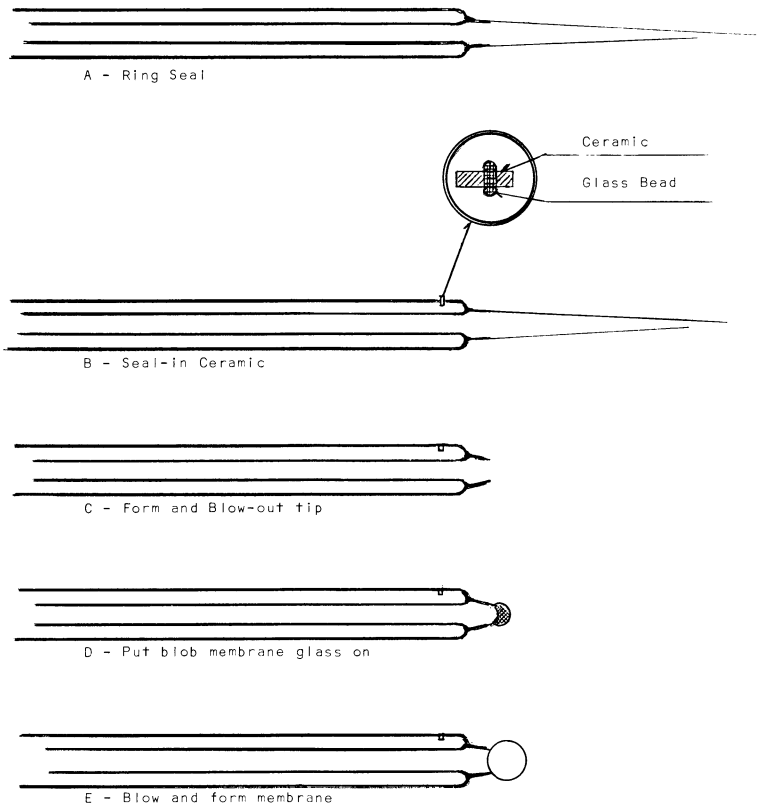

A - Ring Seal
Ceramic
Glass Bead
B - Seal-in Ceramic
C - Form and Blow-out tip
D - Put blob membrane glass on
E - Blow and form membrane

FIGURE 5

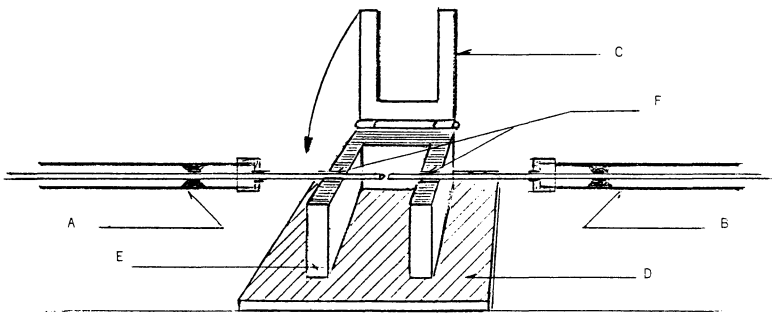


FIGURE 6

Capillary Sealing Support Block

a - Capillary guiding tube/Head Stock.

b - Capillary grinding tube/Tail Stock.

c - Hold-down plate.

d - Base plate for mounting on burner carrier.

e - Horse-shoe capillary support/High Density Carbon.

f - "V" groove.

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