PROCEEDINGS

The Thirtieth Symposium and Exhibition on the Art of Glassblowing

1985

THE AMERICAN SCIENTIFIC GLASSBLOWERS SOCIETY

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CONTENTS

1	Quartz EPR or ESR Flat Cells	*T.Anne Hostetter*
8	Setup, Fixturing and Holding of Glass while on the Cut-Off Machine	*Robert E.Platt II*
23	The Glassblower and His Lamp	*J.A.Frost*
29	Derivation of a Guideline Equation for Hot Cracking Tubing on the Lathe	*Don Lillie*
33	Chromatography	*William H.Shoup*
44	Computers without Computerese	*Gary Coyne*
53	Teflon Clear Coating on Glass, with or without Primer	*I.C.J.Dur*
55	A Concise History of Lampworking	*Frederick R.Birkhill, Jr.*
60	Fatigue Failure of Silica Based Glasses	*Connie Barry and Dr Patrick Nicholson*
67	Calibration of Hot Cathode Ionization Gauges	*Coe Gotoh*
75	Laser Welding of Quartz Brewster Windows	*Czeslaw Deminet*
80	The Effects of Black Light on Silvered and Unsilvered Surfaces	*Allan B. Brown*
83	Manufacturing Glass Containers	*Dr L.H.Gevaert*
84	Thermally Devitrifying Frits for Matching Fused Silica and ULE™ Titania Silica Glasses and Lightweight Mirror Applications	*Henry E.Hagy*
85	Novel IR Sealing	*Dr Josef Francel*
86	Exhibitors	
87	In Attendance	

Quartz EPR or ESR Flat Cells

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When I was first asked to make quartz EPR (electron paramagnetic resonance) or ESR (electron spin resonance) flat cells, the approach was one that many of you are familiar with; "well so and so made them for me at university X". Not to be outdone by so and so, but wondering how on earth one went about actually making one of these things, I did the only thing one could do under the circumstances, put it off as long as possible. Little did I realize at that time how very much I would enjoy this project.

The terms EPR and ESR are often used interchangeably. EPR is typically used by European and inorganic chemists, whereas the term ESR is usually used by organic chemists in North America. However, EPR is becoming the more common term.

With any new problem, some understanding of the subject gives me a sense of perspective and dimension which I can then use in my approach to solving construction or handling problems.

The quartz flat cell was not chosen by the chemist because he wished to aggravate the glassblower. He explains his reasoning thus. EPR spectroscopy is the study of chemical systems containing an unpaired electron in the presence of a large external magnetic field. The sample is held in a cavity (Figure 1) between the pole faces of a large magnet and irradiated with high frequency (microwave region) electromagnetic radiation. EPR spectroscopy yields structural information of the sample containing the unpaired electron by examining the environment of the electron and the manner in which other nuclei interact with the unpaired electron.

Prior to sample analysis the cavity (sample holder) must be properly matched to the impedance of the waveguide, which delivers the microwave radiation to the cavity. This procedure is termed "tuning" the cavity. It should be noted that the impedance of the cavity will be different when a sample is placed within the cavity. Samples that have a high dielectric constant such as water or acetonitrile will cause energy losses to occur in the cavity. For example, aqueous samples can cause losses so great that it is impossible to lock the frequency of the radiation to that of the sample cavity. This problem can be overcome to some extent through the appropriate design of the sample cell.

In order to optimize the design of the sample cell it will be helpful to examine the behaviour of the electromagnetic radiation in the sample cavity (Figure 2).

As the name implies, there are two components to the microwave electromagnetic radiation: magnetic and electric components. Figure 3 depicts the electric field contours (y-z plane) in a rectangular cavity (looking from the top down). Figure 4 depicts the magnetic field contours in the x-z plane (looking from the side).

For aqueous samples, maximum signal-to-noise and cavity coupling (tuning) is achieved by minimizing the amount of sample in the electric component of the microwave radiation (Figure 3) and maximizing the amount of sample in the magnetic portion (Figure 4) of the radiation in the sample cavity.

Quartz has a lower dielectric constant than pyrex and is more transparent to the electric field. Further it actually 'focuses' the magnetic field giving stronger signals. A quartz flat cell takes advantage of both these requirements. The sam-

Figure 1

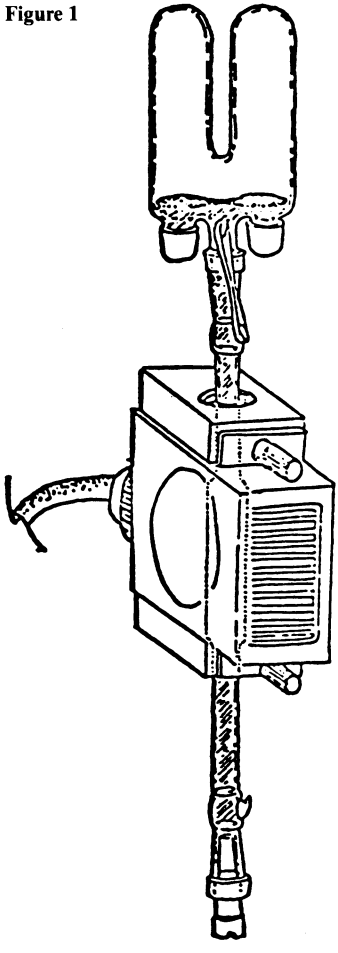


Figure 2
EPR cavity

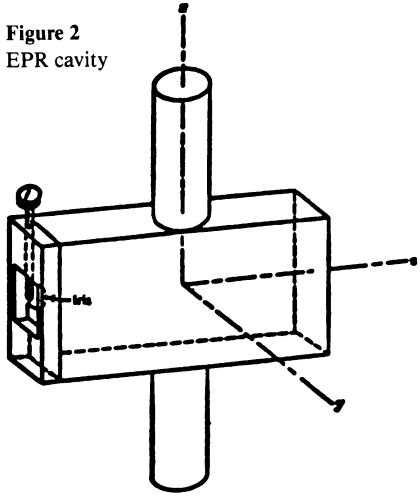


Figure 3
Electric field contours

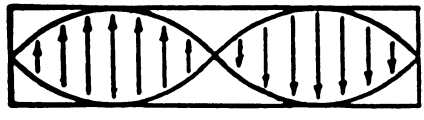
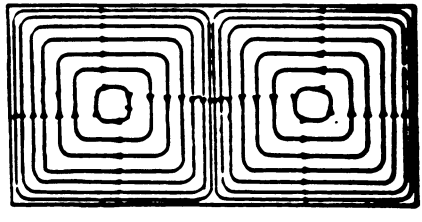


Figure 4
Magnetic field contours



ple resides in the region of highest density of the magnetic field, yet the electric component of the radiation does not interfere with the sample, allowing the sample to be easily tuned in the cavity and analyzed.

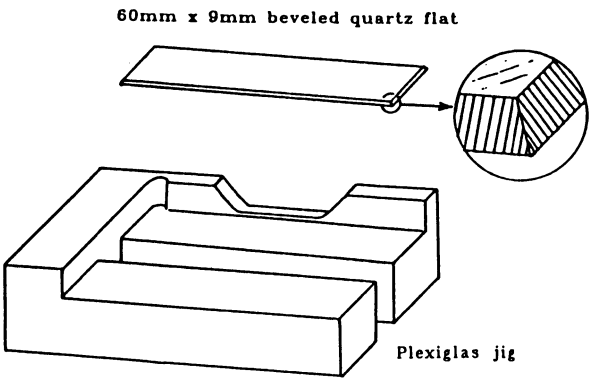
Having qualified the need for a quartz flat cell, it is time to move on to how we are going to make our contribution to the ongoing quest for answers to unsolved scientific riddles.

The main ingredient in the construction of a quartz flat cell is infinite patience. Other basic requirements are a plexiglass jig, 25 mm × 75 mm × 1 mm quartz slide, quartz rod, a number 7/25 and a number 5/20 quartz ground glass joint, 2 mm I.D. × 6 mm O.D. quartz capillary and some 7 mm O.D. quartz tubing. To prepare the quartz slide to the required dimensions, a new, soft silicon carbide blade is used. Most of the flame work is done with a Purox hand torch using natural gas and oxygen. The Sharp Flame hand torch is also used to remove some of the silicon dioxide which results when the joints are attached to the flat. Nitrogen is used for blowing to reduce the build up of silicon dioxide. The cleaning process uses diluted hydrofluoric acid (about 5%) and lots of tap and distilled water. This process will subsequently be referred to simply as wash. Cleaning in this manner is repeated often during the construction of the cell, but at no time is the quartz soaked in HF. Dipping only is usually sufficient. Dust is removed with mild soap if necessary (Ivory Liquid) and fingerprints with a good grade of acetone before the HF wash.

The width of the flat cell is determined by the size of the holder in the cavity. The finished width is preferably 10.25 mm with a maximum of 10.5 mm when built for the Varian Tm_{110}EPR cavity which is used with a Varian E 104 Century Series Spectrometer. This will leave about a 1.5 mm space in the cavity holder for inserting and removing the flat cell.

Using the jig (Figure 5) the slide is cut into two pieces, each to a length of 60 mm and a width of 9 mm. It is imperative that these two pieces are exactly the same size if you wish to avoid problems when sealing the sides of the flats

Figure 5



together later. Using the side of the saw, each piece is then bevelled to a 45° angle on all four edges. The flats are washed, dried with acetone, and set aside in a Kimwipe. Quartz rod for sealing the two flats together must be drawn. Ideally it should be 1.25 mm in diameter for 0.25 mm space between the flats. To prepare the 5/20 quartz joint, the tubing is cut off to within 1 cm of the joint. The joint is washed and then the capillary is joined on after which the assembly is washed again. The length from the top of the ground glass joint to the end of the capillary should be whatever length you require plus 0.5 cm. The same general procedure applies to the 7/25 joint but in this case 7 mm O.D. tubing is used rather than capillary tubing (Figure 6).

We now return to the two flats that have been set aside. Using tweezers, select one flat and attach a 3 mm O.D. quartz rod to one end. Lightly fire polish the bevelled edges and then attach a similar rod to the opposite end. Repeat this process for the second flat. Now that there is something to hold on to the flats can easily be handled. Clamp one rod of one of these pieces in a retort stand (Figure 7). Place two thicknesses of .005 in. asbestos paper on each end of the flat and lay the second flat on top. At the same time seal the two flats together

Figure 6

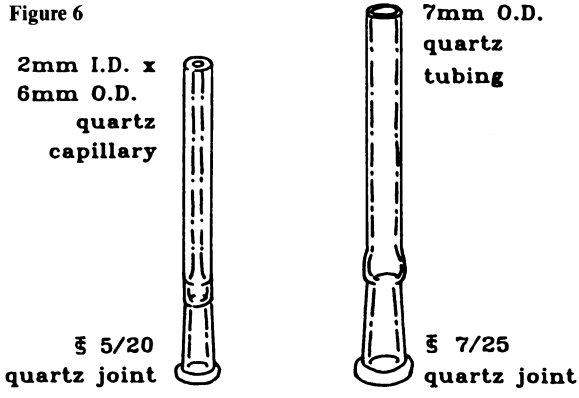
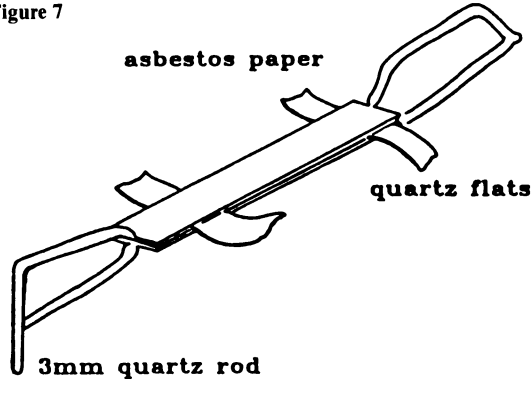


Figure 7



where the 3 mm rods meet at one end and square up the flats. Holding the centres of the flats together with tweezers, seal the 3 mm rods together at the other end. Still holding the centre together, return to the first end you sealed and heat it to remove the tension. Remove the tweezers and gently slide the asbestos back and forth between the flats. You will be able to feel any high or low spots. These are elimated by heating the ends of the rods and making the necessary adjustments.

When you think all looks well, slide the paper back and forth again. A light tension should give you the space you are looking for. Remove the paper and hold the flats up to the light looking through the space. This will be your final chance to gauge the spacing of the flats. Wash the assembly before proceeding to the next step.

Sealing the sides is not difficult but close attention to the procedure used will help to eliminate pin holes. Clamp the flats by the 3 mm rod in a retort stand. Looking down from the top so that the open edges are up, attach the predrawn 1.25 mm rod to one end (Figure 8) and lay the rod along the length. Now seal the rod to the other end. With a fairly hot flame, and using the tip of the cone, begin to heat the rod at one end. As it begins to melt, wipe the rod with a carbon round. Continue doing this to the end. This will flatten out the rod and push it into the space. Immediately return to the end you started at and holding the flame at a 45° angle to the vertical and a 45° angle to the perpendicular (Figure 9) start on one side and slowly move the flame forward. You can observe the sealing by watching for the disappearance of the reflected white light. Do the same on the opposite side. Remove the silicon dioxide deposit with the flame. Turn the flats 180° and repeat these steps.

Figure 8

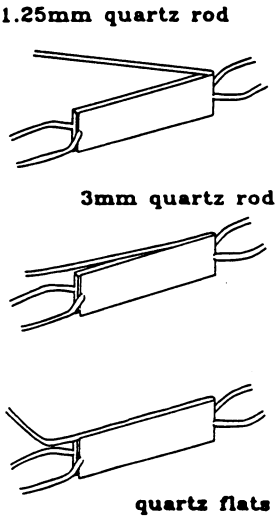
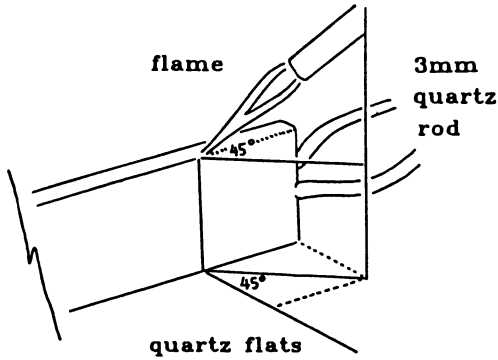


Figure 9



Turn the flats end up and remove the two 3 mm rods that held that end together. With a carbon flat, about 4 mm × 0.25 mm, heat one side of the flat at the open end and push it outward about 1 mm. Do the same for the other side.

Taking the prepared 7/25 quartz ground glass joint, shape the end of the tubing to fit the shape on the end on the flat (Figure 10) and tack it in place. Turn the unit end for end and follow the same procedure using the 5/20 joint but seal it on permanently rather than tacking it on. Remove the silicon dioxide from the area where the 5/20 joint was sealed. Returning to the 7/25, complete the seal and remove the bloom. Finally hooks are attached just below the joints making sure that they do not extend beyond our present boundaries (Figure 11).

Occasionally you will find that some 5/20 joints have a very heavy lip. Always check this with respect to the cavity opening and remove any excess by grinding it off on the saw before adding the 2 mm I.D. × 6 mm O.D. quartz capillary.

While it is well known that quartz must be worked in the cleanest possible

Figure 10

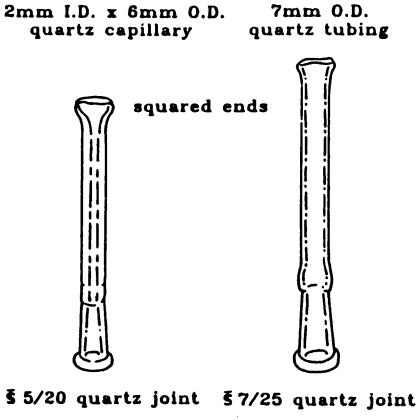


Figure 11
Quartz flat cell

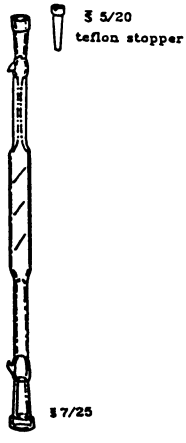
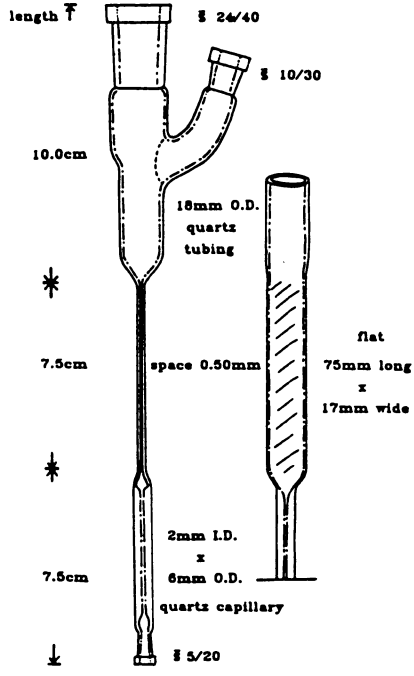


Figure 12
Quartz flat cell



conditions, I would caution you against too much use of HF. Use a soft flame when removing the silicon dioxide. When joining the ends to the flat, if you can avoid heating the "bejeezus" out of the quartz, you won't have to spend forever trying to remove the bloom. This is a little cell. Treat it gently and with respect.

Although this flat cell is the one most commonly used at the University of Guelph the need arose for a larger cell (Figure 12). With a larger Tm^{110} cavity and consequently a larger flat cell, the maximum signal-to-noise ratio could be realized. Figure 13 and 14 show some variations on the original flat cell.

While I have been making these cells for several years now, each one is a new challenge. Can this one be made just a little better than the last one? The challenge in the case of flat cells is definitely in the medium.

ACKNOWLEDGEMENTS

I would like to thank Dr Henry J. Stronks of Bruker Spectrospin (Canada) Ltd., Dr Henry Blount of the National Science Foundation in Washington, D.C., and Uwe Oehler, a graduate student in the department of Chemistry and Biochemistry at the University of Guelph, for their contribution in discussing the research applications of flat cells. I would like to express a further word of thanks to Uwe Oehler for preparing the illustrations.

Figure 13
Quartz flat cell

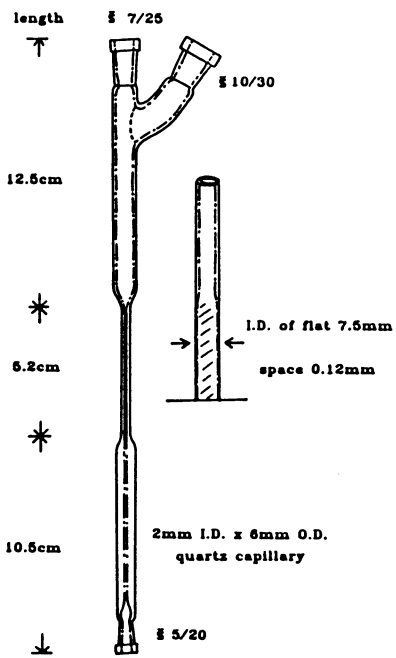
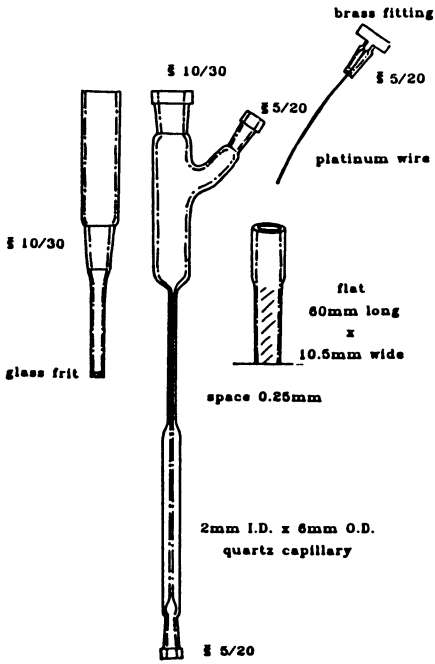


Figure 14
Quartz flat cell



Setup, Fixturing and Holding of Glass while on the Cut-Off Machine

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ABSTRACT

A two part paper, first covering basics of geometry involved in cutting and fundamentals of the cut-off machine. Testing procedures and simple equipment to evaluate potential accuracy. Testing by cutting, procedure to produce square cuts. Angle cutting, effect of geometry and setup, and helpful suggestions for more consistent angle cuts. Corrective procedures on machines with accuracy problems.

The second portion covers simple cutting problems that everyone encounters, and simple methods and materials to assist in producing accurate cuts with safety. Considerations in cutting peculiar to glass. Feed rates and power feed considerations. Handling large diameter work, long work, crooked work. Rotary cutting problems discussed. Protecting both work and machine. Special fixtures for production, with examples and explanation of design considerations.

INTRODUCTION

In the first part of this paper I will cover some basic subjects that are constantly coming under discussion. I will not be able to go into much depth on some of them, but will cover a lot of small items in order to make you aware of these problems, and help you see where certain difficulties are getting into your cutting jobs.

In the second part, I hope to give you some ideas on how to hold work more securely, and how to increase production with some special fixtures.I am not a glassblower, and most of my cutting is in metals, but over the years I have been asked to solve many weird cutting problems, and I hope that some of my ideas can help you.

BASICS TO GOOD SETUP

Before we can even think of special setups for tricky work, we should check our understanding of the basic geometry involved in cut-off machines of any kind, and what must be done to ensure that the most basic cut of all can be made, i.e. Cutting Square. If the machine will not cut square, even after all proper adjustments are made, there is no point in trying to make it cut accurate angles,

split tubing, or anything else. If it won't cut square, it really is not much good for anything.

Table travel must be square with the centerline of the spindle and the centerline of the spindle must be parallel with the surface of the table. These are the only factors, other than spindle condition, vibration, and general machine setup that are necessary for accurate cutting of the type that the Scientific Glassblower normally encounters. We are assuming that all work is being done on a single axis machine, i.e. a machine where movement in one direction only is controlled, and there never is motion in any other direction while the cut is taking place, as would occur in a grinding operation where the work is rotated, the work fed into the wheel, and the wheel moved along the work, all at the same time.

To understand the geometry involved it is sometimes best to look at an extreme case (Figure 1).

Some testing procedures are helpful: A test bar is a great aid in setups. Use it with a dial indicator mounted on table to test table travel squareness with spindle (Figure 2).

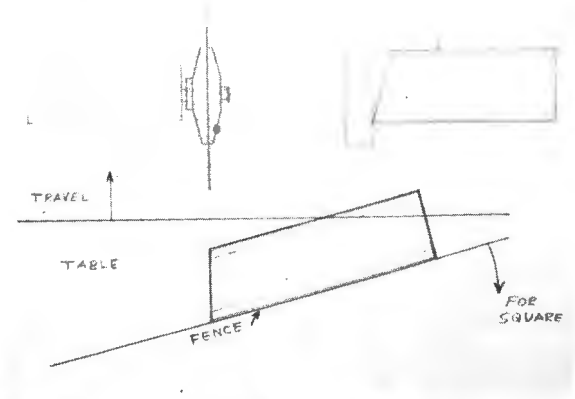


Figure 1

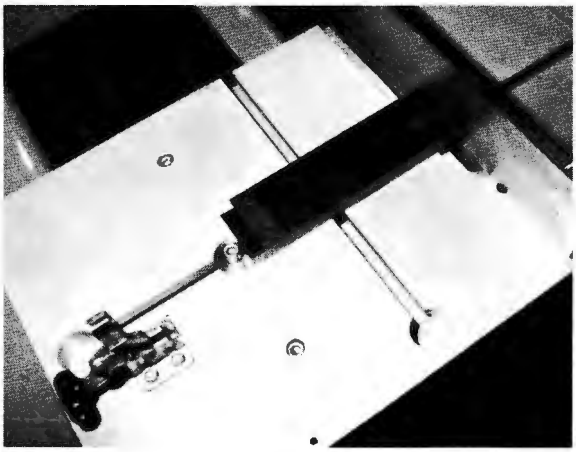


Figure 2

MISALIGNMENT CONDITIONS

Misalignment conditions are best discovered by use of a test bar (as in Figure 2) to test parallel of spindle centerline with table top. A setup with test bar and a precision square is made, and the test bar rotated through 45 degrees with the square clamped to the table surface. A feeler gauge is used between the test bar and the square to show if a wheel mounted on the spindle would be square with the table. Remember, what actually governs accuracy is the plane of rotation of the spindle bearings, and the closer you can come to actually determining where this plane is in relation to the table surface and the table travel, the better your understanding of the machines's potential accuracy.

Without going through the above procedure, you are just guessing at the cause of inaccurate cutting, and the use of a square against the side of a non-rotating wheel is a complete waste of time because the wheels are almost always slightly dished until stretched by the force of rotation at high speed. All diamond wheels have this "dish" built into them for this purpose, and hence cannot be trusted for squaring during setup.

Test cutting to prove square setup: This procedure will save a lot of time once it is mastered, and again the basic geometry involved should be studied so that the method is fully understood.

1 Pick a piece of tubing that is as straight as possible, about 1.5 in. to 2 in. diam.
2 Make a mark along the "top" of the tube with black, and a mark along the "bottom" with red.
3 With top up, trim end off, about ½ in. Discard this piece.
4 Rotate tube 180 degrees, with bottom up.
5 Make a cut about ¼ in. to ⅜ in wide.
6 Measure the width of this ring, checking all the way around. The maximum variation in the width dimension will be twice the error in squareness of the setup.
7 Without the rotation, you will cut rings that have parallel faces, but with ends that are not square with the centerline of the material (Figure 3).

CUTTING ANGLES

Cutting angles is complicated by several factors. The first is in the measuring of the angle to be cut. This is affected by type and size of wheel, dress of wheel, and by the cutting geometry. Second, a large wheel will slide along the work more readily than a smaller wheel, as will a thin wheel. A diamond wheel will slide less, as will a wheel with sharp corners. The larger the angle, (further from square) the harder it will be to hold tolerance on the angle.

Because of these factors, I recommend the use of plastic triangles, such as a draftsman uses for the approximate setting of angles. You will find that you will have to add or subtract from the initial setting, depending on these factors, but once you get a setup working properly, you can often cut a piece of material

to save for the next time you set up and run that same exact job, WITH THE EXACT SAME WHEEL.

Better cutting geometry can often be incorporated into the setup to aid in angle cutting, but this is not always possible because of the type of machine and the quantities involved. Large quantities can often justify special fixtures, or even a special machine to achieve the accuracy and/or costs desired (Figure 4, 5).

Figure 3

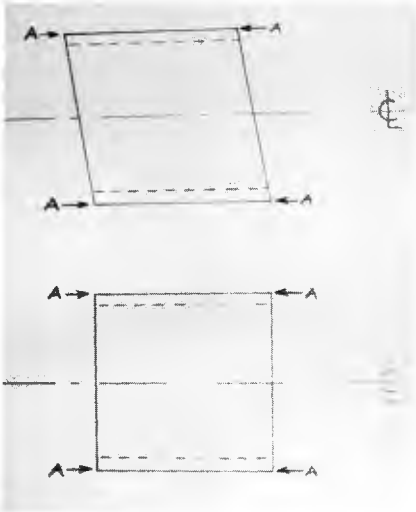


Figure 4

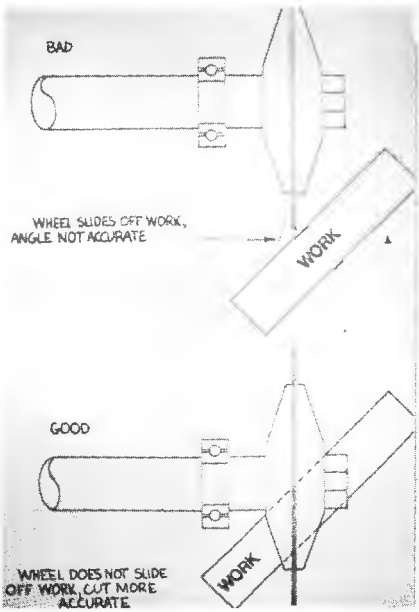
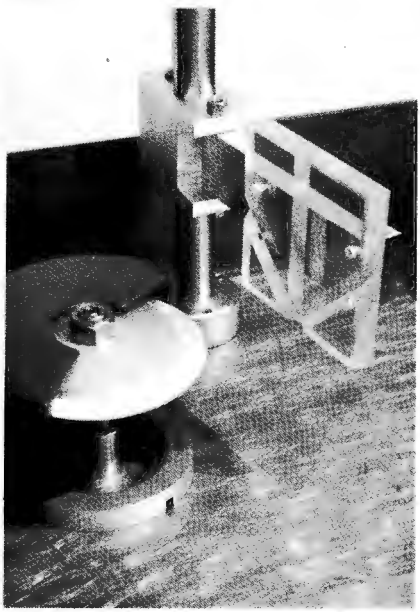


Figure 5



Guiding the wheel to start the cut straight without any sliding along the work is sometimes feasible. We often do this with a block of wood on single cuts, but have done it with solid tungsten carbide blocks mounted into the fixture itself. Air and water jets are used in other industries for this kind of guiding, but we have not had to go this far.

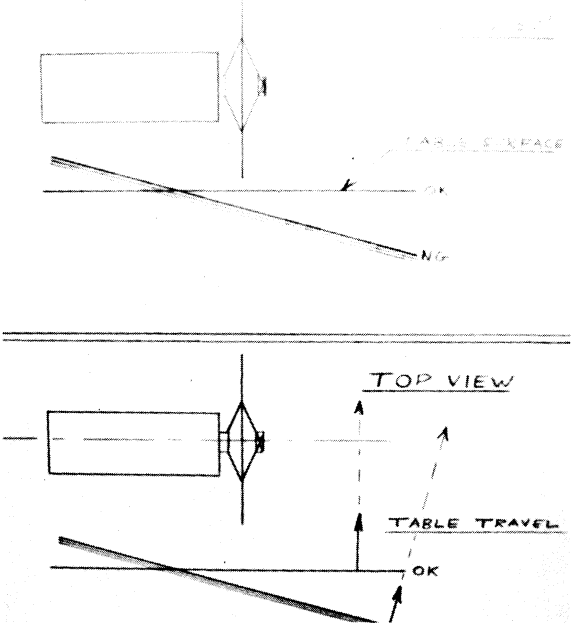
The break out chipping is almost always worse on angle cutting because one corner of the wheel comes through the work ahead of the other, and the work will try to move before the cut is completed. Secure holding of the workpiece, along with very slow cutting at this stage is the best insurance that this problem be kept to a minimum. Again, note that angle cutting will almost always wear one side of the wheel more than the other, and so cause a poor dress. Dress the wheel often when cutting angles.

A constant problem is that of work that is not straight, which if bad enough, will often cause the work to break because of unsupported weight or vibration. Some thin, low angle wedges cut from fine grain wood are very helpful to slip under or behind crooked work. In some cases adjustment for crooked work must be built into special fixtures.

CORRECTIVE PROCEDURES ON MACHINES WITH ACCURACY PROBLEMS

1 Level machine. Lightweight machines are very sensitive to being out of level. This condition can actually bend the machine causing binding and mis-alignment of the table (Figure 6).

Figure 6



2 Adjust alignment of table to spindle. Without proper instruments, this is generally a waste of time, but with a test bar as previously described, a good six inch machinist's square, and a 0.0001 or 0.0005 reading indicator much improvement can be made.
3 Shimming table. Some tables can get out of flat, and small shims placed under mounting screws can help. Check your table with a straight edge and feeler gauges. Remember also that very little glass is straight, so be realistic in your expectations as to how flat the table needs to be.
4 Regrind of table. This is often the least expensive way to improve accuracy. Dragging abrasive grit across the surface will eventually wear a gutter down the middle, so check for this condition with a straight edge, or, mount a sacrifice piece of material onto the table to lay the work against - wear this piece out instead of the table.
5 Rebuild of machine. This can often restore machine to better than new condition. Ask the people who built it.
6 Spindle bearings can be cause of trouble, particularly axial float. The wheel starts out OK, but then the whole spindle moves either left or right in the bearings due to the load of the cut, a floating motor drive shaft, heat build up, or poor dress on the wheel. New spindle bearings, or a change in the type of spindle bearing, can give accuracy a big boost (Figure 7).

FIXTURING - HOLDING GLASS WHILE CUTTING

The biggest problem in cutting glass and similar materials, such as quartz, some ceramics, etc. is that they cannot stand the high clamping forces that most metal and other materials will tolerate without failure. The best hold on glass seems to be with the most common clamps of all - your fingers. This is nothing new to glassblowers, and all successful clamping seems to at least try to mimic the action of hands and fingers. Probably the most commonly used material that comes closest to finger pressure is sponge rubber of some kind.

Common materials such as modeling clay, wax, epoxy cement, plaster of

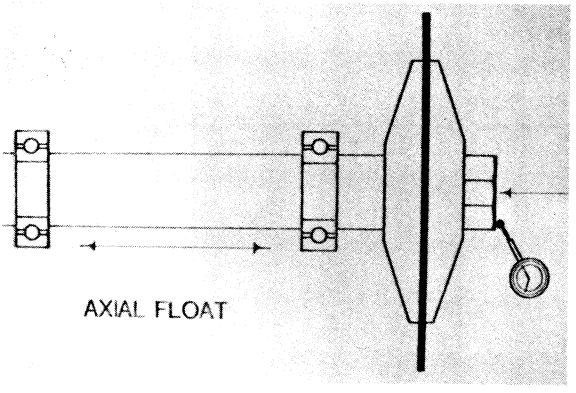


Figure 7

paris, tapes of various kinds, can also be used to hold work while cutting.

Velcro should be a very good material for flexible holding means, but we have not tried it for this purpose. We do know that it is reliable when used under wet, dirty conditions. We would suggest several straps to pull the work into secure contact whith the cutting fence, or down to the table.

We have used a resinous material called Crystalbond 509 to hold glass for cutting. No stress is imparted by this method, and a very secure hold is obtained. This material melts readily at a fairly low temperature, and can be washed off with acetone or MEK.

Soft rope can be used to obtain a gentle but firm hold on glass. In this fixture, used to hold a pressed glass part for gang cutting into 3 pieces, nylon rope has been glued into grooves cut into the fixture. When the fixture is closed and locked, the part will not move – yet the hold is quite gentle. The hold can be adjusted by either closing the fixture more or less, or using larger or smaller rope (Figures 8, 9, 10).

One of the most common problems is cutting an object of several diameters, such as a joint. Aside from the fact that the cut would be out of square if the

Figure 8

Figure 9

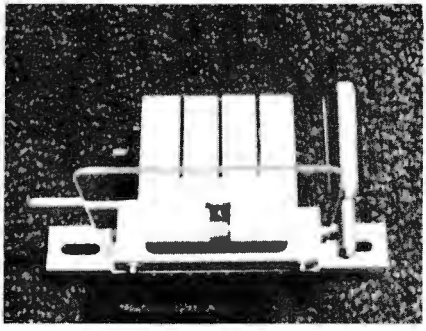


Figure 10

piece was merely laid on the table and cut, the piece will almost always break because the work is not supported under the cut. In metal cutting, the most common cause of wheel breakage is this kind of cut – no support under the cut. Cut the head off a bolt without support under the body of the bolt, and you will break the wheel. A "V" block of some kind is the cure. This can be of any material; wood does fine for many of these cuts. Always use the straightest grain pieces you can find as they will not warp under the wet conditions. We find that redwood or cypress are the best. We have recently been experimenting with a hot melt glue gun for making this type of fixture and this looks like a very promising method for building simple jigs very quickly. Make sure you use waterproof glue.

It is often necessary to protect either the machine, the fixture or the work from the severe abrasion encountered in cutting. We have found that UHMW (Ultra High Molecular Weight) polyethylene tape is very good for this purpose. It may be applied to any surface for protection and is fairly easily removed and replaced when it becomes worn. Better to wear out the tape than scratch the machine table, fence, or the work. "Hardcoating" on aluminum is another approach. This is a chemical conversion coating to the aluminum surface, creating a hardness approaching that of sapphire, a couple of thousandths thick.

CLAMPS

Common clamping setups are often used, however mostly for materials other than glass. We often use vise-grips in cutting metal, but their use in glass cutting would usually be limited to holding a fixture or guide block in place. Figure 11 shows how I am trying to hold a very tight tolerance on the length and squareness of cut on ¼ in. tungsten rod. With this ugly setup I held ± .002 in. without any problem. To do this you must clamp both sides of the cut.

In the past we have tried acrylic plastic for fence facings, and found that it

Figure 11

is not stable, in fact we have traced some very bad problems to the use of this material. It absorbs water rather rapidly and swells. If one side is protected and the other side exposed to water, the effect is bad warping. If you use plastic around the cut off machine, make sure of the water absorption characteristics before use. Bakelite or glass filled micarta is the best. PVC may also be worth looking into.

"V" SLOT FIXTURES

(Figure 12) This is a most common fixture but one that can help produce very close length tolerances on small glass tube cuts. In use, the work is laid in the "V" slot with the stop rod held against the end of the work. After positioning the work the stop rod is moved out of the way, so that the fingers can restrain the work piece from either spinning or moving as the wheel breaks through the rear wall. After the piece is removed the stop rod is again positioned. This setup also allows all grit, etc. to be washed out down the "V" track. We have also used this fixture with a small lever clamp to hold the work, the clamp faced with a small piece of polyurethane rubber. We have also bundle cut small diameter tubes (3 mm OD), 5 pieces at a time in this jig. The tubes were laid in a piece of aluminum angle, arranged in a perfect nesting pattern, and then the ends glued together with Crystalbond 509, this material forming a triangular plug holding all the tubes together. A little silicone release agent was used on the aluminum to keep the resin from sticking. The bundle is then transferred to the "V" slot fixture for cutting. Considerable down pressure must be used here to keep all 5 pieces of glass from moving during the cut, and you cannot hold as close a tolerance on lenth as with single piece cutting.

Figure 13 illustrates another version of a "V" slot fixture, one where gravity is used to feed a 12 in. stick of glass down to a stop. The piece is cut, then a lever is pressed that drops the cut piece and then allows the stock to feed down. The small tubes are additional water feeds to keep the internal workings of the fixture clean.

FANCY FIXTURES

Figure 14 illustrates a very simple, straightforward type of fixture - a purchased air operated toggle clamp, modified very slightly. The operator places the work under the clamping shoe and turns on the air. This rig would probably smash any glassware except solid rod that was placed in it. But the concept can be used to hold delicate material. You merely use very small cylinders and very low pressures. Because glass exhibits strain characteristics it would be best to keep the clamps as far away from the cut as possible, and to use the clamp to hold the glass in contact with some kind of friction material for grip, rather than holding by brute force.

There are many kinds of quick acting vises on the market, but most are too large for glassblower type work and would have to be cut down or modified for

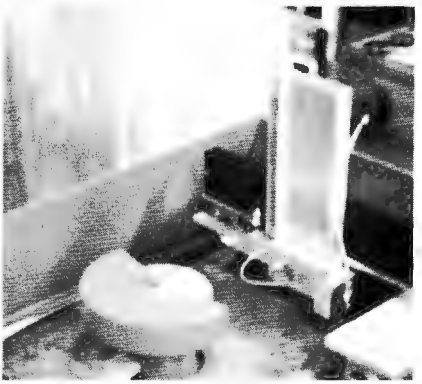
use. A good source of ideas on both tools and holding devices is in the lapidary field as those making jewelry and polishing gems are confronted with many of the same problems: holding and precisely cutting fragile materials (Figure 15).

POWER FEED

Anyone who has attempted to make a perfect cut on a piece of glass tubing very quickly learns that you must start the cut rather cautiously, then speed up a bit to get through the first wall or side. You can then again speed up cutting most of the tube, then slow down after touching the rear wall and then practically come to a stop as the cut is finished. The perfect power feed for glass tubing would have to do the same.

Figure 12

Figure 13



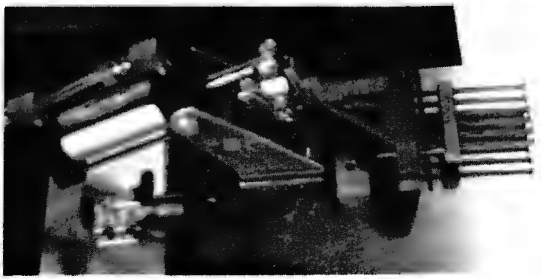


Figure 14

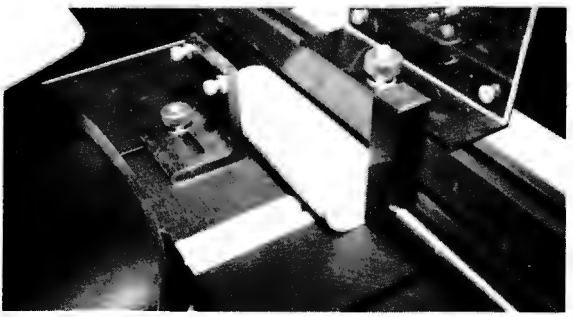


Figure 15

As the chart in Figure 16 shows, such a system would tend to be complicated in its control circuits for ONE size, and more complicated as the number of sizes, wall thicknesses, etc. was increased. About fifteen years ago I built such a controller using a mechanical approach to the problem, and while I could make it cut rather well, it would not out-perform an experienced operator. Today, with microprocessors performing this function, the possibilities look much better for a reliable power feed controller for glass cutting. The problem at this point is sensing both the position of the wheel in relation to the work, and also the pressure exerted upon the work. In cutting thin tubing you are often using pressures down in the ounces.

A compromise feed may be one that can be set for the most critical part of the cut – the final breakthrough. In use, the operator would cut in the usual way up to perhaps the last 10%, and then engage the feed. Once set, the feed would do this portion of the cut the same way each time.

Where speed or production is not the main consideration, a sensitive air/oil power feed can be used with good results. In this machine a combination power feed and a clamping fixture are used.

(Figures 17, 18) In this fixture, used to cut 20 Alumina tubes for high intensity lamps, clamping is done on both sides of the cut by means of aluminum bars faced with sponge rubber tape. The tubes rest in "V" blocks, and as the tubes are not very straight, the operator must rotate each tube to properly nest it into

Figure 16

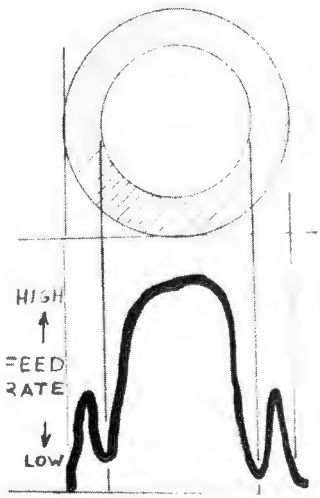


Figure 17

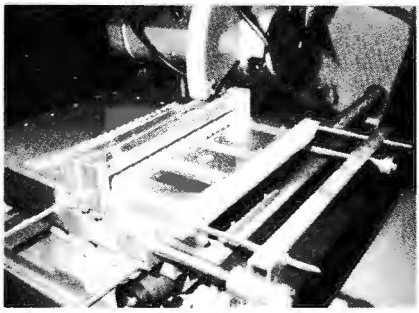


Figure 18

position. The cut is being made with a 10 in. Resin Bond diamond wheel. An air cylinder under the table, adjustable for quite low pressure, operates both clamping bars simultaneously. If we were to build this fixture today, we would consider the use of thin wall rubber or polyurethane tubing in place of the sponge rubber strip to hold the work. The rubber tubing would be mounted in such a way as to be inflatable, thereby giving an adjustment on both the softness of the hold, and also a size adjustment to handle other sizes of material.

Some of the problems to keep in mind when considering power feed on glass are: stress and strain, straightness, length, size/diameter. Any extraordinary feature in these areas are sure to defeat you in power feed cutting of your material.

Even though power feed may not be applicable in very many cases today, the key to good quality and accuracy does lie in good fixturing, even fixturing that includes power clamping. An operator can become fatigued, distracted, etc. but a small air clamp doesn't, and its clamping pressure is always the same, from the first cut in the morning to the last at quitting time. The clamp doesn't worry about cutting its fingers on the work, either!

If you feel that this discussion has been a bit negative concerning power feed cutting of glass, then I have been successful in getting my message across. However, I would encourage experimentation.

ROTARY CUTTING

Rotary cutting of glass is a bit different. We have done this both manually and under power.

Figure 19 shows a machine which was built originally to trim broken bottoms on bell jars and it has several advantages in this work. First, only a wheel big enough to get through the wall of the material is needed. Second, half the work can be done from inside the jar, cutting down on the wetness. Third, because

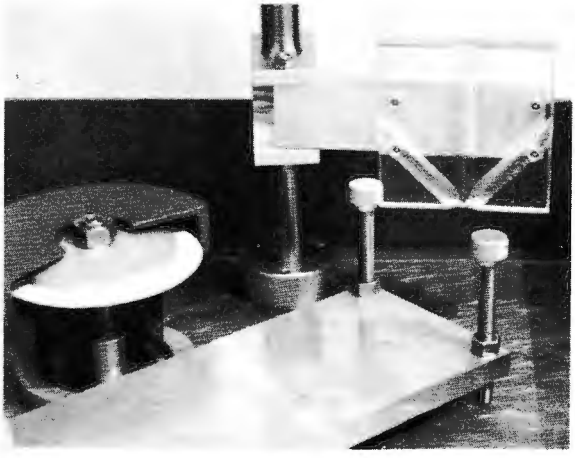


Figure 19

of the cutting geometry, you always come back to the starting point after going around the work with the wheel.

Another type of rotary cutting is shown on a silicon crystal (Figures 20, 21). Because the finish of the cut does not have to be in the exact center of the piece, slight out of roundness can be tolerated. The set up to cut glass tubing would have to be a bit better, but this fixture was built with 4 chucks rotating in syncronization, so you can grip both sides of the cut, thereby generating a perfectly square cut edge. Non-straight work would be the worst enemy with this fixture.

Figure 22 illustrates a fixture which is used to support long work of all kinds, the fixture moving with the cut-off machine table. The fixture is adjustable for squareness and level.

Figure 23 shows a type of fixture which has been equipped with rollers so that long, large diameter work can be held and rotated to effect a square, trim cut on the ends. Because this type of work is almost never straight, and will walk

Figure 20

Figure 21

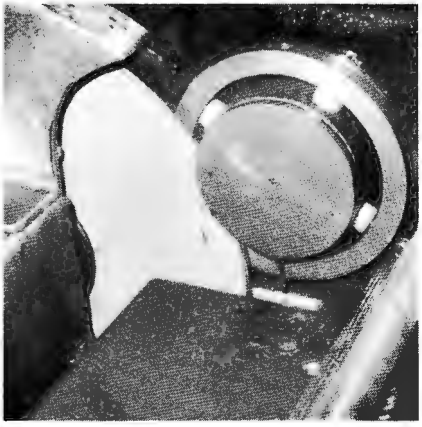


Figure 22

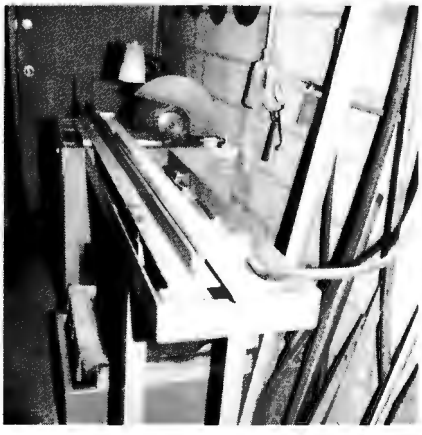


Figure 23

one way or the other during cutting, the cut is, more often than not, a spiral groove. To eliminate this problem a center must be held against a stop. The center can most easily be made by inserting stoppers in each end and running a length of metal rod through the stoppers, with suitable collars in place to keep the work from falling apart during the cut. Round and polish the end of the rod that will be held against the stop. As you cut, keep a light pressure on the assembly against a stop that is solidly attached to the cut-off machine table. Because you are only going through the wall of the tube you never reach the center rod while cutting. With this setup manual rotary cuts will always come out square.

OTHER TYPES OF MACHINES

At this point I would like to point out some general design concepts that apply to cutting machines. When really odd work is encountered it is sometimes best to consider what type of machine would be ideal for the job, and then design the fixture accordingly, or possibly consider building a special machine.

The first design (Type A) is the general purpose cut-off machine, where a moving table moves into the wheel on the approximate center of the wheel. The spindle is fixed in machine frame.

Advantages

a Because cutting force is generally downward vertically, hand holding of work is possible.

b Because table can be light in weight, or mounted on ball bearings, a fine feel of the cut is obtained.

c Solid, rigid design lends itself to accuracy.

Disadvantages

a Limited depth of cut equal to approximately one third of the wheel diameter.

b Long or heavy work must be supported on a moving table.

c Position of wheel in angle cutting leads to skidding of wheel and inaccurate angles.

In Type B Machines the work table moves generally below the wheel. The spindle has a vertical adjustment for depth of cut.

Advantages

a Length of cut only limited by travel of table and configuration of machine.

b Table can be mounted on very solid bed.

Disadvantages

a Cutting force tends to drag work under wheel, precluding hand held work for many jobs.

b Because of **a** above, feel of cut is reduced.

c Mounting of drive to spindle and vertical adjustment tends to limit horsepower available, and makes design complex and expensive.

On the Type C machine the work is clamped to the machine bed or the floor and the spindle travels on rails above the work. The entire rail assembly can be adjusted vertically for depth of cut.

Advantages

a Very long cuts can be made, limited only by length of rail the spindle rides on.
b Suitable for heavy work such as plate glass, slate and stone cutting.
c Motor and rails generally above dirt and debris of cutting.

Disadvantages

a Most expensive of all designs.
b No feel of cut at all, not suitable for light delicate work.
c Cutting force tends to either drag wheel into work or resist penetration into work, depending on direction of feed.

On the Type D machine the work is held or clamped to the table with the spindle mounted above on a swinging mount. Cutting is accomplished by forcing spindle and wheel down through work.

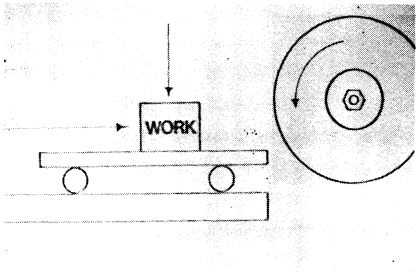
Advantages

a Simplest, cheapest design of cut-off machine.
b Long work easily handled as work does not move.
c Angle cuts made accurately as wheel has little tendency to skid on work.

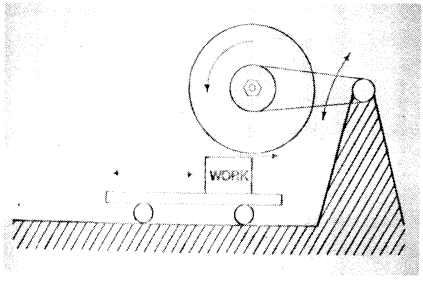
Disadvantage:

a Cutting force tends to drag work under wheel.
b Clamping of work absolutely necessary.
c Little feel to cut.

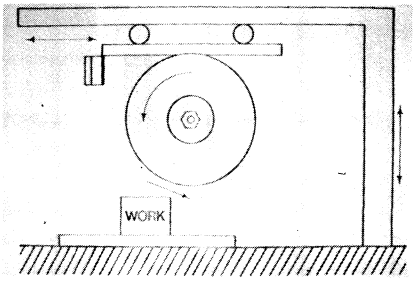
Type A



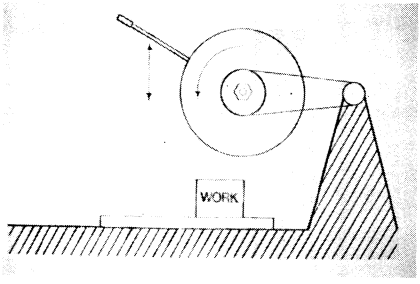
Type B



Type C



Type D



The Glassblower and His "Lamp"

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From early times alchemists, and later experimentors and scientists, have utilised glass vessels of many sizes, shapes and forms to help them in their experiments and researches. The earlier vessels, alembics, aludels or udells, retorts and other distillation equipment, were blown and formed directly from molten glass taken from the glass furnace by the glassblower. Later, more sophisticated and complex equipment and apparatus was, and still is, made or fabricated from glass tubing and rod by manipulation in a flame by the glassblower-lampworker. Italian supremacy in science in the 16th and 17th centuries owed a great deal to the availability of glass vessels and tubes from a flourishing glass industry which employed many craftsmen skilled in the production and manipulation of glass. These men were the heirs to the Roman tradition of glassmaking. Galileo's thermoscope, precursor to the thermometer and barometer was a product of their skill. Evangelista Torricelli said, 'We live submerged at the bottom of an ocean of air' and then he 'weighed' this air by using a glass tube closed at one end and filled with mercury. Closing the open end with a finger and inverting it under the surface of some mercury contained in a bowl, he noted that while the height of the column fell, the mercury did not all run out of the tube. He therefore deduced that the weight of the air exerting a pressure on the surface of the mercury in the bowl was supporting the column of mercury in the glass tube. This experiment, which is now so extremely well known, was only possible because of the availability of a glass tube and a clear glass at that.

Glass had been made at Murano as early as the 13th century, and by about 1300 the glassmakers were able to produce a colourless glass. Italian glass was also exported and Blaise Pascal, the French mathematician and philosopher, in 1646 used glass tubes from Italy in an experiment which was, in effect, a repeat of Torrecelli's, but using water instead of mercury; these tubes were 46 feet long! I speculate on their transport; they could hardly have been 46 feet in one piece, so they must have been much shorter and then joined together and made air-tight, but with what? Leather? There must have been then as now, quite a few experimental problems.

Robert Hooke, in 1691 described a deep sea thermometer made of glass. It is illustrated in a book published in 1726, and consists of an outer tube with a large bulb at one end. The other end of this tube is open, but inserted into this open end is a smaller tube which is a sliding fit. This smaller tube has a simple

valve contained within it so that when the whole apparatus is lowered into the sea the liquid in the bulb will contract and draw down this inner tube. On being raised from the sea, and the temperature then rising, the liquid will expand, the valve will lift leaving the inner tube static, so that the amount that has been drawn into the tube can be seen, and thus the temperature calculated. Although it is doubtful if this thermometer was ever constructed, it would seem reasonable to assume that Hooke had enough knowledge and confidence in the glassworkers of his time to know that they would be able to make it.

The term lampworker and the description of the burner as a lamp can be traced back at least to the 17th century when the flame used to soften the glass was produced by an oil lamp. These lamps burnt fuels such as colza oil, oil of turpentine or tallow, and would therefore produce a smokey luminous flame, and certainly in the case of tallow, a smelly one. The relatively high temperatures needed to soften the glass were obtained by blowing a stream of air across or through the flame, variations in the flame size or spread, and direction could be obtained by moving the whole lamp about in relation to the horizontal air jet. The steady and continuous blast of air for the jet was produced from a large pair of double acting bellows operated by the foot to leave the hands free for the manipulation of the glass. Bellows of this type were almost identical in design to those still used by some blacksmiths and farriers. Oil lamps were in use until the beginning of this century and foot operated bellows lingered on for another 30 years.

Modern lampworking still depends greatly on the manipulative skill of the craftsman-technician although a great deal of more sophisticated equipment is now available to supplement and extend this skill and therefore the craftsman's range and capabilities.

The 17th century lampworker used very few tools and equipment and relied almost entirely on his or her manipulative skill. In these days of the equal opportunity acts and women's liberation movements it is worth noting that in an edition of the book Ars Vitraria Experimentalis, by Johann Kunckel and published in 1756, there is clearly shown women carrying out lampworking. The fuel used in these lamps was oil or fat of animal or vegetable origin, and the restrained description of one early writer when he says that, 'the odour from the tallow lamp is regarded as unfragrant by many' can be appreciated. The lamps were made of metal or possibly clay, in some illustrations they resemble Roman lamps used for illumination. The shapes varied considerably, but it is a fundamental necessity that they should be broad and rather flat so that while they were stable on the bench top and would not overturn easily, at the same time they could be readily moved in relation to the air blast. A hole or groove at one end supported the wick and allowed it to project the requisite amount above the oil, or fat. Provision also had to be made for trimming and skewering the wick up or down. The lamp shown in Figure 1 is a very simple illustration of this type. A horseshoe shape stands with the top completely open and at the front there is a spoon-like nose with a depression like a gutter to support

the wick. The wick could be controlled by a flat piece of metal with a semi-circular depression cut into it. This piece of metal was just laid lightly on top of the wick and otherwise unsupported and by moving it about with tweezers, different sizes, heights and widths of flame could be obtained.

Other lamps were shaped roughly like the body of a violin with a hole or small tube at one end from which the wick projected, whilst others which were of a similar shape, had a large opening at one end. This would be packed with a mass of fibrous material, acting as a wick and this would give a much broader flame than a single wick. A lamp of this type is shown in Figure 2. The small metal cylinder projecting above the top of the lamp was possibly a flame guard, a shield to protect the worker from radiated heat and to obscure or screen off the luminous portion of the flame, to prevent eyestrain and at the same time to render the upper or non-luminous part of the flame more readily visible. Much later and up to or even after the advent of coal gas as a fuel, oil lamps were still in use and as one would expect from their long history the latter models achieved a high degree of sophistication. The one in Figure 3, although it is of the conventional horseshoe shape has a screw device for adjusting the wick.

Improvements took place gradually and Figure 4 shows a lamp which begins to look like a gaseous fuel burner but it was in fact intended to burn oil. It is of tubular construction and has a circular wick somewhat similar to some modern domestic oil heaters. The air blast passes through the centre of the lamp

Figure 1
Open top horseshoe shaped lamp

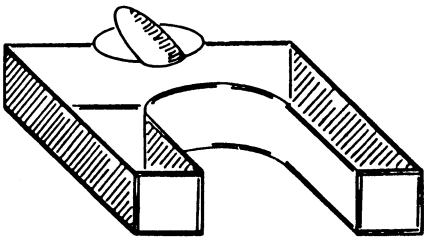


Figure 2
Closed top lamp with guard

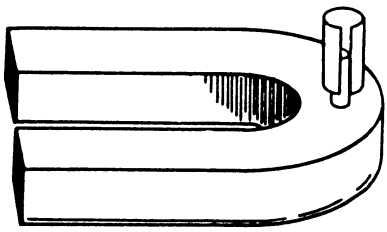
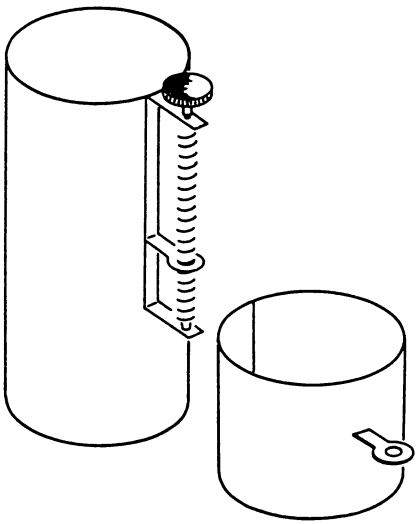


Figure 3
Screw device for adjusting wick



and therefore through the centre of the wick. There is an oil reservoir with a means of controlling the flow of (gravity fed) oil to the wick. The designer (this is about 1898), mentions that it is for use with paraffin oil, oil of turpentine or even methylated spirit, although this with a word of caution, "the latter fuel is not very suitable". This warning is uttered because of methylated spirit possessing two or three properties which other liquid fuels do not have. It was rather too highly inflammable, and it will burn without a wick so that if it were spilled or escaped from the lamp, as it burns with a non-luminous, almost invisible flame the results could be rather unfortunate.

Figure 5 shows a glassworking lamp whose details were published in 1822. It is "fired" by boiling alcohol; the alcohol in the upper chamber being boiled by the flame from the lower, the resultant jet of vapour ignited and according to the illustration is then directed at the operator. I have a feeling that anyone using this apparatus would have more than burned fingers to show for his

Figure 4
Tubular oil lamp

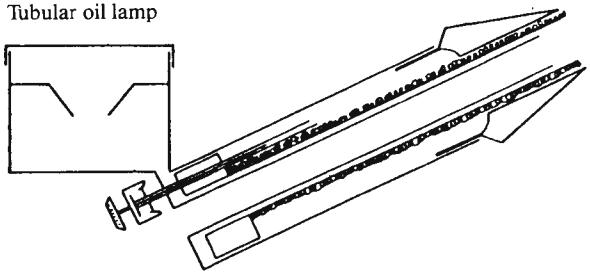
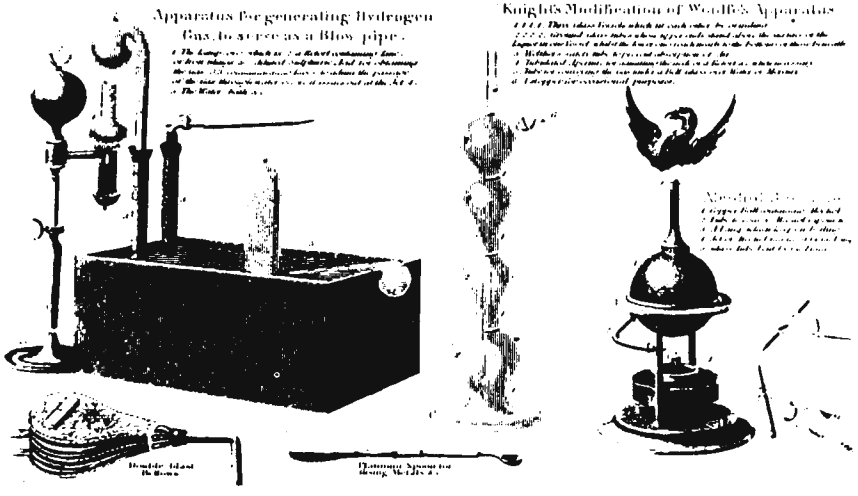


Figure 5



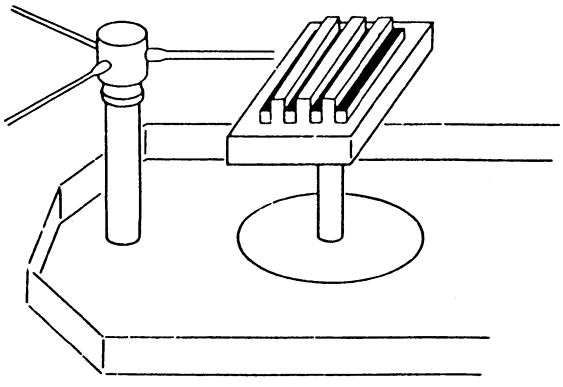
efforts. The Phoenix-like bird on the top? Pure ornament? An embellishment by the engraver of the plate? or a symbolic representation of the fate of anyone using the apparatus; who knows.

At the present time we take much for granted in the ready availability of a supply of fuel of consistent composition. Our earliest predecessors had a wider choice of fuels both liquid and solid (rather than gaseous) but of variable quality and low thermal capacity.

The pros and cons of liquid fuels versus "solid" fuels were much debated. Solid fuels such as tallow, in spite of their lack of fragrance, had some things to commend their use. When the lamp was not in use and needed to be stored, the hot "oil"on cooling would solidify and the lamp could then be put away without any danger of spillage. With the advent of the wax candle and paraffin wax as a fuel, more sophisticated solid fuel lamps were evolved. One of the more complex lamps is shown in Figure 6. It consists of a small open iron tray through which pass three or more flat tubes and between these tubes are placed small flat pieces of wick, the fit between them being so arranged that the pieces of wick may be easily adjusted by means of a pair of pointed tweezers. The resulting flame is broken or divided so that the combustion is concentrated into a smaller area and the air blast which is directed across the flame carries the flame with it more completely than is the case with an ordinary single flame. The wicks may be easily removed and larger or smaller wicks inserted according to the kind of glasswork which needs to be carried out and relating this to the size of air jet which has been selected. Two small pieces of tin plate are used as side covers, to adjust the flame within certain limits. A larger cover is provided, designed to fit over the whole lamp and it also serves as an extinguisher. The capstan-like object with three arms or tubes is a quick change air jet which may be rotated to select any one of three different sizes of jet.

William Murdoch introduced coal gas as an illuminant in 1792 and by 1798 he had installed a gas plant for lighting the factory of Boulton and Watt at Soho

Figure 6



near Birmingham. By 1807 or 1808 gas lamps were lighting Pall Mall, and in the early 1820's gasworks were constructed in many cities. As gas became available, lampworkers gradually used it. Although as late as 1921 books on glassblowing were still describing oil lamps with wicks as being suitable for glassblowing, gas had several advantages over oil as a fuel. One of its advantages was that it gave a more constant flame in relation to the air jets than any flame dependent on the burning of a wick, so all but the most conservative changed to gas as a fuel. The early gas-air "blowlamp" was a relatively simple affair, two concentric metal tubes with gas supplied to the outer tube and compressed air to the inner, mounted on a heavy base, with taps or other simple controls to vary the volume of the gas or air (Figure 7).

Figure 8 shows a vacuum tube made by lampworking from tubing by Dr A. Geissler in 1876 for studying electrical discharges in gases at low pressure. Geissler tubes showing the luminous effects of a discharge of electricity through various rarified gases were popular in the late 19th century and were found in various sizes and shapes.

Figure 7

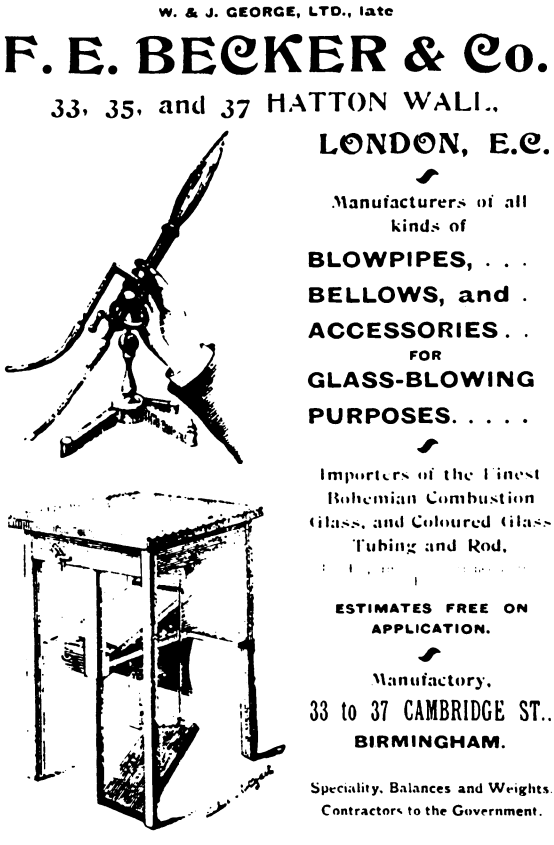


Figure 8

Derivation of a Guideline Equation for Hot Cracking Tubing on the Lathe

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Since antiquity, glassblowers have used the phenomenon of hot cracking to remove the blowpipe or the pontil from the finished piece. The furnace worker would be lost without the technique to remove the last vestige of fabrication. The old German films on window glass show a worker hot cracking a large tube with a cold iron rod both circumferentially and longitudinally. A World War II story tells about the extreme shortage of drinking glasses in North Africa and how the natives made them by taking coke bottles, filling them to a certain height with water, then adding a small volume of oil. A red hot metal rod was immersed quickly in the bottle; the oil having a higher heat conductivity, thermally cracked the vessel at the desired level. The liquid was emptied onto the concrete and the glass ground smooth to become a functional piece. An old English book on glass described a method to hot crack an opaque lamp globe. The object was scored at the desired length, a string soaked in lamp oil was tied around the score mark and ignited. The resulting stresses would cause the globe to sever at the mark.

In our glass shops, there are various devices and methods to cut glass by hot cracking. Hot cracking can be defined as the method of severing cylindrical objects by mechanically scoring the surface, then applying thermal shock either with a concentrated source such as a hot rod or wire or a rotational heating followed by point quenching. A Corning engineer at Big Flats once told me that the most desirable and successful method to hot crack was to score the tube internally and apply the heat source to obtain the cleanest cut. This is probably the most difficult to do since many configurations render it almost impossible. Figure 1 shows a roller device with an adjustable scoring arm to be inserted

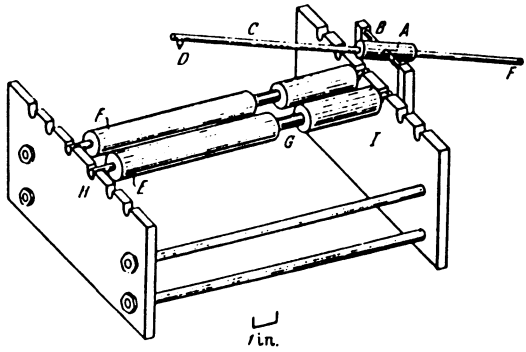


Figure 1
Tube cutting device

inside the tube. The tube is rotated with one hand while the other holds the arm against the glass. A device designed by the Corning Glass Works for field cutting of pipe and drainline over 2 inches in diameter is shown in Figure 2. This cumbersome creation did an exceptional job. The jaws are adjusted to confine the tube, then the center arm is set to the desired length and locked. Rotation of the crank caused the scissors arrangement to expand and allow the scoring wheel to contact the internal wall with a predetermined force, depending on the pipe diameter. Subsequent flaming would produce a clean severance. One had to be careful that the scoring wheel would not walk and cause an internal score that would not meet. This would result in a tip or residual lip on flaming. Figure 3 depicts an old standby; the hot wire device for bench "snap offs". After scoring the tube is arranged in a cradle, the Nichrome wire looped around the score and current applied.

Before glass lathes, hot cracking was done at the bench. Barr and Anhorn as well as Wheeler treat the subject very superficially. I am sure many employ this technique without really thinking about any descriptions or teaching of the technique. The amateur will usually employ a rod too small for the job, linger too long before applying and will indiscriminately cram the hot blob into a mushroom configuration with little success. I have compiled a simple graph,

Figure 2

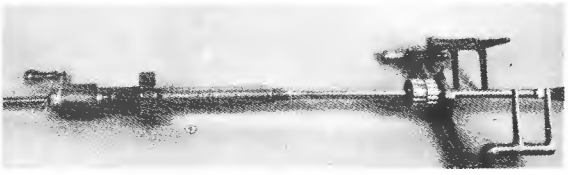
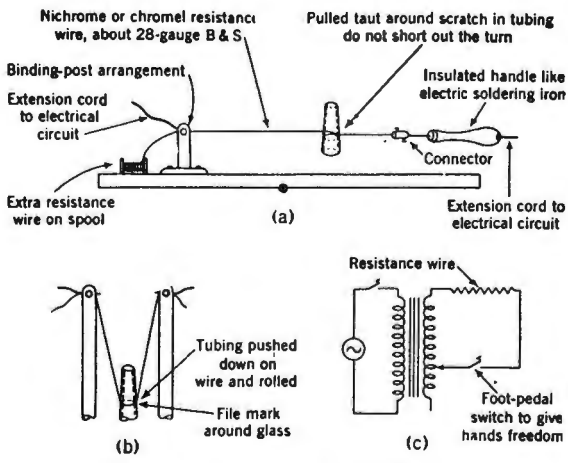


Figure 3



Hot-wire glass cutters. (a) Loop method. (b) Half loop. (c) Diagrammatic electrical circuit. The variable transformer can be replaced by a rheostat.

Figure 4, showing some guidelines for hot cracking at the bench. After rod selection the piece should be positioned so that the hot rod can be applied immediately to the score mark. It should be applied at an end of the score mark rather than the center. This will cause the crack to propagate in one direction whereas applying to the center of the mark will result in two cracks proceeding oppositely with a higher probability of them not meeting concentrically. End application will create one crack and result in a square snap.

Let us now consider the main object of this paper; hot cracking on the lathe. The glass lathe is an essential component of the modern glassblowing laboratory and one of the most utilized techniques on the lathe is hot cracking. Some glassblowers use a diamond scribe and score the tube all the way around. One has to secure the tool and be sure to join the score otherwise a clean snap is not obtained. I simply use a tungsten carbide knife and create a short score mark from ¾ in. to 1 in. in length. Figure 5 shows some specifics of a snap. "Hackle marks" occur in the scored zone and a residual line indicates where the circumferential cracks meet. The ideal method after scoring is to apply heat, then stop the lathe and shock with a wet towel thus producing the cleanest snap. If a tube is bowed or misaligned or if one end is heavy, a deflection will occur which if the lathe is rotating will produce chipping on the cracked interface. With these prerequisities, I have derived an equation which will present guidelines to be implemented. Figure 6 shows the equation

Figure 4

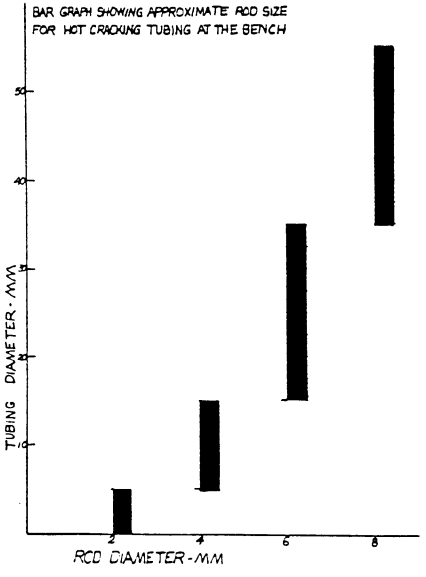
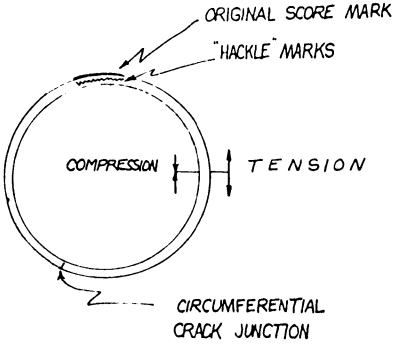


Figure 5



$$N = K \frac{WD}{2}$$

where

N = number of revolutions of the lathe required.

K = frθ = a constant where f is the flame width and length, r is the rotational velocity of the lathe and θ is the coefficient of expansion of the glass. In this case f = 1 mm × 5 cm, r = 120 rpm and θ = 32×10^{-7} and for simplicity resolve the product to equal unity or 1 since all are constant.

W = wall thickness, in millimeters.

D = diameter (outside, in millimeters).

Also in Figure 6 is a graph of the equation. If w = 2, 3, and 4 mm for various wall thicknesses, then a resultant number of revolutions can be calculated for a given D. All tests were done on a S-3 Heathway Bench Lathe using a Carlisle Universal torch with a No. 1 tip. Score mark was made and the lathe turned on. The torch is held as steady as possible with the inner blue cone next to the periphery of the tube. The revolutions are counted as the illuminated score mark passes the tip. Upon completion, a wet paper towel is applied to the score mark. If the surface checks appear the tube has been heated too much and will not crack. If underheated, a crack will appear but must be coaxed round to meet at the origin.

Since larger lathes turn slower and r is a factor, the graph terminates with 100 mm diameter. However, a general rule which works at slower rpm's is simply d/2 in mm equals the approximate revolutions required. For example, a 150 mm standard wall tube would crack acceptably at 75 revolutions.

I have attempted to take a rather simple technique and formulate some guidelines to convey a direction to someone less familiar with the technique and procedure.

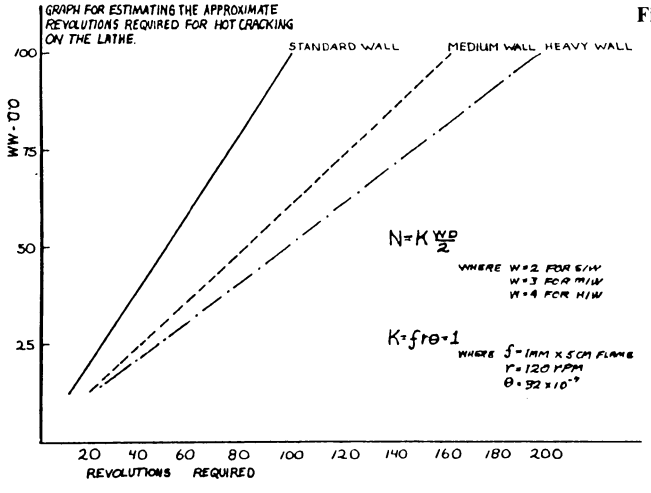


Figure 6

Chromatography

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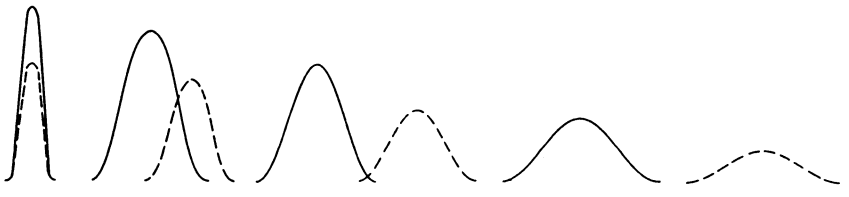
For the glassblower to create superior apparatus, he should understand as much as possible about the functions of the apparatus and the physical and chemical forces involved. This knowledge will also be a help to the researcher, who will be able to look to the glassblower as a resource for design and problem solving rather than simply as a glass mechanic. A complete knowledge of any of the disciplines of chromatography would be a full-time job in itself. Therefore, the purpose of this paper is to explain some of the forces and processes involved with the various methods of chromatography, and to present a method for fabricating and operating a flash chromatography column.

The word chromatography derives from the Greek word *khromatos*, meaning color, and *graphos*, meaning written[1]. Chromatography is a scientific tool used to separate a mixture of two or more substances through differential migration. To achieve this the intrinsic natures of the substances are used to allow one of them to be moved in relation to the other. The separation of the wheat from the chaff is an analogous illustration of the dynamics of chromatography. Here is a mixture which needs to be separated. The mobile phase is the wind; the stationary phase is gravity. When the mixture is thrown into the air there is a partition between the chaff and the wind, which carries the chaff off, while gravity keeps the seed in place (Figure 1). The wind does not make a significant enough partition with every bit of chaff to carry it off on the very first throw. Rather there is a stop-start behavior in which some of the chaff moves somewhat downwind yet still falls back with the seed. To achieve a complete separation

Figure 1 *Peak Disengagement*
As the mixture of the two components (dotted line and solid line) move through the chromatographic device, they separate from each other because of differential migration.



there must be many opportunities for the chaff to achieve partition with the mobile phase, the wind. This system is at the mercy of many variables such as the strength of the wind, the degree of density difference between the seed and chaff and the strength and skill of the operator. Chromatography is similarily replete with variables. The selection of the types of variables which will be most effective in achieving the desired separation is the basis of chromatography.

In any chromatographic system there is always a mobile phase and a stationary phase. The mobile phase may be a gas or a liquid while the stationary phase may be a liquid or a solid. When a chromatography system is designed and operated correctly the mixture separates so that each portion or fraction is apart from the next without overlap. Since the apparent fractions are not discrete but diffuse zones, it is important that the zones be as compact as possible to their centers, and that the centers be well disengaged from each other. To achieve the best differential migration a variety of methods may be used: adsorption chromatography, including liquid-solid and gas-solid chromatography; partition chromatography, including liquid-liquid and gas-liquid chromatography; ion exchange chromatography; exclusion chromatography; and electrophoresis or zone electromigration. In addition a variety of equipment and materials can be used for one or more of these methods. Chromatography columns, capillaries, gels, adsorbents, supports, solvents, thin layers, papers, power supplies, heaters and coolers are some.

FORCES OF CHROMATOGRAPHY

The distribution of components of any solute mixture between the stationary and mobile phases of a chromatographic device is the result of the interactive and intraactive forces involved. These forces act not only between the solute and the mobile and stationary phases individually but between these two phases exclusive of the solute, as well as within each phase. The scientific fields which address these forces are hydrodynamics, solution theory, surface chemistry, adsorption kinetics, diffusion theory and the science of porous media.

Polarity or the dipole-dipole interaction is the attraction of opposite charges and the repulsion of similar charges. That is to say that if there is a permanent charge distribution disparity within a molecule its positively charged end will be attracted to the negatively charged end of its neighbor. The result is that there will be little solvation if the solute does not possess similar charge disparity as the solvent, for the polar solvent would rather adhere to itself than include something non-polar into its structure[2]. This is the reason for the saying 'like dissolves like' as a prediction of solubility. Molecules which include nitrogen, oxygen or halogens are frequently polar in nature.

The dispersion interaction is also known as London's forces[3]. This is caused by a sort of micro dipole interaction because the electron is in motion in its orbital cloud. Sometimes electrons are on opposite sides of adjacent electron clouds and sometimes they are on the same side. This phenomenon causes

attractions and repulsions. Because they are short lived, these events are extremely weak. Since the force of the dispersion interaction is very weak intermingling or solvation with other nonpolar species is acceptable with a non-polar solvent.

Hydrogen bond[4] interactions are very complex due to the unique nature of hydrogen. Because hydrogen is so small and because the innermost electron orbital is not full there is a strong dipole-dipole attraction between the hydrogen in one molecule and the unshared electron pair on a highly electronegative atom on an adjacent molecule or site on the same molecule. These forces are much stronger than those of London's forces. Water has a strong tendency to hydrogen bonding resulting in clustering of four or five molecules. Solutes which can ionize or form hydrogen bonds are very favored for solvation in water.

Covalent bonding[5] occurs when two or more atomic nuclei share an electron pair. Ionic bonding[6] occurs when an electron is tranferred from one nucleus to another atomic nucleus.

All these bondings and interactions are dependent upon temperature, pressure, and concentrations, and result in viscosities, and solubilities. Each of these dependencies and their results act as variables in the dynamics of chromatographic operations.

In each method of chromatography there is a mobile and a stationary phase. When the mixture being separated interacts between these two phases the various constituents or fractions of the mixture interact unequally. It is by optimising and taking advantage of these inequalities that the desired chromatographic separations occur.

ADSORPTION CHROMATOGRAPHY

Adsorption chromatography (Figure 2) includes both liquid-solid and gas-solid chromatography and is the oldest form of chromatography. The American David Day[7] published a paper in 1897 about petroleum origins and in 1903 the Russian, Mikhail Tsvet[8] published a paper on plant pigments. But as early as 1893 the British chemist Lester Reed[9] had used chromatographic methods to separate organic and inorganic salts. All used liquid-solid chromatography techniques. In adsorption chromatography the stationary phase is a solid. The mobile phase is a solvent or combination of solvents. Separation of the mixture occurs because the components are adsorbed onto the surface of the stationary phase to varying degrees due to one or more of the forces mentioned above. Since adsorption is a surface process the adsorbed portion is exposed resolvation by the mobile phase. So, the molecules of each fraction are not simply swept through the chromatography device, rather they stop and start many times as they proceed through. Those fractions of the mixture which are more strongly attracted to the stationary phase make more and longer lasting adsorptions onto the stationary phase. Thus their progress is more impeded than those fractions which are more strongly attracted to the mobile phase.

PARTITION CHROMATOGRAPHY

Partition chromatography (Figure 2) includes both liquid-liquid and gas-liquid chromatography. This method uses immiscible liquids as both the mobile and the stationary phases in the chromatographic device. To get the stationary phase to stay in place it is attached to an inert support. It is fixed so tenaciously that when it contacts the mobile phase, it does not move. As in adsorption chromatography those molecules more attracted to the stationary phase elute more slowly and make more and longer lasting partitions with the stationary phase. Those more attracted to the mobile phase move through more rapidly and elute first. Frequently, the support is silica gel and the stationary phase is water. Martin and Synge[3] developed this method of chromatography in 1941. As in adsorption chromatography the goal is maximum disengagement of the zone centers and reduction of zone spreading to a minimum.

EXCLUSION CHROMATOGRAPHY

Exclusion (Figure 2) is a form of liquid chromatography and was developed under two independent systems called gel filtration for aqueous systems and gel permeation for non-aqueous systems. The dynamics of both systems are the same. Exclusion chromatography is like picking dirt up with a sponge. The smallest particles go in the deepest and the larger ones stay near the surface.

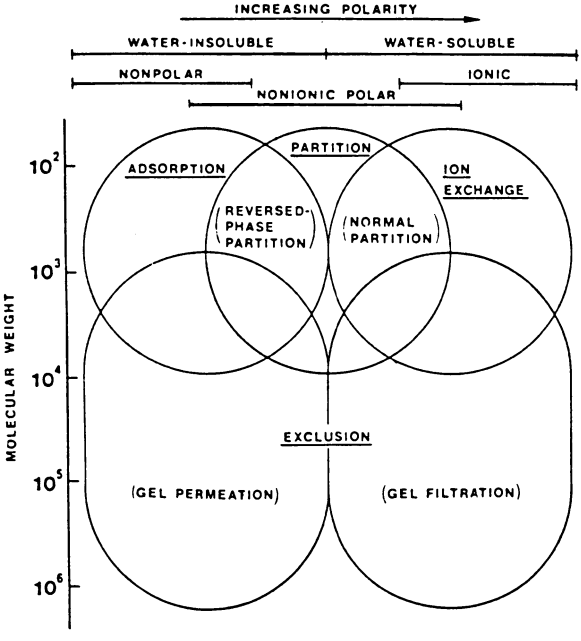


Figure 2 *Techniques of Liquid Chromatography* (reproduced by permission of VanNostrad Reinhold Company, New York)

When the sponge (stationary phase) is rinsed (mobile phase) the larger pieces come off first while the smaller ones are retained in the pores. The range of molecular size is controlled by the pore size and distribution of the stationary phase. There are many kinds of materials which are used as stationary phase: cross linked dextran gels, agar and agarose gels, polyacrilimide gels and polystyrene gels. Exclusion chromatography is useful for the separation of molecules ranging from 500 to 1,000,000 in molecular weight.

ION EXCHANGE CHROMATOGRAPHY

In ion exchange chromatography (Figure 2) the insoluble stationary phase has ion pairs on the surface. One of the pair must stay attached to the stationary phase, usually a polymer resin. The oppositely charged ion is free to be exchanged for a similarly charged ion being carried in the mobile phase. Separation of similarly charged species will take place because those of lesser charge will be displaced by those of greater charge, and thus the least charged species will elute first. Careful manipulation of ionic strengths, buffers of precise pH and selection of mobile and stationary phase materials make this method applicable to biochemical operations as well as metals chemistry.

ELECTROPHORESIS

Electrophoresis is the movement of charged particles through a fluid under the influence of a high potential (voltage), direct current electrical field. Electrophoresis is primarily used for biochemical separations such as proteins and nucleic acids. The conductor is usually a buffer solution which is contained on a support of paper or gel. The separation takes place because the charged end of the molecules being separated are attracted to the opposite pole in the electrophoresis device. The differential migration rate of the molecules being separated depends on many variables. These include the electrochemical nature of the molecules being separated as well as that of the buffer solution and the support, the potentials being used in the apparatus, the pore size of the support, the temperature as well as the densities and shapes of the molecules.

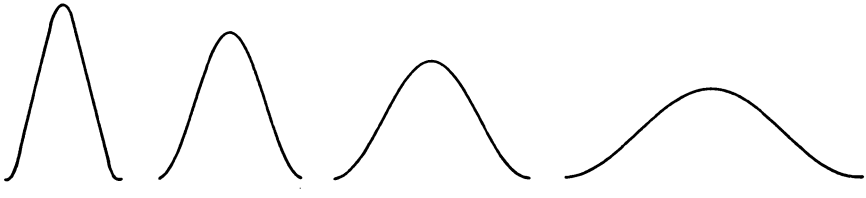
ZONE SPREADING

Keeping the stop-start behavior of chromatographic separations in mind the causes of zone spreading or band broadening (Figure 3) should be addressed. Notice in Figure 1 above that as the peaks disengage they also exhibit band broadening. There are three general causes to zone spreading: ordinary diffusion, eddy diffusion and local nonequilibrium. In ordinary diffusion there is a normal migration of the solute from areas of higher concentration to those of lower concentration. The greater the amount of time the solute mixture spends on the column the greater this effect. Eddy diffusion occurs because the pores in a

porous medium are never exactly uniform. The stationary phase must always be porous to the mobile phase. Some pathways will run straight through while others are more tortuous. Those molecules in the longer pathways will lag behind the center or average and those in the shorter pathways will move ahead of center of concentration. Since tightest packing of the stationary phase is not likely where the packing material meets the column wall, there is frequently a higher mobile phase velocity at the column-packing interface. Local nonequilibrium is a general category for a number of phenomena which are related. Considering the bell-shaped curve, the highest point on the curve represents the highest concentration of the specie being transferred. On the downstream side of the center more couplings are taking place with the stationary phase. In the trailing or upstream side of the center more decouplings or resolvations are taking place. If the leading forces of adsorption onto the stationary and the trailing forces of resolvation with the mobile phase aren't equal the distribution will be skewed away from center. Increasing the velocity of the mobile phase only further broadens the band.

Consider an electroporetic problem of an ion which can exist with two interconvertible electrical states. Unless the mobilities of the two charged forms of the same specie are the same (which is unlikely) nonequilibrium spreading will occur as the specie changes back and forth from the faster to the slower charged form. In a tube with no packing (as gas chromatography), the viscous interactions of the mobile phase and the wall of the tube cause a velocity gradient. The flow is faster at the center than at the wall causing a nonequilibrium situation. Where a column is coiled or bent there is also a velocity gradient. Since the velocity is greater on the inside of the curve a nonequilibrium of concentration will exist between the inside and outside of the curve. A common problem, especially for large preparative columns, is uneven packing. As mentioned in eddy diffusion this leads to uneven mobile phase velocity and thus band broadening.

Figure 3 *Zone Spreading or Band Broadening*
The concentration (vertical component) is gradually lessened as the fraction moves through the chromatographic device (horizontal component). Notice this same effect in Figure 1 where the third pair of peaks shows the best separation without excessive band broadening as found in the fourth set of peaks.



SOLUTIONS TO ZONE SPREADING

Steps can be taken to minimize the three causes of band broadening or zone spreading: ordinary diffusion, eddy diffusion and local nonequilibrium. To minimize ordinary diffusion it is necessary to optimize the mobile phase flow rate so that it is not too slow. This causes the forces of diffusion to be overcome by the hydrodynamic forces of the chromatographic column. To minimize eddy diffusion it is necessary to optimize the amount of the stationary phase to the minimum amount which will still give good peak separation. Careful preparation of the column helps insure even packing. When making glass columns select tubing which is as free of irregularities as possible. Striae, stones, splice seals, sealed cracks, dirt, bends, etc. can all lead to problems of eddy diffusion. If fritted discs are used to support column packing a minimum of dead volume below the fritted disc is helpful in reducing the remixing of closely located fractions as they elute.

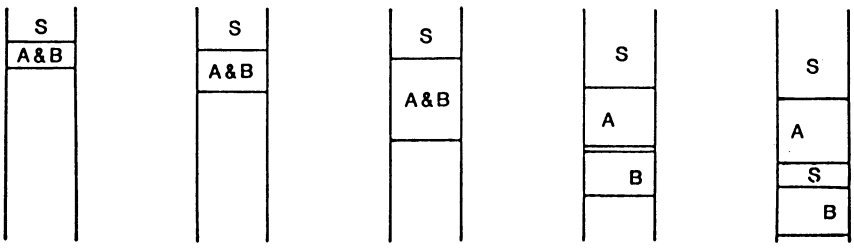
The solutions to the general class of problems known as local nonequilibrium must be addressed individually. In the case of the velocity gradient of the curved column, if the radius of the curve is made as large as can be accomodated, this gradient will be minimized. Other problems such as unequal couplings and decouplings and interconvertible electrophoresis species are the responsibility of the researcher since he must be familiar with the natures of the mixtures in order to avoid these problems of local nonequillibria.

SEPARATION TECHNIQUES

There are three distinct techniques used in chromatograpy: elution development, frontal analysis, and displacement development[1].

Elution development (Figure 4) is the most common method used. The mixture is introduced in one dose at the top of the column. As the solution of the mobile phases sweeps down the column the fractions of the mixture which come out first are those with the least interaction with the stationary phase. If done correctly the fractions are distinctly apart from one another and they are compact to their centers.

Figure 4 *Elution Development*



In frontal analysis (Figure 5) the sample is added continuously onto the column. The more tightly held component of the mixture will build up on the stationary phase. The more weakly held will be the first to elute. However, both substances will subsequently begin eluting. This is not a widely used technique. One use is to determine the adsorptive capacity of a chromatographic column.

In displacement development (Figure 6) a solvent is more attracted to the stationary phase than either of the fractions being separated. As it proceeds down the column it strips the column of both fractions. The elution will be similar to frontal analysis in that the less tightly held fraction will elute first in pure form followed by a mixture of both. If there is a reasonable disparity in their attractions for the stationary phase this band where both parts elute at the same time will be narrow and will be followed by the more tightly held will elute in pure form. For example, in metals chemistry this process can be done repeatedly on the same column.

FLASH CHROMATOGRAPHY

Recently there have been some innovations in chromatography which have been the basis of the technique flash chromatography. The work was initiated by B.J. Hunt and W. Rigby[12], and was developed by W.B. Clark Still[13]. The variables of band broadening have been optimized to give rapid separations of samples

Figure 5 *Frontal Analysis*

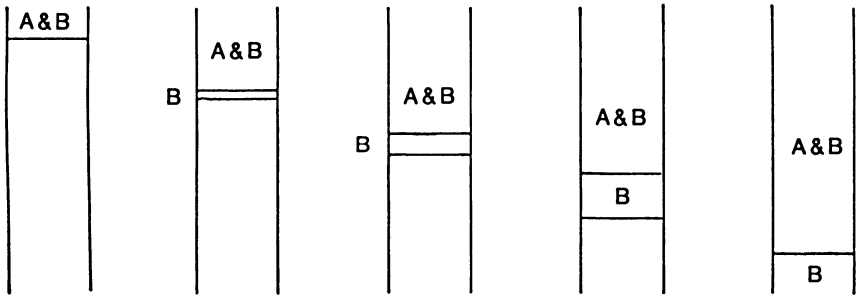
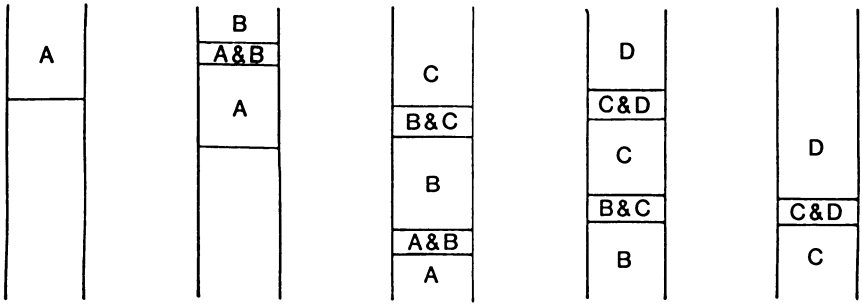


Figure 6 *Displacement Development*

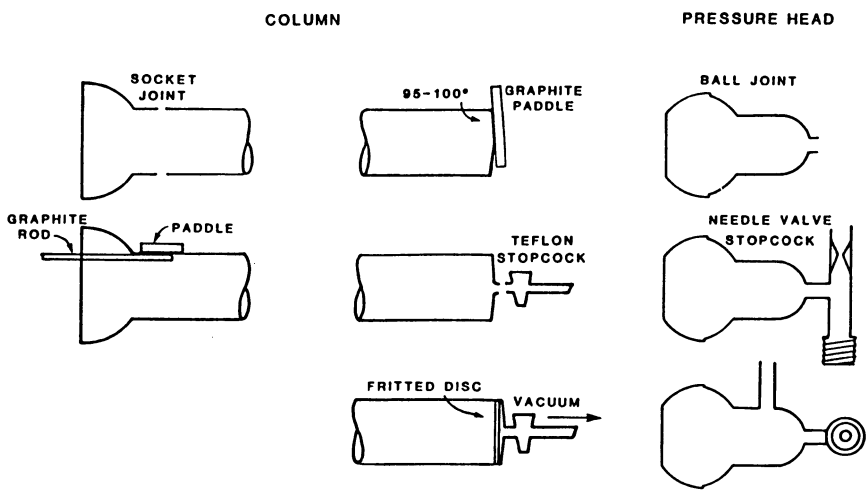


up to ten grams or more. Generally the chromatographic device is a short wide column which is pressurized to optimize ordinary and eddy diffusions. This column has a flat bottom with a teflon stopcock, an optional coarse fritted disc on the bottom, and either a socket joint or standrd taper joint on top. A column length of fifty centimeters will accomodate most needs regardless of column diameter. Below is a technique I have found effective for creating a flash chromatograhic column.

COLUMN

To make low dead-volume column (Figure 7) prepare the bottom for sealing in the fritted disc. Make bottom not quite flat with inside corners sharp enough to accomodate the corners of the fritted disc. Be sure the frit fits all the way to the bottom of the column. Seal the stopcock onto the bottom. Without removing the column from the lathe move the fritted disc into position and apply vacuum from the stopcock end to hold the fritted disc in place. It should fit very well and be very square to the bottom of the column. The distance of the stopcock tubulation from the surface of the frit should not exceed three or four millimeters. After preheating the whole area very well, seal the edge of the fritted disc by paddling but not blowing. Remember to disconnect the vacuum as soon as the frit is stuck to the glass to avoid moving the glass from the bottom onto the face of the frit. Do not overwork or eddy diffusions will result. Put the piece into a hot oven and anneal. After annealing attach the socket joint. The most successful designs have the inside diameter of the socket joint larger than or equal to that of the column. The reason for this is that it accomodates

Figure 7 *Flash Chromatography Column and Pressure Head*



good column packing which is extremely important for the successful use of this device. For sealing large ground joints onto columns with the same tubulation size (greater than fifty millimeters in diameter) the following method can be used. First, prepare the ends of both the socket joint and the column by hot crack-off on a diamond scratch. Keep the length of the tubulation on the ground joint short (one-half to one diameter's length below the ground portion. With both pieces chucked in the glass lathe use the underfire burners adjusted to minimize flame spash and heat a narrow band on the two tubes. Seal the ends together and pull slightly to thin the seal. Remove the heat. As soon as the glass is rigid release the socket joint from the lathe chuck. Separate the chucks well to accomodate the proceeding steps. Reheat the seal while reaming with a graphite rod from the inside to ensure a smooth seal. If the seal thickens, it can be paddled smooth from the outside while reaming from the inside. The seal should be done quickly. The graphite tools can be quenched in Aquadag solution to keep the graphite from becoming overheated. This also prevents the graphite from becoming granular. Anneal with a flame and put in cold oven to anneal so the fritted disc does not crack from thermal shock.

The pressure head flow controller on the mating ball joint is then fabricated and added to the hot oven. The flow controller consists of a needle valve rotary stopcock for pressure regulation on top and a tube or hose connector on the side for inlet of pressurizing gas.

OPERATION OF A MEDIUM PRESSURE FLASH CHROMATOGRAPHY COLUMN

The packing of the column is extremely important and most bad results are caused by mistakes made here. If no fritted disc is installed then a glass wool plug must be inserted at the bottom of the column into the stopcock tubulation. Sand is then added (50-100 mesh) 3-4 mm thick and this must be VERY FLAT. Sand is optional if a fritted disc has been installed in the manner described has been installed. Dry silica gel 40-63 micron (400-230 mesh) is poured in to a depth of about 15cm. The stopcock is opened and the column is tapped and rotated in a vertical orientation to get rid of any air pockets and encourage tightest packing. A layer of sand 3-4 mm thick is added to the top of the very flat column packing. The chosen combination of solvents is carefully poured onto the column, filling it, and the pressure head is clamped to the top of the column. The column is pressurized to drive the solvent through the packing material. It is very important that the column have all the air expelled and that the column be cool before stopping ths solvent flow by relieving the pressure. The excess solvent on the column is then forced out at lower pressure but the top of the column packing must not be allowed to dry. The introduction of the sample to be separated must be done with great care. A 25 percent sample-eluent solution is gently pipetted onto the top of the column. This is gently forced just onto the top of the column with the pressure head. The walls are gently rinsed

with a little more of the solvent eluted previously and this too is forced just onto the top of the column packing. The eluted solvent is then carefully added to the top of the column to fill it without disturbing the column packing, and the pressure cap is attached. The pressure in the column should be adjusted to provide for 5 cm drop in liquid level per minute. The correct fraction size in milliliters should be about equal in number to ½ to 1 times the inside diameter of the column in millimeters. The location of the target fractions should be done by thin-layer chromatography. The column may be reused if not allowed to dry by stripping it with the more polar solvent used in the column and then with the mixture used in the column. This method of flash chromatography yields about 95 percent of the sample and can do gram samples in minutes rather than days. This is a medium resolution technique which can be very useful in the lab.

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Computers without Computerese

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INTRODUCTION

The demands in the glass shop are continuous. We are called upon to make, redesign, alter, and occasionally invent apparatus. However, we are often responsible for other, more mundane matters, that have nothing directly to do with glass. By this I refer to writing letters, filing and inventorying glassware, maintaining a proper stock of supplies, and sometimes the drawing of items we make. Unless we have some aide in the shop, or access to a secretary, we must do it ourselves.

At some point, one may wish to check out computers to see if they can help as much as is claimed. The answer is that computers can help in the glass shop. Unfortunately, many people shy away from computers because it is felt they will have to program everything. There is also a fear that they will have to learn some computer language to do anything. However, it is not necessary for one to learn a computer language and, because of new technologies, it is far easier to use computers than in the past. There is little to avoid, but much to gain.

USES

For example, word processors are fantastic. When one is as poor a typist as I am, the ability to easily correct errors as they appear (or as they are discovered on later proofreadings) can be immediately appreciated. It is easy to rearrange words, sentences, or paragraphs to see if an alternate order provides a better form and structure for the document. The computer can keep track of how much material can be kept on a single page. Therefore on the computer screen, and in printouts, pages can be self-numbered and even self-dated. There are spelling correctors, and one can pre-code the computer so that every time an abbreviation is typed, a complete phrase will be printed. (For example, if one were to type ASGS, American Scientific Glassblowers Society). Even the structure and look of what you write can be aided by the variety of different styles of letters and the different settings that permit paragraphs or sentences to be justified or aligned to the left, right, or center.

Also, graphic systems can be fun and useful. It can be nice to design and develop a personal letterhead, but sometimes it is critical to include a drawing in a letter to point out the details of your written description. In our work, a

sketch is usually more than sufficient in providing the materials needed and the sizes required. However, when we have specific size or containment limitations, the ability to design in accurate size to scale on the computer screen can save us hours of time.

Even filing systems in computers can do more than just list items or names. One can easily select items from within specific groups, based on specific criteria. For example, one might look at one's entire inventory, a specific inventory, the materials used by a particular customer, or the purchasing that a customer has made. One can even cross-reference any of these partial possibilities. Since one can design one's own filing system, one can file information on any subjects or matters that are of concern to the particular computer user.

THE PROBLEM WITH COMPUTERS

However, the computer has a poor reputation with some people. Part of that reputation is deserved; much of it is not. Although the cost of computers has gone down dramatically, they are still thought to be expensive. Computers are also thought to be very difficult to learn how to use. Common opinions are:

"Word processing is great, but I can't remember which keycode will do what command!"

"Why balance a checkbook on a computer when an inexpensive calculator can do that?"

"Programs that can draw spirals are cute, but what does that mean to a glass shop?"

"How can a filing system program be made for something as esoteric as a glass shop?"

"There isn't enough time to learn how to use a computer!"

There is also the unspoken reality that, for some people, the computer's very power and impersonal nature can be intimidating.

Worse still, people who are 'into' computers are perceived as a bit of a drag: as they talk, words such as 'hard disc', 'kilo bytes', 'RAM', and 'ROM' keep on entering the conversation. One ends up feeling as if he were dropped into a foreign country where there were no common links between languages. As if that were not enough, the stereotypic computer user is 14 years old and busy trying to hack into the Pentagon's computer system, or worse, one's bank!

INTRODUCTION TO COMPUTERS

Obviously, the preceding statements are not true as a whole, but there is enough validity in them to keep some people from computers. There is a language and terminology to computers that one needs to know. Just as glass and autos have their own language, so do computers. Most of the terms are straightforward and logical, and are divided into two major categories: hardware and software.

Hardware is the mechanical parts of a computer; the things that you can hold

and see. The following, as seen in Figure 1, are the basic components:
1 The *computer* is the actual electronics. The *screen* illustrates what one puts into the computer or what the computer tells one. The computer and screen can be one solid piece; often they are separate components.
2 The *keyboard* is the device for communicating with and directing the computer. This is accomplished by normal typing or by the use of specific keys which are pre-coded to operate specific commands.
3 The *mouse* is a new "computer access" device. By sliding it around a flat surface and pressing a button on it, one can activate commands and/or move and create images on the screen.

The following are some of the *peripherals* available, and are not essential to the operation of the computer.
1 *Floppy discs* or *micro-floppy discs* store information or programs on a metal-oxide affixed to a plastic base. Like audio tapes, information is recorded on the discs, which are erasable and reusable.
2 The *disc drive* and the *second disc drive* are the devices by which programs and information that are stored on the micro-discs are inserted. The second disc drive is important if one needs separate discs for storage of information or if the amount of information for a program is greater than can be held on one micro-disc. Often two drives are incorporated in the same computer rather than a separate unit as seen here.
3 The *dot matrix printer* provides a paper print-out of whatever one has developed in the computer. Each dot is a called a *pixel*. By printing everything with dots, the printer is not limited in the types of shapes or sizes of letters or drawings it can reproduce. This allows one to provide drawings directly on one's correspondence, for example without having to hand draw them later.

Software, by comparison, is intangible. It is the series of commands that the computer blindly follows to accomplish the desired task. This series of commands is called a *program*. An example of a simple program to compute the sales tax and purchase price of items sold in California would go as follows:

Figure 1

Figure 2

"1. Write down a number ....."
"2. Multiply that number by 0.065 (6.5%) ....."
"3. Round to two places ....."
"4. Add that to the original number ....."
"5. Print the answer ..... _____"

Only the first of the four steps above would need to be done by the computer user; the software instructs the computer to continue with the remaining steps automatically. So, for the above example, the computer user might type into the keyboard:
"$1.00", then press the "enter" button on the keyboard, and "$1.07" would appear on the screen.

The hardware of the computer is totally ignorant until it is provided with the information necessary to do a task. Fortunately, there is no need for users of computers to program anything. There are many companies that offer a wide diversity of prepared programs. All the user needs to do is take a prepared program disc, insert that disc into the computer, and perform the operations of which that program is capable. When one introduces a program into the computer, the computer can do only the work of that program. If the computer is set up for word processing, one can do only word processing. If one wants to do a graphics program, he will be thwarted. A program will only recognize data that was made by that specific program. If one wants to write a letter that will also include illustrations, both a word processing program and a graphics program must be entered in the computer.

Although it wasn't mentioned earlier, there is a third major division of computer language; *fleshware*. It is the user of the computer (and probable cause of most of the problems) (see Figure 2).

One of the main operational problems with computers is communication with the computer. This problem can be divided into two major categories: The first is spelling and/or typographical errors. If one is trying to recall a previously made document or a specific program, one must request it exactly as it was originally recorded. The computer will not second guess you. For example, if one is trying to retrieve a letter and the code:
"letter to Wilbur"
was originally used, the computer would respond with
"nothing entered by that name"
if one was asking for any of the following possibilities:
"Letter to Wilbur"
"leter to Wilbur"
"letter to wilbur"
"ltr to Wilbur", etc.

The second operational complication of computers is remembering which keycodes correspond to what command. If one wishes to type in a bold face print, a command key on the keyboard must be pressed at the same time the letter "B"

is pressed. Now anything that is typed would be in bold print. Aside from bold face, there can also be commands for underline, superscript, subscript, changing the type size, changing the margins, setting the tabs, and so on. Remembering which key pressed with the command key will do what command can become very confusing and frustrating. The mouse, described earlier, solves most of these problems. This allows a non-computer person the ability to use, and enjoy, a computer.

THE NEW TECHNOLOGY OF COMPUTERS

The mouse (Figure 3) is a small electronic device that is about the size of a package of cigarettes. There is a button on it. By pressing the button, one can control events on the screen and in the computer. A ball on the bottom makes contact against several electrodes as one slides the mouse across a table. The rolling ball activates the electrodes, controlling an arrow on the screen that moves in a like manner. Thus, if one wants the arrow to move right, one slides the mouse to the right.

Icons, or pictures that represent specific symbols, are used to represent programs, systems, and previously-made documents. By using the mouse, one can maneuver the arrow to a specific icon on the computer screen. When the button is pressed, it is as if one is saying "give me that one".

On the top of the screen are a series of *menus* of commands. If one wishes to have the computer follow a command, by guiding the mouse, one goes to the appropriate menu and presses the mouse button to cause available commands from that menu to appear on the screen. Then by sliding the arrow down the list until it points to the desired command and then releasing the mouse button, one activates that command.

A DEMONSTRATED EXAMPLE

This process is best demonstrated by the sequence of entering a program, doing something, exiting the program, and *discarding* an unwanted document. In this example, a word processing program demonstrates how, by using the mouse, complex commands can be initiated by simply pointing arrows and

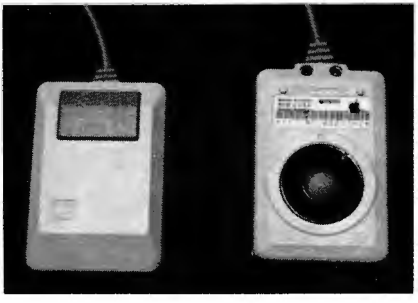


Figure 3

pressing buttons. For an example, one could correct a letter in which a name occurs many times but has now changed:

1 after pushing a micro-disc into the computer, the screen will show a few menu titles on the top of the screen, a disc icon (with that disc's name underneath it), and a trash can.

2 to see what is on that disc, one brings the arrow on the screen to the icon (by sliding the mouse on the table), and clicks the button on the mouse twice in succession to "open" the disc up.

3 as the disc opens (Figure 4) one sees a variety of icons, each representing a particular application. The example disc contains a word processing program and previously written letters.

4 to use a specific program, or to see a previously-made document, again one places the mouse over the icon and presses the button twice in succession to "open" the document up. Since the letter is on the same disc as the original program from which it was made, all of the program functions are available to alter, edit or rewrite the letter.

To change a word or a name that occurs many times, the most efficient method would be as follows: one moves the arrow to the menu listing of *search*. Pressing the button, the full menu opens to show (Figure 5):

"*Find...*
Find Next
Change...
and *Go To Page* © "

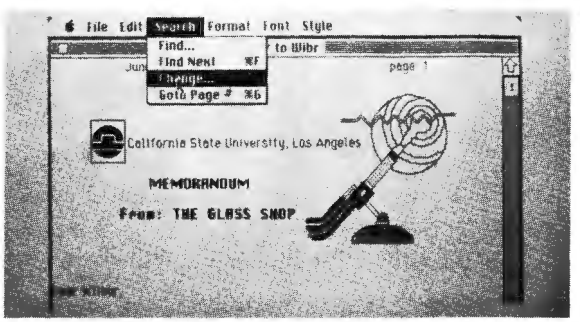


Figure 4

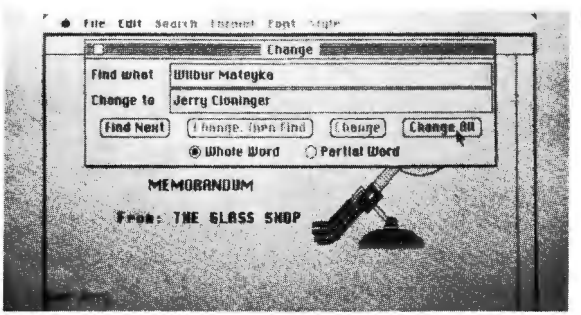


Figure 5

Pulling the mouse to the word *Change*, and releasing the button will generate a box (Figure 6) in which one can type the error and then the correction. The box also provides options on what one can do to make the changes one desires.
To demonstrate
a First one types the word as it was originally typed.
b One then moves the typing cursor (by moving the mouse) to the second box, presses the button, and then types the new word.
c By moving the mouse (arrow) to the *Change All* box and clicking the button, every entry of that name will now be corrected.
d Finally, moving the mouse (arrow) to the small square box on the upper left of the *search* box will "close" this command box, and allow one to continue with the document.

Earlier it was mentioned how, in a standard computer, one could touch the "command" key with the "B" key and the computer would start printing in bold type. By repeating that command, the computer would go back to printing in standard type. This keyboard command ability is not lost in computers that have the mouse. However, the same process can also be accomplished by using the mouse and the arrow by going to the menu titled *style* with the mouse dragging to "bold", and releasing the button to put the computer in bold type. Repeating this process puts the computer back in normal type.

These options provide the computer user with a choice. Since it can be difficult to remember which key (when pressed with the command key) will do what command, one does not have to remember it; one can just as easily use the mouse. Thus the computer user can successfully use the computer before learning the locations of the various command keys. In fact, the computer user need never even learn any key code.

5 To print a letter, one enters the *file* menu (Figure 7a), drags to print, and releases the mouse button. A box appears with instructions to the user (Figure 7b). In this example, the computer user has instructed that he wants an average quality print, continuous paper, and that the whole letter, including the graphics, be printed. The computer user then brings the arrow to the box that says *OK*, presses the mouse button, and a letter is generated out of the printer.

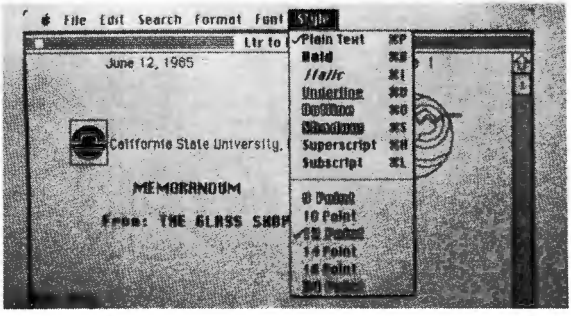


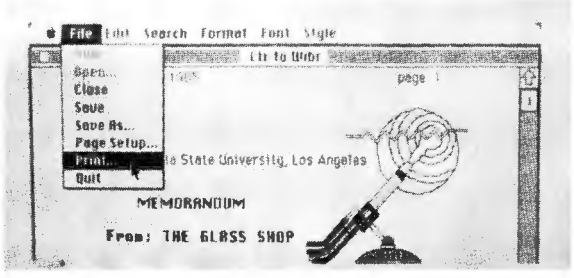
Figure 6

6 After the letter is printed, and one is ready to leave the computer, the micro-disc must be removed before the computer is turned off. First, one returns the arrow back to the *file* menu, drags to *quit*, and the screen appears as it did when the disc was first inserted. Now, if this letter is no longer needed, one can "throw it away" in the literal sense by bringing the arrow to the Icon of that letter and pressing and holding the mouse button as the Icon is dragged over to cover the trash can in the lower right side of the screen. When one releases the button, the Icon is no longer on the screen; it is in the trash can. By moving the arrow to the menu titled *special* and dragging to the *empty trash* entry, the letter is removed from the computer.
7 Finally, by moving the arrow back to the *special* menu and dragging it to *shut down*, the micro disc is popped out of the computer like an audio cassette from a car stereo.

Note that throughout this example, no typed commands were used; rather, all commands were made by pointing an arrow and asking for "that one".

Conclusion: The Benefits of the New Technology

The benefits of using a system with a mouse can be detailed three ways:
a it is easy to use. By using the menus to initiate commands, not only is the learning time significantly cut, but also the occasional user need never worry about forgetting how to give critical commands.
b it is easy to locate and recall any previous document on a micro-disc. There is no need to remember how the title was originally spelled, and since all the documents are displayed on the screen, there is no question of whether a given document is stored on a given micro-disc.



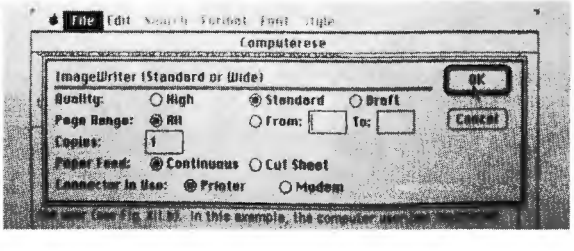


Figure 7a, 7b

c although it is easy to throw out items that are no longer wanted, it is an operation that requires two deliberate acts as a failsafe step to protect one if a second chance is needed.

It should be pointed out that this paper is based on Apples' Macintosh Computer. The Macintosh is not the only computer system that has developed this technology, nor is this paper intended to promote the Macintosh over any other system. Rather, the Macintosh has been used as an example of the technology because it is what this glassblower owns.

This new computer technology, part of the computer revolution, is not an end, but a major step for the user. The glass lathe was originally invented so that one with far less experience than a master glassblower, could make radio tubes and other glass items. However, now there are items made on lathes that could never be made on the bench. What began as a crutch has become a valuable tool.

As busy glassblowers, many of us feel we do not have time, or the desire, to learn how to operate computers. Fortunately technology has given us a "computer lathe" so that we can work with the best of them. However, the mouse technology has aided computer graphics to a point that no keyboard commands can duplicate.

This paper dealt primarily with word processing because it is very easy to relate to. The form and manner that computer graphics operate cannot be adequately presented in a written form. It is suggested that one go to a computer store and talk to a salesperson specifically knowledgeable in computer graphics for further information.

Teflon Clear Coating on Glass, with and without Primer

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To overcome inherent disadvantages of glass, attempts have been made for several years to prepare teflon coated glass. Coated glass has several advantages: it is easy to clean, the surface is homogeneous and non-absorbing and consequently less heterogeneities occur; no scratches or small glass fragments hamper experiments. The coating has a low conductivity of electricity thus preventing the absorption of charged particles. Furthermore, the coating is chemically inert thus experiments using conditions of high pH or high concentrations of hydrofluoric acid can easily be performed, which otherwise would affect the surface of the glass. A disadvantage is the impossibility to repair a teflon glass object: prior to the repair, the coating needs to be completely removed by a process of burning-in. However, in this process, teflon particles remain in the pores of the glass. In preparing teflon coatings, strict safety precautions should be taken (below).

Starting from standard procedures for teflon coating on metal surfaces[1], a method was developed for the preparation of glass coatings. Prior to coating, the glass object is roughened, thoroughly cleaned, and dried with methanol. A thin layer of primer is sprayed onto the glass; after drying in the air, one or two layers of coating are sprayed onto the first layer. The resulting thickness (high-build) can be varied. Thin layers are essential to prevent cracking of the final coating. The object is now slowly heated in an oven and sintered for 15-20 minutes (Figure 1). Control of sintering temperature is important to obtain coatings; too high a temperature results in porous coatings.

The final coating is transparent with a slightly greenish color. The color is due to the presence of the primer. Omission of the primer results in a transparent, white-colored coating which however, is less solid. Extensive tests showed that although not absolutely necessary, it is preferable to employ a primer.

Glass, coated with teflon using this procedure, displays several of the above mentioned advantages in experiments. Subsequent research is in progress to further improve procedures.

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SAFETY precautions to be taken when preparing teflon coatings:

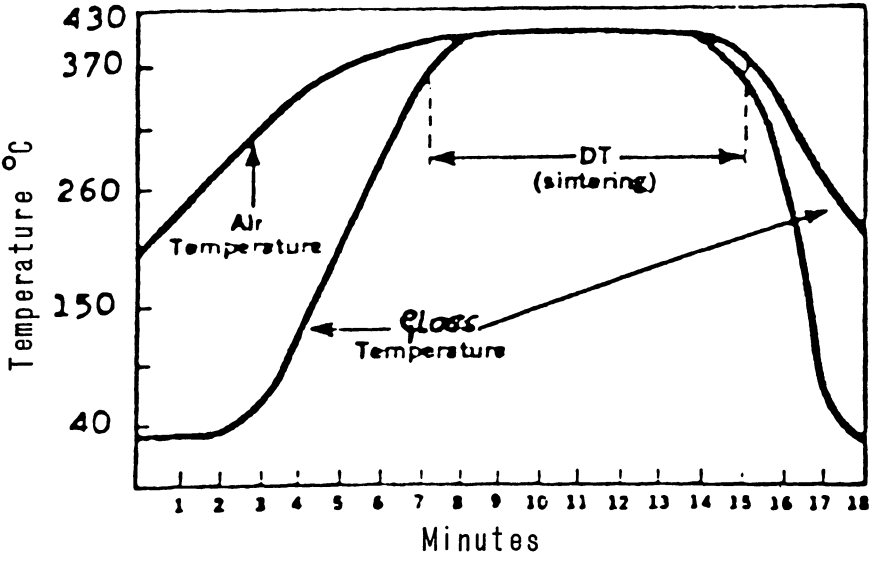
1 use adequate ventilation, avoid inhalation of spray dust and oven vapors
2 keep away from heat and open flames
3 no smoking in spray area
4 wash hands before eating or smoking
5 avoid coming into contact; especially skin and eyes
6 in case of contact, rinse thoroughly with plenty of water.

References

1 Technical details, copyright Du Pont de Nemours, Mechelen, Belgium

The author wishes to thank Menning B.V., Soest, The Netherlands, for their aid in the spraying of coatings.

Figure 1 *Typical Oven Profile*
(with permission of DuPont de Nemours, Belgium)



A Concise History of Lampworking

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It cannot be accurately determined when the use of an oil lamp or torch was first used as a technique to manipulate glass. Probably, lampworking in one form or another is as old as the working of glass itself. There are several fragments of complicated patterns on early Roman glass in the Corning Museum Collection which suggest that lampworking may have been employed as a technique. In the third and fourth centuries lampworking was used to decorate so called snake thread vessels in the Rhineland and in the Near East. The technique may also have been used to apply trails to the surface of the glass, then blown to form a blank for later fabrication of cameo glass.[1]

The next major technical development in flameworking of glass came about during the Venetian Renaissance. Perhaps the finest examples of the use of lampworking, at this time, are in the Accademia Cimento in Florence, Italy. These, originally designed by Galileo Galilei at the end of the 17th century, were the forerunners of the modern thermometer and were used for measuring the expansion of liquids.[2] These early examples of laboratory apparatus are probably the first use of lampworking as we know it today, using a bench burner to manipulate the glass tubing and rod. By the time these early thermometers were executed, much of the great technical advances, originally from Venice, had spread throughout Europe.

According to one source, some of the original glassworkers from Venice eventually emigrated and settled in the black forest of Germany. This area was chosen because of the abundant wood supply desperately needed to satisfy the tremendous energy demands of the glass industry. Eventually, even this thickly wooded area began thinning out and restrictions on the use of timber for glass working had to be imposed. This created periods when individual craftsmen were not allowed to work at the furnace. To provide work during these restricted periods, glass tubing and rod were drawn for lampworking to produce small objects such as animals and Christmas ornaments. This not only provided work but also saved energy in the form of conserved timber; lampworking, then as now, uses far less energy than furnace glassworking.

One may question the accuracy of this scenario, however, it does provide a plausible historical link between the transferring of the technology of lampworking from Venice to Germany. One thing that can be said with certainty is that

there were a number of areas in Germany where lampworking was practiced during the 18th, 19th and 20th centuries.

One center that has been of special significance is the quaint village of Lauscha located about 60 miles north of Nurmberg in the Thuringian Forest, in what is now East Germany. Glassworking in Lauscha dates back as far as 1596, but it was not until the late 18th century that extensive lampworking was practiced. At this time, lampworked scientific instruments, glass eyes for toy dolls and humans, and glass toys were manufactured. Eventually, Christmas ornaments provided the staple industry for the village.

Historically, this area has nurtured such artists as Karl Muller, who was Wilhelm Roentgen's scientific glassblower and who was credited with producing extraordinary reproductions of 17th century dragon stemmed goblets in his Hamburg workshop. So convincing were his reproductions that up until 1978, the Corning Museum believed that the Muller Venetian styled goblet was a genuine example of Renaissance glass.

Another significant artist was Friedrick Zitzmann, who was also born in Lauscha, but worked in Wiesbaden in collaboration with Karl Koepping. These two artists, working together, created some of the finest examples of Art Nouveau Glass in the period.

The most significant contemporary artist to come from this region is Albin Schaedel. In 1958, he developed the glass montage technique in which numerous individual tubes of glass are fused together at various angles to form one complete piece.

Today, from its early humble beginnings, Lauscha has cultivated eight artists of international reputation. These Artists are Albrecht Greiner-Mai, Otto Schindhelm, Volkhard Precht, Hartmut Bechman, Walter Baz-Dolle, Hubert koch, Walter Schwarg, Gunter Knye.[3]

Though Lauscha has been historically one of the most fertile areas for lampworking, it is not the only one. In Dresden lived the Blaskas, perhaps the best known family to work in this medium. Leopold and his son Rudolf Blaskas undertook a lifelong involvement with the production of the Ware Collection which is now in the Harvard Botanical Museum. Truly, this monumental accomplishment was a tour de force of any period in the history of lampworking. Using just very simple tools and a primitive cross fired burner, fueled by solid paraffin with air provided by a foot powered bellows, glass flowers with exquisite realism were created.[4]

It is beyond the scope of this essay to mention all of the artists that use flameworking as a method of working with glass, but for historical perspective, a few contemporary individuals shall be mentioned to represent the whole.

Just as Albin Schaedel, after WWII, developed lampworking to a high level of sophistication in East Germany, Kurt Walstab is now a leading figure to carry on the traditions of this technique in West Germany. Recently, he has been exhibiting and demonstrating throughout the USA.

Pavel Molnar is another artist working in West Germany. His use of subtle, delicate pastel colors is incorporated in the imagery of the vessel, as if to suggest distant cloud formations. In addition, Pavel is now using both lampworked glass and furnace glass together as one coherent, unified form.

Rosemarie Lierke, has also used the vessel format in a rather curious way with incompatable glasses to achieve a highly unusual crackled effect.

Perhaps one of the most significant sculptural lampworking done today is that of the Czech artist Vera Liskova. Her use of blown sections of glass fused together, often suspended in mid-air, or precariously poised on points resting on the ground, is a sublime statement revealing the potential of this medium.

Ginny Ruffner is another artist to use lampworking as a sculptural medium. She has employed solid glass to create impressive free standing forms. Often the glass is sandblasted which imparts the impression of sensualness with a simultaneous threatening quality by using pointed forms incorporated into her compositions.

The Italian artist, Gianni Toso, has created humorously expressionistic rabbinical figures which are some of the most unique solid lampworked glass made today; one can make a vague comparison between this work and the Nevers Figurines of 17th century France.

One would expect the use of the lamp, as well as other techniques of glassworking to establish itself in the United States. Indeed, many lampworkers did

Figure 1
Mark Pieser. Torch decorating a large vessel.

emigrate from Europe to the U.S. during the 19th and 20th centuries. The primary use of flameworking has been for scientific purposes, and as such has excelled. Unfortunately, on the whole, there has not been a simultaneous level of excellence in its artistic application. However, coming from the scientific traditions are two significant artists. Hans Frabel's playful, pop-art forms, represented in crystal borosilicate glass, has established sculptural lampworking as a significant art form. Paul Stankard's scientific background has enabled him to execute extraordinary life like floral forms, frozen in optical crystal.

Some of the old world techniques of using soda lime and lead glass tubing in conjunction with cross fired burners survive to a limited extent in this country. Such individuals as Jean and Donavon Boutz still use this approach.

Perhaps the finest use of lampworking in the U.S. is in conjunction with furnace glass working. Such artists as Joel Philip Myers and Mark Pieser have used propane torches to apply decorative elements to large vessels (Figure 1).

It will be interesting to see the future development of lampworking in the U.S. Will a tradition be established as in Europe or will the scientific use of the technique remain supreme? Ultimately, this will be determined by you, the future artists of this country.

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4 Schultes, Davis, Burger. *The Glass Flowers at Harvard*. E.P. Dutton, Inc., N.Y., 1982

The basic technique of lampworking is covered extensively in numerous technical books on the subject. Probably the best book from a purely technical standpoint for the glass artist is: Creative Glassblowing, by James E. Hammesfahr and Clair L. Stong (W.H. Freeman and Co.).

It would be redundant to go into detail on specific methods of flameworking. However, since the technique of glass montage is not covered in any other source material, I thought it would be informative to mention the method. A word of caution is added to preserve your sanity! I would not attempt this approach of lampworking without mastery of the skill.

This essay would not be complete if I did not comment on my own work in glass. I have had a varied background with the material. My first experience with glass can be traced to a scientific lampworking course I took in the early 1970's at Eastern Michigan University. This formed the basis for my work today. I have also worked in stained glass with Patrick Reyntiens and in furnace glass with Herbert Babcock at the Center for Creative Studies in Detroit.

After leaving CCS in 1981, to build my own studio in the country, I had the desire to continue working with glass but did not have the studio space. During construction of a new studio, I had to improvise. Since the equipment is simple, requires so little space, and is relatively inexpensive, I set up a small lampworking studio in my basement, primarily making Christmas ornaments.

My skill developed, with the help of Kurt Walstabe, to the point that I can work successfully with the vessel format. I am currently using both soda lime and lead glass in the form of 25mm neon sign tubing. I also use Kugler and Zimmermann rods and powder. To achieve a patina finish, I sometimes use black copper oxide, applied to the glass tubing with a paint brush, before it is worked in the flame. With some modification, I can use most of the major techniques of furnace glass with those of lampworking.

Figure 2
Montage technique (a)

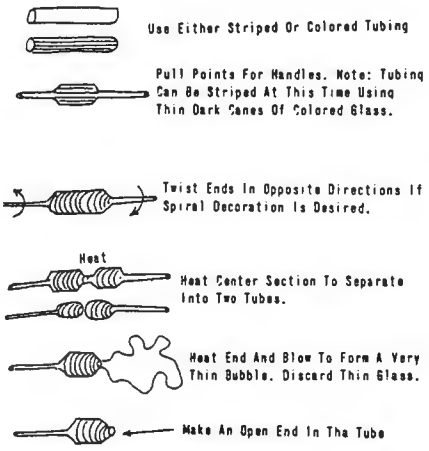

Figure 3
Montage technique (b)

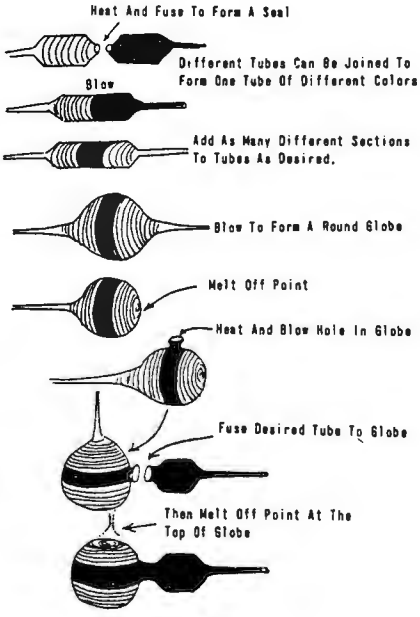

Figure 4
Montage technique (c)

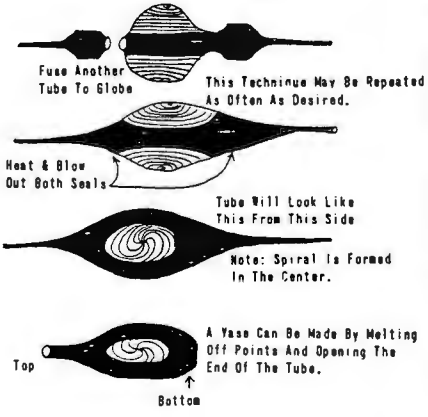

Figure 5
A Birkhill vessel; montage technique.

Fatigue Failure of Silica Based Glasses

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Static fatigue of glass may be described as the time dependant loss of strength of the glass under load. This behaviour is a result of a corrosive environment acting to enhance flow growth in the glass piece. Under certain conditions, catastrophic failure may occur. In consideration of this, stress corrosion behaviour is an important criterion in the design or use of a glass in a load bearing application.

Considerable effort has been extended to the study of stress corrosion in glasses. Glasses studied range from that used in the space shuttle windows[1] to a glass used for orthopaedic prosthesis[2]. Of perhaps greater import to the glassblower is the stress corrosion behaviour of the more familiar silica based glasses. Reaction or pressure vessels and glass laboratory systems made by the glassblower commonly encounter corrosive environments in service. Perhaps the most damaging, and yet least expected, corrosive species is water.

Ten different mechanisms of corrosion have been identified for the soda-lime-silica glass/water system. Most of these are also applicable to other glasses. The mechanisms as outlined by Hench[3] are listed in Table 1.

Table 1 *Mechanisms of Glass Corrosion*

1	Ion Exchange or Selective Leaching	Stage 1 Corrosion
2	Network Dissolution	Stage 2 Corrosion
3	Pitting	Localized Network Dissolution
4	Solution Concentration	Concentration of glass constituents in solution retarding specific corrosion
5	Precipitation	Insoluble compounds in solution precipitate onto surface of glass
6	Stable Film Formation	Surface layer formed from initial corrosion more corrosion resistant than bulk
7	Surface Layer Exfoliation	Large strains induced in surface layer lead to cracking and exfoliation
8	Weathering	Reaction to environment of water vapour and reactive gases (i.e. CO_2)
9	Stress Corrosion	Enhanced corrosion due to stress field
10	Erosion Corrosion	Physical erosion of surface layer

Of these ten, numbers 1 and 2 are basic corrosion processes. Numbers 3 through 10 list mechanisms of corrosion dependent on, or consequences of, 1 and 2 occurring under certain conditions. Excellent reviews of the basic corrosion processes, selective leaching and network dissolution may be found in *Corrosion of Glass* by Clark, Pantano and Hench, and "Physical Chemistry of Glass Surfaces", *Journal of Non-Crystalline Solids* 28(1978)83-105 by Hench and Clark.

Clark[4] gives an illustrative description of Stage 1 and Stage 2 glass corrosion for soda-silica glasses. Using a 2-dimensional schematic representation of the 3-dimensional random network structure of glass, the basic mechanisms of glass corrosion are presented in a simplified form. Clark's representation is given in Figure 1.

The initial stage of corrosion involves ion exchange between the alkali ions of the glass and hydrogen ions of solution.

$$SiONa(g) + H_2O \rightleftharpoons SiOH(g) + NaOH$$

Stage 1 corrosion is predominant at low pH values. For pH > 9 the contribution from Stage 2 corrosion becomes greater (both Stage 1 and Stage 2 corrosion may occur simultaneously over a wide range of pH values. The relative magnitude of each mechanism will change with changing pH of the corrosive environment).

$$\begin{array}{l} \text{O} \quad \text{O} \\ \text{-O-Si-O-Si-OH} \\ \text{O} \quad \text{O (g)} \end{array} + 4OH^-(\text{sol'n}) \rightleftharpoons \begin{array}{l} \text{O} \\ \text{-O-Si-OH} \\ \text{O (g)} \end{array} + \begin{array}{l} \text{OH} \\ \text{OH-Si-OH} \\ \text{OH (sol'n)} \end{array}$$

Stress corrosion, or static fatigue of glass results when these corrosion mechanisms occur under a localized stress field. On the surface of a glass there are numerous small flaws or cracks. The brittle nature of the material leads to extremely high stresses being induced at the tips of these flaws when the glass body is subjected to a tensile, or crack opening, force.

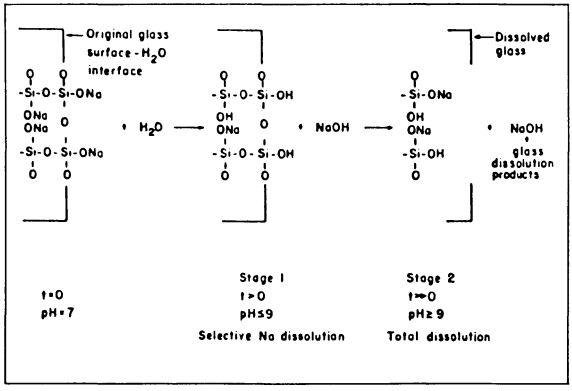


Figure 1 *Corrosion of Glass* D.E. Clark, G.G. Pantano Jr., L.L. Hench, Magazines for Industry.

Inglis[5], considering a surface crack in a large glass plate, developed an expression to calculate the magnitude of the crack tip stress (Figure 2).

The radius of curvature of the crack tip is considered to be of the same order of magnitude as the bond distances in the silicate glass network. By Inglis' analysis, this means tremendous magnification in the stress level acting at the very tip of the flaw.

Lawn[6] and Michalski's[7] views differ from Inglis' in their representation of the crack tip in glass. The "atomistic crack" model represents the brittle crack as slits joined at the tip by "linkage" bonds. Under the influence of the high stress fields, the bond at the crack tip, represented as a spring element, is stretched beyond its linear response range (shown in Figure 3).

Michalski performed molecular orbital calculations to estimate bond energies and electron distributions which would give information of bond strength and reactivity as a function of bond strain. He found that strain could weaken and transform the Si-O bond to a reactive adsorption site.

The stress corrosion model of Michalski and Freiman[8] details a three stage reaction of water with the strained Si-O-Si bond (Figure 4).

Stage 1 Strained crack tip bond adsorbs water molecule. Lone pair of electrons on oxygen of water interacts with silica, hydrogen proton forms hydrogen bond with bridging oxygen of glass.

Stage 2 Cleaving of Si-O and O-H bonds to form two hydrogen bonded silanol groups.

Stage 3 Cleaving of hydrogen bond and consequential fracture at crack tip.

Sequential rupturing of freshly exposed crack tip bonds leads to crack growth and static fatigue of the glass.

Figure 2
$\sigma_m = 2\sigma(c/\rho)^{1/2}$
σ_m = crack tip stress
σ = applied stress
ρ = radius of curvature of crack tip

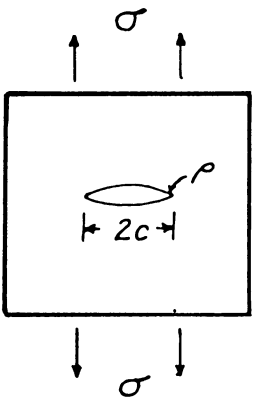
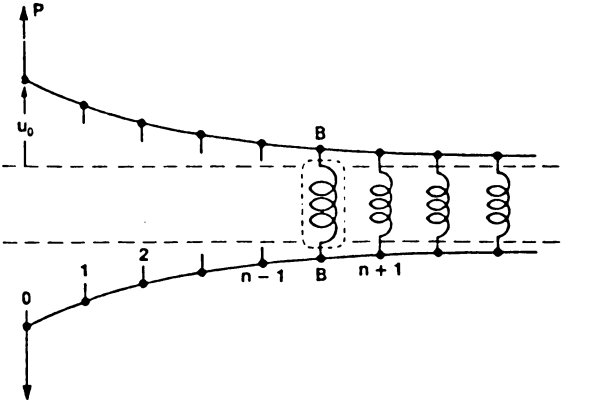


Figure 3 "Physics of Fracture"
B.R. Lawn, J. Amer. Ceram. Soc., Vol 66, No.2, 1983.



The accelerating influence of water vapour on crack growth is apparent on comparison of the two curves plotted in Figure 5 for the stress corrosion of a soda-lime silica glass. The curve for 100% relative humidity test environment is displaced to considerably higher crack velocities in relation to the results for a "dry" environment at a given stress intensity. (K, the stress intensity, is a function of the applied stress, σ, the flaw size, c, and a geometric factor, Y, for the particular test configuration).

In Figure 5, three distinct regions of crack growth behaviour are evident, these labelled I, II, and III. In Region I at low stress intensities, the rate of crack growth is limited by the corrosive reaction occurring at the crack tip. With increasing stress intensity, the crack velocity reaches the plateau of Region II. In this stress independent region the crack growth rate is limited by the time for migration of the corrosive species to the crack tip. At these stress levels the corrosion reaction itself is rapid. In Region III, the contribution to fracture by the corrosion process is negligible. The stress levels are sufficiently high to break the Si-O network bonds. Crack velocities in Region III are very high and quickly approach the limit of catastrophic failure with increasing stress.

Figure 4
Slow Fracture Model Based on Strained Silicate Structures
T.A. Michalske, B.C. Bunker. J. Appl. Phys. 56(10), 1984.

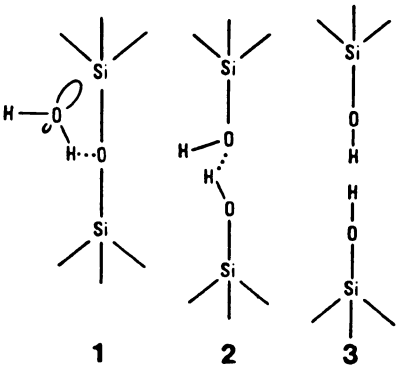


Figure 5
Influence of Water Vapor on Crack Propagation in Soda-Lime Glass
S.M. Wiederhorn, J. Amer. Ceram. Soc., Vol 50, No.8, 1967.

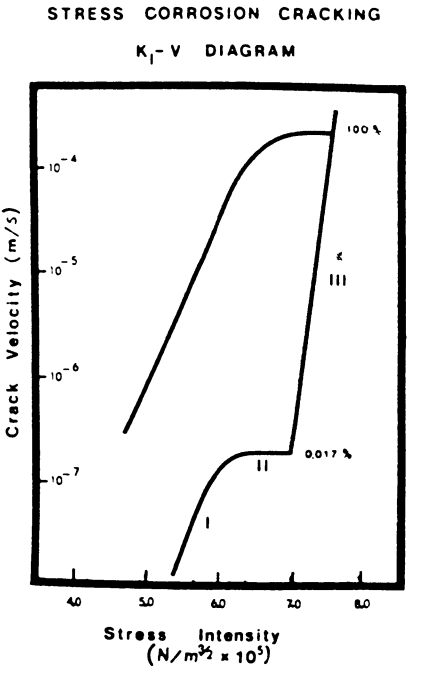


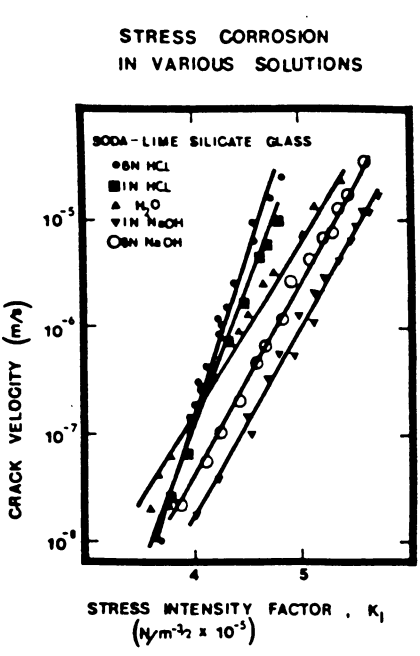
Figure 6 displays Weiderhorn's results for the stress corrosion experiments of a number of silica based glasses. The effects of composition of stress corrosion behaviour are illustrated by the differing slopes and positions of the curves of Region I behaviour. Increased alkali content enhances stress corrosion; compare the curves for silica and soda-lime silica glasses. Leaching of the alkali by Stage 1 corrosion leaves an open and weakened glass structure susceptible to static fatigue. Corrosion resistance is enhanced with the addition of alumina by the formation of a stable surface layer. The curve of log v versus K for the aluminosilicate glass is of lower slope than that of pure silica.

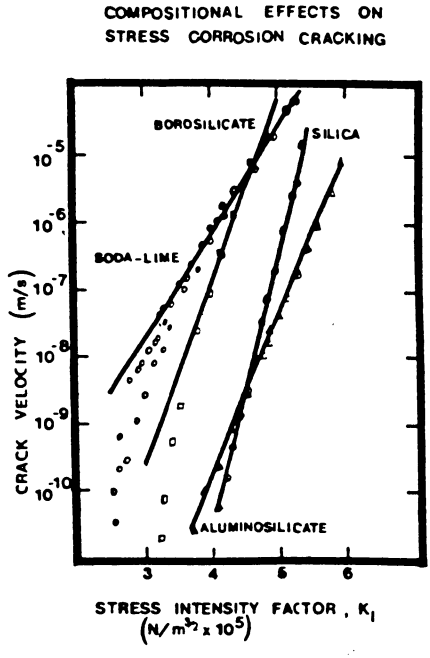
Besides water, other common laboratory solutions cause static fatigue of glass. Examples of these are presented in Figure 7. Other reactive species include NH_3, CH_3, NH_2, CH_3OH, and BCl_3. Michalski[8] proposes that species having lone pair orbitals opposite proton donor sites can chemisorb on the strained Si-O bonds and promote stress corrosion.

The K-v curves generated from stress corrosion experiments can be used to make predictions of the lifetime of a glass in service. Important parameters are

Figure 6 *Environmental Stress Corrosion Cracking of Glass*
S.M. Wiederhorn, *Corrosion Fatigue Chemistry, Mechanics and Microstructure*, June 1071, U. of Connecticut.

Figure 7 *Effect of Electrolyte pH on Crack Propagation in Glass*
S.M. Wiederhorn, H.Johnson, J. Amer. Ceram. Soc., Vol.56, No.6, 1973.





noted on the general K-v curve of Figure 8. In Region I crack velocity can be expressed $v = AK^n$. In this empirical relationship A and n are constants dependent on glass composition and the corrosive species. K_{1c}, the critical stress intensity is the stress required for fracture in a perfectly dry atmosphere under Mode 1, or purely crack opening, stress.

Using $K_1 = \sigma Y\sqrt{c}$ and $v = AK^n$ and integrating $v = da/dt$ from initial conditions to failure for a constant applied load σa, one obtains the time to failure:

$$t_f = 2K_{1i}^{2-n} / A\sigma a^2 Y^2 (n-2)$$

substituting: $K_{1i} = \frac{\sigma_a}{\sigma_{1c}} K_{1c}$

$$t_f = 2\sigma a^{-n}(K_{1c}/\sigma_{1c})^{2-n} / AY^2(n-2)$$

It must be stressed that the above equation may be used only as an estimate of failure time. This is due to the dependence of t_f on the initial crack size, a value which varies considerably between glass pieces. Statistical analysis must be per-

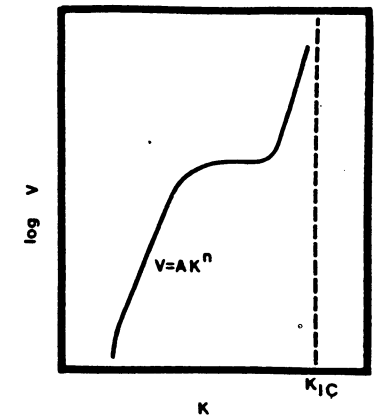


Figure 8 *Lifetime Prediction*

Time to failure under constant load

using $K_I = \sigma Y\sqrt{a}$ $V = AK^n$

integrating $V = \frac{da}{dt}$

$$t_f = 2K_{Ii}^{2-n} / A\sigma_a^2 Y^2 (n-2)$$

substituting $K_{Ii} = \left(\frac{\sigma_a}{\sigma_{IC}}\right) K_{IC}$

$$t_f = 2\sigma_a^{-n} (K_{IC}/\sigma_{IC})^{2-n} / AY^2 (n-2)$$

formed to obtain a more confident estimate of lifetime. The reader is referred to articles on Weibull analysis and prooftesting for comprehensive discussion of this subject.

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Calibration of Hot Cathode Ionization Gauges

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ABSTRACT

To assure accurate measurement of pressures ranging from 10^{-4} torr to 10^{-12} torr by hot cathode ionization gauges, it is necessary to study the linearity of positive ion current vs. pressure, which is defined as the calibration. Usually the calibration is done against the standard McLeod gauge. This method requires good equipment and experience so that the secondary standard ionization gauge method can be employed for the calibration of other ionization gauges in accordance with the Japanese Industrial Standard (JIS-Z-8570). Calibration of Bayard-Alpert type ionization gauges was made against the secondary standard gauge in the pressure range 10^{-4} torr to 10^{-7} torr. The positive ion current vs. pressure of the Bayard-Alpert gauge yielded linearity. One of the gauges had a sensitivity of 10.6 $torr^{-1}$.

INTRODUCTION

Following the original work of Buckley[1] in 1916, various types of hot cathode ionization gauges were developed by Soh[2], Found and Dushman[3] and others[4-8]. Today it is the most widely used instrument for the measurement of pressures ranging from 10^{-3} torr to 10-12 torr. However, accurate calibration requires the determination of the dependence of ion current on pressure.

Usually the calibration is made in the 10^{-4} torr range using a McLeod gauge. At pressures lower than 10^{-4} torr the conductance[9] and other methods[10-16] are employed. These methods require sophisticated equipment and expertise and often are time consuming. These methods, therefore, are not convenient. Nor are they recognized as acceptable national standards.

In 1958 the Vacuum Society of Japan developed a McLeod gauge of high accuracy. It was approved by the MITI (Ministry of International Trade and Industry) as the acceptable national standard gauge and was installed at one of the government testing laboratories.

In 1962 the calibration method for the ionization gauges was standardized and published as the Japanese Industrial Standard. One of its features is that various types of ionization gauges are calibrated against the secondary standard ionization gauge which were in turn calibrated against the standard McLeod

gauge within an accuracy of ± 3%. This paper deals with the calibration of hot cathode ionization gauges by using these secondary standard gauges.

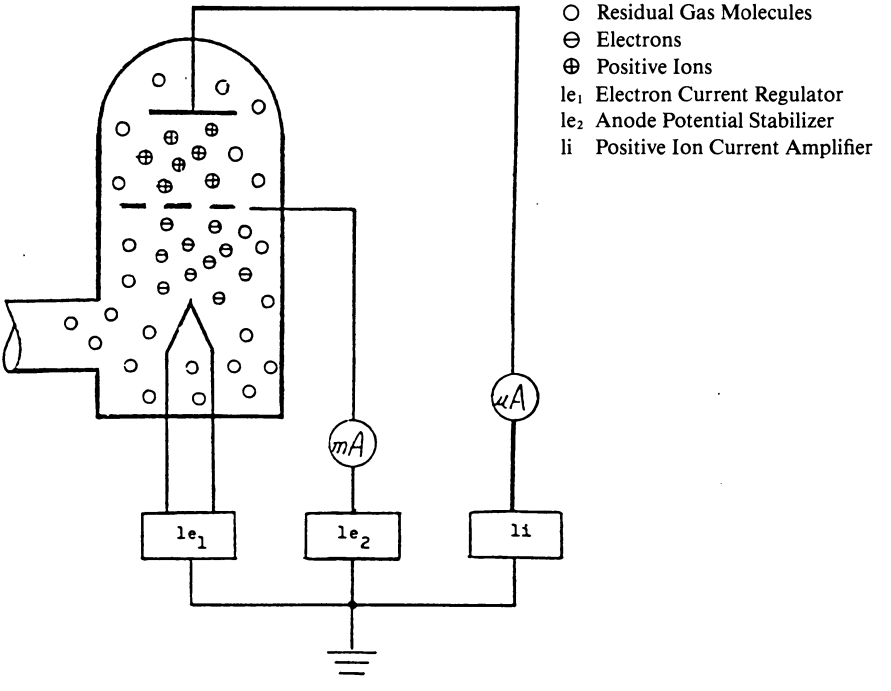
PRELIMINARY REMARKS

In any type of hot cathode ionization gauge, electrons produced at pressures lower than 10^{-3} torr by the hot cathode or filament will collide with the residual gas molecules and ionize them in the space between the cathode and anode as shown in Figure 1. In the ionization gauge, at a given anode potential, the positive ions, Ii, produced by a given number of electrons, Ie, are directly proportional to the number of residual gas molecules. Then pressure, P, to be measured by the gauge may be written in the form as follow:

$$Ii = S \times le \times P \text{ or } P = \frac{li}{S \times le} \qquad (1)$$

where S is a constant which depends upon the geometry of the gauge, the anode potential and the nature of gas. This factor S is defined as the sensitivity, and is expressed as the ratio of the positive ion current, in amperes, to the pressure of the residual gas in torr per ampere of the electron current, that is

Figure 1
Principal of the hot cathode ionization gauge



$$S = \frac{li}{P \times le} \qquad (2)$$

The sensitivity S is one of the important parameters and a number of attempts have been made to calculate it from the basic ionization data present, notably by Morgulis and Reynolds[17]. However, the complicated electron trajectories make the calculation most difficult and no successful literature reference has been found.

For newly built ionization gauges, the relationship between positive ion current and pressure over a wide range is studied by varying the anode and the collector potentials, and the electron current. The most suitable potentials are obtained from these data.

THE SECONDARY STANDARD IONIZATION GAUGE

The secondary standard ionization gauge is shown in Figure 2. It is one of the conventional hot cathode ionization gauges whose electrodes are mounted on Kovar leads sealed by the sintering method. The gauges are calibrated against the standard McLeod gauge for N_2 gas at pressures ranging from 5×10^{-3} torr to 10^{-4} torr which is available for that purpose from the Vacuum Society of Japan.

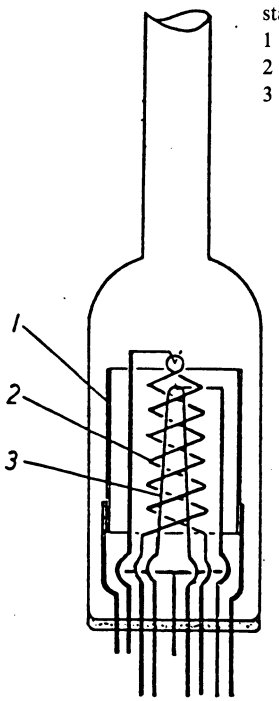


Figure 2
Schematic, VS-1 secondary standard ionization gauge
1 Positive Ion Collector
2 Anode (Grid)
3 Cathode (Filament)

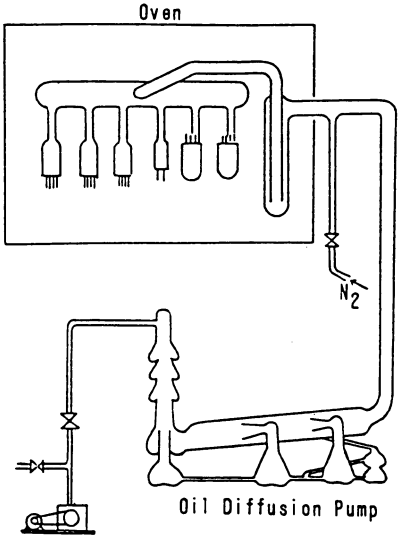
Under normal operation, the potentials applied to the electrodes of the gauge are + 125 V for the anode and − 25 V for the positive ion collector with an electron current of 1 mA. The sensitivities of the gauges are between 19 $torr^{-1}$ and 20 $torr^{-1}$ for N_2 gas and are very stable at pressures ranging from 5×10^{-3} torr to 5×10^{-7} torr.

CALIBRATION PROCEDURE

The calibration of other ionization gauges against the secondary standard gauge is carried out for N_2 gas at pressures ranging from 10^{-3} torr to 10^{-7} torr in accordance with the Japanese Industrial Standard (JIS-Z-8570) "Method of Calibration for Vacuum Gauges".

Figure 3 shows a suitable apparatus for laboratory use. The ionization gauges are sealed onto the manifold in the oven by glassblowing. Prior to the calibration, they are baked at a temperature of 400°C for a few hours to drive out the adsorbed gases, mostly water vapour. While cooling, further degassing of the gauge electrodes is carried out by electron bombardment or by high-frequency heating. The gauges are then turned on and the electronics are allowed an hour to stabilize. The purity of the N_2 gas and a stable room temperature are important factors for accurate calibration. N_2 gas is introduced by a controlling needle valve on the manifold. Quasi-static pressures of N_2 gas for the calibration are attained and can be expressed in the form

Figure 3
Calibration apparatus



$$P = \frac{\text{throughput (torr lit. sec}^{-1})}{\text{pumping speed (lit. sec}^{-1})} \qquad (3)$$

Under quasi-static condition, the positive ion currents of the gauges are measured by dc microammeters of high accuracy. The sensitivities of the calibrating gauges are calculated from the positive ion current by equation (2). As the sensitivity of the secondary standard gauge is known, the pressure, P, at each calibration is easily calculated from the positive ion current of the secondary standard gauge as follows:

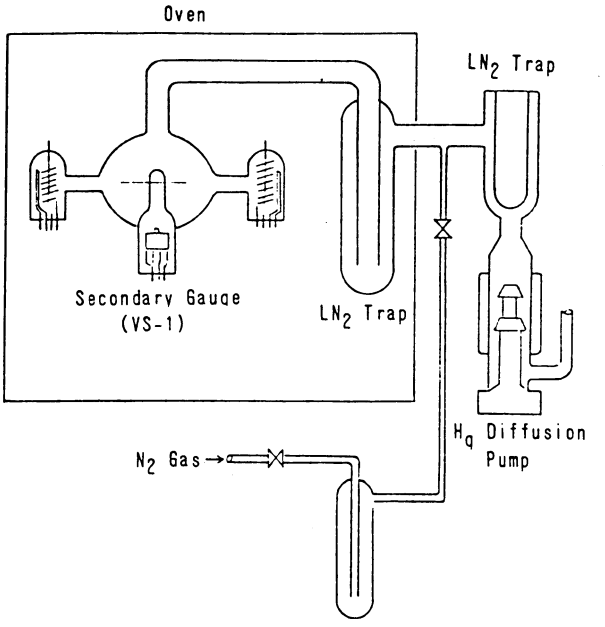
$$P = \frac{\text{ion current } (\times 10^{-6} \text{ ampere})}{\text{sensitivity (torr}^{-1}) \times 1 \times 10^{-3} \text{ ampere}} \qquad (4)$$

CALIBRATION OF BAYARD-ALPERT GAUGES

Calibration of Bayard-Alpert type ionization gauges was done using the secondary standard gauge method.

The secondary standard gauge obtained from the Vacuum Society of Japan had a sensitivity of 19 $torr^{-1}$ for N_2 gas. The Bayard-Alpert gauges were of conventional type and obtained from a dealer of vacuum apparatus. They were sealed by a hand torch onto the manifold of the calibration apparatus shown in Figure 4. The manifold was fabricated from borosilicate glass. The manifold

Figure 4
Bayard-Alpert gauge calibration apparatus



with cold trap was installed in an electric oven. In order to avoid the contamination of an oil diffusion pump, a vertical type of two-stage mercury diffusion pump was used with a pumping capacity of 50 lit. sec^{-1}.

Following a leakage check by Tesla coil, the ionization gauges were baked at a temperature of 400°C for 4 hours. While cooling, the gauge electrodes were degassed for 30 minutes by electron bombardment at potentials of 850 V dc at

Table 1
Calibration of a Bayard-Alpert type ionization gauge

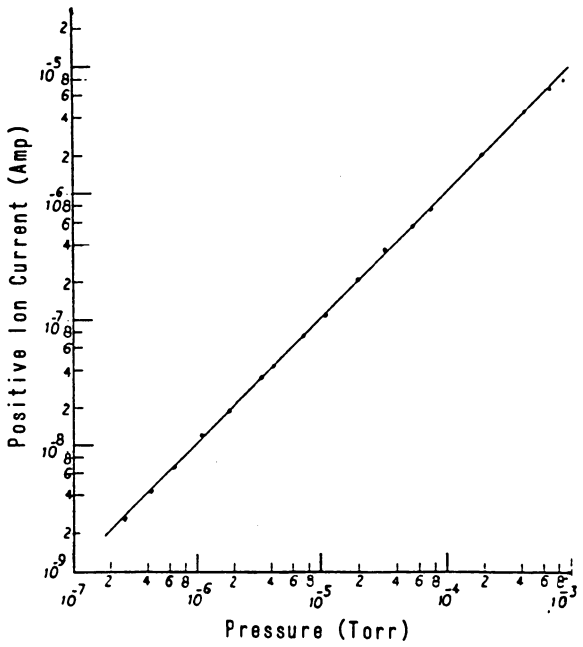
Secondary Standard Ionization Gauge		Bayard-Alpert Type Ionization Gauge	
Ii (Amp)	P (Torr)	Ii (Amp)	Sensitivity
1.6×10^{-5}	8.4×10^{-4}	7.8×10^{-6}	9.3
1.2×10^{-5}	6.3×10^{-4}	6.5×10^{-6}	10.3
8.0×10^{-6}	4.2×10^{-4}	4.5×10^{-6}	10.7
3.7×10^{-6}	1.9×10^{-4}	2.0×10^{-6}	10.3
1.4×10^{-6}	7.4×10^{-5}	7.6×10^{-7}	10.3
1.0×10^{-6}	5.3×10^{-5}	5.6×10^{-7}	10.6
6.2×10^{-7}	3.2×10^{-5}	3.4×10^{-7}	10.5
3.8×10^{-7}	2.0×10^{-5}	2.1×10^{-7}	10.5
2.1×10^{-7}	1.1×10^{-5}	1.1×10^{-7}	10.0
1.4×10^{-7}	7.4×10^{-6}	7.8×10^{-8}	10.6
8.0×10^{-8}	4.2×10^{-6}	4.5×10^{-8}	10.7
6.6×10^{-8}	3.5×10^{-6}	3.5×10^{-8}	10.1
3.6×10^{-8}	1.9×10^{-6}	1.9×10^{-8}	10.0
2.1×10^{-8}	1.1×10^{-6}	1.2×10^{-8}	10.9
1.3×10^{-8}	6.6×10^{-7}	6.6×10^{-9}	10.0
8.0×10^{-9}	4.2×10^{-7}	4.3×10^{-9}	10.2
5.0×10^{-9}	2.6×10^{-7}	2.7×10^{-9}	10.2

100 mA. The final pressures obtained at room temperature were 3×10^{-8} torr by one of the Bayard-Alpert gauges.

The potentials applied to the electrodes of the Bayard-Alpert gauges were + 150 V for the anode and − 40 V for the positive ion collector with an electron current of 1 mA. The operating circuits were allowed to stabilize for one hour. Then, N_2 gas was introduced through a needle valve via a liquid nitrogen cooled trap to eliminate any condensable vapours. By adjusting the needle valve, the desired quasi-static pressures for the calibration were attained. At each calibration they were recorded by a mV recorder of high accuracy connected to one of the measuring circuits.

Positive ion current vs. pressure of the secondary standard gauge and the Bayard-Alpert gauges was measured using multi-range dc microammeters. Table 1 shows one example of a calibration. Pressure readings of the secondary standard gauge were calculated from the positive ion current of the gauge by equation (1), while sensitivities of the Bayard-Alpert gauge were calculated from the positive ion current of the gauge by equation (2). The average value of the sensitivity was 10.6 $torr^{-1}$. Figure 5 shows the plot of the positive ion current of the Bayard-Alpert gauge vs. pressure of the secondary standard gauge, which yields linearity in the pressure range between 10^{-4} torr and 10^{-7} torr.

Figure 5
Calibration curve of a Bayard-Alpert gauge for N_2



CONCLUSION

The conclusion of the study on the calibration of hot cathode ionization gauges by using the secondary standard gauge method is as follow:

1 the secondary standard ionization gauge is an acceptable standard instrument for the calibration of other ionization gauges for scientific and industrial uses.

2 the sensitivity of the Bayard-Alpert gauges at pressures higher than 10^{-4} torr is unreliable since the gauge is non-linear.

3 the ionization gauge can act as a gas source and proper degassing procedures should be adopted in the course of the measurement. It can also act as a pump and these facts should be understood.

4 as the sensitivity of ionization gauges depends upon the nature of gas, the purity of the calibrating gas should be checked by a mass spectrometer.

5 condensable vapours in the calibrating gas should be removed by a liquid nitrogen cooled trap.

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Laser Welding of Quartz Brewster Windows

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ABSTRACT

A technique is described whereby quartz windows can be sealed to quartz tubing at the Brewster angle by laser welding. This technique allows the windows to be sealed without destroying their interferometric accuracy and without the loss of UV transmission associated with flame welding.

HISTORICAL BACKGROUND

The Brewster angle for quartz is 34.5 degrees. There have been several previous approaches to sealing quartz Brewster windows. Those reported in the literature include:

1 a sphere can be blown on the end of a piece of quartz tubing, cut at 34.5 degrees and then mounted on the lathe at the same angle. In this manner the window can be sealed perpendicular to the lathe spindle.

2 a second method, described by Baird, Taylor and Turner[1], and demonstrated a few times at ASGS symposiums, is referred to as diffusion bonding. The elliptical surface of the tubing cut at the Brewster angle is polished to optical flatness. The window of the same shape is held flat against the tubing and force is applied by vacuum or weight. The seal is made with a small sharp flame.

3 seals for ultraviolet use can be made without the flame by using pressure-sensitive glass frit tape as described by Kitty Ettre at the 15th Annual ASGS Symposium in 1970[2]. Suprasil quartz has a very good transmission in the UV (50% at 1700 nanometers). This is due to the absence of water in the quartz structure. When Suprasil is heated in a flame, water of combustion is absorbed, cutting the UV transmission. A simple tooling for making this type of seal is shown in Figures 1, 2, and 3. It consists of a quartz cradle, such that the seal can be made in a horizontal position under the quartz weights. Glass in the tape is Corning #7070 which will withstand 600°C.

LASER WELDING

CO_2 laser welding avoids the water of combustion, and can be performed in an inert atmosphere, if needed. A very small area of the edge of the window is heated so there is minimal optical distortion. The pioneering work of CO_2 laser

welding of quartz was done by Pfitzer and Turner of the National Research Council in Ottawa, Canada[3].

To secure an inexpensive Suprasil window having good optical quality for use on 12 mm diameter tubing, 5 cm × 5 cm × 1.5 mm thick squares were used. Interferograms of the transmission quality of each square were made (Figure 4). The squares were then cut in a manner so that the best optical flats of rectangular shape could be selected. Ten rectangular pieces were then waxed to a metal shaft 12 mm in diameter cut at 34.5 degrees. The batch was then ground to the size of the shaft with a diamond tool. Interferograms were made again of each window so that the best could be selected.

The laser welding facility was built around a Coherent Radiation Co. CO_2 continuous-wave laser. The laser, Model 41, is capable of delivering 200 watts

Figure 1

Figure 2

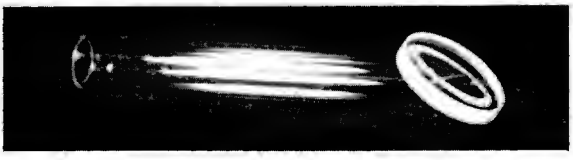

Figure 3

of beam power. To be able to direct the invisible CO_2 laser beam, an optical integration system using a small HeNe laser as a designator was constructed using a zinc selenide beamsplitter. The beamsplitter is also used to divert 2% of the CO_2 beam for use in laser alignment. This also allows optimization of the shape of the beam, since no lens is used in this procedure. The beam is then reflected by molybdenum mirrors via a Lucite tube into the containment box (Figure 5). The Lucite provides a safety shield. The vertical control of the beam position is outside of the Lucite box; allowing the beam to be moved up and down during the welding. The containment box (Figure 6) is lined with refractory bricks. Mirrors in the box allow the operator to see through the Lucite

Figure 4

Figure 5

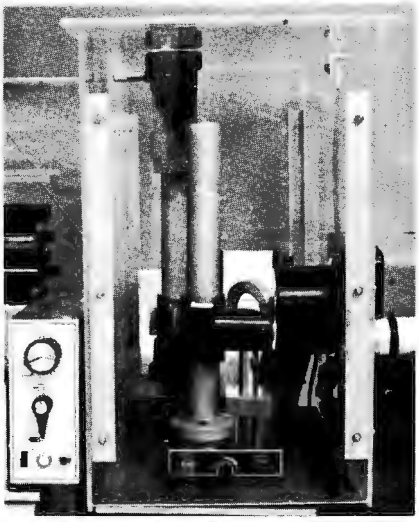

Figure 6

window on the box cover and follow the welding progress from different angles. The containment box holds the drive mechanism operating the turntable. A DC motor, operating through the drive train, turns the table at a minimum speed of 1 rpm. The operator can control the speed, the direction of the tuntable, and the vertical flame. The support on the turntable holds the tubing at an angle such that the window can be welded in a horizontal position. The HeNe beam is used to position the beam precisely on the window edge. Typically, welding is performed with 60-80 watts of beam power and at a rotational speed of 1.5 rpm.

To weld a window in the middle of the tubing (Figure 7) is more complicated.

Figure 7

Figure 8

Figure 9

First, the window is tacked lightly to the tubing in the holder. A short piece of tubing is then placed on the window. The length of this second piece must be such that it stands by itself since there is no provision for support as it rotates in front of the laser beam. Figure 7 illustrates a dual Brewster window assembly employed as insulation for a high temperature laser experiment. Interferograms of the sealed windows on the end of a tube (Figure 8) and in the middle of a tube (Figure 9) show that the optical quality of the window is preserved. For repairing pinholes or breaks in laser welded seals, a paste consisting of pure silica sand suspended in amylacetate has been used successfully.

ACKNOWLEDGEMENTS

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The Effect of Black Light on Silvered and Unsilvered Vessels

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ABSTRACT

This paper discusses the reasons for evacuating and silvering a dewar flask. The insulating properties vary according to whether you use a silvered dewar flask, taped and not taped. Details are provided showing what was used and how the outcome affects us in industry.

INTRODUCTION

It is well known that a silvered and evacuated dewar flask works best for holding liquid N_2. A study to determine if one type of dewar flask would be better than another was made. What changes if any could be undertaken to make the flasks better or worse? Let us take a look and see the results.

APPARATUS

1 Nine dewar flasks made of pyrex brand tubing, using 84 and 64 mm outside diameter tubing. All dewars were 24 cm long.

2 One scale with horizontal lines spaced 1 cm apart and numbered one through 14 cm.

3 Two holders made of 11 mm O.D. tubing with three side arms made of 5 mm rod.

4 Two pointers, made from red plastic drinking straws.

5 Two pieces of styrofoam, 1 cm^3.

6 12 liters of liquid nitrogen.

7 Amber stain by Ceramic Coatings, Inc.

8 Silvering solution by London Labs, Ltd. For silvering technique, refer to my paper in the Proceedings of the 22nd Symposium (1977), pp.85 through 90.

9 One roll black electrician's tape.

10 One roll white tape.

11 A forty-five minute clock.

A comparison was made of each of the nine flasks, two at a time. Liquid nitrogen was placed in each dewar until it was approximately half full. The styrofoam floats with the straw pointers were set into the dewar flasks. Next on top went the 11 mm O.D. holders with the 5 mm rod acting as a guide for the pointers in

the center of the flasks, allowing free up and down movement. Enough liquid nitrogen was then added to each flask so that the top of each pointer was even with the 14 cm mark on the scale. Using the 45 minute clock, changes were recorded every five minutes until the clock reached 0 minutes. A graph was then made showing the evaporation rate of each dewar. Readings at the five minute intervals were plotted, showing the evaporation rate of the liquid nitrogen in each flask.

The first two flasks were both clear with one being evacuated and one open to atmosphere. After five minutes the vacuum clear was 13.5 cm, the open clear was 10 cm. After ten minutes the vacuum clear was 12.5 cm, the open clear was 7 cm. After fifteen minutes the vacuum clear was 12 cm, the open clear was 5 cm. After twenty minutes the vacuum clear was 11 cm, the open clear was 3.5 cm. After twenty-five minutes the vacuum clear was 10.5 cm, the open clear was 2 cm. After thirty minutes, the vacuum clear was 9.75 cm, the open clear was 1 cm. After thirty-five minutes the vacuum clear was 9 cm, the open clear was 0 cm. After forty minutes the vacuum clear was 8.5 cm. After forty-five minutes, the vacuum clear was 8 cm.

The next two flasks used were vacuum strip silvered and open strip silvered. After five minutes the vacuum strip silvered was 13.75 cm, the open strip silvered was 10.5 cm. After ten minutes the vacuum strip silvered was 13.5 cm, the open strip silvered was 8 cm. After fifteen minutes the vacuum strip silvered was 13 cm, the open strip silvered 6 cm. After twenty minutes the vacuum strip silvered was 13 cm, the open strip silvered was 4.5 cm. After twenty-five minutes the vacuum strip silvered was 12.5 cm, the open strip silvered was 3 cm. After thirty minutes the vacuum strip silvered was 12.25 cm, the open strip silvered was 2 cm. After thirty-five minutes the vacuum strip silvered was 12 cm, the open strip silvered was 1 cm. After forty minutes the vacuum strip silvered was 11.75 cm, the open strip silvered was 0 cm. After forty-five minutes the vacuum strip silvered was 11.5 cm.

The next two flasks were vacuum silvered and open silvered. After five minutes the vacuum silvered was 14 cm, the open silvered was 10.5 cm. After ten minutes the vacuum silvered was 13.75 cm, the open silvered was 7.5 cm. After fifteen minutes the vacuum silvered was 13.5 cm, the open silvered was 5.5 cm. After twenty minutes the vacuum silvered was 13.5 cm, the open silvered was 4 cm. After twenty-five minutes the vacuum silvered was 13.5 cm, the open silvered was 2.75 cm. After thirty minutes the vacuum silvered was 13.25 cm, the open silvered was 1.5 cm. After thirty-five minutes the vacuum silvered was 13.25 cm, the open silvered was 0 cm. After forty minutes the vacuum silvered was 13.25 cm, and after forty-five minutes it was 13.25 cm.

The next two flasks were vacuum silvered with black tape and vacuum amber stained. After five minutes the vacuum silvered with black tape was 14 cm, the vacuum amber stained was 13.5 cm. After ten minutes the vacuum silvered with black tape was 13.5 cm, the vacuum amber stained was 12.5 cm. After fifteen minutes the vacuum silvered with black tape was 13.5 cm, the vacuum amber

stained was 11.75 cm. After twenty minutes the vacuum silvered with black tape was 13.5 cm, the vacuum amber stained was 11 cm. After twenty-five minutes the vacuum silvered with black tape was 13.25 cm, the vacuum amber stained was 10.5 cm. After thirty minutes the vacuum silvered with black tape was 13.25 cm, the vacuum amber stained was 9.5 cm. After thirty-five minutes the vacuum silvered with black tape was 13.25 cm, the vacuum amber stained was 9 cm. After forty minutes the vacuum silvered with black tape was 13 cm, the vacuum amber stained was 8.5 cm. After forty-five minutes the vacuum silvered with black tape was 13 cm, the vacuum amber stained was 7.75 cm.

The last dewar flask was vacuum silvered with white tape. After five minutes the vacuum silvered with white tape was 14 cm. After ten minutes it was 14 cm. After fifteen minutes it was 13.75 cm. After twenty minutes it was 13.75 cm. After twenty-five minutes it was 13.5 cm. After thirty minutes it was 13.5 cm. After thirty-five minutes it was 13.5 cm. After forty minutes it was 13.5 cm. And after forty-five minutes it was 13.5 cm.

CONCLUSION

The results of the experiment are indicated on the graph (Figure 1). The differences are interesting. It is obvious that vacuum makes the most difference. Then, the silvered interior is the next most important. What was surprising however was the difference made by black or white tape. The best dewar flask therefore would be silvered and evacuated and wrapped with white tape.

The results in order of performance are:

Best	vacuum silvered with white tape	6th	vacuum amber stained
2nd	vacuum silvered	7th	open strip silvered
3rd	vacuum silvered with black tape	8th	open silvered
4th	vacuum strip silvered	9th	open clear
5th	vacuum clear		

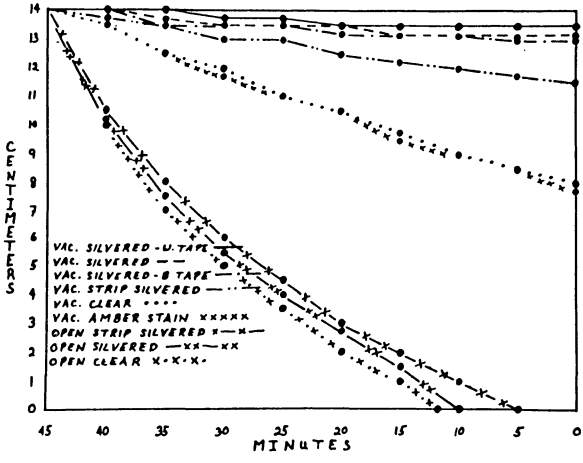

Figure 1
LN_2 Evaporation Rate

Manufacturing Glass Containers

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ABSTRACT
This paper presented present methods of manufacturing glass containers, including raw material handling, melting, and container formation.

Thermally Devitrifying Frits for Matching Fused Silica and ULE™ Titania Silica Glasses and Lightweight Mirror Applications

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ABSTRACT

Two thermally devitrifying frits that closely match the expansions of fused silica and ULE™ titania silica glasses were discussed. Physical properties were presented with particular attention to expansion and bond strength for which special tests were developed. Applications to lightweight mirror fabrication were covered.

Novel IR Sealing

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ABSTRACT

This novel technique is a fast, clean and controllable method for glass to glass, glass to metal and glass to ceramic sealing. It is based on preferential IR heating of the absorbing glass in the 1 micron region. The desired absorption is achieved by the addition of iron oxide and pigments to the ground glass substrate.

The optimized processes were discussed in detail and several applications useful to glassblowers were described.

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