PROCEEDINGS

THE THIRTY-SEVENTH
SYMPOSIUM
ON THE

ART OF SCIENTIFIC GLASSBLOWING

1992

THE
AMERICAN SCIENTIFIC GLASSBLOWERS SOCIETY

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The Thirty-seventh

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Sponsored by
The American Scientific
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THE
AMERICAN SCIENTIFIC GLASSBLOWERS SOCIETY
Toledo, Ohio

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40 Years of Expert Glassblowing Support

James L. Dye
Professor of Chemistry
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Keynote Address to
The American Scientific Glassblowers Society
Dearborn, MI - June 23, 1992

When Keki Mistry asked me nearly two years ago if I would be willing to give a talk to this group, I agreed immediately for two reasons. First, he asked me so far in advance that it caused me no concern at the time; but now I have to deliver! Second, I am so grateful to glassblowers for making my research possible over the past forty years that this is the least I can do to repay the profession for its help. However, I asked myself, "Why me?" Certainly not because I fabricate glassware occasionally - all you have to do is look at my work and it comes through loud and clear that I am no glassblower! On the other hand, ever since my freshman year, when I burned my fingers while making bends in glass, I have had a fascination with glass. More importantly, complex glassware has been a requirement for my research from the beginning. Even as a graduate student when I was heating mercury alloys to the boiling point, the glassblower, "Breezy" Jones, was able to make thin glass diaphragms to sense the pressure. I relied on their high quality to protect me from mercury poisoning!

Over the years I have interacted with more than 20 glassblowers at MSU and on sabbatical leave. The training and skill that they achieve is remarkable. I wish that auto mechanics had the same degree of competence! There have been very few times in nearly 40 years that I have received a defective piece of glassware from the shop.

From the very beginning of my research at MSU I have relied on home-built glassware. There are two features of our work that require this. First, we work with solutions of the alkali metals (lithium, sodium, potassium, rubidium and cesium). Many of you may have seen the result of adding sodium or potassium to water — *immediate reaction*, and in the case of potassium, hydrogen forms and ignites with a bang! Cesium metal is even more reactive. I once accidentally dropped a thin-walled glass bulb containing a small amount of cesium; when it broke it looked as if a flash bulb had gone off! The second reason that glass works so well is that the solutions and solids we study must be kept *cold* at all times. We need to see what is going on, so transparency is critical and we need to work with dry-ice baths, liquid nitrogen, etc. In addition, we need to seal-off samples under vacuum for cold storage. For all of these properties nothing can compete with glass.

Researchers for many years have worked with reactive materials by using a method known as the Schlenk technique. With this method, one evacuates a vessel and fills it with nitrogen or argon to exclude air and moisture. This works well with most air-sensitive reactions, but the method wasn't "clean" enough for our work. So we started work with vacuum lines and completely air-free manipulations, with the added necessity to keep samples cold. The core of this technique is the vacuum line. For years we used conventional greased high vacuum lines and "good-old-Apiezon grease". I had to keep telling graduate students, "Don't over-grease; grease traps organic vapors and can lead to trouble". It seemed sometimes that I "bathed" in benzene in the early days while cleaning stopcocks - and now benzene is on the cancer-suspect list!

We had some interesting times working with vacuum lines but probably nothing beats the entry in the notebook of one of my first graduate students. It seems that Gale Smith had just cleaned and reassembled his vacuum line and was about to pump it out when he noticed a live ant walking along inside. His notebook gives the following advice under the heading, *How to remove a live ant from a vacuum manifold.* The description goes on, "Gently heat the glass at some distance from the ant. When he starts walking in the opposite direction, move the torch past the first tee and take out the stopcock. When the ant gets into the intersection, he will make a right turn and walk right out of the stopcock hole." That procedure was preferable to re-cleaning the manifold!

Although greased lines worked well, when decent, O-ring-sealed Teflon vacuum valves became available, we switched to them and never went back. We now have seven vacuum lines in continuous operation for synthesis. A bit later I will describe what I believe to be the best and most versatile vacuum system available for air-free synthesis.

Without glassblowers like Keki Mistry, Manfred Langer, and Scott Bankroff in our glass shop, our work would be impossible. Oh, we could order a vacuum line, but as you will see, that is not the end of the story. Our syntheses by their nature require that the apparatus be rebuilt after every use. Since glassblowers have played such a crucial role in our work, let me say a little about some I have known.

When I started research and teaching at MSU in 1953, fresh out of graduate school, we had one glassblower named Gene Hood. The name was appropriate, for Gene repaired cars in his free time. It was easy to get him to go out of his way to make complicated glassware if you also trusted him under the hood of your car! He even continued to talk to me after I had him make a seal-off in my lab that ended in a terrific explosion that sent both of us out for first aid. That was when I realized that a glassblower is a colleague, not some nameless person in a shop who makes things according to a drawing submitted through a third party.

The first of five sabbatical leaves that I have taken was in Göttingen, Germany, where they had four glassblowers in the shop. This was in 1961 and most of their work was done with soft glass. Although I may not have understood the German well enough, I think the head glassblower's statement was something like, "Soft glass separates the true professional from the beginner". But, I had to make vacuum seal-offs with sensitive stuff like cesium inside. They probably still refer to me as the "Ugly American" who insisted that they use borosilicate glass. But they were so skillful, and turned work back so fast, that I had little need to do my own glassblowing.

The story was a bit different in 1975-76 when I was on sabbatical leave in Strasbourg, France. For one thing, my French wasn't nearly as good as my German and I couldn't speak Alsacian at all! So I was faced with a language barrier — how do you say "graded seal", "breakable bulb" or "fritted disk" in French? My dictionary didn't have these words! The other problem was the time delay. I didn't have time to wait! What a feeling of desolation — to be forced to do you own glassblowing! Figure 1 shows the kind of thing I was faced with. We call this apparatus the "triple trombone" and I had to make my own! On the right is a commercial break-seal ampoule of an alkali metal such as potassium. Our glassblowers could make the wells just the right size so that 5 grams of melted alkali metal would exactly fill all three. After evacuation to 10^{-5} torr, the bottom of the apparatus and the metal sample were heated, the break-seal was broken, and the metal flowed out to fill the wells. Each "trombone", which contained three tubes, each with a different, pre-measured inside diameter was then sealed off at the constriction and then the metal was melted and coaxed into the tubes, each of which ended up being about half-full. These in turn were divided into a number of small tubes, each containing some potassium. By measuring the length, we could get a good idea of how much metal was

used in each synthesis. Of course, this doesn't work with lithium, which reacts with glass.

The lithium-glass reaction reminds me of another incident on a vacuum line that really gave me a helpless feeling. I once wanted to purify lithium metal by dissolving the lithium metal in liquid ammonia, filtering the solution through a glass frit, and later evaporating the ammonia. I put some chunks of clean lithium, covered with hexane, on top of a frit, evaporated the hexane under vacuum and pumped on the lithium. To be sure it was dry, I heated the lithium gently (I thought) with an external heat lamp. I neglected to recognize that the lithium would absorb the infrared radiation and get hot, but in a good vacuum the heat had no place to go! So the lithium melted and began to react with the glass frit; it "caught fire" inside the vacuum system! Now that's a dilemma — a fire without air, and no way to get at the fire. It got so hot that it burned through the glass - end of experiment!

Oh, yes, I did get a few triple trombones made in France and there is now a story in the lab about this crazy American Full Professor who not only worked in the lab but made funny little pieces of glass equipment that looked awful but seemed to work! After about four months in France, I went to Professor Lehn, with whom I was working, and complained that I spent so much time blowing glass that I couldn't get anything else done. He said "That's easy to fix, I will pay a bonus and you get to have the full services of a glassblower all afternoon every Friday." Money talks - even in French!

I never cease to be amazed at what glassblowers can do - and often it is much more than just working glass with a torch. I remember when we needed a vacuum-tight flow cell to study the optical spectra of solutions just a thousandth of a second after mixing. Previous flow cells had been drilled out of Plexiglas with cemented windows or were made of metal, but we needed them made out of fused silica. To insure complete mixing and turbulent flow without cavitation, we needed the design shown in Figure 2. This required four tangential jets of 1 mm ID each coming into a 2 mm ID fused silica capillary. Then we wanted to separate the streams into four streams and re-combine them again *in a total distance of only 10 mm.* The flow then had to go to the ends of a tube of length 10 mm and through the tube, with windows sealed on the end. The method used by the glassblowers was to "drill" the holes with an Airbrasive® unit, which used a supersonic jet of fine alundum powder, and then to seal in plugs, seal on side-arms, etc. The final product was vacuum-tight and gave absolutely complete mixing. We used these cells successfully for more than 10 years.

Let me turn now to a brief discussion of the research that has attracted so much attention, and how specialized glassware made it possible. As early as 1954 we began to work with alkali metal solutions in liquid ammonia. This required good glassware, but the field had been developed for some time and the methods were rather standard. We also tried using solvents other than liquid ammonia, but the solubility of the alkali metals was so low as to be useless for the synthesis of new materials. Then in the late 60's, several organic chemists, who in 1987 shared the Nobel Prize for their discovery, came up with some remarkable molecules that have a ferocious appetite for the positive alkali metal *cations* such as Na^+, the common ion in table salt. These organic complexants, called *cryptands* and *crown ethers*, completely revolutionized the study of alkali metal solutions. We already knew that an alkali metal such as potassium while not very soluble in a solvent such as ether, dissolved to a *slight* extent to yield K^+ and K^- in solution (K is the symbol for potassium). Without the cryptand present, potassium is virtually insoluble in a solvent such as dimethyl ether. The solubility is less than 0.4 parts per million. The addition of cryptand, however, just "sucks it in" and the solubility increases by a factor of at least 30,000. Now we had *real* metal solutions to work with. We made this discovery in 1970 and it led to the development of two new classes of compounds called

alkalides and *electrides*. Alkalides are crystalline salts in which the negative ion is an *alkali metal anion*, M^-. Electrides are crystalline salts in which the anions are *trapped electrons*. Electrons are the same little charged particles that paint the picture on your TV tube and carry current through the wires to your home — except that, in electrides they are trapped inside of a crystal in equal amounts to the positive ions. There are as many trapped electrons in a one inch cube of an electride as pass through the filament of a 50 watt light bulb in one minute.

In 1974 we were able to produce nicely-formed crystals of a *sodide*. Chemistry textbooks for more than 100 years had said that sodium (Na) could only form the positive ion, Na^+, or the neutral reactive metal, Na, but certainly not an *anion*, Na^-. Now, textbooks have been forced to change, and there is even a color photograph of sodide crystals in a Freshman Chemistry text by Chang that is used at Michigan State and at many other universities.

More recently, we have made crystalline *electrides*. These are so novel that a representation of the crystal structure was used for the cover of *Scientific American* in 1987 and for the cover of one of the world's most prestigious journals, *Nature*, in 1988.

Now, how does glassware fit into this story? Twenty years after our first use of greaseless vacuum lines, things haven't changed a lot, but the details are different. The type of vacuum line now used in our lab is much more convenient than the earlier models. The design, shown schematically in Figure 3, was worked out when I was on sabbatical leave at AT&T Bell Labs in 1982-83, working with Andrea Wayda. She had been brought up on Schlenk-ware and was very adept at using it and at carrying out reactions in an inert atmosphere glove box. We put our heads together and came up with a double manifold, flexible stainless steel vacuum tubing and Ultra Torr® connectors that combined the flexibility of Schlenk-ware with the high-vacuum capabilities of our synthesis methods.

Since the paper we wrote about this design in the Journal of Chemical Education (April 1985) was authored by Andrea Wayda and James L. Dye it has come to be known jokingly as the "Wayda-Dye" vacuum line! Manifolds of this type are now available from several glass companies. We have also written a book chapter that describes the method in detail [A.L. Wayda, P.A. Bianconi and J.L. Dye, in Experimental Organometallic Chemistry, A.L. Wayda and M.Y. Darensbourg, Eds., ACS Symposium 357 (1987) pp. 116-135].

While we think Teflon valves are very useful, they have one drawback that some of you clever people might think about overcoming. When you assemble the valve, air gets trapped between the O-rings, with the result that the initial pump down takes several days until, with an ion gauge, you can avoid a detectable "burp" every time you open the valve. Vincent Nicely in our lab in the late '60's found a solution to the problem by using a double valve but it proved to be too expensive for routine use. I think there are ways around the problem if someone who makes such valves wants to develop the perfect high-vacuum greaseless valve.

In early studies, we went to great lengths to prepare electrides by the only method that worked until 1983 - evaporation of the solvent. This lead to glassware of various types such as "the spider", which had multiple arms for lots of different measurements on powders made from the same solution. The first *crystalline* samples were made in various types of apparatus that permitted sample tubes to be sealed-off under vacuum. but the "work-horse" has been the "K-cell" shown in Figure 4. Keki Mistry, Manfred Langer and Scott Bankroff have made so many of these that they can make them in their sleep!

A photo of this type of cell appeared in the Scientific American article which acknowledged Keki for making it. The Associate Editor was so intrigued by the artistic beauty, yet utility, of this piece of glassware that he had Keki make one to hang as a "mobile" in his office. In this apparatus we can isolate crystalline samples in the four glass (or fused silica) "fingers" into which the sample is finally poured. If you look at this diagram upside down, you can see why it is referred to as the "cow"! Every time we use it, we make vacuum seal offs of the two side-arms, and each of the four fingers. Then it needs to be cleaned and returned to the glass shop for repair. I estimate that since 1975 the shop has made or repaired about 4,250 "cows"!

The K-cells are attached to our vacuum lines by flexible stainless steel tubing. Since the first compounds were made in the mid 70's we have prepared and studied over 40 different types of alkalides and 10 or 12 electrides. We use a lot of dry ice in cooling the cells to make samples. For example, in the fiscal year '90-'91 we used 3,771 lbs. of dry ice - nearly two tons!

What about the future? Times are changing; the "new kids on the block" may not use glass as much as we "old timers" do. Indeed, we have also changed our ways to some extent. We found some years ago that we could make films of alkalides and electrides with a simple piece of glass apparatus. Now we study films in earnest in a complex bell jar apparatus with lots of electronic gadgetry that doesn't look much like the traditional stuff in a chemistry lab! Another simple glass prototype was followed by a complex piece of apparatus that is now used to measure the photoelectric response of alkalides and electrides. The new apparatus has three vacuum pumps (a mechanical pump, a cryopump and a turbomolecular pump). To really pump effectively we use a metal gate valve with an eight-inch opening. These changes in the way we make measurements have not diminished our need for glass synthesis apparatus, as a look at a day's "dirty dishes" would confirm. Indeed, our appetite for K-cells is bigger than ever, now that we need to make so many measurements on the products of synthesis.

In spite of the move to metal systems, plastic ware, disposable pipettes and syringes, etc., there will always be a need for glassblowers. However, the mix of work has already changed and will change more in the future. There will be more metal-to-glass seals, fabrication with special cements, use of machinable ceramic and glass, etc. Glassblowers will expand their expertise to include machine shop work, instrument design and more integration of glass, quartz, sapphire, etc. into finished apparatus. As many of you already do, glassblowers will need to find out more about the objectives of the researchers and get out in the labs and offer help and advice where appropriate. In fact, I have the feeling that the imaginative use of glassware can expand beyond chemistry into many fields that now rely on non-glass equipment.

I hope that I have been able to give you a view of our research that is understandable. When speaking to an audience of non-chemists, I am always afraid that my comments will be too technical. I am reminded of my attempts to describe our research to my 94 year old mother — a mentally sharp lady who is not easily impressed! Recently, I wrote her a letter describing our preparation of small metal particles by using alkalides and electrides. I noted that, "these particles are so small that a trillion of them would fit on the head of a pin." She responded by writing, "That sounds like very difficult research. By the time you count to a trillion your eyes must get awfully tired!"

It has been a real pleasure to attend this interesting meeting. I wish you every success for the rest of the week and a happy 40th birthday to the American Scientific Glassblowers Society.

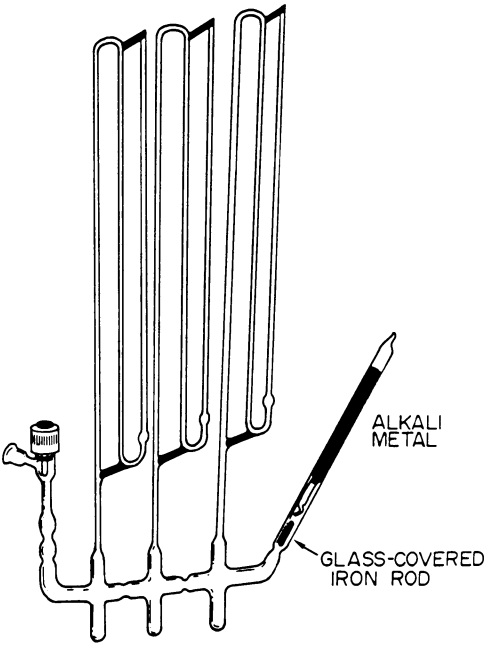

Figure 1. "Triple Trombone" apparatus for distribution of alkali metals into three sizes of pre-measured tubing. Each tube yields 6-8 samples of alkali metal for later use in synthesis.

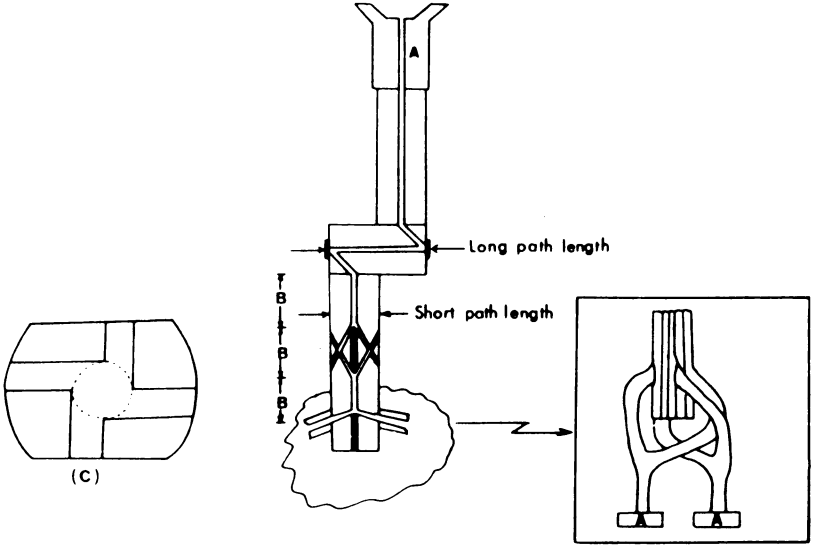

Figure 2. Fused silica flow cell for spectroscopic studies of solutions 1/1000 second after mixing of two reagent solutions. The holes were "drilled" with an ultrasonic stream of alundum powder driven by high pressure helium with an Airbrasive® unit in such a way that tangential mixing would occur without a change in overall cross-sectional area.

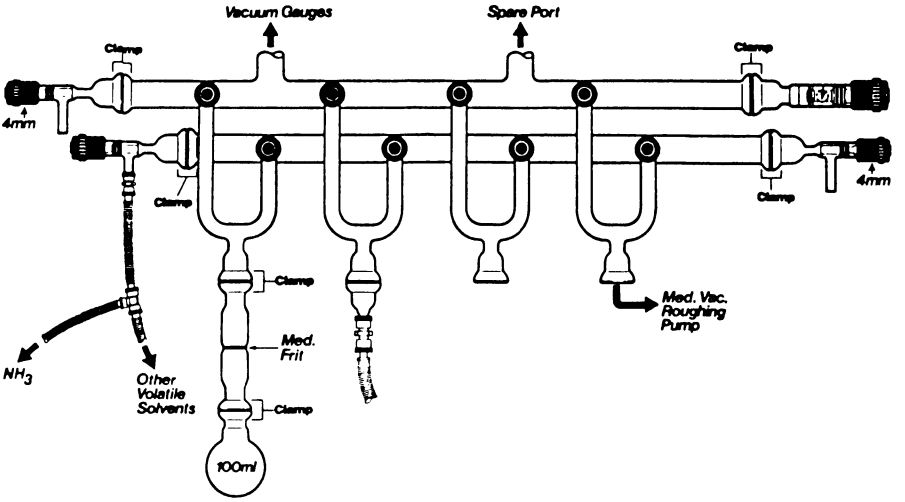

Figure 3. "Wayda-Dye" vacuum manifold with high-vacuum and low-vacuum capabilities. Flexible stainless steel tubing and UltraTorr® connectors are used to attached to the glass synthesis apparatus.

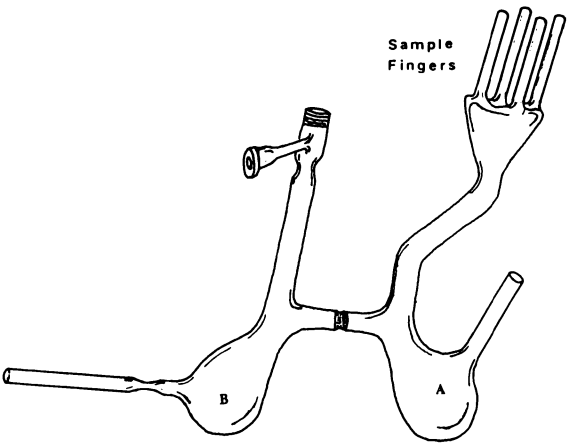

Figure 4. K-cell ("cow") used to synthesize alkalides and electrides. Alkali metal samples are introduced into the side-arms on B and complexant is put into A in a helium filled inert atmosphere box. The open tubes are capped with the aid of UltraTorr® connectors. After evacuation, the side arms are sealed off, the metal is distilled into B and solvent is distilled into A. The complexant solution is then poured into B to dissolve the metal and the blue solution is poured back into A, where crystals are grown. After washing the crystals and vacuum-drying them, they are poured into the "fingers" which are sealed off with a flame. At no time is the sample temperature allowed to rise above -40° C.

Ancient to Modern Bead Making Techniques

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Special Thanks to: Gail Bardhan
Audio-Visual Librarian, Corning Museum of Glass
for her historical information and slide duplication and Jean Birkhill for photography.

The topic of my lecture concerns the technique of making lampworked beads. I have been teaching lampworking now for about eight years. I think that most of the people in this audience would agree that learning the skill of lampworking glass is a very tedious process. We, as glassworkers, all have experienced the endless hours of practice needed to master the craft. Teaching this skill has been very frustrating to me because I realize the amount of work that my students will have to go through to learn this technique. Many of my students often give up before they even give the exercises I have provided them with a chance. As a consequence, I have had to provide my students beginning exercises that they can master relatively quickly.

An ideal exercise to teach beginning students basic lampworking skills is beadmaking. Students learn the skill quickly and therefore gain confidence to go on to more difficult techniques. An equally important skill which is learned in the process is creative thought. Learning to make lampworked beads provides for an endless opportunity to work with color, form, technique, and basic design.

It is because of these advantages that I encourage all of you who teach this craft to expose your students to these techniques. It is for this reason that I now wish to share with all of you what I have learned through my own research in the fundamental techniques of making lampworked beads.

First, I wish to show you a chart which illustrates various types of glasses used for making beads. (Illustration #1) Note that there are four basic types of glasses which I recommend.

At the top of my list is Moretti Soda Lime. This glass is one of the easiest to use. It has a very high expansion. Because of its high expansion, it is limited in the number of glasses it can be worked with. If you wish to incorporate powdered glass with this material you may use copper enamel powders with Moretti glass. They both should have the same expansion.

I have also included other glasses which I have found to be useful. These are Lead glass (i.e. Corning 0010), Kugler Colored Glasses, and Soda Lime (i.e. Corning 0080). Note that all of these glasses have roughly the same expansion. I have not found any problems due to incompatibility when using all of these glasses together.

There is one important point I wish to make concerning the use of Lead Glass, (this includes Kugler Colored Glass); because these glasses contain lead they must be worked in an oxidizing flame (more oxygen than gas) or they will turn black, due to the metallic lead reducing on the surface of the bead. To the best of my knowledge, there is no

significant danger of lead poisoning when working with these glasses. This is because these materials are in a vitreous form and not in a metallic form.

Although it is possible to make glass beads using Borosilicate (Pyrex) Glass, because of the high temperatures required to melt this material and because of the relative few colors available, I do not recommend using this glass for making beads.

COMMON GLASSES USED FOR MAKING BEADS

TYPE OF GLASS	EXPANSION ($X10^{-7}$ cm/cm/°C)
MORETTI SODA LIME	104
LEAD clear neon sign tubing Corning 0010 or equivalent	93.5
KUGLER COLORED GLASS	91
SODA-LIME Corning 0080 or equivalent	93.5

These three glasses have roughly the same expansion.

These two glasses contain lead, and therefore must be worked in an oxidizing flame (more oxygen than gas).

HOW TO MAKE A SIMPLE BEAD

I recommend using 9/64 inch stainless steel welding rod. Cut the rod into one foot segments. File the end of the rod to form a taper. This will help when sliding the bead off of the rod. A separator must be used on the rod so the glass does not permanently fuse to the rod. We recommend one of Freds special bead separator formulas. Mix the following proportionally:

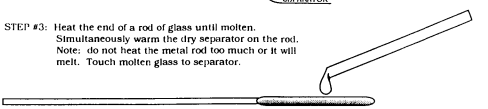
FORMULA #1	FORMULA #2
1 cup Ed Hoy Kiln Wash	1/4 cup Silica Flour
1/2 cup water	3/4 cup EPK Kaolin
5 to 10 drops Sodium Silicate	1/2 cup water
	5 to 10 drops Sodium Silicate

Mix well so that solution is free of lumps. Additonal water may be added to achieve the consistency of heavy cream.

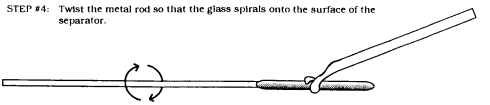
STEP #1: Begin with a brass or steel rod. Taper approximately one inch of the end of the rod so that the bead will slide off easily.

STEP #2: Dip the rod into the bead separator and let it dry. Heat the rod in a pure gas flame to burn off the water and sodium silicate.

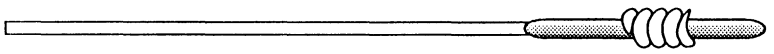
STEP #3: Heat the end of a rod of glass until molten. Simultaneously warm the dry separator on the rod. Note: do not heat the metal rod too much or it will melt. Touch molten glass to separator.



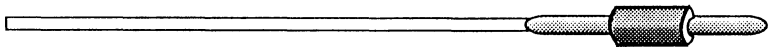
STEP #4: Twist the metal rod so that the glass spirals onto the surface of the separator.



STEP #5: Make the spiral as even as possible when putting the glass on.



STEP #6: Heat the glass evenly and flatten on a graphite or steel paddle.



STEP #7: All beads should be annealed or they could crack. Annealing is the process of relieving strain in the glass. This is accomplished by allowing the glass to cool slowly. Once the bead is made, the easiest method for annealing is by first reheating the glass evenly until it glows a dull orange color. Then allow the bead to cool in the air until it looses its glowing color and place it in a bucket of vermiculite. (Vermiculite can be obtained from a garden supply store and is commonly used as a soil conditioner.) Let the bead cool for at least one half hour.

STEP #8: When the bead is cool it should easily slide off the metal rod.

TROUBLE SHOOTING:
If bead separator is exploding off of the rod when you try to heat it the separator is not completely dry. If you live in a humid climate drying can take up to 30 minutes. Sometimes drying the separator on the rod for a few seconds in a gas flame can speed drying.
If bead separator is flaking off of the rod while you attempt to apply glass, clean the rod with sandpaper. Make sure that your bead separator is mixed well before applying it to the rod.
The most common problem is not being able to remove your bead from the rod. This problem can be caused by a variety of things including waking up on the wrong side of the bed.
Here are some hints:

•If the bead separator cracks and some of the glass sticks to the rod the bead will probably crack. If bead separator is cracking often try adding a few extra drops of sodium silicate.
•If too little bead separator is used, the bead will not separate from the rod.
•Try soaking the bead and rod in water for a few minutes. This will sometimes loosen the separator.
•Try freezing the bead for 15 minutes in the freezer and then attempt to pull off the bead.
•Hold the metal rod with a pair of pliers while attempting to twist off the bead. File two flat spots at the bottom of the metal rod for the pliers to hold onto so that the rod doesn't twist.
•Put rod through a hole in a piece of wood that is C-clamped to a sturdy table and hammer on top of the rod carefully forcing the bead off. (shown ➶)

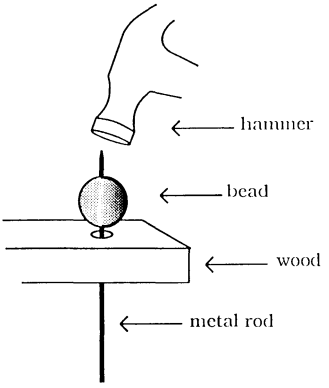
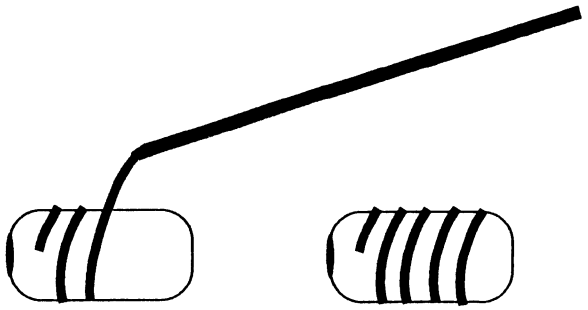


Figure 3A

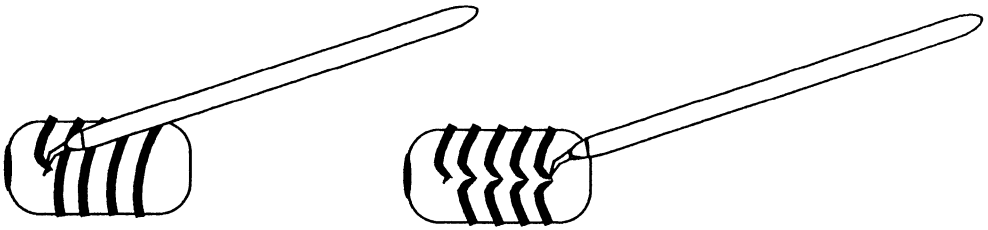
WAYS TO DECORATE BEADS

Once you have successfully made a bead out of clear glass, try coloring and decorating the bead. To make a colored bead you can either start out by using colored glass rod or you may wish to make the bead out of clear glass rod and then color the bead by rolling it in colored kugler glass powder. I like to use powders because I find them to be a more economical method of coloring the glass.

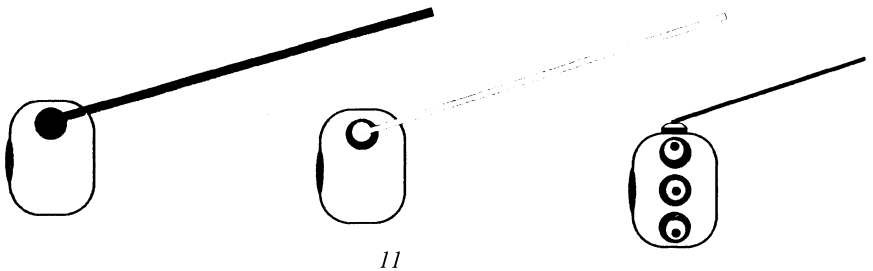
Using a small cane of solid color heat and spiral on bead.



After spiraling on a thread of colored glass you can use an ice pick or a dental tool to feather the stripes. Heat the stripes one the side you wish to feather then pull the point of the tool through the stripes.



Polka dots can be added with the color cane. Different colors applied on top of eachother creates a bulls-eye effect.



The Manufacture of ESR Dewars on the Bench

by
Allan B. Brown

This paper details the manufacture of an Electron Spin Resonance Dewar at the glassblower's workbench without the aid of rollers or a lathe. The procedures and techniques for this hands-on process make it possible for a glassblower to easily manufacture this dewar without expensive equipment.

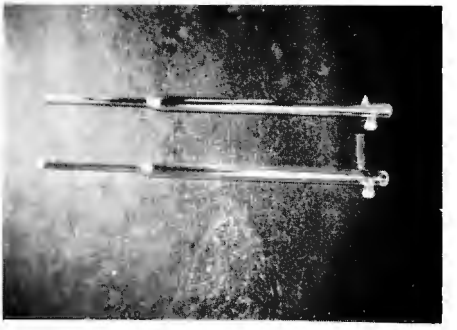
According to Webster's Dictionary, a dewar is "a glass container that has an evacuated space between the walls (and) is often silvered on the innermost surface to prevent heat transfer." It is named after the Scottish Chemist and Physicist, Sir James Dewar (1842-1923). The Electron Spin Resonance (ESR) Dewar is a specialized version which is widely used by chemists today.

Before embarking on a detailed description of the actual construction of this dewar, let us review the individual glass parts and the necessary tools (Photograph #1).

Photograph 1

Let us start from left to right. The first two pieces (15mm and 8mm tubing) will be used for the outside jacket; the next two (10mm and 4mm) are for the inside jacket and the fifth piece (7mm) will become the side arm and the pump-out stem. Above this last piece is a 12/5 ball joint that has been cut to length. To the right of these items are the tools required. First is a 1/4" graphite rod with one end sharpened on four sides to a point. The next one is an old tapered reamer that has been ground down. The third piece is a standard tapered reamer and the fourth is a small flat paddle. A tungsten pick will also be used.

The second photograph (Photograph #2) shows two dewars.



Photograph 2

The one on the right is ready to be silvered, whereas the one on the left has been silvered and evacuated.

Carefully select the tubing for the dewar so as to ensure that there are no striations or stones that could possibly cause a vacuum leak; then cut them to length. Blow out all ends of the tubes prior to sealing; this will help guarantee a tight vacuum seal (Photograph #3).

Photograph 3

Taking the 15mm tube and the 8mm tube, make a shoulder seal and work it well (Photographs #4 & #5).

Photograph 4

Photograph 5

When this seal is cool, use the short carbon to flare the top lip of the outside jacket, approximately 3/4mm (Photograph #6).

Photograph 6

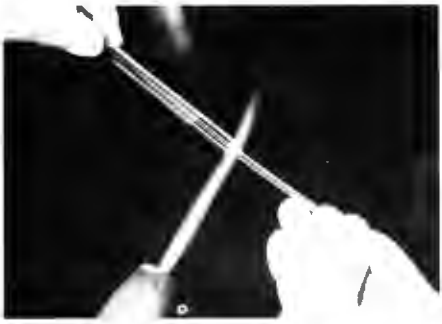
Using the 10mm tubing, heat and tool what will become the top of the inside piece, to hold a 10/18 standard tapered joint. You will notice a white line on the tapered reamer which is my reference mark for a 10/18 size joint (Photograph #7).

Photograph 7

Flare the top of the inside tooled piece approximately 3/4mm which will act as a seat when making the dewar seal (Photograph #8).

Photograph 8

When the tooled end of the 10mm is cool enough to handle, seal a piece of 4mm tubing to the opposite end (Photographs #9 & #10).



Photograph 9

Photograph 10

Measure the overall length and cut off the excess 4mm tubing. Now, using a 1/4" graphite rod, flare the 4mm tubing end so that it will just fit inside the 8mm tubing, which is the bottom of the outside jacket (Photograph #11).

Photograph 11

Locate, heat, and blow a hole in the 10mm tube just below the taper (Photograph #12); then seal a piece of 7mm tubing in place for the side arm (Photograph #13).

Photograph 12

Photograph 13

Finish the side arm seal using a #2 tip on a Carlisle hand torch making sure that it is well worked and not thin (Photograph #14). This is a very important step, because if this side arm is not added (even though it may only be 2mm long when finished), there will not be enough material to make a good ring seal.

Photograph 14

Cut and grind down the side arm (Photograph #15) so that it will fit inside the 15mm tube.

Photograph 15

Insert the 10mm tube into the 15mm tube and check for fit (Photograph #16).

Photograph 16

If it fits well, take the pieces and clean them thoroughly with Alconox and water. Make the final insertion - you will notice that the two flared pieces seat and that the flare at the bottom of the 4mm tube centers itself.

Holding the 10mm tube snugly in place, locate and mark the spot for the pump-out stem (Photograph #17).

Photograph 17

Turning the dewar 180 degrees, heat and seal the 15mm tube to the side arm (Photographs #18 & #19).

Photograph 18

Photograph 19

This will now hold the inside tube firmly in place in preparation for making the dewar seal at the top. Warm the whole tube so that the tacked ring seal will not crack (Photograph #20).

Photograph 20

Using the short carbon, make the top dewar seal (Photograph #21).

Photograph 21

Next, heat the ring seal that was tacked and blow the center of it out (Photograph #22).

Photograph 22

Add the 12/5 ball joint to the side arm seal (Photograph #23), working the seal well (Photograph #24), and then warm it out (Photograph #25).

Photograph 23

Photograph 24

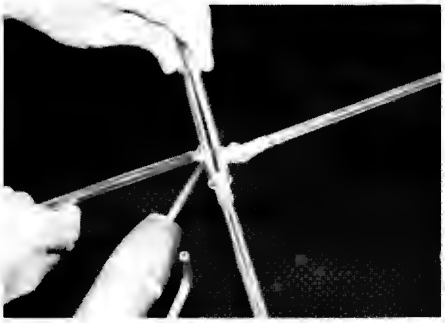
Photograph 25

The spot marked for the pump-out stem is heated and blown out (Photograph #26).

Photograph 26

Seal on the 7mm pump-out stem (Photograph #27) and work it well (Photograph #28).

Photograph 27



Photograph 28

Using a knife, cut off the outside 8mm tube approximately 1mm below the flared end of the 4mm inside tube (Photograph # 29).

Photograph 29

Using the 1/4" carbon, make a dewar seal on the bottom of the dewar (Photograph #30).

Photograph 30

Continue to heat the bottom of the dewar until it heavies, while using a tungsten pick that has been soaked in bees wax to keep the hole open (Photograph #31).

Photograph 31

The reason for the heaviness is that the bottom of the dewar has to be ground round.
The dewar should now be annealed (Photograph #32). When cool, grind both ends to finish size, before silvering and evacuating (Photograph #33).

Photograph 32

Photograph 33

If you follow these procedures to build this ESR dewar on the bench, you will find that you have saved a considerable amount of time. Excluding silvering and evacuating, the entire construction should not take more than twenty minutes.

Modifying Volumetric Flasks to Deliver Known Volumes of Liquid

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The technique of calibrating a vessel is one which calls for care and experience, if a reasonable degree of accuracy is to be achieved. Most small glassblowing shops lack the facilities to perform this task. There is nothing worse, both for the shop and the customer, than having to decline an order.

It is not the purpose of this paper to intrude upon the techniques employed in those establishments where calibration of vessels either to contain or deliver a known volume of liquid is routine. Rather the purpose is to offer a procedure which is of practical value to those who must perform this task on an occasional basis. The technique is based on having available precalibrated vessels which will deliver a known volume. Three types of vessels are used - pipettes, burettes, and flasks. Measuring cylinders are not included in this list for the simple reason that most of them are little better than a cheap kitchen measuring jug.

As a first step it is important to establish the degree of accuracy required! This is a very misunderstood specification. One, in fact, which can border on the bizarre. An example for personal experience will help to illustrate this point. A request was made to supply a 4 litre vessel with an accuracy of plus or minus 0.01 ml. Let's see just what this figure means. The specifications laid down for Class A 100 ml pipettes is plus or minus 0.08 ml, very close to 0.1 ml. The request would have involved calibrating the vessel, which is 40 times larger than the pipette, to 10 times the degree of accuracy. The closest large volume flask is of 2 litres capacity with a guaranteed accuracy of plus or minus 0.5 ml. If both errors fall on the same side two such flasks would have an error of 1.0 ml, or 100 times greater. Be it ignorance or folly, the reply to such a request demands a very diplomatic approach, no easy thing.

Certain steps must be taken in order to maximize the successful outcome of the procedure. Firstly, there is the problem of temperature. By international agreement the specific temperature at which measurements of liquids is made is 20° Celcius. Now 20° C is relatively cool compared to the ambient level of most laboratories; and significantly below the average encountered in glass shops. The problem compounds itself in tropical locations. Some operators take a cavalier attitude, "Oh, the temperature feels about right! It's not all that important." Let's take a close look at this question of temperature. Put succinctly even very small variations in temperature during the process of calibration has a profound effect upon the accuracy thereof. In order to clarify this point let's consider in general terms the significant difference between the cubical expansion of most solids and those of liquids. The expansion coefficient of all liquids is 5 to 10 times greater than that of solids. For water and most aqueous solutions at low concentration levels this is 0.0003 per ° Celcius, 3 in 10000 - what's all the fuss about. Consider 1 litre of water in a vessel at 20° C. A rise in temperature to 21° C means that we now have 1000.3 mls. This increase is sufficient to comprise the accuracy of a 1 litre Class A volumetric flask. If the unobserved temperature creeps up to 25° C or 77° Fahrenheit the volume increases to 1001.5, 25° C is a comfortable working temperature. Lucky are they in a glass shop to labour in such comfort. If you would aspire to succeed, watch the thermometer, which by the way should be an accurate one. Cheapies of student quality

are not good enough. Often the water in the faucet is well below 20° C and affords a simple means to cool down the vessels and their contents. Bear in mind, especially in a warm location, that speed must be combined with care so that calibrations are completed before any significant error can creep in. When the water supply is above 20° C ice will be needed to maintain control.

It is appropriate to compare the cost of purchasing first class vessels in terms of accuracy with Class B vessels. Table 1 sets out the differences in accuracy and price for Class A and Class B volumetric flasks.

The information contained in Table 1 clearly indicates that for a very modest extra cost accuracy is doubled from Class B to Class A, so-called several purpose, or student ware is a waste of money. A similar price ration exists for pipettes and again as shown in Table 2 the accuracy of Class A is double that of Class B.

The accuracy relationship for measuring pipettes is compared in Table 3.

Now to describe the procedure to modify volumetric flask to deliver a known volume as well as to contain that volume. For the sake of expediency, figures are given to cover 500 ml, 1 litre and 2 litre flasks. The reasoning behind this choice is that, firstly a smaller volume than 500 ml is more accurately determined by using pipettes, and secondly it is possible to purchase small capacity flasks already graduated to both contain and deliver a specific volume. Assuming that 500 ml, 1 litre and 2 litre Class A flasks are available together with delivery pipettes 0-2 ml and 0-5 ml, and supported by distilled water with or without ice depending on the ambient temperature, the calibration is ready to be made.

Firstly, chill the distilled water and the flasks to 20° C until the flasks are to within 5 mm of the graduation mark, and check the temperature. When the operator is satisfied that the temperature is steady at 20° very carefully fill to the graduation mark, using a simple shop made pipette -6 or 7 mm tube with a fine tip. Now, referring to Table 4, add water using the appropriate pipette as indicated by the table.

It should be noted that for round 2 litre flasks and for the 500 ml and 1 litre ones, irrespective of shape, the volume of water to be added is less than 2.0 ml. It is, therefore, desirable to select a 2.00 ml pipette graduated in 1/100 ml rather than one calibrated in 1/10 ml in order to take advantage of the improved accuracy. Pay close attention to the temperature during this step in the procedure. Having added its specified volume of water place a thin straight line decal around the neck of the flask to coincide with the new level of liquid within the flask. Make sure that the decal remains in place, now empty the flask and fire the decal onto the glass. Keep the temperature of the oven about 20° - 25° C below the annealing point to avoid any risk of altering the dimensions of the flask. The conversion of the vessel from that which only contains a known volume to one which will now deliver that same volume is complete. There remains one final step to follow. In order to allow liquid to drain away from the walls of the flasks a drain time of 20 seconds has been chosen and this factor has been included in the calibration technique. Failure to observe this 20 second drain will result in a low volume delivery.

For those who might be curious, the reason why square flasks require more additional water than round ones, is because of the physical fact that spherical shapes required less surface area to encompass a given volume than do square ones.

TABLE 1

	Class A		Class B	
Capacity ml	Accuracy	Price	Accuracy	Price
10	± 0.02	7.34	± 0.04	6.61
25	0.03	7.86	0.06	7.20
50	0.05	8.06	0.10	7.37
100	0.08	9.41	0.16	8.47
500	0.20	14.20	0.40	12.71
1000	0.30	18.05	0.60	16.29
2000	0.50	27.22	1.00	25.15

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TABLE 2

Capacity	Class A	Class B
0.5, 1.0, 2.0 ml	± 0.006 ml	0.012
3.0, 5.0	0.01	0.02
10	0.02	0.04
15, 20, 25	0.03	0.06
50	0.05	0.10
100	0.08	0.16

The accuracy relationship for measuring pipettes is compared in Table 3.

TABLE 3

Capacity	Subdivision	Class A	Class B
1.2 ml	1/10	± 0.01 ml	0.02 ml
5	1/10	0.02	0.04
10	1/10	0.03	0.06

Now to describe the procedure to modify volumetric flask to deliver a known volume as well as to contain that volume. For the sake of expediency, figures are given to cover 500 ml 1 litre and 2 litre flasks. The reasoning behind this choice is that, firstly a smaller volume than 500 ml is more accurately determined by using pipettes, and secondly it is possible to purchase small capacity flasks already graduated to both contain and deliver a specific volume. Assuming that 500 ml, 1 litre and 2 litre Class A flasks are available together with delivery pipettes 0-2 ml and 0-5 ml, and supported by distilled water with or without ice depending on the ambient temperature, the calibration is ready to be made.

Firstly, chill the distilled water and the flasks to 20° C until the flasks are to within 5 mm of the graduation mark, and check the temperature. When the operator is satisfied that the temperature is steady at 20° until very carefully fill to the graduation mark, using a simple shop made pipette -6 or 7 mm tube with a fine tip. Now referring to Table 4, add water using the appropriate pipette as indicated by the table.

TABLE 4

Capacity	Square Flasks	Round Flasks
500 ml	0.92 ml	0.60 ml
1000	1.30	0.75
2000	2.18	1.90

It should be noted that for round 2 litre flasks and for the 500 ml and 1 litre ones, irrespective of shape, the volume of water to be added is less than 2.0 ml. It is, therefore, desirable to select a 2.00 ml pipette graduated in 1/100 ml rather than one calibrated in 1/10 ml in order to take advantage of the improved accuracy. Pay close attention to the temperature during this step in the procedure. Having added its

Various Unique Vacuum Holders

By
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Many glassblowers use vacuum holding devices to support a flat plate in the glassblowing lathe to seal onto the end of, or inside of, a glass cylinder. This is done through a tube of some sort attached to a swivel and terminating at a vacuum pump. Although glassblowing blowhose swivels can be used for this purpose, they tend to leak excessively. To combat this I incorporate a rotating union instead of the typical swivel. These unions are readily available from the hydraulics industry, and are also recommended for vacuum service. I incorporate a simple valve system allowing me to isolate the vacuum supply and pressure release which allows for a quick and simple removal of the glass parts. A stainless steel tube is fit to the union by means of soldering a pipe coupling with the matching thread size as the union connection. Care should be taken to select a coupling and stainless steel tube that will rotate very straight. These together now offer you the main body of the vacuum holder; see Figure 1. With the holder constructed, your imagination can now run wild designing small graphite holders to fit into the end of the stainless steel tube for holding various glass parts.

Most of the graphite holders are designed to securely hold specific parts to facilitate the ease of glassblowing fabrication. This is necessary because many of these parts and assemblies are hard to hold for the sealing operations. The important factors when machining these holders is to make a snug fit to join inside the stainless tube, and a good square shoulder to rest up against the end of the stainless steel tube. When making the graphite holders the use of the machine lathe is imperative because it allows one to maintain faces and shoulders perfectly straight and square.

The first graphite holder is shown in Figure 2. This is the most familiar type to scientific glassblowers. It has been used to hold flat discs that are intended to be sealed on the end of, or inside the end of a glass tube. The narrow end on the bottom fits snugly into the end of the stainless steel tube with its shoulder butting up squarely against the end of the stainless tube. When the valves are in the "vacuum orientation", a glass disc can be held on the end of the graphite holder. You will notice that the bore is straight and narrow. Discs up to two and three times the bore diameter may be held with great success. If larger discs are to be held, a graphite holder can be made with a larger surface area on the end. Note that the vacuum space is much larger on this holder shown in Figure 3.

Now it is time to proceed to more sophisticated holders. Often we are asked to seal discs with a hole in the center (doughnut shaped). Obviously, the holders mentioned to this point would not accommodate this application. Often we drill the hole after the sealing operation, but we all know that drilling is a very delicate operation and many times the discs crack. A more acceptable procedure would be to drill the hole first and then seal the disc into a tube with the holder shown in Figures 4 and 5. Holes are drilled on angles from a grooved radial vacuum space until they meet at the center vacuum hole. This center hole is not drilled completely through the front end of the graphite holder.

As you probably realize, the ideas for these holders were not all conceived at one time. They were designed and constructed as the various jobs warranted. One of my favorites that I have found endless uses for is this next holder. It was designed to hold a cylindrical cell body on its circumference to facilitate the addition of smaller side tubes. The simplistic design of this holder, shown in Figures 6 and 7, made it invaluable when side tubes needed to be precisely aligned under crowded conditions. Figure 8 shows an example of this. A milling machine was used to create a concave curvature in the end of the basic holder. The radius of the curvature should exactly match the diameter of the glass tube to be held. If you do not have a milling machine available, one can grind the

soft graphite on the side of the glass tubing with silicon carbide grinding compound. Continue grinding until the graphite holder rests securely on the tube as shown in Figure 9. Care must be taken to assure the graphite is held very straight during grinding so the glass tube is ultimately held correctly at 90 degrees with respect to the holder.

You can go one step further with this holder and add a second angle as shown in Figure 10. By machining or grinding the groove at a 45 degree angle, tubes can be added to a piece precisely and with great ease. The tubes you add this way must also be cut at 45 degrees in order to match up with the parent tube when it is mounted in a glass lathe; see Figure 11.

The vacuum holding technique that incorporates the rotating vacuum swivel has been used for many years. However, the graphite holding inserts I developed are new and have proven to be most successful and beneficial.

A short note here to inform you that this technique is also useful in production. An example of this is to firepolish the ends of large quantities of shell covers (short test tubes). One can be attached quickly onto the graphite vacuum chuck, firepolished, and rapidly removed and replaced with the next tube. This technique is fast and requires very little movement. You can also hold hemispheres or flasks to add joints or polish ends quickly; see Figure 12.

And so, with a vacuum pump, rotating union, graphite, and imagination, you can accomplish many complicated requirements (like the apparatus shown in Figure 13) faster, easier, and with greater accuracy. I am pleased I was allowed to share the different types of graphite holders I have designed and used.

Acknowledgements

I would like to acknowledge certain individuals who have helped me to create both the graphite holders and this paper. They are: Mr. Wes Prucnal, Tool Maker at Argonne National Laboratory Machine Shops; Mr. William Mulac who helped to create the computer schematics; and Mrs. Cathy Carbaugh, who provided computer assistance. I would also like to thank Argonne National Laboratory and the Chemistry Division for allowing me to be innovative and creative in my employment and profession. This work was performed under the auspices of the Office of Basic Energy Sciences, Division of Chemical Sciences, U.S. Department of Energy, under contract #W-31-109-ENG-38.

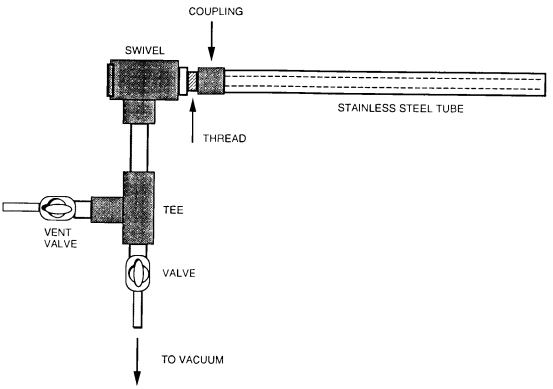


Figure 1. Body configuration of rotating vacuum holder.

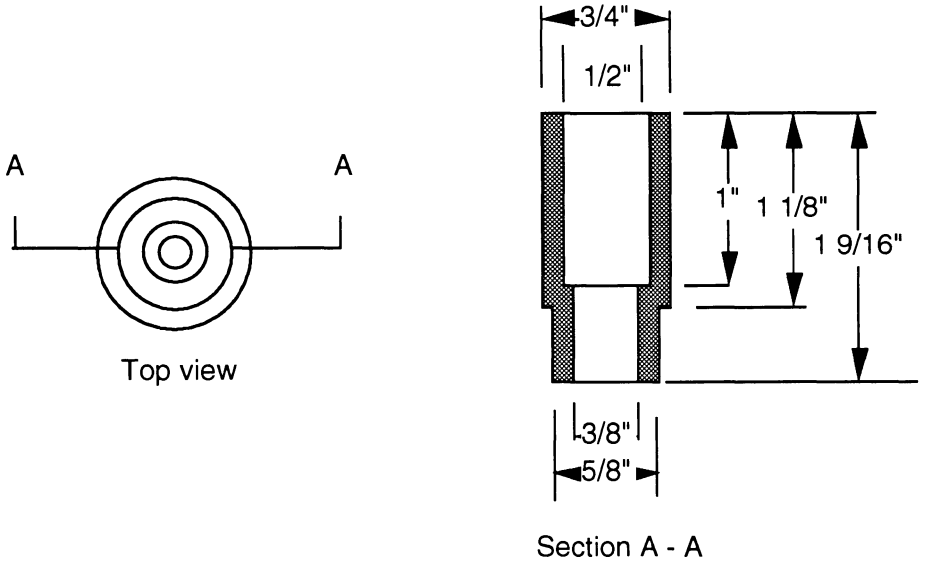


Figure 2. Graphite holder for small and medium discs and hemispherical shapes.

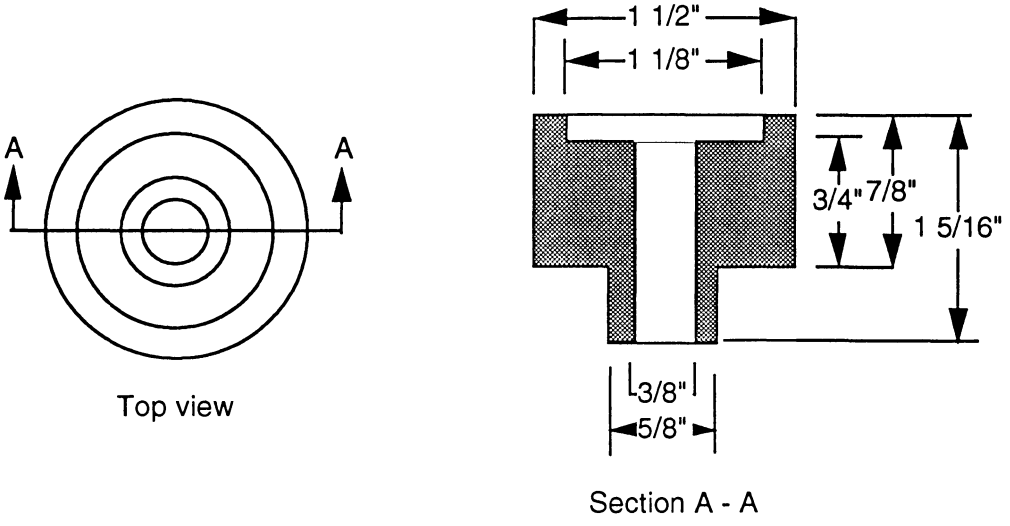


Figure 3. Graphite holder for larger discs and flasks.

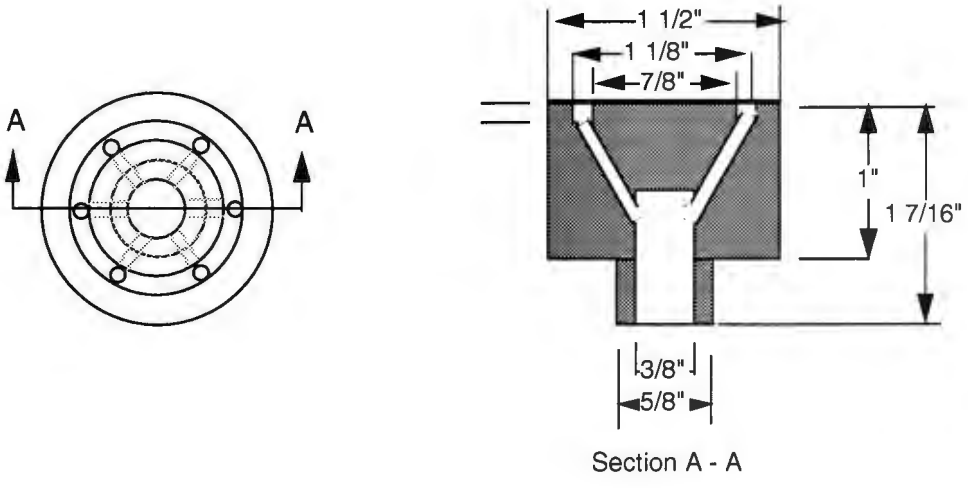


Figure 4. Graphite holder for discs with holes in center.

(figure 5)

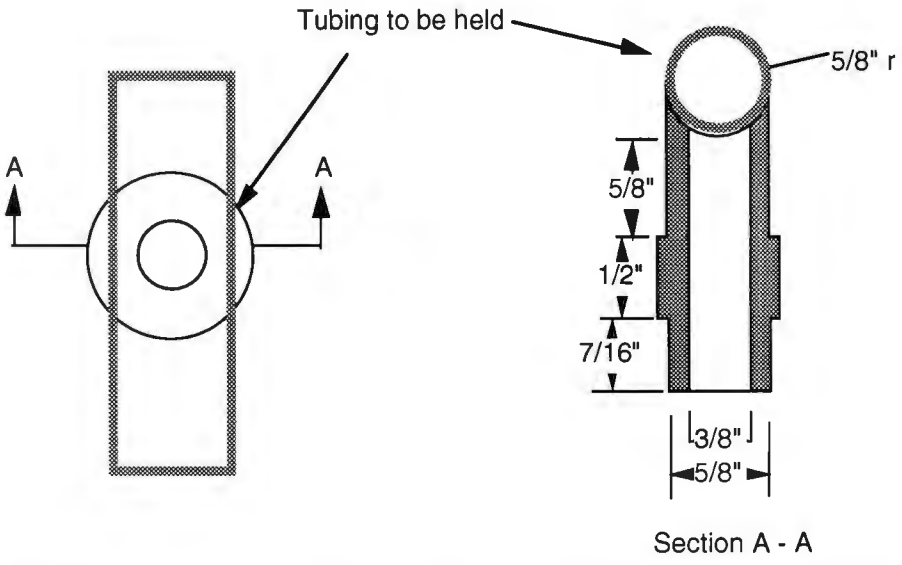
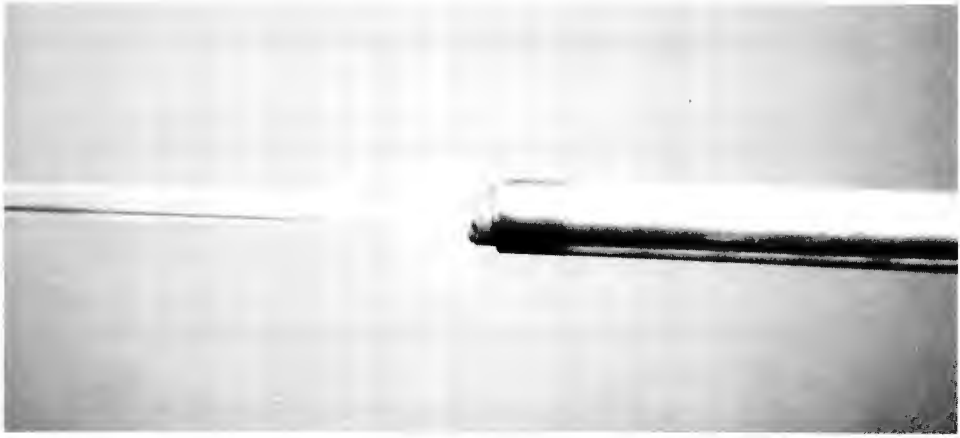


Figure 6. Graphite holder for holding tubes on their circumference

(figure 7)



(figure 8)

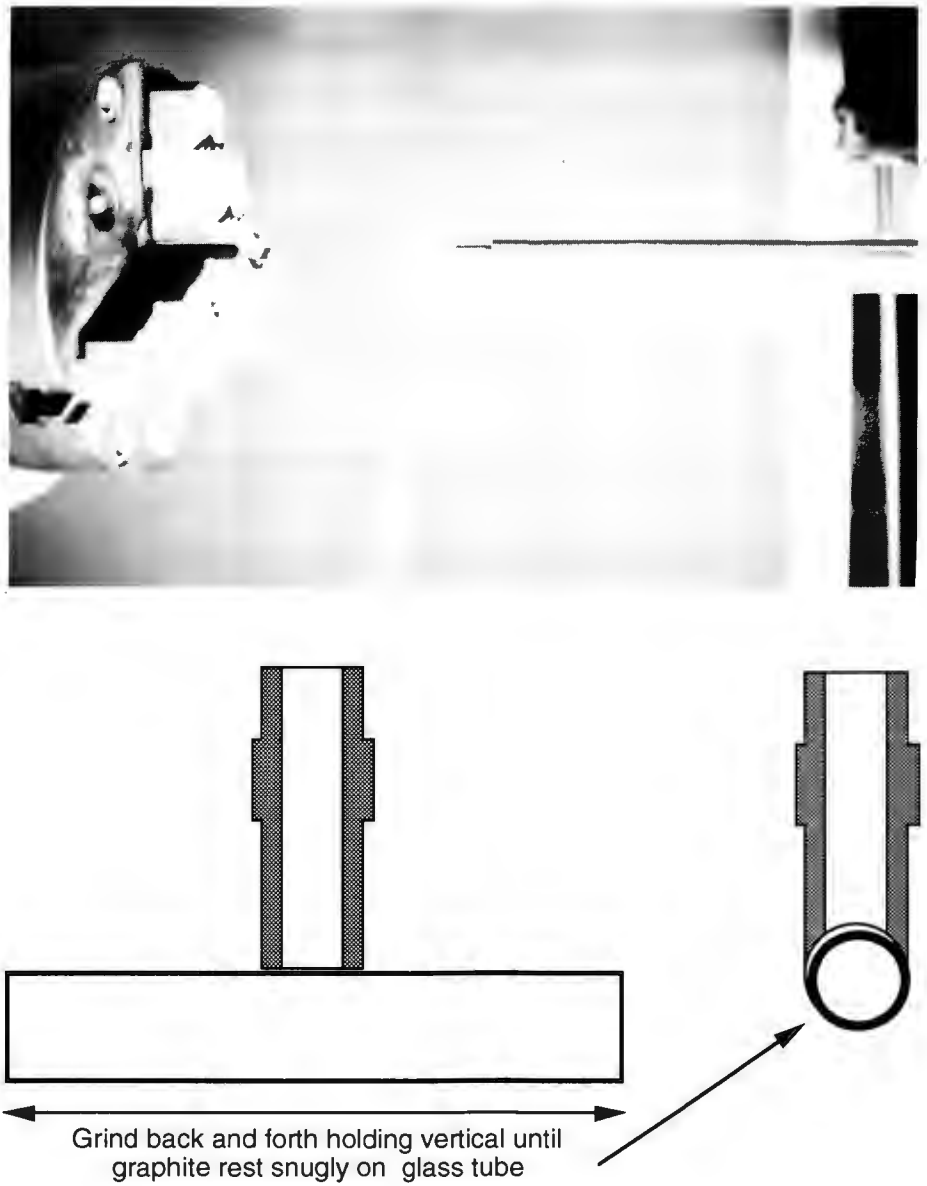


Figure 9. Set up for grinding radius in graphite holder.

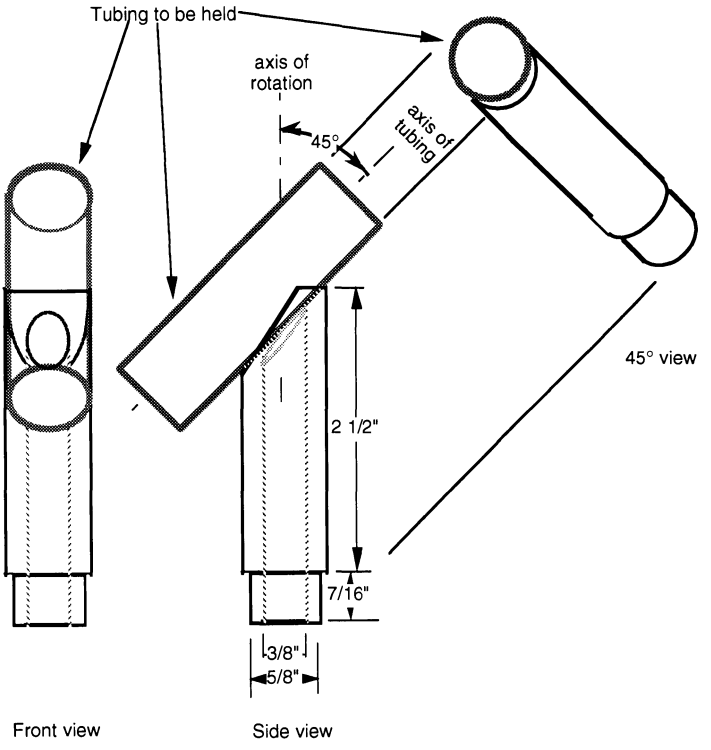


Figure 10. Graphite holder for holding tubes at 45 degrees.

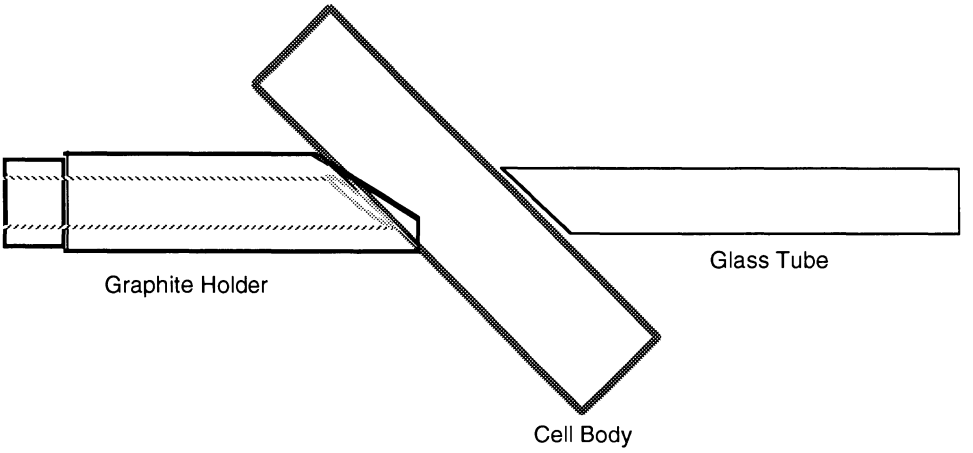
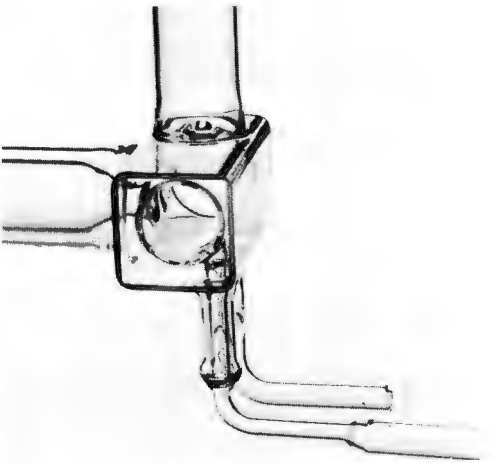


Figure 11. Graphite holder for holding tubes at 45 degree angles with both glass tubes in place.

(figure 12)

(figure 13)



Temperature Induced Stress Build-Up At Glass-Metal Boundaries inTungsten Halogen Lamps

H.E. Hagy, Consultant
Corning Incorporated

Abstract: Failure analyses of tungsten halogen lamps made with Corning Code 1724 glass show substantial development of axial tension in the glass with time at the interface with molybdenum leads in the lamp base during operation. Experimental studies with lamps at an operating temperature of 550°C show an exponential-like stress build-up with times up to 1000 hours. The mechanism is attributed to density compaction in the glass, the magnitude of which should be reduced in a glass with higher annealing point, such as Corning Code 1725. Comparative stress build-up and density measurements between glass Codes 1724 and 1725 support this diagnosis.

1. Introduction

A recent problem with tungsten halogen lamps had an interesting diagnosis in which part of the causality was due to glass transformation range behavior. The suspect behavior has been verified in an experimental program described in this paper.

Tungsten halogen lamps manufactured with Corning Code 1724 glass experienced cracking at the molybdenum leads during life testing to 1000 hours. In the life testing experimental format the lamps are inverted, and in this orientation the lamp base attains a high temperature. Measurements with small thermocouples indicate that the base temperature can reach 550°C.

Initial photoelastic measurements provided information that prompted the theory that was followed in this investigation. Optical retardations measured at the molybdenum lead-glass interface showed that life-tested lamps had a significant stress change toward axial tension as compared to lamps from the same production lot not subjected to the life test.

The writer proposed that the stress change was due to density compaction at 550°C and the associated stresses would explain the type of fractures observed.

2. Fracture details

A sketch of a tungsten halogen lamp is shown in Figure 1. A magnified sketch is also shown of the lead-glass details at the upper portion of the lamp base. One of the contributory factors in the fracture process is the re-entrant geometry of the glass-metal seal. This tends to be a problem with tungsten halogen lamps due to the high softening points of the glasses used, forced upon the system due to high operating temperatures.

One can see that a crack can start at the re-entrancy as the glass contracts if a significant density change in the glass occurs. The crack cannot propagate along the lead however because the glass contraction produces radial compression in the glass at the interface. The axial tension in the glass associated with the glass contraction, turns the crack normal to the lead axis as shown.

3. Glass Transformation Range Compaction

When glass is cooled, the dynamics of cooling, coupled with the continuum of relaxation rates decreasing rapidly towards lower temperatures, forces the glass to depart signifi-

cantly from equilibrium conditions as shown in Figure 2. Tungsten halogen lamps are generally cooled rapidly through the transformation range in the manufacturing process, permitted by their small size and low glass expansion. Faster cooling forces the glass further from equilibrium.

Upon reheating, the glass will attempt to reach equilibrium with time, as shown by the dotted arrow. Although 550°C is 125°C below the strain point of glass Code 1724, it is well know that glass structural changes can occur at temperatures far below the strain point of glass.

If it could be proven that glass density compaction occurs in glass Code 1724, it would be reasonable that a glass with a higher strain point, like Corning Code 1725, would experience less compaction.

Experimental Program

4.1 The Glasses

Two glasses were chosen for study, Corning Codes 1724 and 1725. These are aluminosilicate glasses with pertinent physical properties given below:

	Code 1724	**Code 1725**
Annealing Point, °C	725	780
Strain Point, °C	675	725
Density, g/cm^3	2.64	2.70

4.2 Experimental Lamps - Stress Change Measurements

Lamps were manufactured by the same process using both glasses. Two lamps for each glass were selected for the determination of stress retardation change at 550°C over a period of 1000 hours.

At the start of this investigation, the optical retardation was measured in the glass at the glass-lead interface. Measurements were made at both leads in each lamp, giving four data (averaged) for each glass. All four lamps were heated together, held at 550°C, and periodically removed to determine stress retardation change at room temperature. Optical retardation measurements were made with a Friedel polarimeter using white light in accordance with ASTM Designation F218, "Analyzing Stress in Glass."

Results of these measurements with time at 550°C are shown on Figure 3. Significant stress retardation occurs for both glasses with Code 1725 having less change, as expected. The shapes of the curves behave exponentially, typical of transformation range behavior, reinforcing the premise that glass density change is the cause.

4.3 Density Change Measurements

In a separate measurement program, pieces of both glasses were cut from cast patties, laboratory melted. Starting densities and those with time at 550°C were measured in accordance with ASTM Designation C-693, "Standard Method of Test for Density of Glass By Buoyancy."

Before subjecting these density specimens to heat treatment at 550°C, they were annealed on a schedule intended to approximate production downrates. The schedule used is shown on Figure 4, the downrate produced in a laboratory furnace in an unpowered state. Actual production annealing rates for lamps are more rapid. This will have the effect of making the measured density changes conservative for this study.

Experimental results of measured density change with time are plotted in Figure 5. The change for glass Code 1724 is twice that for glass Code 1725. Due to the fact that the cooling rate falls off with decreasing temperature in the pre-anneal, the comparison is biased against Code 1725 since it received a faster rate at its annealing point.

4.4 Stress Release

The density change specimen for glass Code 1724 was ideal for measuring stress release with time at 550°C. This specimen was nearly a centimeter thick, so that considerable (~600nm) central plane tensile optical retardation was present following the pre-anneal. The specimen for glass Code 1725 was thinner and had complex bends, making it impossible for stress release measurements.

Accordingly, at those periods of time when the specimens were removed for density measurements, the central plane tensile retardation was measured for the Code 1724 specimen. Results are plotted on Figure 6, showing stress retardation remaining with time at 550°C. Very modest stress release takes place, the data predicting about 12% at 1000 hours. Of course, glass Code 1725 will release less than this, since it has a higher strain point.

5. Analysis

Time and budget did not permit a finite element analysis of the stress system in the lamp base. However, such a study was not necessary to convince one that reasonably disturbing stress magnitudes build up in the glass with time.

The optical retardation changes in Figure 3 can be converted to stress with the following equation

$$\sigma = \frac{\Lambda\lambda}{PK} \qquad (1)$$

where: σ = Tensile stress build-up, psi
$\Lambda\lambda$ = Maximum retardation change, nm
K = Stress-optical constant, 0.191nm/cm/psi for both glasses, and
P = Optical path, cm.

When measuring the optical retardation in the study lamps, it was observed that retardations fell off rapidly as one scanned normal to and away from the glass-metal interface. This led to the conclusion that a reasonable optical path would be molybdenum lead diameter, 0.06 cm. Equation (1) then gives these stress build-up values:

Glass	**Stress Build-up, psi**
Code 1724	3500
Code 1725	1600

Density changes can be converted to stress build-up, as well. Firstly, the density change can be quantified as a mismatch in strain units:

$$\delta = \frac{\Lambda\rho F}{PK} 10^6 \qquad (2)$$

Where: δ = Mismatch strain, ppm,
$\Lambda\rho$ = Maximum density change, g/cm^3,
ρ = Nominal glass density, gcm^3, and
F = Stress release factor 0.88 for glass Code 1724, 0.95 (estimated) for glass Code 1725.

Equation (2) then gives these results:

Glass	δ,ppm
Code 1724	444
Code 1725	235

Stress build-up then can be calculated using the equation given below, which assumes equal strain split between the glass and molybdenum:

$$\sigma = \frac{\delta E}{2(1-v)} \qquad (3)$$

Where: v = Poisson's ratio, 0.24 for both glasses, and
E = Elastic modulus, 10^6 psi, 12.0 for both glasses.

Equation (3) yields these results:

Glass	σ,psi
Code 1724	3500 (3500)
Code 1725	1900 (1600)

Stress values in parentheses are those calculated from retardation change measurements. Agreement is excellent.

One might quarrel with some of the assumptions made for both approaches for stress build-up calculations. However, the mismatch strains from density changes are not open to question and can stand by themselves. Over the years a rule has been established to limit sealing mismatch to a maximum of 100 ppm for high reliability. Even Code 1725 exceeds this conservative limit somewhat, but the lamp can probably tolerate the 200 ppm level. The improvement over Code 1724 is substantial.

6. Conclusion

Optical retardation measurements in test tungsten halogen lamps and glass density measurements show with good correlation that significant stresses can build up with time at operating temperatures of 550°C.

This study suggests three ways to counter breakage from stress build-up:

1. Increase the annealing point of the glass.
2. Reduce seal re-entrancy
3. Adjust the glass thermal expansion relative to molybdenum to attain modest axial compression at room temperature. See Figure 7, which represents a "target curve" for glass Code 1725.

7. Acknowledgement

The writer is indebted to Mr. Scott E. Posey for his carefully carried out and prompt glass density measurements. Mr. Posey is a member of the Physical Properties Analysis Department at Corning Incorporated, Corning, NY.

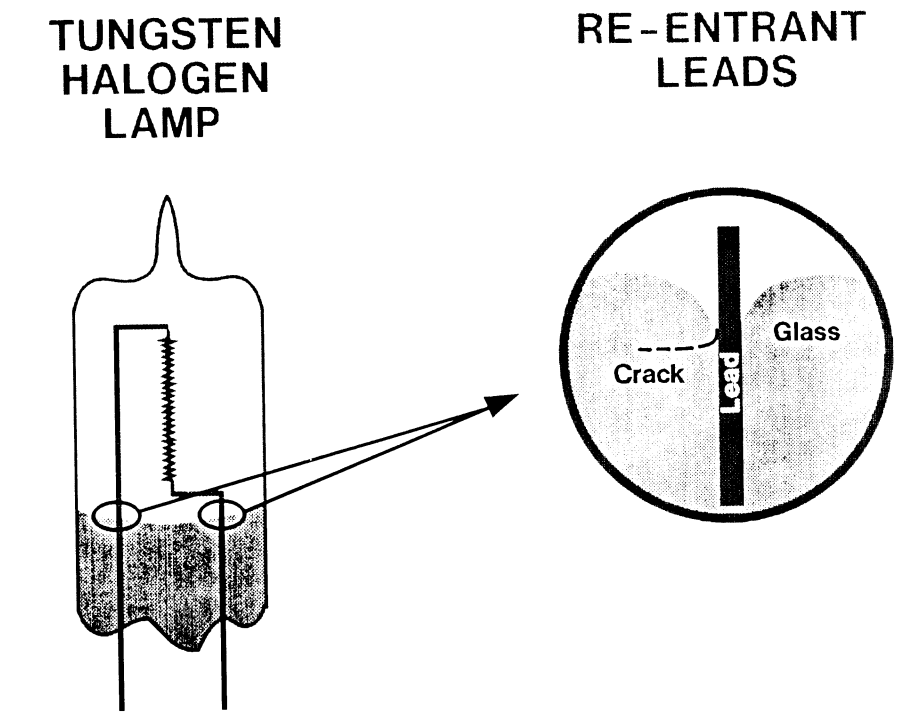


Figure 1. Sketch of a Tungsten Halogen Lamp Showing Seal Re-entrancy and Cracking

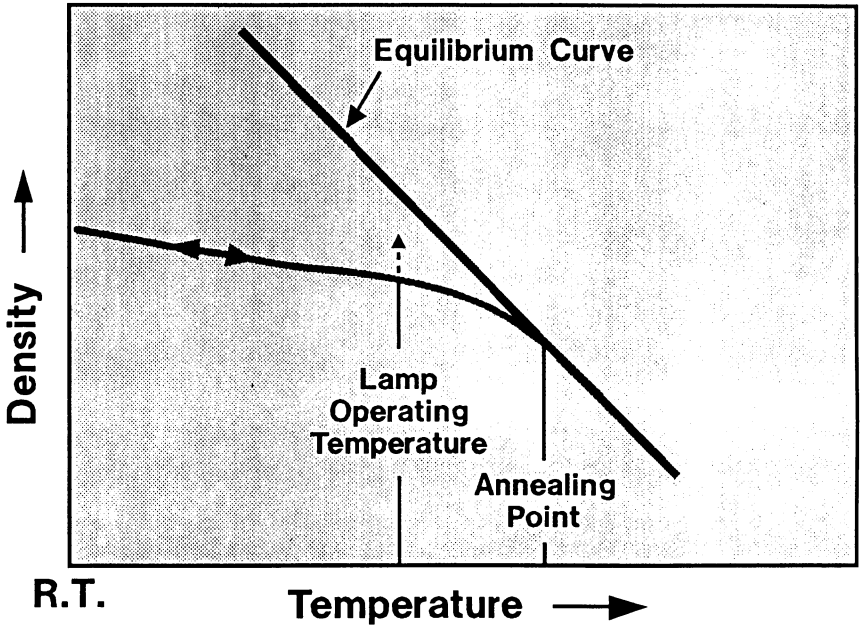


Figure 2. Glass Density Behavior in the Lower End of the Transformation Range

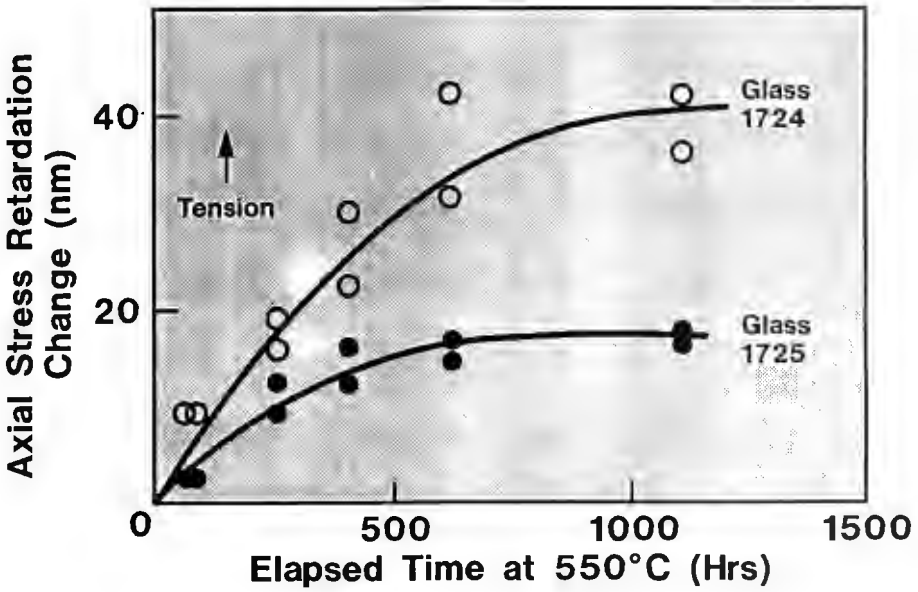


Figure 3. Axial Stress Retardation Change with Time at 550°C for Glasses Codes 1724 and 1725

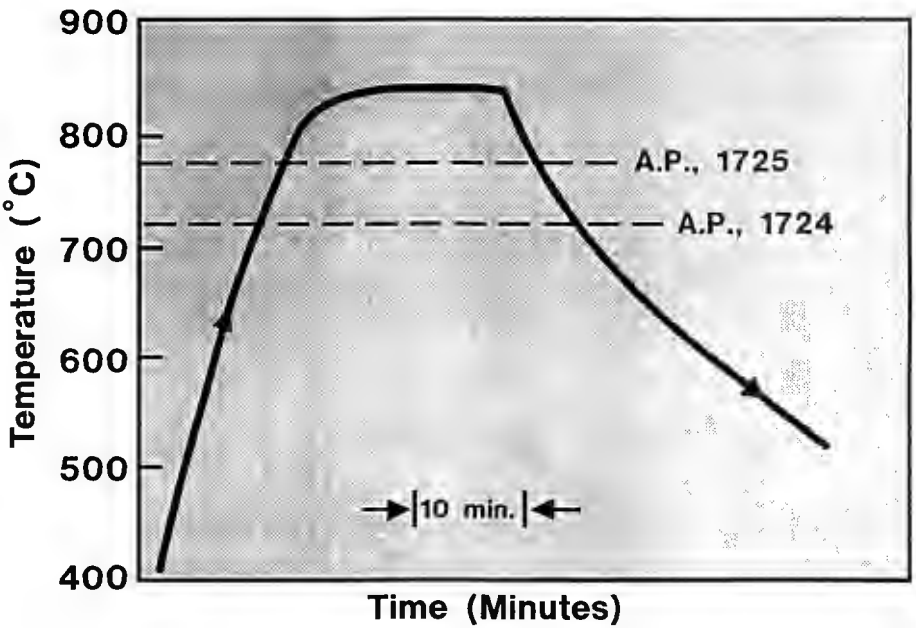


Figure 4. Pre-anneal for Glass Density Specimens

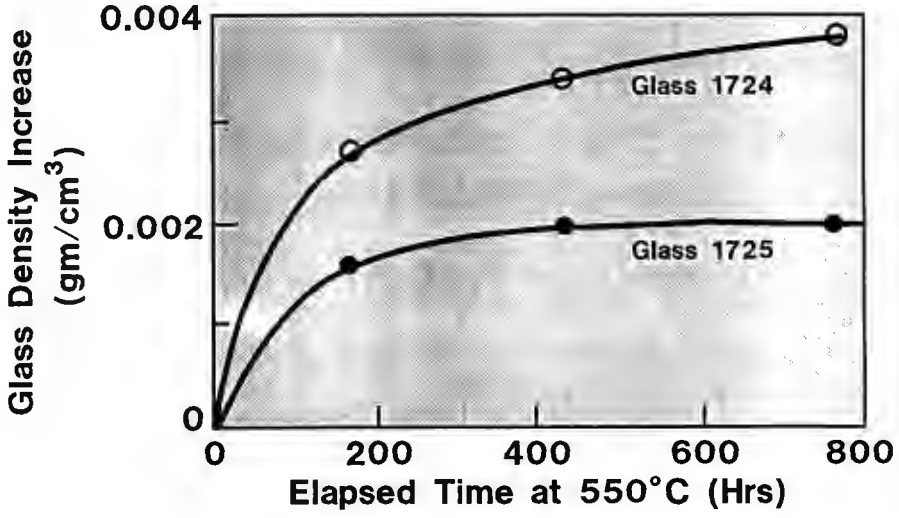


Figure 5. Measured Glass Density Changes with Time at 550°C for Glasses Codes 1724 and 1725

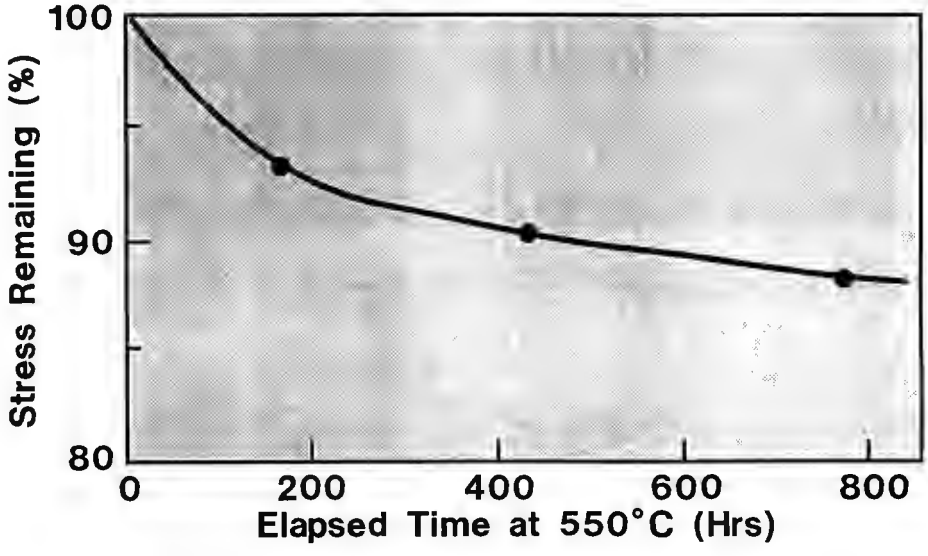


Figure 6. Stress Release Data for Glass Code 1724

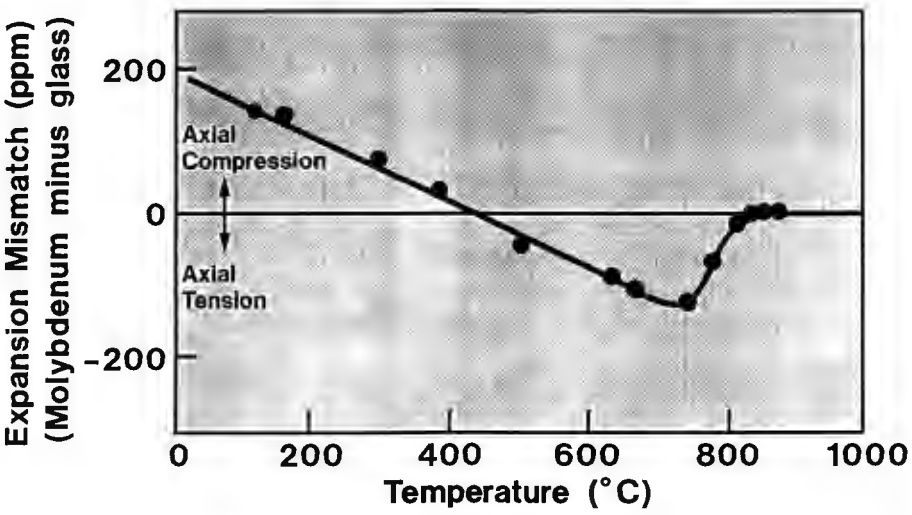


Figure 7. "Target" Expansion Mismatch-Temperature Curve for Glass Code 1725

Materials and Methods Manual: Your Chief Source of Information

Timothy E. Landers
Office of Research Services
University of Massachusetts, Amherst, Massachusetts

Introduction

I am thankful to be able to present a technical paper at this, the 37th Annual Symposium and Exposition of Scientific Glassblowing. Writing this paper has been a positive experience for me.

Recently, my co-workers and myself appeared on the cover of the Wale Apparatus Co. catalog. Since the catalog came out, many people have made comments which I would like to respond to. One comment was, "Do you really wear ties at work?" Yes, we wear ties. "If you wear ties, don't they create a safety problem?" We feel they don't since we have never came close to getting them caught in the lathe. I can't claim that we haven't put burn marks on them. Another comment was "Is Sally making coffee?" No, she is pouring liquid from a beaker to an erlenmeyer flask. Sally wanted me to add that she had never made or drank a cup of coffee in her life.

Now the serious stuff. Where do you turn when you have a glassblowing question? Do you look at all your books on your bookshelf and wonder which one has the answer to your question? Or maybe you grab every book and pamphlet you have and start going through them one by one until the answer is found, or just maybe you call a friend like Joe Walas, the glassbuster, because you know your friend has the answer to your question.

One thing I would like to suggest is that whenever we have a question or a glassblowing problem one of the first places we should turn to is the Materials, Methods, Safety and Hazards Manual. This manual was created to be our chief source of information.

Some of you may be saying, "I don't know much about this manual" or you may be saying, "How could this manual be my chief source of information?" Hopefully, this paper will explain how this manual can be a chief source of information. If you're not acquainted with this manual, hopefully after hearing this paper you will discover how valuable this manual is.

How the manual evolved:

Seeing how the manual evolved can help us know the reason for its creation and how it could benefit us today.

The following information I received from George Sites, Randolph H. Searle and Arthur Dolenga. I am grateful for their help.

Around 1950, Corning made available to A.S.G.S. basic information concerning glass composition and methods for its manufacture. The problem the Society faced was what to do with this information to make it most valuable to the Society members.

After considerable negotiations with Corning, Corning agreed to reproduce the information and design and supply loose leaf binders if the Society would distribute them to its

regular members. This the Society agreed to, and thus the Materials and Methods Manual was created.

Kimble Glass gave information to be included in the manual. They also underwrote the cost of manufacturing more manuals as they were needed.

The Methods and Materials Committee solicited other companies for information that could be included in the manual. As new information was received, it was distributed in sheet form to members. New members were included as their names and addresses were sent to the Methods and Materials Committee.

In 1958, J. Allen Alexander and Randolph H. Searle had the concept of a safety manual. They felt glassblowers were exposed to hazards and unsafe conditions which could lead to injury and possible death.

The A.S.G.S. Board of Directors agreed that the Society members needed a safety manual but decided to combine it with the Materials and Methods Manual. The binders were re-designed to include safety and hazards.

Randloph H. Searle, the first Safety and Hazards Chair, and his committee developed the information in the manual about safety and hazards as it pertains to glassblowers. Arthur Dolenga and staff from General Motors Research Laboratories added to their information and did the final editing.

Over the years, material in the manual has been added and deleted.

If you would like a more precise account of how the manual evolved, look through the Board of Directors minutes.

About the manual:

The manual is presented to anyone who becomes a member of the A.S.G.S. A manual can also be obtained through the A.S.G.S. home office for $45.00.

The desire of the A.S.G.S., as stated in the manual, is that "in time it will become your chief source of information concerning safety, hazards, materials, and methods."

At one time, the manual was divided into two binders. Now the manual has all the information in one binder.

Each manual has two parts. The first part is on safety and hazards and can be identified by its red tabbed dividers. The second part is on materials and methods and can be identified by its white tabbed dividers.

The contents of the first part (safety and hazards) are:

Part I General Concepts of Safety

Part II Unsafe Condition or Hazards Associated with Glassblowing, and the Safety Practices which Control Them

Section 1. Torches and Burners

- Operation of bench burner, using the following fuels:
  A. Propane and oxygen

B. Propane and air
C. Hydrogen and oxygen

- Operation of hand torch
- Operation of hot air torch for plastic welding
- Noise resulting from operation of certain blast burners, plasma jets, air compressors, etc.

Section 2. Compressed Gases

- Handling and using compressed gas cylinders

Section 3. Heating, Furnaces, Ovens

- Operation of glass annealing furnace
- Operation of laboratory-sized hydrogen atmosphere furnace
- Operation of small, high temperature furnace
- Drying or heating materials in drying oven

Section 4. Welding, soldering, sealing

- Operation of spot welder
- Soft and hard soldering
- Seal-off glassware under positive pressure
- Suck seals

Section 5. Drilling, cutting, grinding

- Boring holes in cork and rubber stoppers
- Boring or trepanning holes in glass
- Use of abrasive cut-off saw
- Abrasive blasting for cutting or engraving of glass
- Cutting flat glass
- Cutting cylindrical or spherical glassware
- Drilling with the drill press
- Operation of high-speed grinder
- Using portable engraver or slit saw
- Using the hand file
- Operation of flat horizontal grinder and polisher
- Power sawing with hole saw
- Hand sawing with hack saw

Section 6. Unclassified Unit Operations

- Drawing and handling glass stems
- Inserting glass tubes into cork and rubber stoppers
- Repairing glassware (general)

Section 7. Mechanical Safety

- Operation of vertical chuck
- Use of various clamping devices: bench vise, hose clamp, "C" clamp, and apparatus support clamp
- Machining fluorinated hydrocarbon resins
- Shop housekeeping
- Glass lathe operation
- Operation of the shop high-vacuum manifold
- Operation of hydraulic
- Storage of glass tubing, rod, and raw materials
- Procedures for safe handling of laboratory glassware
- Ten points of lifting

Section 8. Electrical Safety
- Operation of setups using 110 and/or 440 volts
- Static electricity buildup in plastics
- Vacuum leak testing with high frequency tester
- Operation of polariscope
- Operation of variable transformer

Section 9. Chemical Safety
- Cleaning with acids, alkalis, and solvents
- Handling of mercury
- Chemical silvering
- Use of liquid nitrogen refrigerant
- Glassblowing in presence of radiation and radioactive material
- Heating fused silica
- General references

Section 10. Addenda
- Fire and fire extinguishers

The contents of the second part (materials and methods) are:

Section 1. Ceramic Seals
- Sealing glass to ceramic furnace tubes

Section 2. Distillation Apparatus
- Section without articles

Section 3. Foreign Glass
- Section without articles

Section 4. Glass to Metal
- Section without articles

Section 5. Ground Joints and Stopcocks
- Section without articles

Section 6. Lubricants
- Section without articles

Section 7. Miscellaneous
- Section without articles

Section 8. Solder Glass
- Section without articles

Section 9. Tables
- Section without articles

Section 10. Vacuum Techniques
- Section without articles

Section 11. Index
- Outline of vacuum technology

David Chandler, the chair of the Methods and Material Committee, sent me a new copy of the manual. After reading it, I discovered a few things. In general, information in the manual was accurate even though most of it was written around 1962 or earlier. Second, I saw that a lot of sections in the manual had no information in them. Last I realized that there are sections that need to be added because of advances in our field.

Most of the information in the manual is accurate. However, some of the terminology is outdated. One example is the advice to use asbestos gloves "for loading and unloading glassware from the furnace when temperature is above 50°C (p. 33). Another example is the suggestion that cotton wool ear plugs may be helpful in taking away sharp or loud sounds (p. 29).

Some sections in the manual have no information in them. In past manuals these section may have had information in them. Now they don't. One reason for this may be that the information was outdated. Another reason may be the source for the information was no longer willing to donate materials with the information on it.

How this manual can become more valuable.

I feel the manual is valuable now. The question is how can we help it become even more valuable.

One thing we can all do is to customize our personal manuals. By customizing, I mean put the information that we use all the time into the manual if it's not already there. Then our manuals will be our chief source of information. This takes work, but I believe it would benefit us.

Another thing we can do as a Society is to help the Method and Material Committee by giving them suggestions on what information needs to be added to the manual and what information needs to be deleted. At present, they are updating the manual and trying to make it relevant to members' needs.

Conclusion:

The purpose of this presentation has been to show how the Materials, Methods, Safety, and Hazards Manual can be our chief source of information. The manual is not perfect, but I feel it has great potential and can achieve its purpose. As stated in the manual, "in time it will become your chief source of information concerning safety, hazards, materials, and methods."

The challenge we have is to make it a chief source of information. Let's get busy.

The Crystal Palace

by
Don Lillie
President, Lillie Glassblowers, Inc., Smyrna, Georgia

For many years during the late 1700's and early 1800's, many nations held industrial fairs and national exhibitions. It was in 1848 that Prince Albert of England, husband of Queen Victoria, spearheaded an idea for an international fair. One must consider the lengthy animosities history records between countries during this period. The Americans still disliked the British for the 1776 & 1812 affairs. France and England had been enemies for centuries so such a proposal was indeed revolutionary. To condense history and circumvent voluminous text, let me simply state that an international show of gigantic proportion was to be held from May 1 to October 15, 1851, to be located in Hyde Park, and to be known as the Great Exhibition of 1851. It was to be financed by "no taxes on public funds, but by voluntary contributions from industry and private individuals." Proposals for the building included every imaginable shape, size, and composition. Fortunately, Messrs. Joseph Paxton, the designer of the Chatsworth Conservatory, constructed of iron and glass, submitted a revolutionary proposal on a great structure sufficient for the purpose on the space allotted and was dubbed by the London Press as the Crystal Palace.

The footings of the building were to be concrete with a wooden floor consisting of planks spaced 1/4" apart so cleaning would be no problem. (Fig. 1) To test the floor a platoon of guards double timed repeatedly across a test platform. The iron girders would form the main support, the metal bars a gossamer skeleton with a glass skin encapsulating the entire structure. It would cover 20 acres and require over 300,000 panes of handblown glass manufactured by the cylinder method. (Fig. II) This demand required numerous glass factories in Manchester and Birmingham areas to go to round the clock production. The panes would measure 4 feet long by 10 inches wide since the whole construction was based on the number 24 or fraction thereof so wings could be 24 feet in height, the double stories portion would be 48 ft. and the arched transcript about 72 feet tall. Ingenious devices were utilized as shown in (Figure III). This vehicle could carry two glaziers and run in the rain gutters for tracking. This method produced any combination of width and length of structure similar to (Fig. IV). As you can see, this resulted in a greenhouse type structure of unlimited proportions. To the left of Fig. IV is a cross section of the famous Paxton gutter design. The outside precipitation drained into "C" while interior condensation was taken care of by groove "B". These drained into a common downpipe.

As the May 1 deadline approached over 2,000 workers labored 16 hours a day to complete the project. As scheduled, Queen Victoria opened the Exhibition to exhibitors and guests from all over the world (Fig. V). Of interest to us would be the beautiful Fountain of Glass (Fig. VI) by Messrs. Osler of Birmingham. Over 16 feet tall, it was supported by iron bars, but "in no degree interfering with the purity and crystalline effect."

In its five months of operation, over 6 million people visited the Exhibition and three days before closing over 93,000 people attended the last Sunday. This successful venture used its profits to purchase the land now occupied by the Victoria and Albert Museum, Natural History and Science Museum, the Royal College and the Royal Albert Hall in downtown London. To meet the removal date of June 1, 1852, the commission sold the structure to an association who dismantled it and shipped it 25 miles south to Sidingham (Fig. V).

There on the crest of a wide hill with terraced landscaping, an even larger Crystal Palace was constructed. For 80 years this magnificent creation was the showplace of international meetings, annual flower shows, circuses, races, and especially Christmas Programs utilizing the 40 foot pipe organ and choirs on elevated platforms.

Then one night in October 1936, a small fire started in a restroom then spread rapidly to become "out-of-control". This produced one of the most spectacular infernos ever seen with exploding glass and sparking iron. By morning the famous Crystal Palace was no more. How could a building of iron and glass burn? Remember, the floor was wood, the draperies and furniture were combustible and also consider that several wings had research labs located in them, such as one for radar research. Once ignited, the structure and contents were doomed.

Vestiges of its grandeur still remain today. The area is still a park, the footings of the water towers still stand, the terraces, though bare, are still evident. There is even an Active Crystal Palace Foundation dedicated to prolonging its history.

In summary, the objective of this presentation was to familiarize you with the mechanics and intricacies of one of the greatest glass structures ever built, to emphasize its short, first existence and to amplify its second life of grandeur.

So I hope I have added to your cultural knowledge, I hope I have stimulated your historical curiosity and I hope you all strive to become true professionals.

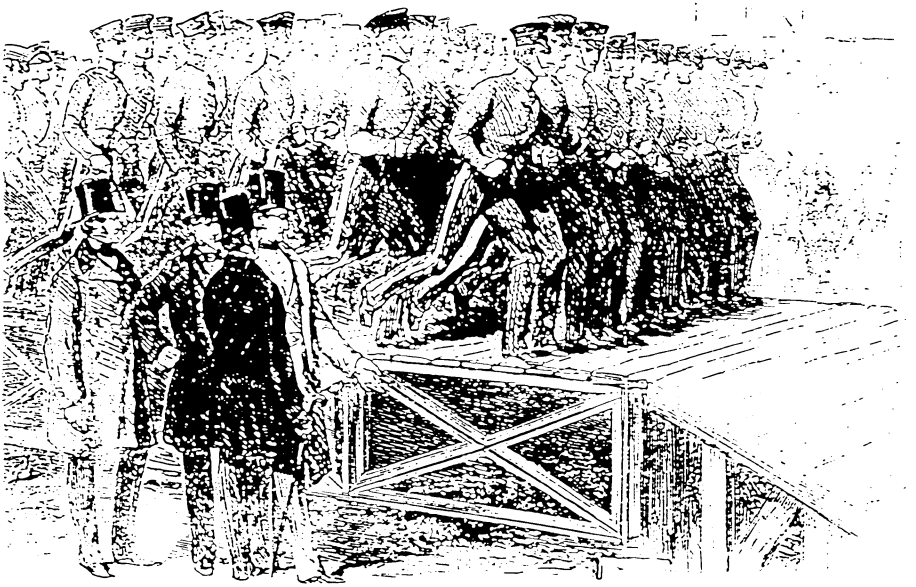


Figure I. Soldiers on platform

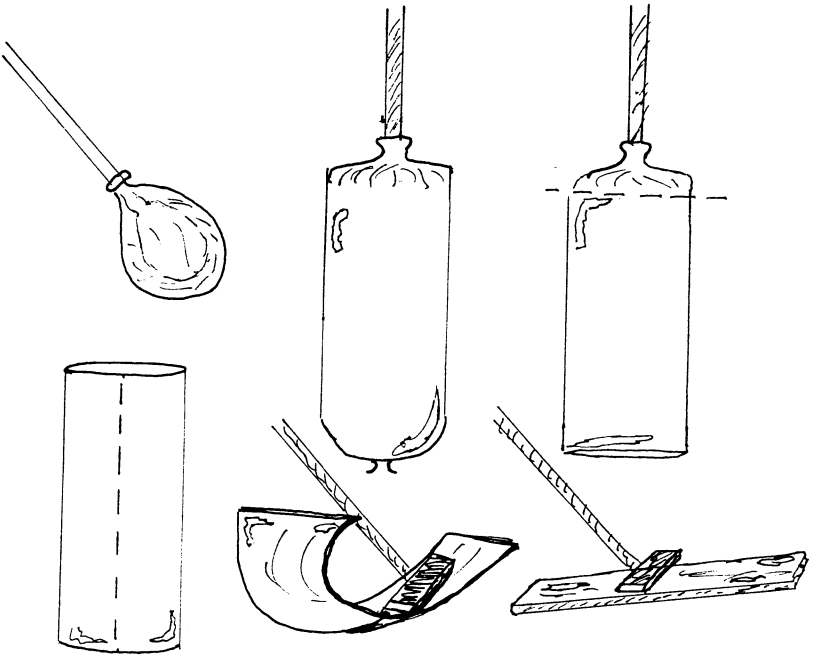


Figure II. Cylinder method

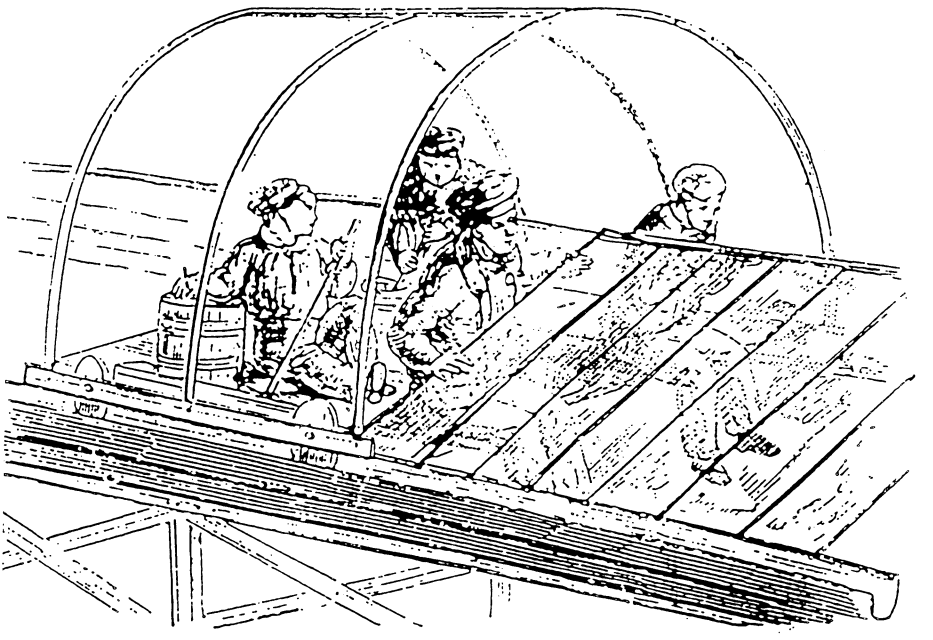


Figure III. Glazier vehicle

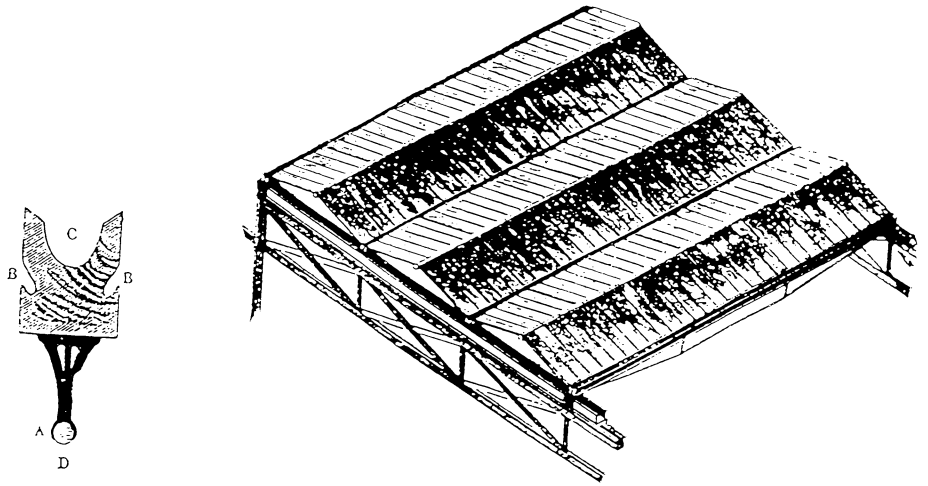


Figure IV. Section of wing and Paxton gutter

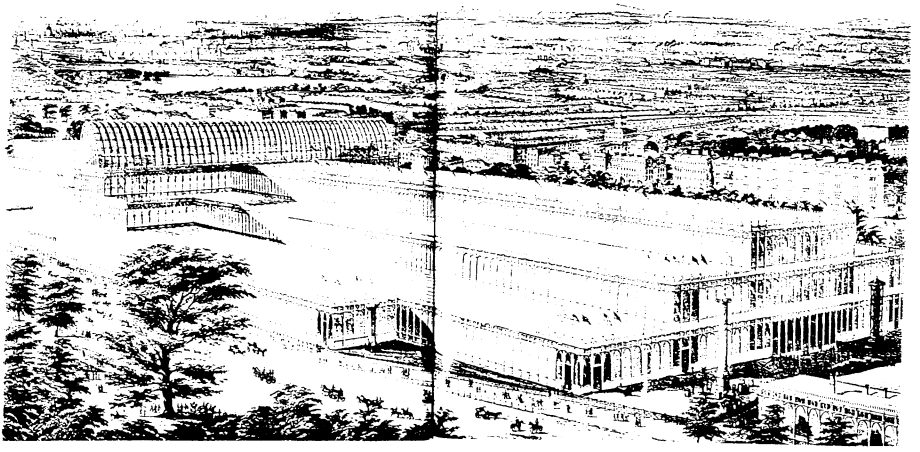


Figure V. Over view of Crystal Palace

Figure VI. Inside view of the transcript & crystal fountain

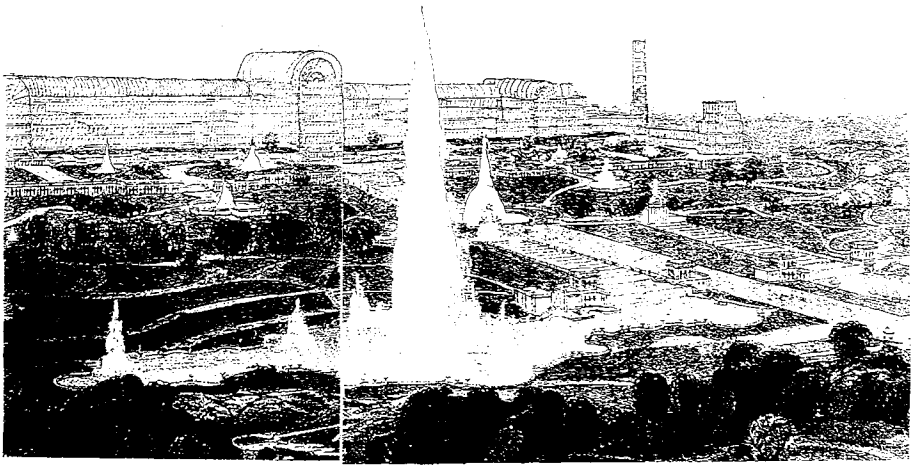


Figure VII. Sidingham Crystal Palace

Modification of Flat Lap Grinders to a Diamond Grinding Surface

by

Don & Thom Lillie

Almost every glass shop has a flat grinder for the purpose of grinding flanges, plate edges and various squaring techniques. Most were designed for use with carborundum to be fed either by hand, V-shaped trough or similar method. Unfortunately, most of the powder is slung across the wheel and is never used. Recycling the grit is alright on roughing jobs, but will incorporate residual grits and glass powder. The messy, dusty resultant environment compelled us to look into the feasibility of a new grinder with a diamond surface.

The simplest approach is to invest $4,000 in a new machine, but still the problem of disposing of the old grinder remains. We decided to convert our 24" Wilt Flat Lap to a more efficient and practical implement.

First, we removed all traces of carborundum by flushing the catch pan and scrubbing the cast iron wheel to eliminate all particles. The cast iron surface was swabbed with acetone to remove any oil and to dry the surface for inspection. The wheel was checked for flatness with a 24" straight edge to make sure no grooves or imperfections had been created through previous use. If the surface is not satisfactory, the wheel will have to be removed and sent to a machine shop for resurfacing.

Diamond disks with adhesive backing can be purchased in diameters from 12" to 24". There is also a Velcro option which would allow easy removal for various grits but we were skeptical about the cushioning effect so we decided on a 24" disc of 220 grit with an adhesive backing (fig. 1). If your old wheel has an opening or recess in the center area like the old Sommer and Maca Units then these centers can be filled and leveled with Bondo or metalized epoxy so a flat continuous disc can be applied. If this sounds too difficult then the center ring can be carefully cut out to fit your existing wheel. We gently heated the cast iron wheel with a heat gun prior to applying the adhesive disc.

To lubricate the diamond surface we purchased a "Little Giant" circulating pump with plastic pan from W.W. Granger, Inc., Model 7P078. This unit has a five gallon capacity and can pump three gallons per minute. The unit is supplied with a brass control valve for control regulation, a 12" plastic beaded "goose neck" so that flow direction (fig. 2) can be obtained and 36" of 5/8" diameter tygon tubing to connect the pump to the machine (fig. 3). The plastic pan reservoir contains slotted sides so a partition can be arranged to create a trap area to capture heavy particles in the recirculation cycle. The pump with the described components costs approximately $150.

Since our grinder has a drain to the rear of the cabinet, the pump assembly was located close to the grinder on the floor (fig. 4). The pump was wired into the existing switch assembly to utilize the foot switch or the continuous operation switch. It has been suggested that if you can rewire the unit to run in reverse, the diamond surface will give extended life.

To protect the diamond surface when the machine is not being used, Thom Lillie designed and fabricated a unique cover. A circle of appropriate diameter is cut out of 1/4" hardboard. A set of 1" x 2" wood pieces are screwed to the underside to form a cross brace (fig. 5). Each center section is removed and notches are formed on each end of

these braces. This creates four hooks which allows the lid to be hung on the splash ring during operation (fig. 6). After use (fig. 7) the lid is replaced to form a cover which sits 3/4" high on the rim to allow the interior to dry. This also allows a flat surface to utilize during adjacent operations.

One of the most important items in the operation of a grinding unit is the coolant or emulsifier. Many types are available, premixed and ready to use, but gallon quantities do not last long in a busy glass shop. We have found that Somaca #265-1000 drilling coolant is an excellent medium (fig. 8). It is inexpensive, concentrated, and has no offensive odor, or caustic ingredients. I strongly recommend an efficient lubricant for longevity of the diamond surface. Our Somaca coolant was purchased in a five gallon container for about $95, but is so concentrated that it will easily treat 500 gallons of water.

We are extremely pleased with our modifications and hope that our experience can give you proper direction.

Fig. 1. Diamond disc cemented to iron plate

Figure 2. Gooseneck allows coolant to be directed

Figure 3. Plastic tank is perfect for modification

Figure 4. Tank positioned behind unit

Figure 5. Specially designed lid for unit

Figure 6. Lid hooks to aid during operation

Figure 7. Lid position for storage

Figure 8. Somaca #265-1000 coolant forms efficient yellowish green libricant

Construction of An Annealing Oven

by
David G. Maul
The Dow Chemical Company, 856 Building, Midland, Michigan 48667

Good morning A.S.G.S., it's good to see everyone here. This slide lecture will start off with some photos of my early glass work. Then I will discuss how I built the annealing oven, and will conclude with some photos of my recent work that was made and annealed in the annealing oven.

The early work, some made at Greenfield Village in the early 1970's and some done later in my studio, are soft glass pieces made from the furnace, some early American tableware, then some lamp worked pieces, and neon sculptures from lead glass.

Here is a slide of some of the scientific glass blowing I've done. This is a 22-liter flask, jacketed with a 50-liter flask. (Figure 1.)

Now lets discuss the construction of the annealing oven. The first thing to consider is you must determine the size and type of chamber you want to use. Will it be a bell type that raises and lowers, a top loader or, as in my case, a front loader? Some people have made annealing ovens from 50-gallon drums or even an old chest freezer.

I choose this large mailbox design. I first saw this type of annealing oven down at Nick Labino's studio in Grand Rapids, Ohio. I really liked the simple design and the way it looked, so I gave him a call and asked him what size he had. He said his was 24" x 36" x 22" high. So that's how I built my first one.

Most of these photos are of the first oven, or Lehr. The new one I'm working on is 36" x 47" x 31" tall. (Figure 2.,3., & 4.)

One thing, when your deciding on your chamber size it's important that you remember the outer dimension minus your wall thickness equals your inside dimension.

OD - WT = ID

When I first built this one, I had great visions that it would be real large inside, but by the time I put the insulation in it, the inside dimension got smaller than planned. The final I.D. was 19-1/2" wide x 31-1/2" deep x 16" high. This is not too bad though, I can anneal a 22-liter flask in it. The new Lehr will have about 7" thick walls, with a final inner dimension of approximately 29" x 34" x 18". The thicker walls should make it even more efficient to operate.

I had the mailbox-shaped chamber built by Dave Thornton, a retired tinsmith. The small one I think was $150.00 and the larger one about $200.00 to $250.00

You can, of course, build it yourself, but for the sake of getting it together promptly, I hired a skilled tinsmith to do the sheet metal. I painted the bare sheet metal with a high temperature aluminized paint inside and out.

Once you get the chamber you are going to use, then you can determine what kind of frame you want and how you are going to build it.

I basically just purchased angle iron and welded the frame together, and I put rollers on it so it would be portable. If you don't have a welder, you can drill and bolt it together if you want. This is a little slower, but you really don't need a welder to do this. You can also have two shelves under the oven for storage.

Now let's discuss the components that will change your box on rollers into a useful annealing oven.

You'll need to get, from a hardware store, eight junction boxes for wiring the terminals, 2 switches, and power input receptacles. Photos show front and back of the power panel that is affixed to the frame with wing nuts for easy removal and servicing. The thermocouple and pyrometer are on top.

Here is a list of all the components I used. They were purchased from Paoli Clay Company, 6879 Paoli Rd., Paoli, Wisconsin, 53508-9743, telephone number 608-845-7000. This is a good source for glassworking equipment.

The components list includes:

- Two 10 amp, 115 volt heating elements
- One pyrometer and thermocouple
- One Dayton® timer
- Two Chromalox® variable switches

and from the local hardware you can get:

- Two ground fault circuit interrupter receptacles
- Two 4" x 4" junction boxes
- Four 2" x 4" metal junction boxes
- Two 2" x 4" open metal junction boxes.

This annealing oven operates on 110 volts. It has two 110 volt circuits. The bottom circuit goes through the Dayton® timer, and the top circuit does not. Each circuit draws 10 to 14 amps. To operate, you plug both circuits into a receptacle that is wired with number 10 wire to a 30 amp circuit breaker in your breaker box.

When I want to get the annealing oven up to temperature, I turn them both on. It takes about an hour to get to 565° C for Pyrex®. If I'm annealing soft glass, I turn the top circuit off when temperature reaches 1000° F and the bottom 110 circuit maintains the oven temperature at 980° F.

The timer used in conjunction with the Chromalox® switch on the bottom circuit is used to control the time and cooling speed when annealing heavy soft glass items.

For annealing Pyrex® I usually just go up to 600° C then off. This seems to work well for Pyrex®. Do not open the door until the oven drops below 450° C. For soda lime and lead glass, keep door closed until room temperature is reached. No peeking, a cool draft can crack hot soft glass.

Regarding elements, if you're a purest and you want to make your own nichrome elements, there is a chapter on building electrical heaters starting at pg. 422 in Wheelers Book on Scientific Glassblowing. It's very informative when you're doing this kind of work.

To insulate the chamber and door, I used 6 or 8 lb. density FibreFrax® or Koawool® ceramic fiber. It comes in 1- or 2-inch thick by 24- or 36- inch wide rolls. They don't give this stuff away, it is quite expensive.

The method of laying these layers in the oven is called stack bonding. Basically, if you are going to have a four-inch thick wall, cut the fiber blanket into 8" wide x 32" long pieces and fold lengthwise. You then end up with a U shaped piece 4" wide x 32" long x 2" thick. Then I just stack them on top of each other starting at the bottom, or from the fire brick up, creating an arch (see photo 5.) I placed metal bands to help support the arch, but if you pack the fiber strips in tight enough you might not need them. It is important to make sure no metal comes in contact with the heating elements.

To carve out the soft brick for the heating elements you can get a ball type grinder at the hardware store and put it in a drill press. You then set your height on the drill press with a stop and you can carve out all these grooves, once you're set up, on all the bricks first. Then it will be easy to run your elements in the grooves. The elements come in a compressed coil and will have to be stretched out to the proper length. Measure the distance from the grooves you have in the soft bricks in the chamber. Attach the coils with V-shape strips of heavy nichrome wire (I think you can buy these from Wilt).

On the door, I held the four 1" thick batts with large penny washers and stainless nuts and bolts, going through to the outside. It seems to work well.

I have a small hole here on the front side of the oven to bring in an evacuation line if you want to evacuate a column or plasma globe at high temperature. (Figure 6.) To attach the elements to the terminal posts, use 3/8" or 5/16" threaded stock to make the posts - these go from inside the oven to the outside of the oven in the junction box. The threaded posts must be shielded or sleeved with a ceramic or thick fused silica tube where it passes through the metal chamber wall. On the outside of the oven in the junction box, you attach the electrical lines to the posts coming from the chromalox switches to the posts. This outside junction box is then covered with screen to keep air cooled. Inside the oven you attach the nichrome elements to the posts with nuts and washers. Don't attempt any of this if you don't feel comfortable with wiring electrical devices. It's basically just black and white. Black is hot [+] and white is ground [-]. 5/16" galvanized threaded stock seems to really hold up well for the terminal posts.

Now, every annealing oven, over time, needs maintenance. If your element breaks off at the post, just turn it off, let it cool and come back a little way on the coil and stretch it out, rewrap around the terminal and bolt again. Every time you shorten your element it will get hotter, so don't shorten it too much.

To install the thermocouple and pyrometer, drill a hole in the top of the oven to place the thermocouple. It should extend into the chamber at least 2 inches. Mount the pyrometer on top, in the front where you can see it. I put a metal clad shield over the wire, going from the thermocouple to the pyrometer. What I like about the pyrometer from Paoli is it reads in centigrade or Fahrenheit. I read in centigrade for Pyrex® and in Fahrenheit for soft glass. This is a dual purpose annealing oven.

To test a thermocouple for accuracy, you can immerse the thermocouple into a boiling liquid of a known boiling point. So if you used boiling water, you could see that it was basically reading in the ball park.

To wire the Chromalox® switches and the timer, you just follow the instruction sheet that comes with these devices, it tells how to do it. You can use these for 110 or 220 volt. The variable Chromalox® switches are great little things. They work by cycling on and off, and they're only $14.00. This method of controlling temperature and timed cooling is a low tech and inexpensive way to effectively anneal glass.

Considering safety, I wired both circuits through Ground Fault Circuit Interrupter (GFCI) receptacles because I wanted to make sure if anything ever went to ground with an element going bad, or possibly the terminal post going through the wall grounding out on the metal wall of the chamber, it would trip this switch. And it worked. It's a safety device that alerts you to problems and it will reduce the chance of an electrical shock - I'm glad I put these on. I got the GFCI receptacles at Builders Square (they have the best price on them).

Another point on safety; wear a respirator when you're carving the grooves in the bricks for the elements, and when stacking the ceramic fiber. It pays to take care of your lungs. You can also get a ceramic fiber rigidizer to keep the dust down after your oven is complete. You just spray it on and it hardens the top layer.

This photo of the timer and chain drive mechanism shows how I mechanically turn down the oven when annealing soft glass. (Figure 7.) By using a juice can, a chain, and a large nut from a 1/2" black pipe union fitting, I let the timer turn down then shut off my oven automatically. This is a time proven method of mechanically controlled cooling that I've used almost daily for 6 years.

Also, note the terminal boxes attached to the outside of the annealing oven - these are air cooled. I have screen on the boxes that can be removed so you have access to the terminal posts. (Figure 8.)

The plugs or power supply cords are # 10 wire extension cords with a male plug on both ends. I plug the one end into the control panel on the annealing oven, frame first, then plug the other end into the power supply on the wall (a 30 amp circuit). I made this oven 110 volt so I could use it many different places, but you could make it 220 if you choose.

After you build your annealing oven, then you can have some fun filling it up. Here is a photo of some production lead glass bells. The beauty of this oven is that you can use it for soft glass or Pyrex®. (FIGURE 9)

Here are some photos of my recent work. All these pieces have been annealed in the annealing oven. (FIGURE 10,11)

At this time I'd like to thank the American Scientific Glassblowers Society for uplifting the glassblowing profession. I appreciate how it's continuing to develop recognition for glassblowers in a professional manner. I would also like to pledge to continue to share technical information on topics of interest to glassblowers by presenting papers at future symposiums and participating in the workshops. I want to challenge some of the glassblowers that have never given a paper to consider sharing some of their pearls of wisdom with fellow glassblowers and in that way we can continue to grow as a group. We are the keepers of the flame. We are, at times, magicians and artists, but above all we are professionals. We are the American Scientific Glassblowers Society. Brothers and Sisters in glass, "Flame On".

Thank you.

You should have all wiring checked by a licensed electrician. Wiring should meet all local and state codes. -David Maul

Figure 1.

Figure 2.

Figure 3.

Figure 4.

Figure 5.

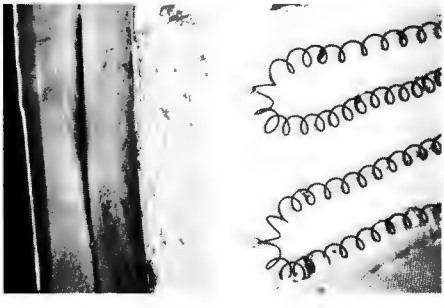


Figure 6.

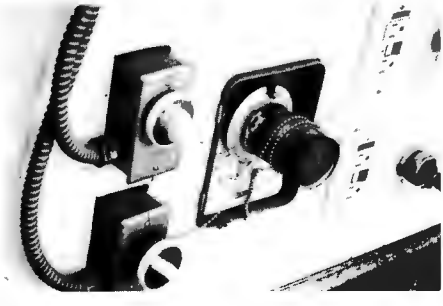


Figure 7.

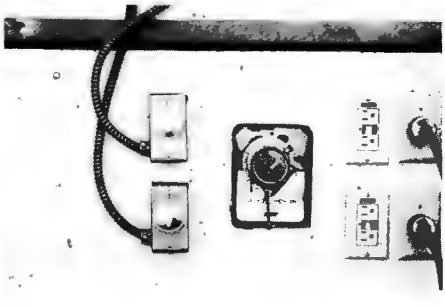


Figure 8.

Figure 9.

Figure 10.

Figure 11.

Marketing Your Glass Shop

by
Frank Meints
The Upjohn Company, Dept. 7287-25-1, Kalamazoo, Michigan

It makes a lot of sense to advertise your service, especially in these recessionary times. If your potential customers are not aware of your products and services, your future in glassblowing could be in question. No matter whether you work in a university or large corporation, a production shop, or a specialty shop, your livelihood depends upon a need and demand for glassblowing.

How can you advertise and market your service? You can advertise in journals like Fusion, send direct mail to potential customers, put up signs, use posters, use a booth at the symposium, display your work as artistic glassblowers do in the malls, and produce catalogs and brochures. Your approach for marketing will depend upon whom you are trying to reach and how much money you have to spend.

The specific area of marketing that I would like to share with you is the brochure.

Two years ago, The Upjohn Co. Glass Shop published a brochure. After seeing our brochure, several glassblowers and vendors asked for copies and were very complimentary of the content and impact our brochure had on the readers. The success of our brochure is what prompted me to share my experience even though my training is not in the advertising field.

A brochure can be small or large, low cost or expensive and is appropriate for just about every glass shop that I know of. Here are my suggestions on what to put in the brochure:

The Front Cover

Your title must clearly identify who you are to your potential customers. If you want to catch someone's eye you can use special graphics or typefaces, like glowing letters or torch flames. You may use a picture of your product or products as a background for your title. If you use your company name, logos, department names, address, or phone numbers, remember to use smaller print to avoid detracting from your main title. Keep it simple: it will be more effective.

Inside Summary

Explain in a few simple words what you do. This can be in the form of a list of your services or a mission statement for example "We make scientific glassware for the chemists," or "we make glass insulators for electric power companies." Your department head may want to write the introduction to emphasize costs savings or other special functions of the glass shop.

Explain how your customer can contact you for this service. Does he call, fax, write a letter, or come down the hall to your shop? Is there a special form to fill out?

Optional Items

Some important items to include might be: What are your hours? What about emergency service? What is the expected turn around time? Do you make "house calls"?

Explain your charge rates for time, material, shipping and method of payment. Avoid including hourly rates or other costs that are subject to change unless you plan to regularly update and republish your brochure.

We at Upjohn conducted a study a few years back and found that certain glassware was economical to repair. We made a note of this in our brochure to convince scientists that this could be done without compromising safety. You may also want to include a statement to that effect.

You may want to explain to your customers about how to package and ship glassware to you for repair or modification. I occasionally receive a container with hundreds of pieces of broken glass that started out as an expensive item with only a chip or small crack. Because of poor packing the customer lost the item and I lost the opportunity to repair it.

It should go without saying but people are not always thinking about your health and safety. Include a reminder that the glassware brought to your shop should be as clean as possible, free of radiation or other chemical contamination.

Because my shop is in the middle of several buildings and also some distance from many of my customers, I included a map. This makes it easier for off-site customers to find my shop.

If you desire you may include photographs. These can depict your shop equipment, a glassblowing process, yourself and/or others. I think people like pictures of flames the best.

The Insert

Inserting or stapling your business card is one good method of giving something to your customers for quick reference. I use a 3" x 9" insert that contains a summary of important information. You could list all your services on this insert. Use the back of the card if you need more room.

The insert may include lists of names, phone numbers etc., even prices. If there are changes, you just get a new insert card printed up instead of having to redo the whole brochure. Keep this in mind when you make your brochure. Get your message out but don't be specific on items that are subject to change unless you want to throw away a lot of time and money printing new brochures. When you have your rough draft, let a 12-year-old read it to see if what you put down is understandable. After that, show it to other adults and glassblowers for grammar and completeness.

Final Form

You can make electrostatic copies of your own design, fold them in half or thirds for a low-cost brochure. Copy centers have a variety of paper and colors from which to choose. With a good typewriter or laserprinter you can make an attractive composition. Examine other brochures for ideas. The one for my shop was done inside the company by our own photographers and printers. I would suggest that you make it the very best you can afford. Some people may relate the quality of your brochure to the quality of your product or service.

Front Page

Necessary

- Clear Title

Optional

- Picture of Your Product
- Logos
- Company Name
- Department Name

Inside

Necessary

- Introduction
  - mission statement
  - what you do
- List of Services
- How do we Obtain this Service

Inside

Optional

- Emergency Service Time Requirements
- Costs Breakdown
- Advantages of Repairing Glassware
- How to Ship Glassware
- Safety Considerations
- Map
- Pictures
  - of you
  - your shop
  - work in progress

Insert

Necessary

- Business Card

Optional

- Slip Card
  - phone
  - fax number
  - address
  - map
  - detailed list of services

Early Physics Radio Research at the University of Wisconsin-Madison

Harley A. Nelson
University of Wisconsin, Physics Department, Madison, Wisconsin 53706

There was a time when sharing was not common in research. This is evident in the radio research at the University of Wisconsin early in the 20th century. Professor Earl Terry was experimenting with radio in 1902 in the Physics Department in Science Hall. He and his colleagues were using a spark transmitter to send code.

When he heard that an Iowan named Lee DeForest had invented a triode tube in 1907, he saw tremendous potential for its application to his work. But, when he contacted the manufacturers in hopes of obtaining some of the tubes for his research, he found that they were not sharing the discovery. So, in 1916, he assigned Karl Jansky, one of his students to design and construct three-element power vacuum tubes as his thesis project. He was to study what went on in the tubes and what they could be used for. At this time, Jansky said, "Learning to build vacuum tubes meant learning to be a glassblower. If there is a technique to try the patience of Job, it is glassblowing. Many were the tubes I carried through the various stages of construction and, in some instances, through the process of pumping out the air, only to have a crack develop somewhere. Thus, several weeks' work would end and I would have nothing to show for it except experience. Then I would start over." (Fig. 1)

They went through many experiments in making the tubes. They had to overcome the problems of annealing, glass-to-metal seals, and vacuum pumping. Pumping was a very time consuming process. Their first vacuum pump was a Sprangle, or liquid piston, pump—a mercury reservoir controlled by a stop-cock, which allows mercury to fall down a column. The stop-cock regulates the flow so there is space between the drops of mercury. Molecules of air are trapped between the drops and are expelled out the bottom of the column. The mercury is collected at the bottom and poured back in at the top. This pump is very slow and requires constant supervision. I built a model of this pump for our museum and it took approximately 18 hours to pump 27 inches of mercury. They had problems with mercury backstreaming in the system. This pump is the same type that Thomas Edison used to pump out his light bulbs. One can be seen in Edison's laboratory at Greenfield Village in Dearborn, MI. One of the techniques that Professor Terry used was a hand-operated forepump to back up and improve the pumping speed of this system (Fig 2).

Professor J.R. Roebuck of the University of Wisconsin Physics Department made a great improvement in the vacuum pumping system while doing research on expansion of gases. In about 1917, he built a high pressure liquid air machine, which made possible the use of liquid air traps on the vacuum system. This stopped backstreaming and made the system more efficient because the liquid air did a certain amount of cryo pumping. As the vacuum pumping systems became better, more current was applied to the tubes to drive gases out of the metal prior to sealing the tubes. This was a very delicate procedure because too much current would crack the glass. Even with the advances, these tubes were not reliable because of the inability to get a good vacuum. They could last anywhere from seconds to hours.

The enthusiasm of Professor Terry and his students was not shared by all faculty members in the Physics Department. They could not understand why anyone would want to talk over the air. There may have been valid reasons for their skepticism. On

one occasion, the head blew off the liquid air compressor and they had to amputate a man's leg. Another time, a student went to check the power supply with his earphone wires hanging and he got 3000 volts through the ears. He woke up in the hall with a headache like he never had before. And the first torches they used were made out of pipe fittings and it was not uncommon to have blowback. These may be a few of the reasons why some members of the faculty thought that this research was a waste of time and called it "Terry's Toy."

In early 1917, the University of Wisconsin station 9XM (now known as WHA) was able to broadcast speech (Fig. 3). About this same time, World War I began and the Navy ordered all radio transmitters to cease operation for fear of sabotage. Station 9XM was ordered to continue experimental broadcasting, sending weather forecasts to the Great Lakes Naval Training Station, which relayed them to ships on the Great Lakes.

Since 9XM was able to stay on the air during this period, the researchers were able to make great strides in their glassblowing and vacuum techniques and to make more efficient and powerful tubes. This was the time when they each began to specialize in certain areas. Jansky was doing most of the glassblowing while others concentrated on the metal work. Professor Terry was willing to share the knowledge he had acquired in this research. In 1918, 9XM and the Physics Department were moved to the newly constructed Sterling Hall. It was decided in 1923 that a full time glassblower was needed at the University, to be shared equally by the Physics and Chemistry Departments. His salary was $2400 per year.

During their research, Professor Terry's students, Jansky included, encountered bursts of static coming over the air. The origin of these bursts was a mystery. They thought it was either in their tubes or somewhere in space. It wasn't until Jansky was at Bell Laboratories in 1932 that he made his very important discovery of cosmic radio waves.

Professor Terry was always interested in new ideas. He was corresponding with a Mr. J. Steensland, who had a theory of sending printed copy by radio. If a phonograph record is played at the broadcasting station, anyone with a receiving set, phonograph, cutting needle, and wax record could make a copy of the record being played. If an electro-type was placed on an Edison phonograph, the needle, in passing over the rough and smooth places on the electro-type, would set similar sound waves in motion, which would be recorded at the other end. If the machine were run at the same speed, the markings would be the same, thus transmitting electro-type. At the time, this seemed like an impossible idea, but it was later accomplished.

Professor Terry died of a heart attack in 1929, leaving much of his work unfinished. Shortly thereafter, in 1934, WHA was moved to Engineering and then made a department in itself. It is recognized as the oldest station in the nation. Thanks to people like Professor Terry, Karl Jansky, and Professor Roebuck, who were willing to share ideas and research, great advancements were made.

The people who founded the American Scientific Glassblowers Society saw the need for sharing to advance our skills and knowledge.

Acknowledgements

I wish to thank Ken Mass, Univ. of Wisconsin, for his help in reproducing photographs and gathering information; Prof. Emeritus Ragnar Rollefson, former student of Terry, for his personal information; Laura Hunt, WHA; and Jay Poster for editing assistance.

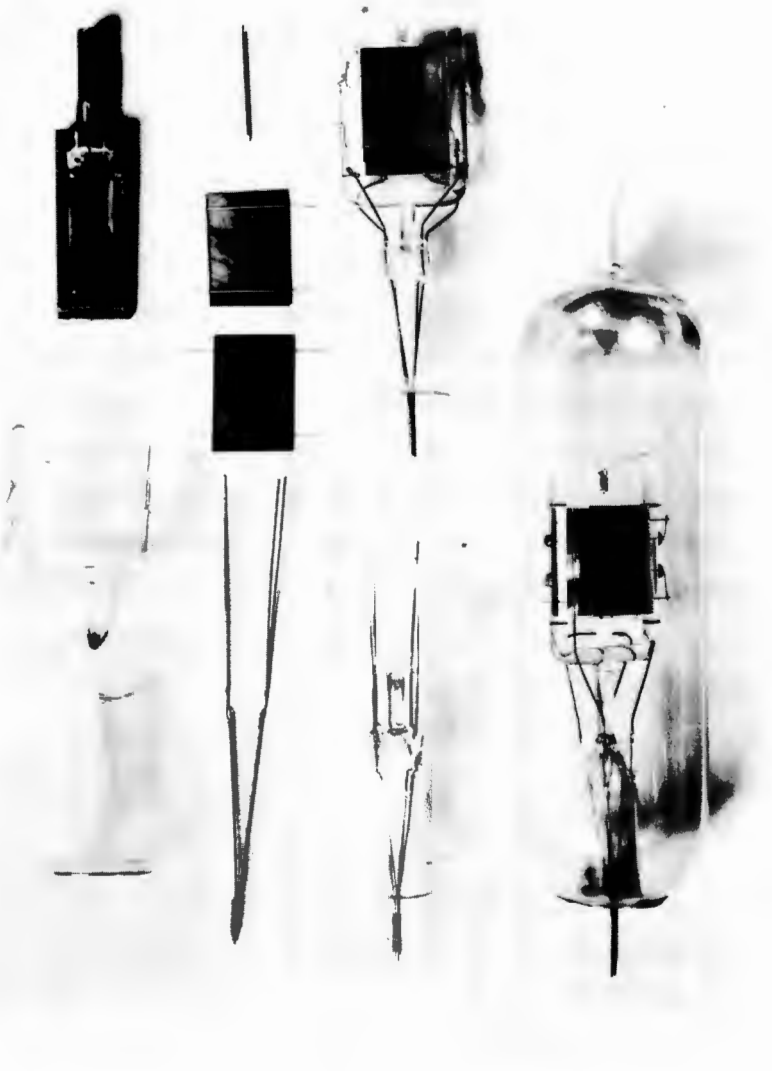


Fig.1. Vacuum tube construction

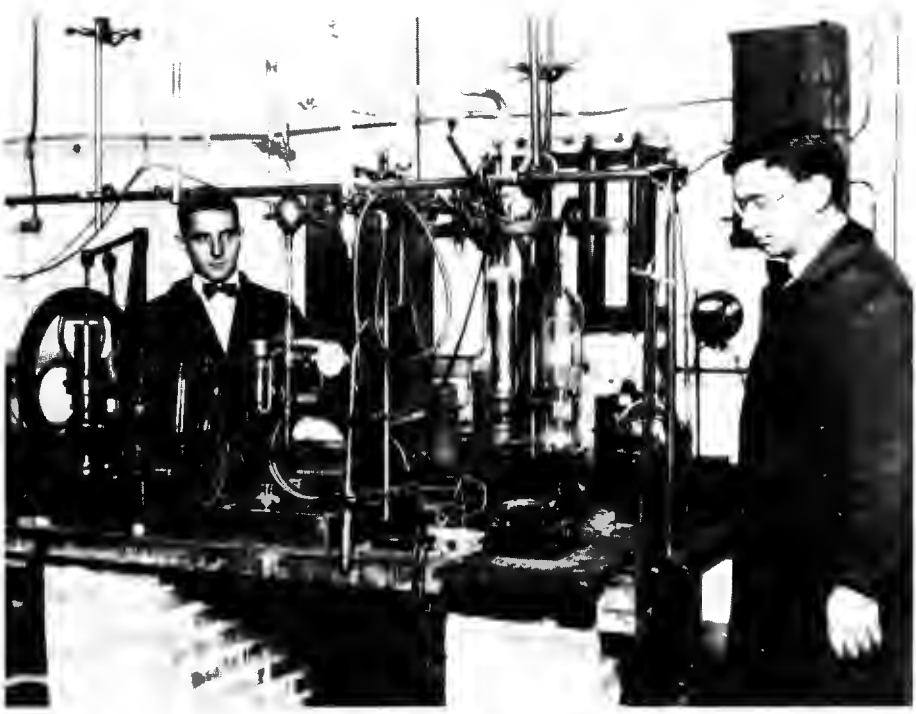


Fig. 2. Professor Terry (right) with his pumping system.

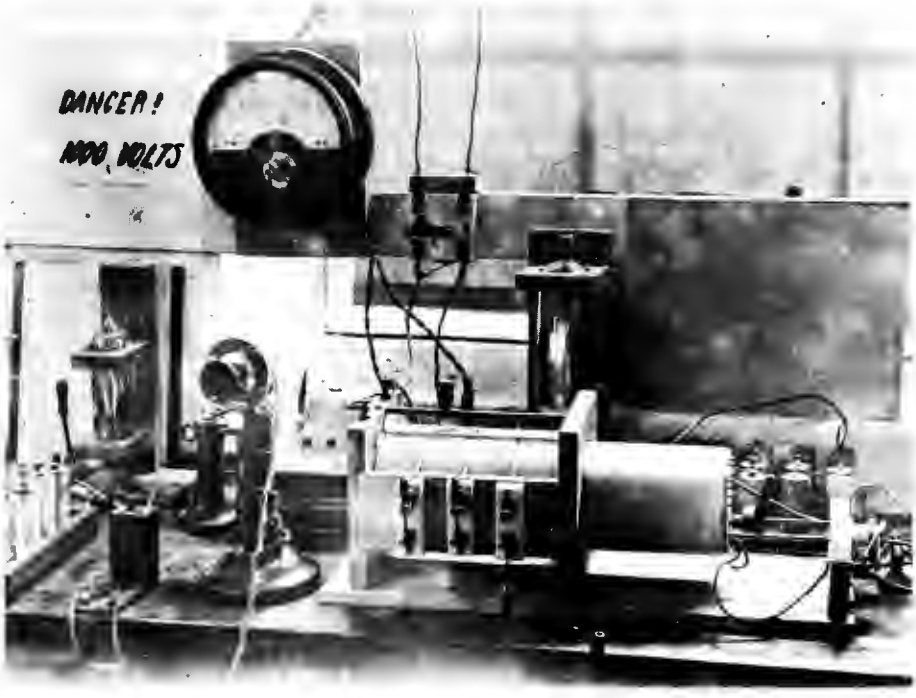


Fig. 3. Original transmitter.

Technical Workshops

Name	Gary Coyne
Company	California State University, Los Angeles
Workshop Topic	CAD Applications for the Glass Shop

Name	Randy Hansen
Company	Dow Chemical Company
Workshop Topic	How to Use Rollers

Name	Dave Maul
Company	Dow Chemical Company
Workshop Topic	Forming Thermometer Wells

Name	Rudolf Schlott
Company	State University of NY at Stony Brook
Workshop Topic	Quartz Transfer Tape Seals

Name	Dumitru Costea
Company	The Glass Menagerie, Inc.
Workshop Topic	Working with Conturax Tubing

Name	Joseph S. Gregar
Company	Argonne National Laboratory
Workshop Topic	Unique and Different Vacuum Holders

Name	Marvin Molodow
Company	Texas Instruments
Workshop Topic	Fabrication with Quartz Surface Mix Burners

Name	A. Ben Seal
Company	Supelco, Inc.
Workshop Topic	Tooling a Small Volume Flask

Technical Posters

Name	Tim Drier and Lisa Malchow
Company	Dow Chemical Company (Drier) and G. Finkenbeiner, Inc. (Malchow)
Poster Title	Holders: Practical, Functional, with a View Toward Safety

Name	Ian B. Duncanson
Company	Notre Dame University
Poster Title	Bubble Traps for Closed Systems

Name	Igno D.J. Dur
Company	Netherlands
Poster Title	Construction of a High Pressure (5-10 bar) Permeation Cell

Name	Ken Everingham
Company	ICI Americas, Inc.
Poster Title	Fabrication of a Micro Reservoir for 3 or 4 Place Multi-channel Micropipets

Name	Doni Hatz
Company	State University of NY at Stony Brook
Poster Title	Borosilicate Optical Windows

Name	Linda Kellc
Company	AT & T Microelectronics
Poster Title	Construction of a Fused Quartz Injection Atmospheric Pressure Manifold, Metallorganic Chemical Vapour Deposition and Process for Semi-conductor Laser

Name	Charles L. Patterson
Company	University of Wisconsin - Madison
Poster Title	Double Line Vacuum Manifold

Name	A. Ben Seal
Company	Supelco, Inc.
Poster Title	Sealing Glass Frits in Gas Chromatography Columns

Name	Joseph West
Company	Eastman Kodak Company
Poster Title	Vacuum Holders

Exhibitors

Ace Glass Inc
1430 N. W. Blvd.
Vineland NJ 08360

Carlisle Machine Works
PO Box 746
Millville NJ 08332

Chemglass Inc
3861 No Mill Rd
Vineland NJ 08360

Corning Inc
HP-AB-01-09
Corning NY 14831

Corning Melting Services
SP-FR-28
Corning NY 14831

Dynacut Inc
PO Box 156
Springtown PA 18081

Ed Hoy's Int'l
1620 Frontenac Rd
Naperville Il 60563-1762

Friedrich & Dimmock
PO Box 230
Millville NJ 08332

GE Quartz Products
21800 Tungsten Rd.
Cleveland OH 44117

Glass Warehouse
800 Orange St.
Millville NJ 08332

Heathway Inc
903 Sheehy Dr
Horsham PA 19044

Kaufman Glass Co.
PO Box 1729 1301 Northeast Blvd
Wilmington DE 19899

Kimble Glass Inc
537 Crystal Ave
Vineland NJ 08360-3257

Litton Engineering
PO Box 950
Grass Valley CA 95945

Nortel Machinery Inc
1051 Clinton St
Buffalo NY 14206

Pegasus Ind. Spec. Ltd.
143 Mills Road
Haax Ont. L1S 2H2
CANADA

Poco Graphite, Inc.
1601 South State Street
Decatur, TX 76234

Richland Glass Co
Tuckahoe Rd
Richland NJ 08350-0404

Schott America
3 Odell Plaza
Yonkers NY 10701

VM Glass Company
580 N.W. Blvd
Vineland NJ 08360

Wale Apparatus Co
400 Front St Box D
Hellertown PA 18055

Wilmad
Route 40 & Oak Road
Buena, NJ 08310

Wilt Industries Inc
Rt 8
Lake Pleasant NY 12108

Witeg Scientific
14235 Commerce Dr
Garden Grove CA 92643

1991 Symposium Attendees

Jo Ann Abbott
George Adams
Sean Adams
Charles Amling
Zalman Ancel
Gary Anderson
Steven Anderson
S. Antczak
John L. Arkebauer
Arnold Arndt
Jurgen Ausborn
Joel J. Babbitt
Joel M. Babbitt
Scott Bankroff
Frank Barrale
Jerry Baumgarten
David Beaubien
Marc Bedard
David Behm
Melanie Behm
Emmanuel Bellantoni
Al Ben-Moche
James Bertwell
Fred Birkhill
Kenneth Bittner
Bryan Bivins
Gwen Bivins
John Bivins
Pat Blake
Theodore Bolan
Ronald Bolnick
Anastacio Bonilla
Martine Boudreault
Hugh Bowe
Ken Brick
Keith Brodnick
Jamieson Brookes
Allan Brown
J. Brown
Lauree Brunfeldt
Robert Brunfeldt
Greg Burns
James Byrnes
Martina Byrnes
Dana Caldwell
Robert M. Caldwell
Robert Calvarese, Sr.
David Campbell
Mike Campbell
Jack Capel
Raymond Carew
Jack Carson
John Carson
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Vera Carter
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M. Castelletti
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David Chandler
Dawn Chandler
Leonard Chandler
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M. Christman
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Gayle Clarke
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Brenda Cloninger
Jerry Cloninger
Patricia Combes
Murray Connors
Paul Cope
Dominic Cordiano
Barbara Costea
Dumitru Costea
Melvin Courtney
Gary Coyne
M. Craig
Robert Crummy
Cory Curtis
Edmund Czohara
Bonnie Daenzer
David Daenzer
Gilbert Daenzer
Katrina Daenzer
Dieter Damrow
Mike DeMasi
Alvan Ditchburn
Darcie Doering
Arthur Dolenga
Carol Dolenga
Mary Dolenga
James Downey
Paul Doyle
Tim Drier
Barbara Duncanson
Ian Duncanson
Igno Dur
Irene Dusek
Leo Dusek
Dr. James L. Dye
Harold Eberhart
Wolfgang Eberhart
Richard Elvin
Diane Everingham
Kenneth Everingham
Pat Fagnani
S. Ferguson
John Ferruzzi
Kevin Fogle
Kim Foley
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John French
Sandy Friedman
Jim Futrell
Dan Gale
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Robert Gant
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Thomas Garling
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Marsha Ginsburg
Carl Goetz
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Mark Gouge
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David N. Gritz
Mark Grozinger
Siegmund Grozinger
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Laura Haddad
Barbara Hagy
Henry Hagy
Bob Halbreiner
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Julia Hammond
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Barbro Hanold
Mats Hanold
Randall Hansen
Larry Harmon
Bruce Harwood
Doni Hatz
Brian Head
Roy Heimbrock
Paul Henrickson

Lona Hill
Winfield Hill
Donald Hodgkins
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S. Hopper
Barbara Hovey
David Hovey
Tommy Howe
Dorothy Hoy
Ed Hoy
Laurie Huff
Darlene Hughes
Joe Kamrad
Gregg Kaufman
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Sharon Kelly
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Timothy Kornahrens
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Janet Kowalczyk
Richard Kowalczyk
Richard Kramme
Brigitte Krmpotic
Milan Krmpotic
R. Kvet
Barbara Lafler
Barry Lafler
Andy LaGrotte
Timothy Landers
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Manfred Langer
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John Legge
Ron Legge
Frederick Leslie
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Linda Lillie
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M. Marusha
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David Maul
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Elaine Meints
Frank Meints
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Keki Mistry
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M.A. Molodow
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Mac Nudd
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Lee Schuldt
Sue Schulze
William Schulze
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Nettie Severn
Peter Severn
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Allan Wootten