Proceedings

THE EIGHTEENTH SYMPOSIUM
ON THE
ART OF GLASSBLOWING

1973

THE

AMERICAN SCIENTIFIC GLASSBLOWERS SOCIETY

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THE EIGHTEENTH SYMPOSIUM
ON THE
ART OF GLASSBLOWING

Sponsored by

THE AMERICAN SCIENTIFIC
GLASSBLOWERS SOCIETY

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THE SCIENTIFIC GLASSBLOWER IN CULTURAL PERSPECTIVE

NORMAN ALEXANDRE

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When one considers any small sector of our high technology culture, there is an *a priori* stance that the particular sector under scrutiny only exists in terms of its interrelatedness to other sectors of society. Scientific glassblowers, despite certain unique characteristics are not excepted. We do not exist for and to ourselves, but make particular and specialized contributions to the larger scientific and technological community, of which we are an integral part.

To be sure, we are a very small minority group — so small in fact that our vocational appelation is seldom listed as a job category in compilations of vocational careers. There is no justification however for a minority group to be considered inferior, either by personal introspection or societal pressures. To the contrary one may find singular support for our uniqueness as specialized craftsmen continuing a 3000 yr. old profession as glassworkers.

A characteristic of contemporary culture is to make significant judgments about a person's identity in terms of what he does and with what degree of efficiency it is done by him. To the layman, upon hearing the answer to his querie — what do you do? Glassblowing evokes a startling response varying from suspicion to awe and followed by interjections about death. Either we are in a dying art — or as "artists" we are about to die ourselves. Everyone subscribes to the demonstrably untrue theory that glassblowers have a brief life expectancy. Dismissing that pernicious theory, I would like to comment on the prognosis of our job description— "scientific glassblowers". Today, economic silicosis is to us as much a threat as the lung disease was to glassblowers 200 years ago, particularly so for anyone who resists change.

For the most obvious fact in society today, all over the world, is that of change. Change is a basic element in life — yet we have a natural resistance, as if that which is inevitable must at least be slowed down to avert certain destruction. Stability is what humans seem to desire most. We accept change only when it is in a direction which we approve — then we call it growth, orderly development or progress. The reason for friction is that not everyone can differentiate between stability and stagnation, development and destruction.

Our 20th century is a time in which change has become so rapid, that the way of life for so many people has caused personality disruption. The thesis of the book "Future Shock" is that the accelerated speed of change is too fast for most people to comfortably adapt themselves. Permanent transition seems to be antithetical to most people's needs. It is not simply that the neighborhood has changed and one does not know

the way around a city because of tearing down and rebuilding — although these are the more obvious and visible signs of change.

Changes are more deeply felt when value systems are questioned and the basic institution of life seems threatened. Every institution has been challenged in the last 20 years — including that of work. Training for work does not guarantee following one vocational pathway for a lifetime. The route to education for life may not be kindergarten through college with traditional courses as the norm. Male/female roles are not determined by the stars. Governments change, new nations emerge, old ones become subsumed, nothing is permanent but change. A basic issue confronting us all is how to nurture in stability while being educated for a life of change. There seems to be only one stance — flexibility with integrity.

To be flexible does not mean a "couldn't care less" or a *laissez faire* attitude; to the contrary it presupposes an ability to analyze the data, recognize viable alternatives and make appropriate choices commensurate with the facts. If that definition is valid we may assume that an inflexible person is either unable or unwilling to become so. A dogmatic assertion to the nonrelative value of one's position is a prediction for a collision course the classic case of the irresistible force confronting the immovable object.

My concern today is directed towards those with the ability for adaptation to change. For every skill each of us have today, every role we play every function we perform — a learning process was the intermediary from inability to ability. The performance principle presupposes the practical and theoretical education and training — whether on the job — in continuum or by formal courses.

The more varied and diverse and the more intense and lengthy the training — the greater become the opportunities for viable choices, in other words the more flexible we potentially may become.

We all know that a college degree, even at the baccalaureate level is not a prerequisite for a scientific glassblower. We all know superb glassblowers who did not even finish high school — for whatever reason. The glassblower to whom I was apprenticed for 7 years left school at age 14 to learn his trade. The point I want to make is that these men from whom we learned our craft lived in a different world than we do today. After learning a trade and profession there was reasonable expectancy that one would do the same thing until retirement. It was a stable and predictable world, the parameters of choice were limited and certainly less anxiety producing than those of today. Higher education was a luxury of the upper middle class and a meritorious reward for the gifted poor.

Today however higher education is viewed as an individual right just for its intrinsic value. With very few exceptions I venture to say that each of us would want to see our children go to college, if the children wanted to go. We would be prepared to make certain financial sacrifices in order to help them achieve their goals. If asked why — we would unvaryingly say something like — "I want my kids to have a better oppor-

tunity than I did". And we say this because we know that they will be able to choose from career options otherwise unavailable to them and because we recognize the value society places on a degree from a University and also because we as taxpayers pick up that tab anyway! We are also suppliant to this trend because of the subtle cultural pressures which suggests that the more meaningful and rewarding careers require a degree.

The puissance of this cultural trend will unfortunately create a judgment ethos where negative pragmatics assert that the non degreed person is bereft of the academic aptitude required to matriculate. Hence he and she is relegated to the less meaningful and less rewarding careers.

Now where does that leave us? It would be fatuous indeed for me to make anything more than a guess. There are no hard data sources of a sociological nature with respect to scientific glassblowers. Very few of us do quite the same kind of work as another. A fairly simple distinction might be between those glassblowers in production circles or those who function in support and service facilities. Now we can plug into economics in terms of goods and services. On the national scale 6 out of 10 workers in the U. S. are in service industries. The G.N.P. (for whatever value it has as an economic index) shows that we are approaching the point where goods and services are 50-50. The larger the part of the G.N.P. the nation, chooses to take in services, the greater the need for higher efficiency and production in the goods producing sector. We need to take note that government employs 20% of the man hours worked in the U. S. economy.

The cry from management is for increased productivity, because they claim, the output per man hour is not high enough (in industry) to meet its environmental, energy conservation, and other goals. Pressures of international competition make this an especially urgent need.

The arguments against higher productivity are legion, a few predominant ones are:

American workers don't want to work anymore
The country has gone soft (not you and me of course)
The protestant work ethic is passé
It doesn't pay to become too productive — you may automate yourself out of a job
Anyway what's the percentage — the boss and the stockholders get the gain.

The rebuttals that seem worthy of merit are that in a general sense higher productivity increases the standard of living for all. Technology does indeed eliminate some jobs — but creates others — usually more interesting and better paid. Salaries increase in a higher rate than dividends.

The argument goes on and the deficit trade balance is computed in billions of dollars. Since our last symposium the president of the United States instituted wage and price controls, which, economist John Kenneth Galbraith affirms will continue as long as we have large corporations and large unions. In agreement with the best 19th century minds the President

promised never to use economic controls. The last third of the 20th century demands them, because of the recognition of the discrepancy between the neo-classical market-oriented economy system which is theoretically accepted in the U. S. and the pragmatic recognition that economic control is in the hands of a few large corporations.

We have always believed in the morality of the open market — that the consumer has the ultimate authority over what will be produced because of the forces of supply and demand. On the national level what we actually have is substantially a planned economy. The open market has been dominated by a system in which the megacompanies exercise control through careful planning and technological organization. The non corporate producers — including most of us are still subject to market conditions and have very little power in the U. S. economy.

The power of the corporate technostructure comes from its access to information and its ability to mold consumer attitudes through advertising. The power within the corporation is shifting and has shifted away from the owners and stockholders and into the hands of corporate management for whom maximization of profit has ceased to be of primary importance. It is in the best interests of corporate management to perpetuate the economic myths of the free market place, the power of the stockholder and that economic well being is reflected by the G.N.P.

Some think it is theologically defensible that St. Peter has asked applicants to heaven what they have done to increase the G.N.P.

In contrast to national economics and its philosophies we need to consider that during the past few years educated manpower in a number of occupations has suffered from unfavorable inbalances in supply and demand. Scientists and engineers have experienced increasingly high unemployment plus slowing growth rates in starting salaries, extended periods in job searches, and significant underemployment. Today as many as 20,000 new science graduates are unable to get work in their field.

The prediction for the supply of Ph.D.s over the next 20 years is likely to exceed the demand by universities and colleges, their major employers.

It is quite clear that efforts are being extended to curb the sharp inflationary rise and every sophomore in economics knows that the way to halt inflation is to increase unemployment. But the most sophisticated economists cannot come up with a credible system to stabilize the economy with full employment and minimal inflation. Our contemporary situation is one of screaming about inflation and screaming about the present solution, particularly as we would rather be a part of the problem than the solution.

So far I have managed to refrain from giving any advice. I am reminded of the schoolboy's essay on Socrates. "Socrates went around the place giving free advice, so they poisoned him".

I do however intend to challenge you in the light of some of the cultural manifestation to which I have briefly addressed myself. The in-

creasing movement towards specialization will without doubt affect most of us. I predict that the major companies producing a wide variety of scientific glass apparatus will drastically curtail their catalogue and concentrate on producing fewer items with increased efficiency and with a higher standard of workmanship approaching excellence. Presumably the management decision will be determined by marketing data showing which items fall below the criteria for continuance. The immediate spin off from such policies will be bread and butter lines for smaller companies who do not choose to specialize. For the glassblowers this trend will create a paradox, on the one hand the generalist may find himself unable to compete with the specialist, while the specialist may find himself economically trapped by the generalist. In other words the era of the prima donna is rapidly diminishing. The person who thinks he can still do any glassblowing function as well or better than another is trapped in the conceit of his own ego.

A more realistic appraisal recognizes the need for humility, where the acknowledgment of another's craftmanship is no cause for the diminishment of one's own. To the contrary, expressed appreciation of the high degree of skill manifested by another is an indirect complement to all glassblowers following our vocation. A new image is very important to our continued survival and growth. Never underestimate the power of public relations. The current projected image of glassblowers is far from desirable. In the local setting we each contribute to that image, either favorably or unfavorably, consciously or unconsciously. The way we are treated is causally connected to the image we have of ourselves and the way we project that image. We have a responsibility to each other not to project the negative image of a professional. E.g., how one dresses, converses, eats lunch has little to do with how one blows glass, but it has a marked impact on the image building process. It just isn't good p.r. for instance to arrive for the day's work in overalls carrying a lunch bucket when the desired projection is one of professionalism.

It seems important to me that there becomes some desirable image of what a scientific glassblower is seeking to become. An image which all of us can embrace in the context of unity and charity. The past significant step in that direction was in the formation of our Society. Every glassblower is singularly indebted to the finding fathers of the A.S.G.S. for providing the first cohesive symbol for our participation. The goals and purposes of the A.S.G.S. are eminently worthwhile despite the deplorable fact that many scientific glassblowers choose to ignore the corporate benefits of membership.

As a symbol, the A.S.G.S. can provide the nucleus for our corporate identity, it can help create the ethos for determining whether we are tradesmen or professionals. I think that the adamant feelings of disaffinity with trade unions bespeaks the fact that we are not members of a trade. I have already stated that rugged individualism manifested as the prima donna complex is passé. That only leaves us with the category of a profession, and we have yet quite a way to go to assume full stature as professionals. Our society has experienced singular difficulty in establishing

criteria for levels of accomplishment with commensurate certification. It will never happen unless we really want it to happen and are willing to work and rethink through our former ideas in the context of flexibility and inexorable change. If we fail to meet these challenges society will pass us by and we will be relegated to a situation analogous to the village blacksmith.

In conclusion I challenge each of you and particularly the Board of Directors and Executive officers to consider these concerns with the utmost seriousness and provide us with the leadership which will make us proud of our Society and proud to be scientific glassblowers.

CONDUCTIVE COATING OF BOROSILICATE GLASS

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INTRODUCTION:

Obtaining electro-conductive coatings on a borosilicate glass (Corning 7740 or Kimax K.G. 33) for laboratory use is a rather simple and inexpensive operation. It's application is frequent in research laboratories and the electronics industries. In many cases substantial savings can be made by using metal coated glass instead of the solid metals in conductivity devices, reference electrodes and others. For such electrodes, the scientist prefers the use of noble metals such as gold and platinum.

In principle the electro-conductive coatings are obtained by firing a chemical solution permanently onto the glass surface. The resistance of the coating will depend on the thickness of the metal layer and the size of the coated surface.

Besides gold and platinum a few other metals can be considered such as silver, tin and others.

PREPARATION OF THE GLASS:

The area to be coated needs to be clean and definitely free of fingerprints. To clean the glass, a strong laboratory glass detergent or a mixture of H. F. (48%) 3½% per Volume and NH_4F, 2.5 oz. per gallon could be used.

After cleaning, rinse the glassware with distilled water and dry it in an oven or in a hot air stream. Do not use alcohol, acetone or other products to speed-up the drying time unless the products are of a high purity. Products of a lesser quality tend to leave a film on the glassware and will contaminate the surface.

CHEMICAL COATING:

The gold or platinum in liquid form are commercially available and applied on the clean glass surface by means of a soft hair brush. When the glass item is round it can rotate in the lathe while an adjustable ring burner can be used for hand support (Figure 1).

Apply an even and thin coat on the glass. Too heavy or too thin coats may either blister or fire out weak. After a few experiments you will obtain satisfactory results. The brushed on solution can be removed from the glass with acetone soaked cotton.

The coating has to be dry prior to start of the firing. To speed-up the drying time, the coated glass can pass through a warm air stream. Natural drying can happen in a few hours.

Figure 1

THE FIRING:

The prepared glass item will be brought into an oven at room temperature and heated up to between 620 and 650°C or above the annealing point (525°C) and below the softening point. Some oils used in the coating solution will produce fumes. These fumes must not be trapped in the furnace, therefore it's recommended to leave the oven door slightly open at the beginning of the firing cycle if no ventilation is present in the oven.

When the coating turns black or "smoked-off" we can close the furnace.

When the required temperature is reached we should maintain this temperature for 5 to 10 minutes prior to switching the oven off and cool to room temperature.

If the coating is not heavy enough or the resistance too high, a second layer can be applied directly on top of the first one. No pre-cleaning is necessary as long as the coated part has not been touched.

The gold and platinum fired film can be removed from the glass with a solution of three parts HCl and one part of HNO_3. This solution is called aqua regia.

INTERNAL GOLD AND PLATINUM COATINGS OF CONTAINERS:

Plating the inside of a borosilicate glass container may cause some problems, as by the firing the fumes are trapped in the container and cause the coating to be of a bad quality.

To obtain a good result, a metal pipe B (Fig. 2) connected to a vacuum pump or aspirator can be inserted in the container A (Fig. 2) to be coated on the inside.

The metal pipe should not touch the bottom of the container. The fumes developed by firing will be exhausted through the metal pipe and the hot airflow coming in on the top of the glass container will guarantee a fairly even temperature inside.

When a vacuum pump is used for exhausting system, the metal pipe can be run through an ice bath C (Fig. 2) to protect the pump.

All other preparations remain the same.

LARGE SURFACE ELECTRODES WITH PERMANENT LEAD-WIRE:

A thin platinum wire (.3 to .4 mm) can be sealed into borosilcate glass with good results. Before sealing the wire into the glass, the platinum is molten into a little bead at one end (Fig. 3, B). This end will be sealed into the glass in such a way that part of the bead is exposed and elevated above the glass surface. (Fig. 3, C). The elevated part can be filed even with the surface afterward. (Fig. 3, D). After coating with platinum or gold a contact will exist between the electrode and the conductive surface (Fig. 3, A). The copper and platinum wire combination is assembled prior to sealing permanently. It is recommended to recoat the electrode area two or three times to assure a good contact.

SOFT SOLDERING OF COPPER LEADS ON PLATINUM COATED SURFACES:

The area where the Copper wire has to be soldered needs to be coated two or three times over to assure a heavier layer of metal. The soldering has to be done using a minimum of solder and heat. This is a fast way of connecting, however the connection is not very strong mechanically and it also takes some practice to perform this technique.

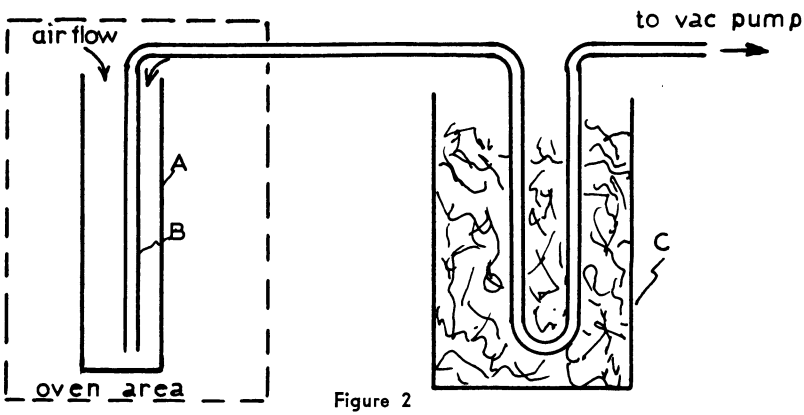


Figure 2

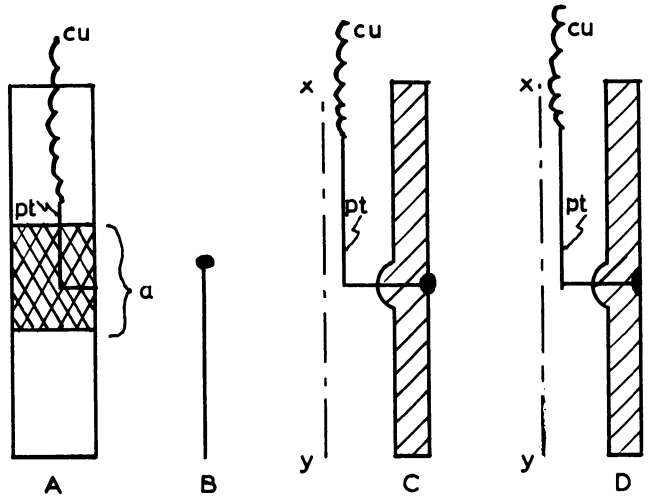


Figure 3

COATING OF PLATINUM OR GOLD LAYER ON GLASS WITH COPPER:

To attach lead wires on a platinum or gold coated surface in a more secure way, part of the platinum or gold surface can be coated with copper prior to soft soldering the lead wires.

A glass tube A (Fig. 4) for example can be gold or platinum coated on the bottom section B with a strip going up to the top.

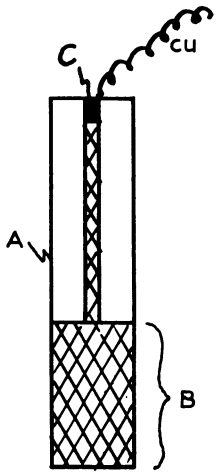


Figure 4

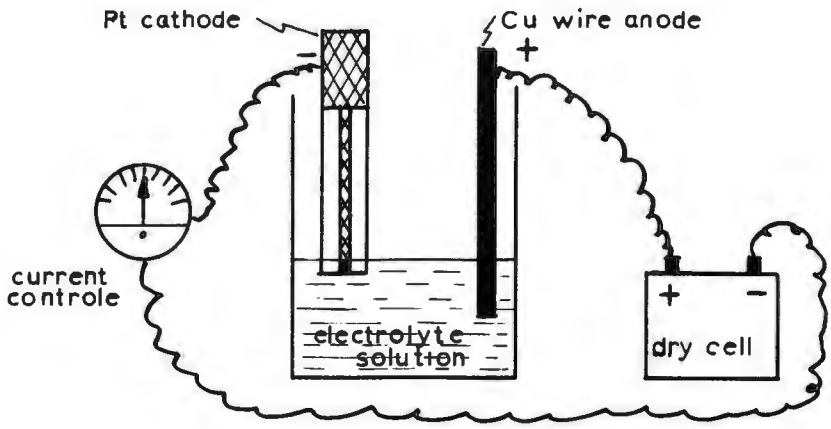


Figure 5

Platinum area C next can be electroplated with copper on which a lead wire can be soldered. This type of connection is mechanically much stronger than the previous method described. The electrocopper plating can be done rather easily as illustrated in Fig. 5 and Fig. 6.

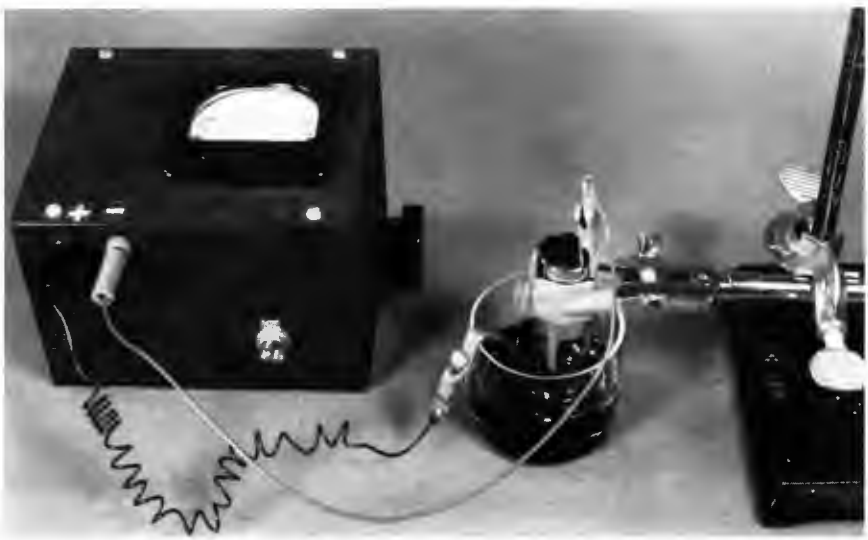


Figure 6

The platinum or gold coated part is immersed into the copper plating solution to the required depth. The plating solution for copper:

50 ml concentrated $CuSO_4$ solution
4 ml concentrated H_2SO_4.
50 ml ethyl alcohol (95%)
35 ml distilled water.

Keep solution in glass bottle with glass stopper.

CONCLUSION:

Besides the subject matter explained and discussed there are a number of other possibilities. Think just about the coating used as an heating element in micro glassware or the decorative purposes involved. Also the possibility exists of soldering metal pipes onto glass pipes. The applications for this technique are numerous.

REFERENCES

Bulletins No. 5 and 9 Hanovia Liquid Gold Division.

DIAMOND DRILLING IN GLASS

J. P. LUNZER, President

Lunzer Industrial, Diamonds, Inc.

Because of its brittle and abrasive nature, glass can pose serious machining problems. This is especially true when it comes to drilling straight, clean holes through glass components. Unless great care is taken, chipping or cracking can happen, as well as drill burn-out or excessive tool wear.

Many people first approach glass drilling with the view that it is essentially similar to drilling metals or wood. A steel twist drill, of course, can be used routinely on these materials. It won't work on glass. And although carbide drills and copper or brass tubes dipped in an abrasive have been successful, the method is slow and laborious.

Without question the ideal tool is a diamond-edged coring drill.

THE DRILL

Basically, the drill is a metal cylinder to which diamonds have been affixed. This can be achieved in three distinct ways.

1. *The impregnated diamond drill.* Here, the diamond section consists of a quantity of natural or synthetic diamond grit, correctly sieved to an exact size, mixed with a holding matrix and bonded to the steel core shank.

2. *The plated diamond drill.* Here, a single layer of diamond particles are plated to the end of the core. The diamond material adheres to the surface of the shank and has far greater concentration than the impregnated type. The principal drawback of the surface-plated drill is that when the diamond surface wears off, the drill must be discarded.

3. *The electro-deposited drill.* Here, the diamond section is built up in layers by an electroplating process. This type of construction has all the advantages of the impregnated drill, but has a much thinner wall.

THE DIAMOND

Long after the advent of synthetic diamond, natural grit was used almost exclusively in the manufacture of all types of diamond core drills. Today, there are specially formulated synthetics — such as De Beers MDA and General Electric MBG-II — that have proved successful in certain glass applications. The choice between natural and synthetic diamond is not a simple one, since each type may be superior according to certain conditions. The important point is, there is a diamond abrasive to suit exactly the requirements of any given application. Hence, the glassmaker must consult his tool supplier in order to determine which type should be used for a specific application.

DIAMOND SIZE

Diamond grit for drills comes in sizes generally ranging from 40 to

240 mesh. Choice of mesh size is largely determined by the size of the drill and the material being drilled. It is highly important that the correct size be used. A 40 mesh diamond drill will not give the same production performance as, let us say, a 140 mesh unit.

DIAMOND CONCENTRATION

Although "diamond concentration" may be familiar to every user of diamond tools, its meaning and significance is not widely understood. Put simply, concentration refers to the quantity of diamond in the drill. For example, consider two drills containing 40 mesh size diamond grit, one having 80 concentration and the other 40. The former drill would contain twice as many diamond particles as the latter. And, understandably, it would be more expensive. But high concentration does not automatically mean better performance. A lot depends on the material being drilled, feed rates, drill condition, etc.

DRILL EQUIPMENT

The basic requisite in successful glass drilling is a sturdy, vibration-free and well-made drill press. Since the ultimate goal is to achieve chip-free holes, every component involved in the drilling process is important. The drill press must be mounted on a firm base and the spindle checked periodically for run-out.

DRILL SPEEDS

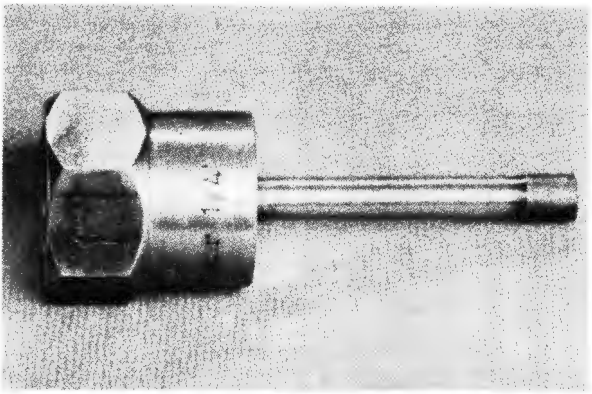
There is a considerable divergence of opinion as to the speed at which drills should be run. The following, however, is generally accepted and recommended:

Drill Diameter (inches)	R.P.M.
Up to 1/2	4,000 - 7,000
9/16 to 1	2,000 - 4,000
1 1/8 to 1 1/2	1,500 - 3,000
1 5/8 to 2	1,000 - 2,000
Over 2	500 - 1,000

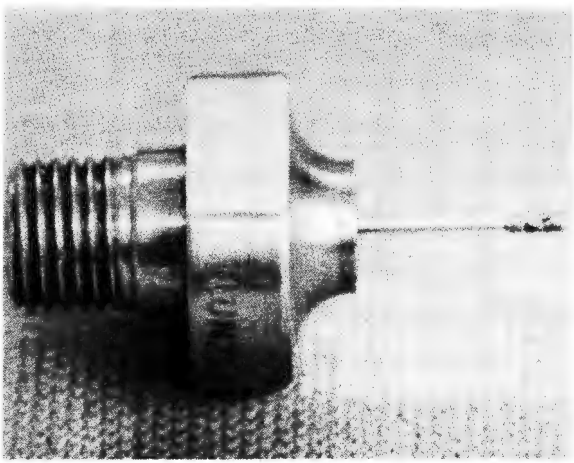
The exact speed of the drill will depend also on the type of drill used, how the coolant is applied, the material being drilled, whether starting or finishing a hole, and the experience of the operator.

COOLANT

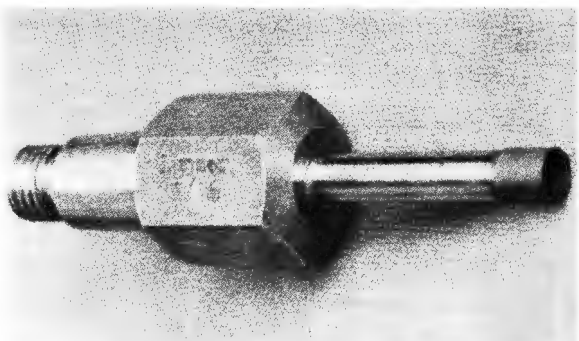
Ideally, drills should be mounted in a collet and attached to a water swivel so that coolant is constantly supplied under pressure through the center of the drill. There are various opinions on the advantages of using oil-based or other type coolants rather than water in drilling. But of prime importance is to see that the drilled is cooled under pressure, thereby freeing the core and preventing core hang-up in the drill.



IMPREGNATED DIAMOND CORE DRILL SET IN STANDARD COLLET FOR WATER SWIVEL.

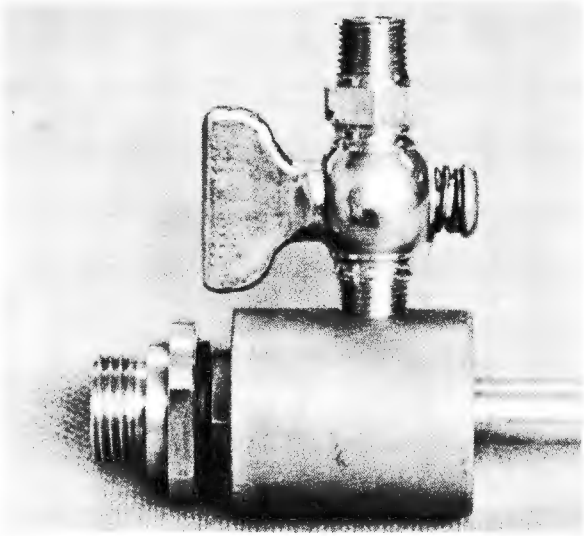


.035" DIAMOND PLATED CORE DRILL SET IN HOLDER FOR BRANSON ULTRASONIC DRILLING.



IMPREGNATED DIAMOND CORE DRILL SET IN TAPERED HOLDER FOR ENGLISH TYPE WATER SWIVEL.

ELECTRO DEPOSITED DIAMOND CORE DRILL.



STANDARD WATER SWIVEL FOR DIAMOND DRILLS.

Recommended pressures are:

Drills up to 1/2 in.	15 - 20 P.S.I.
1/2 to 1 in.	5 P.S.I.
Over 1 in.	5 P.S.I. or less

Coolant can also be supplied externally through a pipe to the drill or hole, or the piece being drilled can be submerged in coolant. Remember that the cooling of the drill is only one of the necessary functions of a coolant. When fluid is applied through the center of the drill, it lubricates the drill and keeps the core from sticking.

METHODS AND TECHNIQUES

Diamond core drilling is an entirely different process from conventional drilling. The diamond drill is actually a grinder, abrading away glass particles. A novice must develop a feel for diamond drills.

Never force a diamond drill. This will only chip the material and clog the drill. It is essential that the drill always be clear and free. As soon as any resistance is felt, clear the drill.

Before using a new drill, run it against a grinding wheel (120/J grit). And whenever the drill feels as if it is going dull or losing its speed, dress it with a grinding wheel. A dull drill can cause core hang-up.

Whenever drilling glass, always mount the workpiece — preferably with epoxy — to a base plate of glass. This helps eliminate chipping at the end of the hole. For perfect holes, drill half way through the workpiece by setting the drill depth gauge for that distance. Then reverse the workpiece and drill from the other side.

DRILLING SMALL HOLES

There is today an ever increasing demand for small holes, down to .020 in. and smaller. Diamond core drills are now available down to .035 in. diameter but, because of their very small core (.018 in.), great care must be taken in their use lest the core become plugged in the drill.

While it would be preferable to use these miniature diamond drills with coolant under pressure, it is by no means uncommon to find them employed with a small precision drill press with the glass to be drilled submerged in water.

One fabricator of glass products, who drills holes in small 1 square inch pieces to a depth of 1/16 in., uses the submerged water technique. At the start of the program, he was getting 40 pieces drilled from each drill. But as his operators gained experience and skill, a drill now gets 700-800 holes at a rate of 30 seconds per hole.

ULTRASONIC ROTARY DRILLING

If it were not for the high cost of the equipment — about $9,000.00 — the ultrasonic rotary drill would be the perfect solution to most glass drilling problems.

By utilizing the Branson UMT-3, or their newer model, the UMT-5, one can drill precision holes effortlessly. Because of the combination of rotary and reciprocating actions, lower pressure can be applied. This results in a decrease in friction and an increase in diamond tool life.

With this equipment, it is possible to drill perfectly straight holes into solid glass that are as small as .042 in. diameter and as long as 12 in.

In conclusion, I would like to express my appreciation to Mr. John Burls of the Industrial Diamond Information Bureau, London, and Mr. William De Angelis of Pensar, Norwalk, Conn., for their valuable help in preparing this paper.

OUTLINE OF THIN FILM DEPOSITIONING AND ION PLATING FOR THE GLASSBLOWER

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INTRODUCTION

The production of thin films for many purposes can be accomplished by one of several methods. In this outline two methods are presented. Both methods require the use of a high vacuum system which includes a vacuum chamber for loading the work piece. The latter is better termed the substrate. Generally standard bell jar equipment is used, especially for small production.

The first method to be covered has been very successfully used since its inception during the second world war. It is termed: "Thin Film Depositioning By Vacuum Evaporation."[1]

The other method we will cover is called ion plating. Actually this method is better defined by calling it "Gas Discharge Assisted Vacuum Evaporation (of Thin Films)."[2] This second method promises in time to supersede the first method. The reasons for this will become apparent before this paper is concluded.

It is suggested, however, that the novice become familiarized with the thin film vacuum deposition method first. Then he will be the better qualified to understand and assume the technique for the ion plating method. (It is my personal feeling, at this point, that the ion plating method will be the one which should interest the scientific glassblower more than the simple vacuum evaporation method.)

PART I.

Thin Film Depositioning by Vacuum Evaporation.

The system shown in Figure No. 1. will evacuate the bell jar down to relatively low pressures, or, in the order of rather less than 5×10^{-7} Torr. (Note: The torr is the newly accepted unit for vacuum pressure and is the equivalent of one millimeter of mercury, absolute pressure.)

For depositions of silver, gold, and aluminum, the stated vacuum is quite acceptable. Aluminum can be worked at somewhat higher pressures but the quality of the film will be observed to decrease. Under the microscope it may even appear dross.

We will now assume that the outlined vacuum system, or its equivalent, is available. However, as shown in Figure No. 2. there are some accessory requirements. They comprise: A power supply with control and current meter; feedthrough terminals which pass up through the base

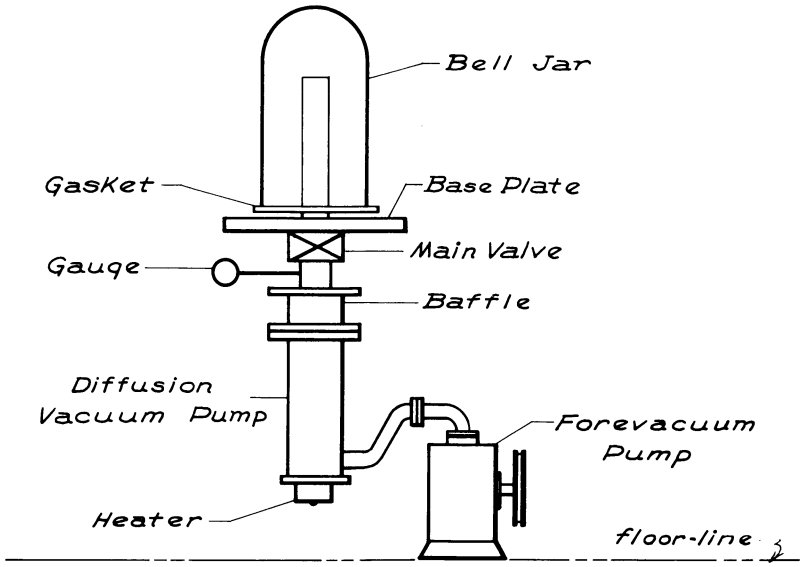

Figure 1
Bell Jar Vacuum System. Schematic.

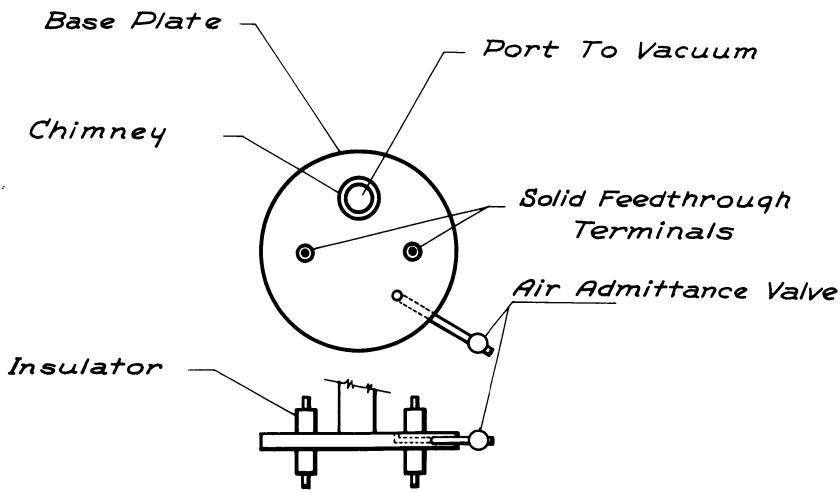

Figure 2
Baseplate.

plate of the vacuum system; bus bar supporting members; and, a heater filament. This filament in many cases is a tungsten wire formed into either a coil or into a looped configuration. It must be made both me-

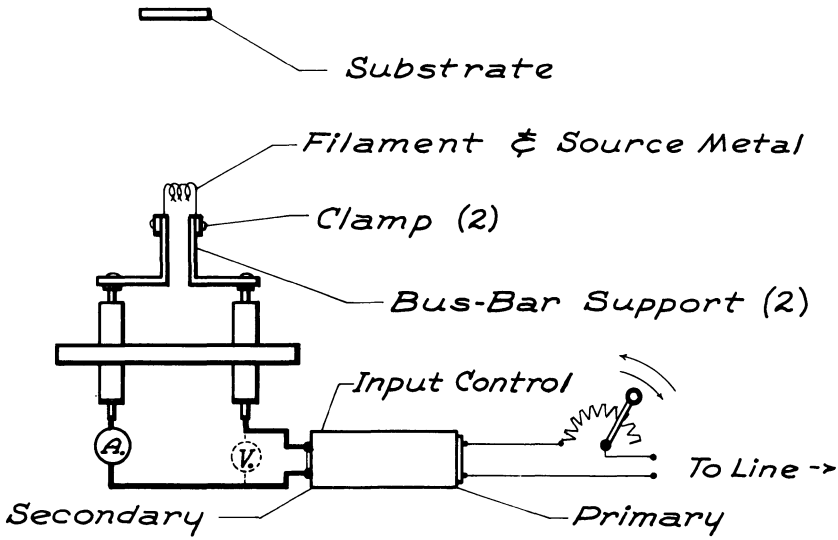

Figure 3
Feedthrough Terminals in Baseplate.

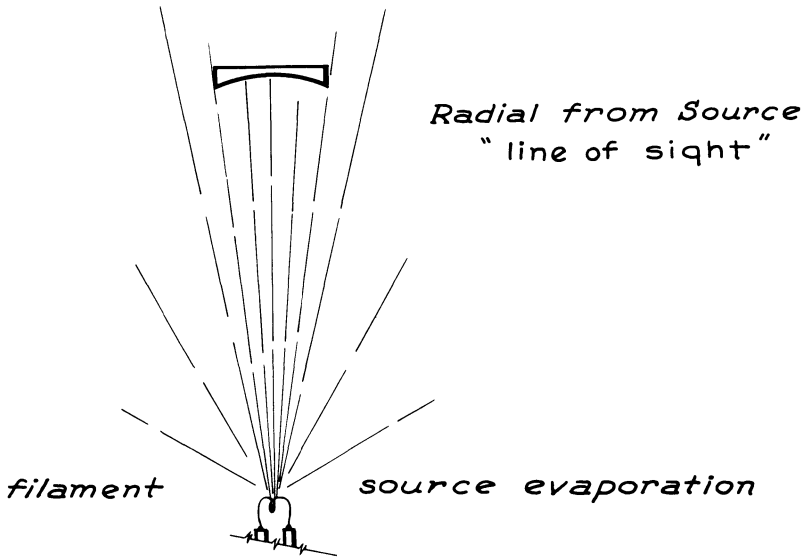

Figure 4
Path of Vapor during Vacuum Evaporation Method.

chanically and electrically secure to the bus bar supports. The power for this filament, or heater, is a "step-down" transformer. Hence we are to be dealing with rather high currents at reduced voltages. This is the primary reason that the bus bar supports are of a relatively massive cross-section. The average rating for the power transformer is about two (2) KVA. This indicates that currents up to 100 amperes may be used — if required.

As presented, this paper can not go into many of the details, all important ones, of high vacuum technology. Many of my readers have such knowledge and skill. On the other hand, at the end of the paper's publication a reference bibliography listing will be offered. Each of the several reference text sources are of interest and helpful. They are authoritative.

Now, the evaporation process of thin film depositioning in a high vacuum environment is accomplished by the simple fact that if one elevates the temperature of any one particular metal high enough it exhibits a usable vapor pressure which, in turn, produces a stream of that metal. It is one which is observed to radiate in all directions from the source location. We may call this "line of sight" for convenience. And, the temperature to which the metal is raised, can be predetermined. There is extensive literature on this.[3]

The author uses one "rule-of-thumb" formula. It is empirical and is a dependent guide. If the melting point (M.P.) of the metal is subtracted from the boiling point (B.P.), then divided by the melting point . . . if the answer, numerically, is equal to or greater than unity (1.0), then that particular metal will evaporate. But if the answer to the same calculation is less than unity, the metal sublimes. (e.i. Passes from the solid state to the vapor or gas state without assuming a liquid state. One example is chromium.)

High purity aluminum is used very extensively for thin film evaporation production. Its melting point is at 659° Centigrade. Its boiling point is well up to about 2447° Centigrade. We have learned that it will furnish an excellent and effective vapor stream at the temperature range of between 1137° to 1195° Centigrade. And, as stated before it radiates in all directions. The vapor stream's high velocity and its constant flow-density when properly controlled will result in high quality reflective films. Even though its useful thickness seldom exceeds 2,000 ÅU it is quite opaque.

The economy of this process becomes obvious as we consider the following: Approximately $15 will cover the cost of a cubic inch of high purity aluminum metal. This amount of source material will cover two million square inches at the useable thickness of 2000 ÅU. (Note: One Angstrom Unit equals 1×10^{-10} meter.)

As one practical example, let's assume that we wish to "silver" the two components of a dewar prior to their final seal-assembly and evacuation. First the geometry; we will be required to have our heater filament with its source material (metal) positioned inside the outer member of the dewar. Too, we will have to provide means to rotate the inner member during its film depositioning — for it will have to be positioned with

the source on the outside. The filament is loaded. Small pieces of the pure metal are placed around the heater loop or coil. But it must not be continuously wound around the filament. Hence, short lengths of the source metal (usually in wire form) are looped, a space then left, and another loop about the filament. We can now lower the vacuum chamber, or bell jar, and evacuate. It requires some time to reduce the bell jar pressure to the proper low level. Even a few hours.

In time our vacuum gauge will indicate the desired high vacuum condition within the bell jar. We can now start some current to flow through the filament. Gradually, as the current is increased, the filament will attain a dull red (glow). It is advisable that the room be a bit darkened in order to see this weak red glow. If left running at this setting, a partial degassing of both the filament and the source metal will take place. This is a good thing, and also it allows a longer period to permit our vacuum pumping system to attain still lower pressures. The next step is to increase the heater filament temperature, gradually, up to the melting point of our source metal. At that time we wish to have the source metal "wet" the filament. In the case of aluminum, this can not be done too rapidly. It can drop away from the tungsten filament. By using a step-by-step procedure the wetting of the filament can be accomplished. The temperature, of course, could be monitored by an optical pyrometer. This would remove any guess work with respect to the temperature; and insure the wetting of the aluminum to the filament. Without the pyrometer, however, one soon learns to judge the desired temperature from the degree of redness of the filament — and the fact that the loops of source metal which are in direct contact with the heated filament also assume the red heat. Several moments can be required at this current level to get the proper condition. The aluminum, when used as the source metal, will first form globules that "hang" from the filament coils. But with a continued heating (at this melting point temperature) the source metal will go upwardly, and surround the tungsten. It may even go atop of each coil. Frequently, it will be observed as rotating — but staying upon the filament. At this stage we are ready to produce the final evaporation.

The author prefers to "be on" the 10^{-7} Torr scale, or better. He, and others, have acquired the concept of vacuum pressures in terms of the "molecular population" . . . for, indeed, the lower the actual pressure in the bell jar the fewer molecules of gas remain.

We now raise the current level sufficient to promote the evaporation of the source metal, or stated in temperature (as before) we elevate the filament and source metal temperature to about 1150° Centigrade. During the temperature rise from melt point to the desired vapor pressure temperature level, the vacuum gauge could be used as an indicator of the temperature level. For, as we approach the 1150° Centigrade level, the pressure will show a marked increase. When this pressure may have advanced, say, a full decade (e.i. from 10^{-7} to 10^{-6} Torr) we should now observe the depositioning, the actual vaporization and recondensing of the source metal upon the glass walls of the bell jar. Were the vapor pressure to fall, during this stage of the game, the filament current must be

advanced a bit — promptly. As soon as the deposit is completed, we can turn off the current to the filament. It will be observed that as we shut off the filament heat, the bell jar pressure almost immediately drops back to the lower pressure scale, or the 10^{-7} Torr range.

The actual time required for the film formation, once all preparations leading up to the vaporization of the source metal are done, is a matter of only several seconds. Thirty seconds would produce a very thick film. Ten to fifteen seconds is a fair average.

After a few minutes the main vacuum valve is closed. Air is slowly admitted into the bell jar, and the latter is lifted away. As we examine our workpiece we may be pleasantly disappointed. Then again we may have an excellent job. More than one or two "pin holes" per square inch is evidence of too rapid an evaporation, or possibly too high a vapor pressure temperature. The condition of the workpiece, prior to the film depositioning, should, of course, be one of faultless surface with respect to possible finger print marks. The film will show these if there are any present prior to the evaporation!

And now, before we present Part II, on the ion plating method, a few remarks may be in order. For it is the writer's feeling that some of my readers may be wondering what interest or what value such information as he attempts to present may have for the scientific glassblower. So far, by inference at least, only mirrors, reflectors, and dewar flask components have been the possible work piece items for thin film depositioning technique. Others, and there are many, fall into the category of novelty glasswork. These could be enhanced by thin film modifications. Also, the well known difficulty of proper photography of clear glass products (for advertising purposes) can be made a relatively easy task were one to deposit a neutral-density film over it prior to the photography. There are certain metal films that may be recommended prior to the sealing together of two unlike glasses. The scale markings of many instruments made of glass would be rendered more visible were the scaled tube to be mirror backed.

But, we will now set aside the limitations that the evaporation method entails and turn our attention to the ion plating method.

PART II

Ion Plating, or

T-F by Inert Gas Discharge Assisted Vacuum Evaporation Method.

INTRODUCTION

The ion plating is superior in most respects. Now the films can be rendered quite thick (e.i. 1.0 MM.) or they can still be controlled for thin film work. The film now adheres tenaciously to the material upon which it is depositioned — whether glass or metal. The film is most uniform, continuous, and its depositioning covers all the surface (inside as well as outside) of the work piece. It is extremely pure. It offers countless

possible improvement for many products, glass items included. Nor is it limited to the production of metal films, as is the first method, generally. Materials such as nylon may be deposited upon a surface or an object work piece. Even special and desirable alloys may be constituted or developed. The process was used both successfully and extensively by NASA, at Cleveland, to develop both metal film and a well-bonded lubricating film upon the entire surface of high speed ball bearing components — or, both inner and outer bearing races and the balls. We will now quickly outline the equipment and the process or technique involved.

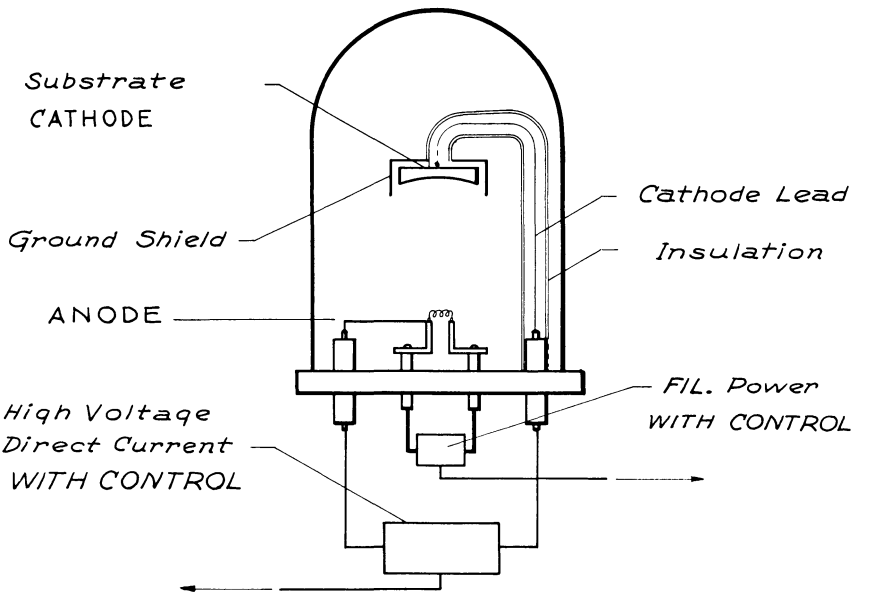

Figure 5
Setup for Ion Plating in Bell Jar System.

The system that is required is illustrated in Figure No. 5. We note that it is quite similar to the vacuum system used for vacuum evaporation. But, we do note the addition of high voltage direct current supply with controls, and metering. However, with respect to the ultimate vacuum that this system will be required to produce for ion plating work, the pressures should be about a decade lower, or down to the mid 10^{-8} Torr range. Such pressure now enters the category of ultra-high vacuum. (NOTE: The system as illustrated for the evaporation method will attain such low pressures, especially were one to use liquid nitrogen on the baffle and/or trap. Or, extending the original pumpdown time by several hours might produce the same results. The true reason for the lower pressure requirement is one which refers to "residual cantaminants.")

The high voltage direct current circuit uses a transformer that has

high impedence, and will deliver upwards of 5 kv. We must have means to regulate the output current, since we are to require a current density approximating from 0.2 to 0.5 milliamperes per square centimeter of cathode area. The latter, of course, will vary from one object work piece to another — or, the actual area of the surface we wish to plate. At any rate, a transformer having a secondary voltage of 7,500 volts at a rated short-circuit current of 60.0 MA is quite satisfactory for small items. The latter can have total areas of: 300 cm^2 for the 0.2 MA current-density; and, 120 cm^2 for the 0.5 MA current-density. (A four inch square is about 100 cm^2.) Voltages will be self-regulated. They will depend entirely upon the geometry of the cathode, the drop from the anode to the cathode, and the gas pressure. The latter will be discussed very shortly.

To ion plate a work piece we arrange things similar to the evaporation method. We even proceed, at first, in the same manner. The filament heater is prepared with the source material wetted to it. Then, at that point when we *could* proceed to evaporate, we now introduce a very low pressure of an inert gas into the vacuum chamber or the bell jar. The suggested gas pressure is quite low. It is about 75 microns (hg) at the most. This is equal to 0.075 millimeters (hg) pressure, absolute. The inert gas, usually, is argon. Other gases can be used such as helium, neon,

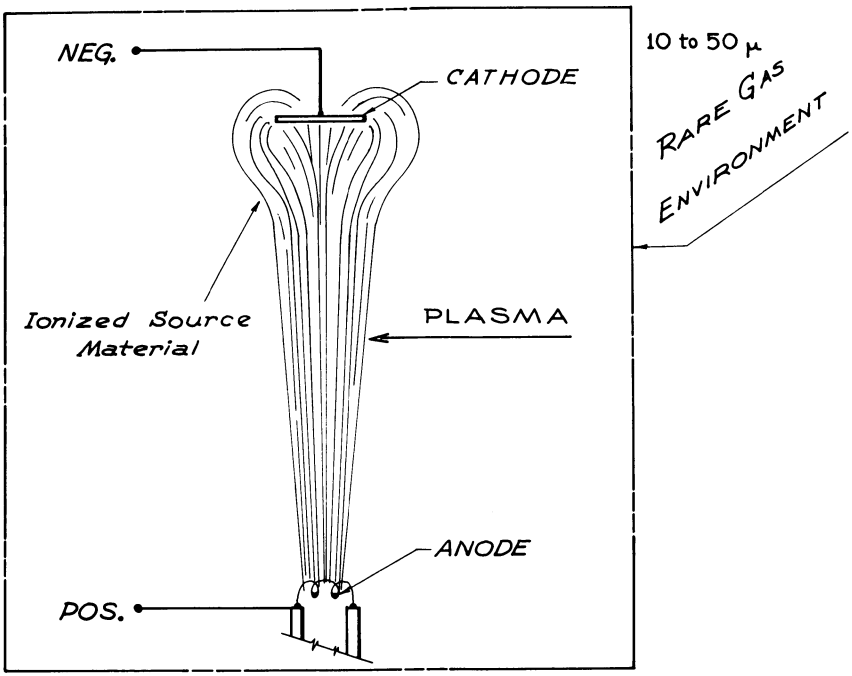

Figure 6
Path of Ionized Vapor during Ion Plating.

and even ultra-pure nitrogen. The author prefers helium. As soon as a steady gas pressure is established at the desired level, we turn on the high voltage and bombard the cathodic work piece. This discharge very quickly renders the work piece ultra clean. The filament current has been allowed to remain at the melting point level during this time. It now can be increased to produce evaporation and depositioning of the source material upon the work piece, or cathode. The high voltage current is watched and manually regulated to maintain one given current level. The vapor stream becomes ionized and (statistically, at least) most of it is attracted to the cathodic work piece. The first film forming on the work piece may be called the interface deposit — and, if it is desired, the bombardment or ionization action may be stopped at this time. The film depositioning may be completed by the ordinary vacuum evaporation method. On the other hand, if real coverage is the aim (on an irregularly shaped work piece) the ion bombardment continues until the desired film is attained.

CONCLUSION

In concluding this outline the author sincerely believes that the ion plating method will affect the glassblower directly. The fusion of two unlike materials or glasses offers one attractive application. It is his intention to pursue this matter. Were he to have definite and useful information following further experimental work, he will be glad to submit it to "FUSION."

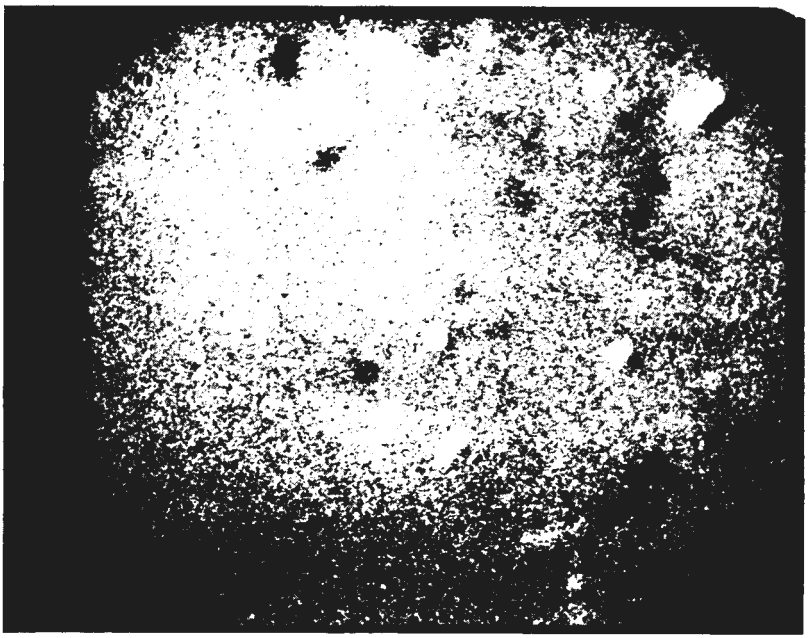

Figure 7

Electron Microscopic Photograph — Vacuum Evaporation.

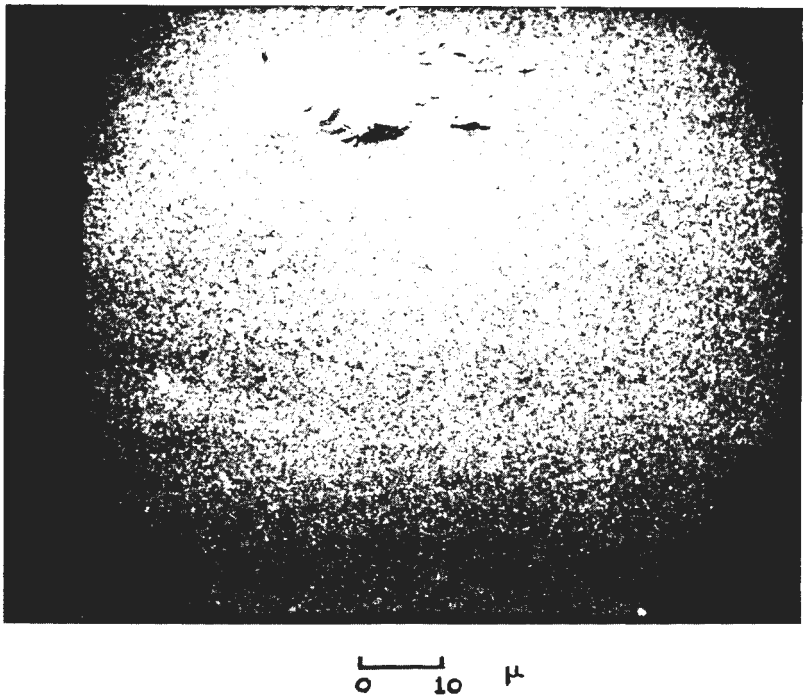

Figure 8
Electron Microscopic Photograph — Ion Plating.

APPENDIX

It was important that the optical quality of the thin films produced by the two methods be investigated. Therefore, measurements were carefully made. These enabled the author to determine the coefficient of reflection (Reflectivity) of the front-surface plano gold reflectors made using both methods.

It was observed that the vacuum evaporated thin film had values of from 0.95+ to 0.96+ for its coefficient of reflection. (Note: The measurements were made in the infrared.)

On the other hand, thin films of the same thicknesses using the ion plating method were observed to have values as high as 0.985 for its coefficient of reflection.

The vacuum evaporation method produces thin film that are known to evidence a decrease of their reflectivity — over time. This, in many cases, can be as much as fifteen to twenty percent of the original.

Since the above reported coefficients of reflection were made when all samples were relatively fresh, it is planned to remeasure and report any possible decreases following a period of about four to six weeks. (e.i. Fall issue of "FUSION.")

ACKNOWLEDGEMENTS

The author wishes to express his sincere appreciation to the following people who made this presentation possible:

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ADJUSTING STRESSES IN GLASS-TO-ALUMINA SEALS BY THERMAL CONDITIONING

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INTRODUCTION

An important and often requested service performed by the glass sealing specialist is to design and successfully produce seals to various types of materials, including metals, ceramics, and other glasses. A frequent requirement for the finished item is that residual stresses be minimal.

Ideally, careful selection of the seal materials will result in satisfactory stress levels. In practice, however, seals with higher stresses than desired are commonly encountered. When this happens, material substitutions are usually made (either in the sealing glass or the material being sealed) to obtain lower stresses. This procedure is time-consuming and expensive, requiring purchase, preparation, or development of substitute materials, and repetition of the evaluation work.

A technique more expedient than searching for substitute materials can sometimes be used to reduce stresses in a sealed product. This technique employs *thermal treatment* of the seal to produce the desired stress level.

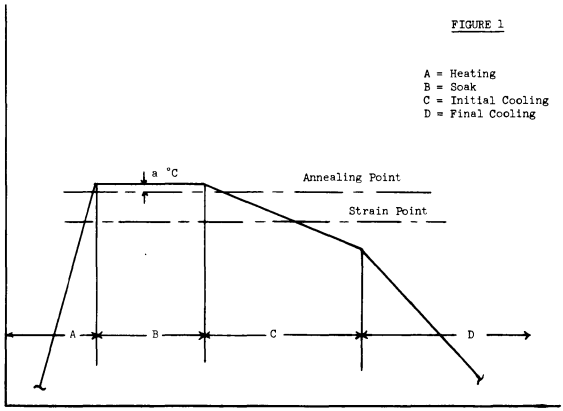
This paper illustrates use of the "stress adjustment" technique, using as examples seals of a glass (Owens-Illinois RP-1) to two slightly different types of alumina. By proper "thermal conditioning" the stress in the seal can be adjusted.

DISCUSSION

When sealing glasses to alumina, the residual interfacial stresses usually depend on the difference in thermal contraction between the two materials from the glass "setting point" down to room temperature. The glass setting point is assumed to be 30-40°C below the annealing point[1] and very near the strain point temperature. If the glass member of the seal displays residual tensile stresses, it is assumed that a lower contraction sealing glass must be substituted in order to obtain a neutral stress condition. However, in many cases seals displaying high glass tensile stresses can be thermally treated to achieve a level of acceptable stress.

Historically, the selection of a thermal treatment or annealing cycle for a seal such as glass-to-alumina is based on experience and tailored to the seal configuration. In general, however, when uniform stresses are the goal, annealing by thermally soaking the seal at a temperature near or above the annealing point is the accepted procedure. The seal is made, then placed in an annealing oven or lehr. The annealing cycle commonly

has a hold temperature 10°C or so above the annealing point of the glass. The soak cycle is typically 15 minutes to two hours. Soaking is followed by cooling at a rate slow enough to avoid unwanted thermal stresses in the glass. The "annealing cycle" removes high thermal stresses induced during sealing and gives the glass member of the seal a definable thermal history. It is known that thermal history of a glass article influences the glass microstructure and properties. In particular, thermal soaking of a glass article at a temperature in the *annealing range*[2] for a period sufficiently long to stabilize the glass results in a structure representative of the soak or stabilization temperature. A term commonly used to describe a unique set of properties and structure of a given glass composition is "fictive temperature". The thermal history of a glass determines its fictive temperature, while not a fixed, well-defined point on the glass viscosity curve, is an important factor in glass mechanics.[3]



At each lower stabilization temperature, the glass structure will be more tightly packed, resulting in measurable increases in density and refractive index. These easily measured changes in properties with thermal history are well documented[4] and show that the properties of a glass are not unique with respect to chemistry but depend within certain limits on its thermal history.

This characteristic of glass . . . the fact that it exhibits properties which correspond to its fictive temperature . . . is the basic factor enabling stress in a seal to be adjusted through thermal conditioning. When the fictive temperature of a glass is reduced by stabilizing it at a lower temperature than its previous stabilization temperature, the effective "setting point", normally about 30°C below the annealing point, is also reduced.

Time Required For Stress Release

Figure 2 shows the approximate time required for stress release in the alumina sealing glass used in these studies, as a function of temperature. Time for stress release is plotted on the ordinate on a logarithmic

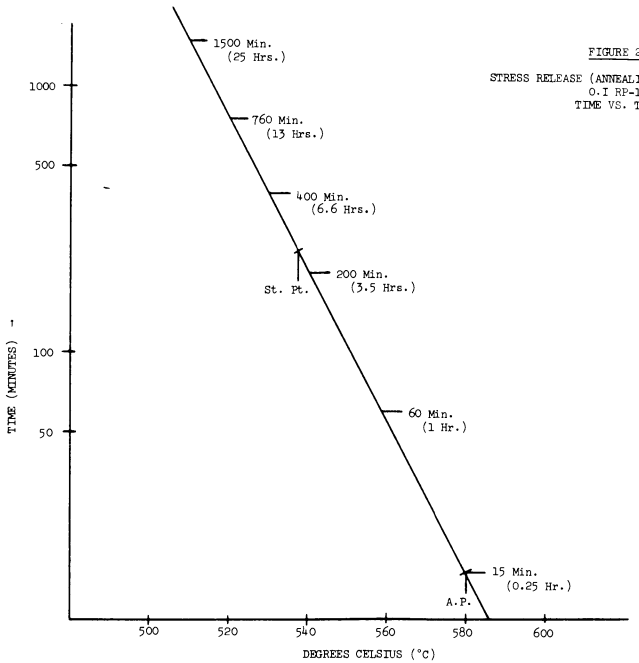


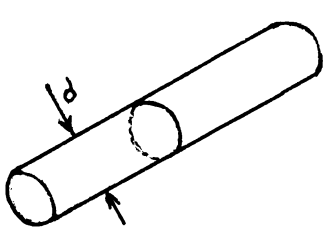
FIGURE 2

STRESS RELEASE (ANNEALING) IN GLASS
0.I RP-1
TIME VS. TEMP.

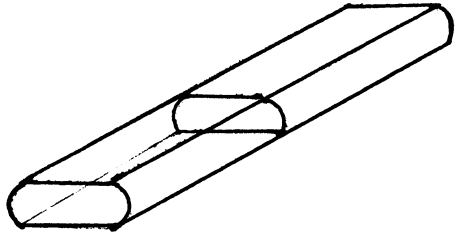
scale, with temperature plotted on the abscissa on a linear scale. Times were obtained from experiments, and depend on glass thickness.

Specimen Seals and Material Thermal Expansions

The specimen glass-to-alumina seals used in this work were quarter-inch butt seals fabricated in a gas-oxygen flame. After sealing, the specimens were annealed. After annealing, opposite sides were ground flat for stress analysis.



BEFORE GRINDING



AFTER GRINDING

Two different alumina lots, with slightly different contraction coefficients as shown in the following table, were used along with the RP-1 glass:

CONTRACTION COEFFICIENT — IN/IN x 10^{-7}/°C

Temp. Range	RP-1 Glass	Alumina Sample 1	Alumina Sample 2
25 - 600°C	92	74	71
25 - 580°C	83	73.5	70.5
25 - 560°C	77	73	70
25 - 540°C	73	72.5	69.5
25 - 520°C	70	72	69
25 - 300°C	64	66	63.5

Method of Stress Adjustment

Initially, the specimen seals were annealed at 580°C for 15 minutes, resulting in a glass stress of 100 psi interfacial glass tension in the seal vs. Alumina Sample 1, and 1950 psi interfacial glass tension vs. Alumina Sample 2.

To illustrate the principle of stress adjustment by thermal treatment, we sought to move the above interfacial stresses in the direction of greater compression in the glass member. One method of accomplishing this would be to reduce the apparent contraction coefficient of the glass by lowering the "setting point". As explained previously, the effective setting point can be lowered by stabilizing the glass at a lower temperature than the original annealing temperature. This thermal treatment lowers the fictive temperature and the setting point, and also erases the previous thermal history. Figure 3 shows the stresses which resulted in the seals

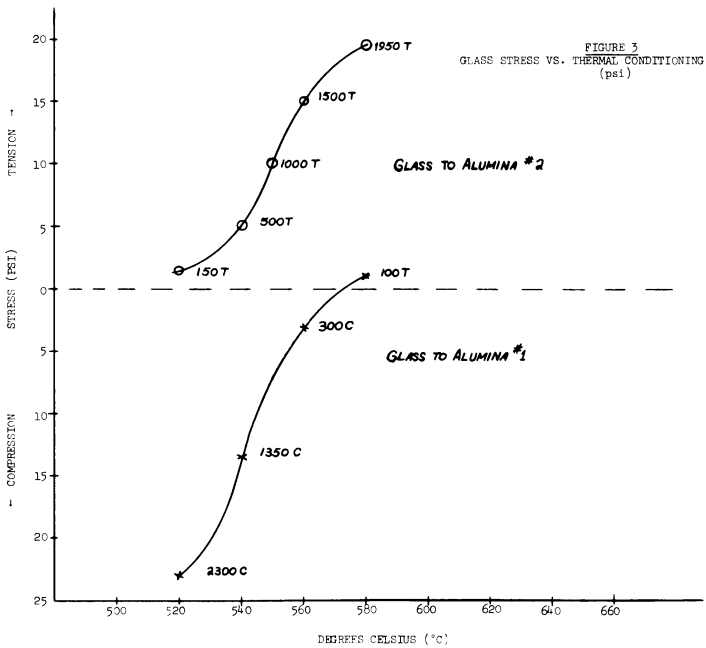


FIGURE 3
GLASS STRESS VS. THERMAL CONDITIONING TEMPERATURE
(psi)

after various stabilization treatments at temperatures below the annealing point.

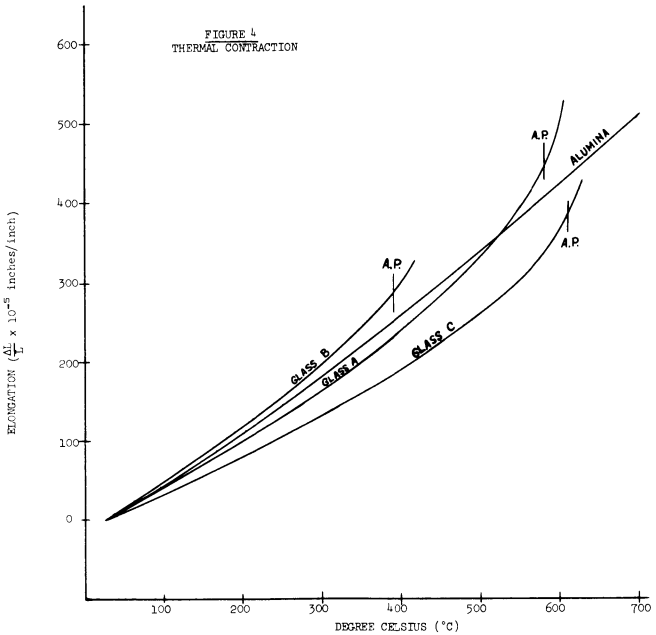
Thus, it can be seen that the residual interfacial stress in certain seals can be adjusted by properly selecting a thermal soak or stabilization cycle and allowing sufficient time for glass structural adjustment to take place (see Fig. 2).

A fact to remember, however, is that all glass-to-alumina seals are not candidates for thermal stress conditioning. In order to determine if the results of thermal treatment will be positive and to what extent, some comparative physical properties measurements are useful. These measurements are *thermal contraction* for both the glass and alumina, and the *annealing point* of the glass. A laboratory test seal can also prove valuable in verifying stresses estimated from the contraction curves and can be used in tests to select a proper thermal conditioning cycle.

How these properties can be used to predict stresses is illustrated in Figure 4.

Figure 4 shows thermal contraction data for three glasses and alumina. Glass "A" is the best candidate for thermal stress conditioning. This glass, when sealed to alumina and annealed normally, would have low tensile stresses at the seal interface (around 200-400 psi).

This glass could easily be adjusted to a lower, near neutral stress. The contraction curves for the glass and alumina cross at 520°C or 10-20°C below the glass setting point. Since at the setting point the glass



contraction (to room temperature) is greater than that of the alumina, the setting point should be adjusted farther down the glass contraction curve toward a lower temperature value. The residual stress will move toward compression (lower tensile stress) in the glass. The conditioning temperature used to obtain neutral stress would be about 20°C below the annealing point temperature, and the time needed for stabilization would be about 60 minutes.

Glass "B" also seals in tension after standard annealing, and like Glass "A" could be thermal conditioned to a lower stress level. Its residual tension stress could not be lowered to less than several hundred psi, however.

The effect of lowering the setting point for Glass "C" would only serve to increase its compressive stresses, so glass "C" would be a poor candidate for this thermal treatment.

As can be seen, the correct conditions are present for easy use of thermal treatment to adjust stresses to neutral in a glass-to-alumina seal when:

1. The glass seals in tension after normal annealing.
2. The contraction curves for the glass and alumina cross in the annealing range of the glass (within 60-80°C below the annealing point).

This can be determined before sealing (by property measurements).

Figure 5 shows a comparison of contraction curves of two different alumina samples and a glass. As can be seen, the glass contraction curve

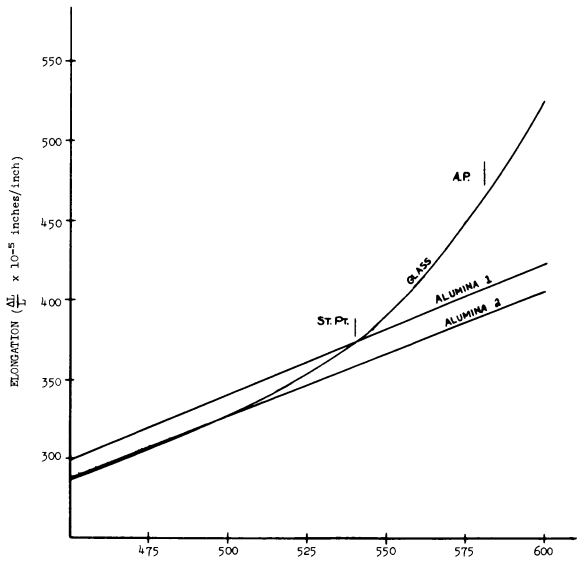


FIGURE 5
THERMAL CONTRACTION

crosses Alumina No. 1 at around 540°C and approaches Alumina No. 2 closely at around 500°C. Figure 3 shows stress reading in seals of these aluminas. The seals were thermally treated at temperatures from 580°C to 520°C in 20°C intervals as estimated stabilization times shown earlier on Figure 2. As evidenced by the stress diagrams, the amount of stress adjustment accomplished for both seals was substantial. These examples show an effective decrease in the apparent contraction of the sealing glass of $\sim$ 10%. The total stress adjustment obtained in the glass-to-Alumina No. 1 seal was 2400 psi. An 1800 psi stress adjustment was realized in the glass-to-Alumina No. 2 seal.

CONCLUSIONS

The procedure for thermal adjustment of glass tensile stresses in alumina-ceramic seals is straightforward, requiring only that the seal be restabilized by holding it at a pre-determined temperature lower than the original annealing stabilization temperature. It should also be mentioned that the effects obtained by any particular thermal adjustment are not permanent but reversible. If a seal is "thermally adjusted" to a stress level which is unacceptable, the adjustment process can be repeated at another stabilization temperature.

The data presented shows that thermal stress conditioning can be a useful alternative to material substitutions. The results can be very rewarding for the little effort required to investigate the variable in a particular sealing problem, and to apply the technique where it appears to be of value.

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A NEW MELTING POINT APPARATUS

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The design of the original melting point apparatus was changed to eliminate some of the problems of this unit. These changes allow for easy dismantling of the unit for cleaning and repairs. A smaller amount of oil means a quicker heating and cooling cycle. Efficient stirring of the oil promotes accurate melting points. The heating coil can be easily removed for repair.

A description of the apparatus follows:

The outside chamber has a 29/42 outer joint at the top, with the glass just below the joint blown to a diameter of 4.8 cm. This section is tapered to 3.5 cm. at a distance of 10 cm. from the joint. The unit is 14 cm. high. The chamber uses about 40 ml. of oil. An area about 1.5 x 5 cm. is blown horizontally 4 cm. from the bottom to hold the heating coil.

The inner tube, 2.2 cm. diameter is sealed to the bottom of an inner 29/42 joint. It is 8 cm. long. Slots are cut into the inner tube using the cut-off machine and are alternated to allow for good circulation of the oil. The top of this joint is cut flush with the top of the outer joint.

A teflon[1] plug is machined to fit the inside taper of the inner 29/42 joint. Holes are drilled vertically in this plug to insert a thermometer and the melting point tubes. The plug is cut 1 cm. above the top of both joints. This allows for easy removal of the plug.

A 1 cm. length of 8 mm. tubing is sealed to each end of the 1.5 x 5 cm. area of the outside chamber. Two 30 mil copper-tungsten-nickel leads[2] are prepared for sealing into the 8 mm. tubing. The nickel section was removed and the tungsten section was tapered on a belt grinder to fit snugly into a 1.5 cm. length of No. 17 needle tubing.

The heater coil is made using 44 in. of No. 26 nichrome[3] wire wound on a ⅜ in. rod chucked in the drill press. The ½ in. sections of needle tubing are then crimped on each end of the heater wire. The coil can then be easily installed thru the outer joint into the main body of the unit. The copper-tungsten leads having been sealed into the 8 mm. tubing in the usual manner, the needle tubing is then slid onto the tungsten tapered ends. The coil is easily removed in case of burnout.

A short length of wire mesh is fitted into a slot cut into the inner tube 1 cm. from its lower end. This mesh acts as a support for the thermometer (o-300) and the melting point tubes.

A teflon covered magnet is used in the bottom of the outside unit for circulating the oil. The stirrer pulls the oil down thru the inner tubes slots and the oil returns between the inner tube and the outer shell.

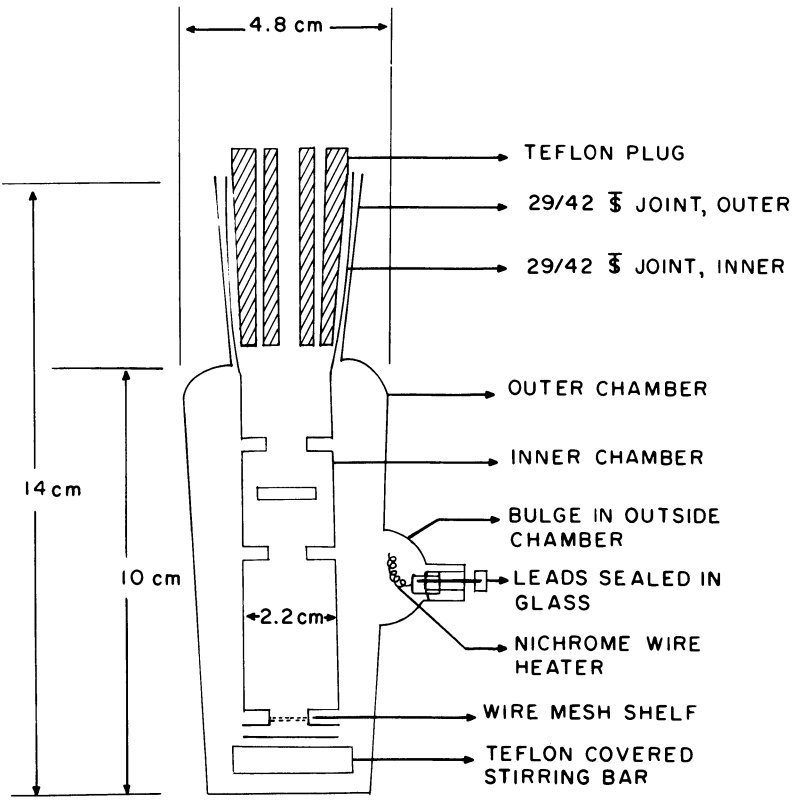

Figure 1

We also used electronic metal caps cemented on the 8 mm. tubing using a silicone cement, RTV 102[4]. Plastic grid caps with a lead attached were used over the metal caps to complete the unit.

In conclusion, this unit is easily made. It has several innovations making it an item of interest to those using this type of unit in their laboratory.

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3. Driver-Harris Company, Harrison, New Jersey
4. RTV-102, made by General Electric Silicone Plant

SOME APPLICATIONS OF SOLDER GLASS

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It is only within recent years that the glass industry has actively pursued the development of glass solders. This interest has mainly been due to the growing market in the electronics industry which utilizes such glass solders in many ways, with perhaps the most significant use being in hermetic (Helium leakproof) packaging.

Much of the work discussed in this article was undertaken as part of the development program at BNR over the last two years on Electro-generated Chemiluminescence (EGCL) display devices[1]. These liquid light displays represent a technology which has only recently emerged from the research laboratory.

Two types of glass solder are commercially available, vitreous and devitreous, and are obtainable in the following forms:

1. powder
2. rod
3. preforms (usually with spacers within)
4. paste, composed of
   a) glass particles (frit)
   b) an organic solvent such as pine oil
   c) a thickening agent such as ethyl cellulose
   d) wetting agents (usually oils or additional solvents)
   e) metal and metal oxides for conductors
5. tapes[2] composed of
   a) a carrier layer (polyethylene)
   b) a glass layer made up of frit, binder (nitrocellulose or poly-n-butylmethacrylate), plasticizer (sucrose-acetate-isobutyrate) and adhesive (acrylic polymer)
   c) protective paper

With the introduction of flat panel displays utilizing plasmas (gas discharge), liquid crystals and other technologies, glass solders have assumed a growing significance and there has been a proliferation of many new and a revival of many old glass solders to meet the specific packaging needs being generated.

The prime considerations for working with glass solders are not dissimilar to those used in the art of glass blowing, namely viscosity and temperature, expansion and contraction.

The unique feature of a solder glass is its low softening temperature[3] (Figure 1). The glass flow necessary to affect a seal depends on the fit

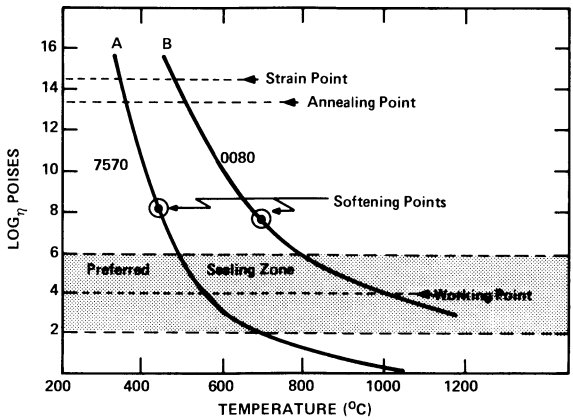

FIG. 1 VISCOSITY TEMPERATURE CURVES FOR ALL GLASS PACKAGE

Code 7570 (corning^R)
Code 0080 (corning^R)

between the parts and the area and geometry of the seal. A good seal is usually made in the viscosity range 10^4 to 10^6P (Poise) but if seal definition and package constituents will permit a higher temperature, a stronger and more reliable seal can be formed at 10^2P. The upper limit of temperature at which a seal can be made, for the type of display shown in Figure 2, is 50°C above the annealing point, but, ideally, the seal should be formed at the annealing temperature of the constituent parts, thus reducing strains in the package.

Perhaps the most critical factor in sealing is the relative thermal expansion or contraction between the solder glass and the body of the package. The figure which is normally used to characterize the expansion and contraction of glasses or ceramics is the slope of the expansion-contraction curves (Figure 3) from 0°C to 300°C. The range of interest on these curves is from the 'setting-up' temperature of the seal down to room temperature. When the solder glass is not severely confined, which is the case in display applications (Figure 4), the setting-up temperature (5°C above the strain point) thus lies well beyond the bend on the expansion-contraction curves. For a solder glass, this bend occurs, of course, at a lower temperature than the glass to be sealed. Therefore, a solder glass should have a lower coefficient of expansion-contraction than the glass being sealed in order to have approximately the same contraction from the setting-up point to room temperature.

There are several types of display packages on the market and these employ fabrication technologies using the following types of seal:

a) glass to glass
b) glass to ceramic
c) glass to metal
d) ceramic to metal

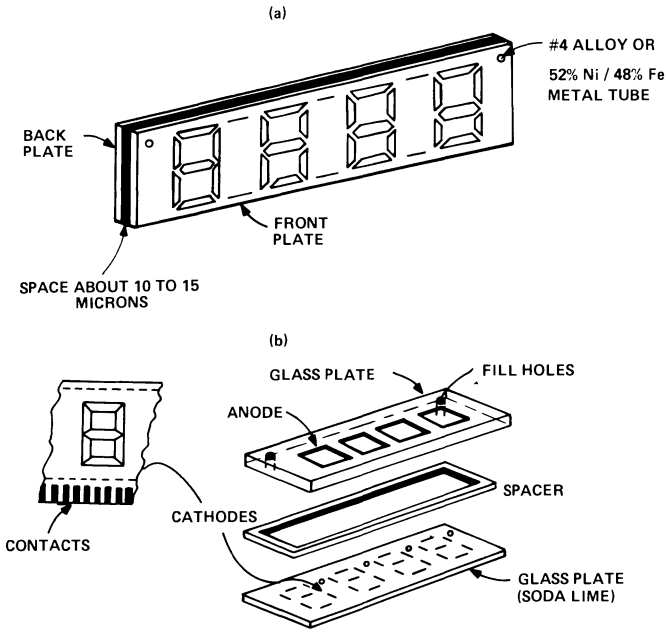

FIG. 2 CONSTRUCTION FOR TRANSMISSIVE LIQUID CRYSTAL DISPLAYS

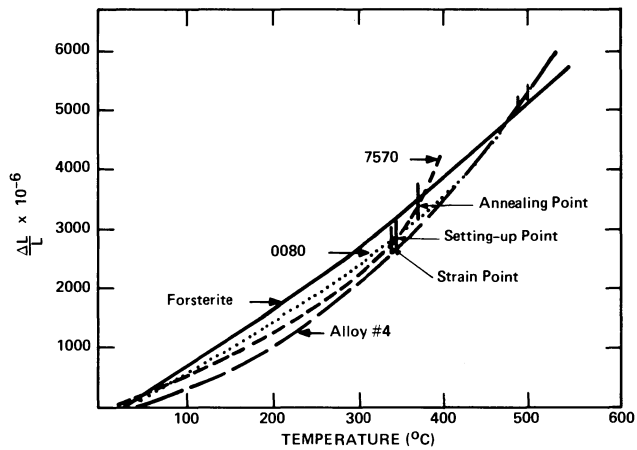

FIG. 3 THERMAL EXPANSION CONTRACTION CURVES FOR GLASS CERAMIC PACKAGE

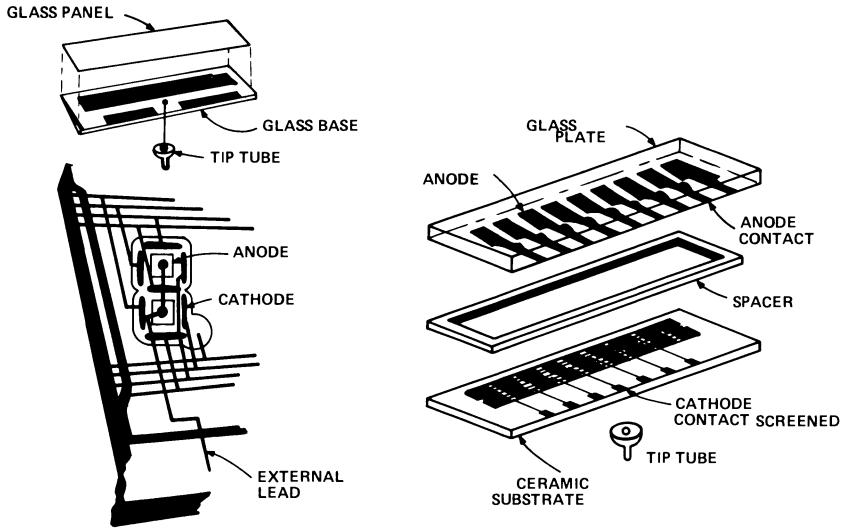

FIG. 4 TWO TYPES OF CONSTRUCTION FOR GAS DISCHARGE DISPLAYS

Two of the more widely known types of display are Gas Discharge and Liquid Crystal. Flat package gas discharge displays use thick film printed electrodes on either glass or ceramic substrates. In either case, a glass faceplate or window is employed with or without transparent tin oxide electrodes. Electrodes are silk screened or electroplated onto the substrate (Figures 5 and 6). If nickel, nickel alloys, molybdenum, or tungsten alloy thick film pastes are used to form the conductor patterns, the substrate must be fired in a hydrogen (reducing) atmosphere at high temperatures to avoid oxidation. A typical furnace profile for multiple atmosphere firing[4] is shown in Figure 7. Hydrogen firing can be eliminated by using electroplated or cast electrodes. The package is then assembled and sealed in an air atmosphere at a temperature low enough to avoid oxidation.

Liquid crystal displays represent a technology which has emerged only recently from the laboratory. Liquid crystal display packages are currently the most economical to produce and are finding extensive use in commercial products.

There are basically two types of liquid crystal devices, reflective and transmissive. The construction of a liquid crystal device begins with two glass plates. The substrate is coated with a transparent layer of tin oxide or indium oxide. Acid resist is screen-printed over this layer, defining electrode patterns, and is dried in a continuous dryer. The oxide is etched, establishing the requisite conductive patterns. If the display is to be transmissive, both front and back plates are treated in the same manner. If the display is reflective, the rear plate is usually printed with a thick film re-

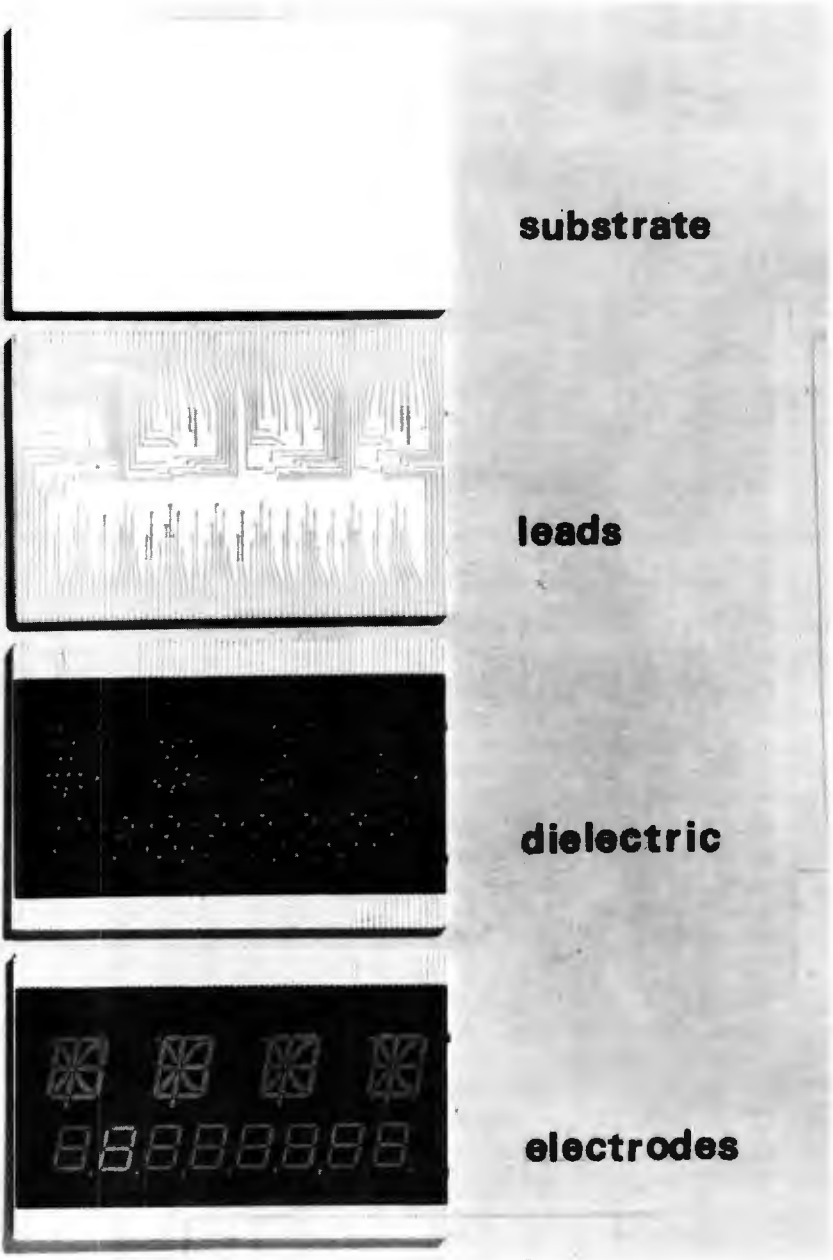

Fig. 5 This is a photograph of a thick film numeric and alphanumeric electrode layout used in an ECL visual display.

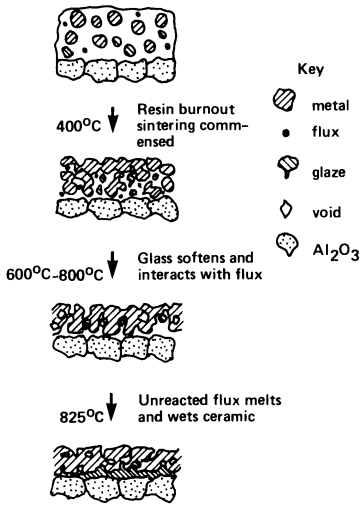

FIG. 6 Stages in Conductor Firing to Substrate Process. 1st is Organic Resin Burn-Out; 2nd is Metallic Sintering Stage; 3rd is When Temperature Reaches Glass Softening Point.

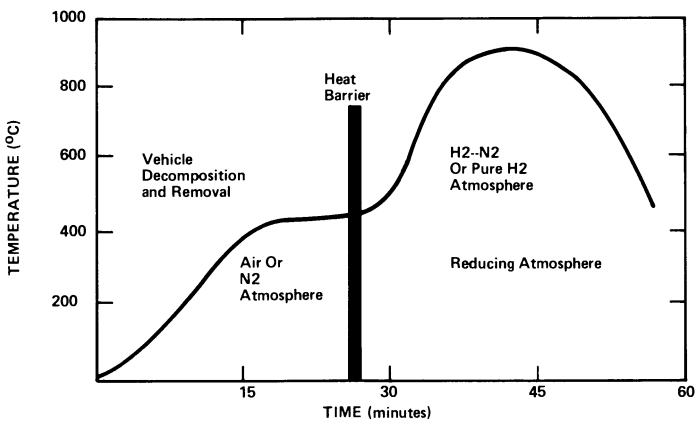

FIG. 7 TYPICAL FIRING PROFILE FOR MULTI ATMOSPHERE FIRING DISCHARGE

flective coating which can withstand the sealing temperatures and does not contaminate the liquid.

The above displays are assembled by using a preform or by printing a solder glass paste as a window frame on the base and face plates and pre-firing to the vitreous phase (Figure 8a). Final sealing is then accomplished by reflowing the solder glass in a belt furnace after the display cell is assembled (Figure 8b). If a crystallizing solder glass is used, the

firing temperature must be raised after reflowing in order to allow crystallization to occur, Figures 9a-c[5]. Typical firing curves are given in Figures 8a-9b, both for vitreous and devitreous glasses covering a range of thermal expansion coefficients.

In summary, most of the profiles given may be accomplished by approximating the firing cycles in a box or tube furnace. One should be wary

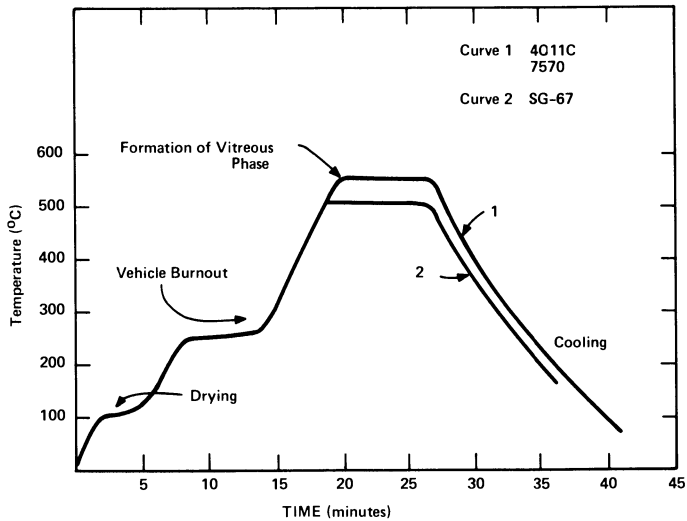

FIG. 8A PRE-GLAZING CYCLE

(4011C) - Electro Science Laboratories
(SG-67) - Owen Illinois

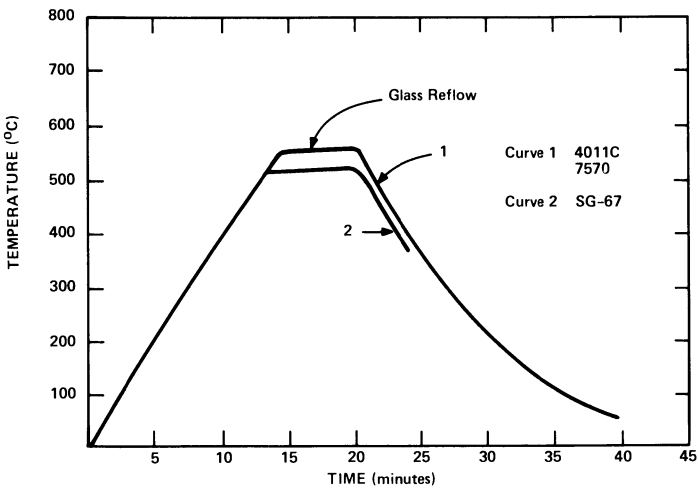

FIG. 8B SEALING CYCLE

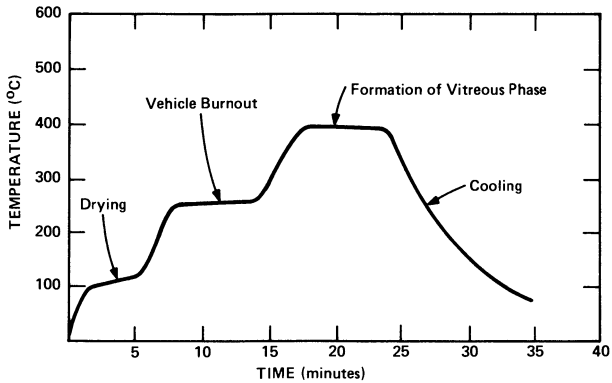

FIG. 9A PRE-GLAZING CYCLE (7572 corning)

of the crystallizing glasses, however, as they are very sensitive to variation in temperature and may over-crystallize. Most of the low temperature seals which are used in flat panel fabrication can be successfully used by the glass blower for simplifying some of his sealing problems.

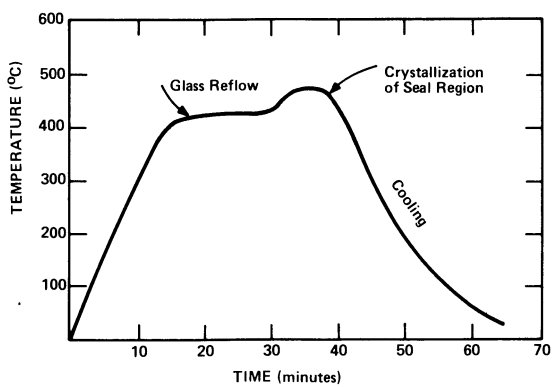

FIG. 9B SEALING AND CRYSTALLIZATION CYCLE (7572 corning)

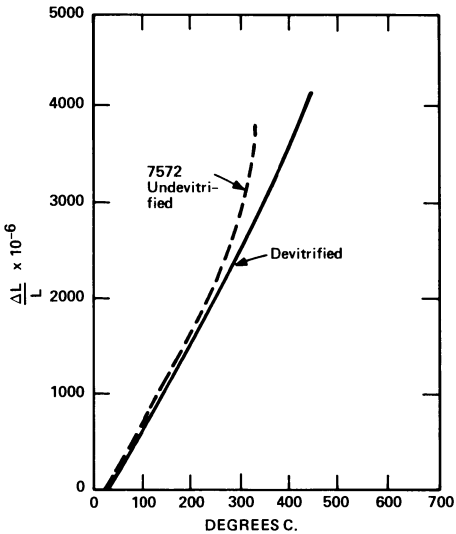

FIG. 9C EXPANSION-CONTRACTION
PYROCERAM® #95 (7572 corning®)

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THE GLASS SEALING CHARACTERISTICS OF COPPER ALLOY C.D.A. #638

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ABSTRACT

Copper alloys in general are difficult to effectively seal to glasses or ceramics due to the nature of the oxide film which forms during firing. A new alloy C.D.A. #638, an aluminum-silicon bronze has been developed to overcome this problem. At elevated temperatures, above 300°C, in the presence of water vapor or oxygen, a surface oxide which is predominantly Al_2O_3 forms. This oxide is tightly adherent to the metal and semi-elastic in nature. It acts as an ideal substrate for glass bonding.

The high bond strengths attainable with this alloy make possible the use of glasses with expansion coefficients different from the metal. However, a new high expansion glass developed by Corning has been found to form an excellent glass-metal bond which is resistant to thermal shock and hermetic in nature. Results are presented on some "in plant" tests of TO5 type semi-conductor headers which can be made using this system. Additional laboratory data on alloy 638 and other glass sealing materials are provided. The results are discussed in light of current bonding theory and glass-metal interfacial properties.

INTRODUCTION

Copper and its alloys are used in a wide variety of applications where appearance, corrosion resistance, solderability, thermal and electrical conductivity are important. One such application is in electronic and electrical devices demanding the superior performance characteristics of copper alloys, in particular, the new high performance alloys.[1,2] However, an area where copper is not extensively used, although it would seem to be an ideal material from a conductivity and corrosion standpoint is in electronic packages and devices requiring an hermetic seal with glass. Huge quantities of metal are used in this area and the market is continually increasing in size. In less rigorous applications, copper alloys have been used in plastic electronic packages[1] which will perform adequately under moderate service conditions. However, to date, little copper is used in metal-glass sealing.

Several reasons account for the reluctance to use copper base materials. These are outlined below.

1. Copper alloys have high expansion coefficients in the order of 170 x 10^{-7}/°C. Most sealing glasses are considerably lower in the order of 40-120 x 10^{-7}/°C.

2. Glasses do not, in general, effectively bond directly with oxide free copper surfaces.
3. The oxides of copper (CuO, Cu_2O) are loosely bonded to the metal and are bulky in nature. Although these oxides will form an effective bond with glass by partial dissolution, the bond fails in the oxide layer or at the metal-oxide interface.

Historically, the problem of thermal expansion coefficient mismatch has been overcome by developing metals with low enough expansion coefficients to match available glasses. This approach, in the past has proven to be somewhat more successful than the development of high expansion glasses with suitable electrical characteristics. The Fe-Ni-Co series of alloys can have expansion coefficients as low as 40 x 10^{-7}/°C. One such material in extensive use in electronic packages is F-15 (Kovar)* (Fe + 29% Ni + 17% Co). The low expansion metals are, however, characterized by their high cost and susceptibility to corrosion and stress corrosion cracking.

Various bonding methods have been tried for metal-glass sealing systems.[3] Of these, the most effective, and the one used in the case of the F-15 alloy, is the development of an oxide film on the metal surface by a pre-treatment of some kind which bonds by chemical action to the glass.

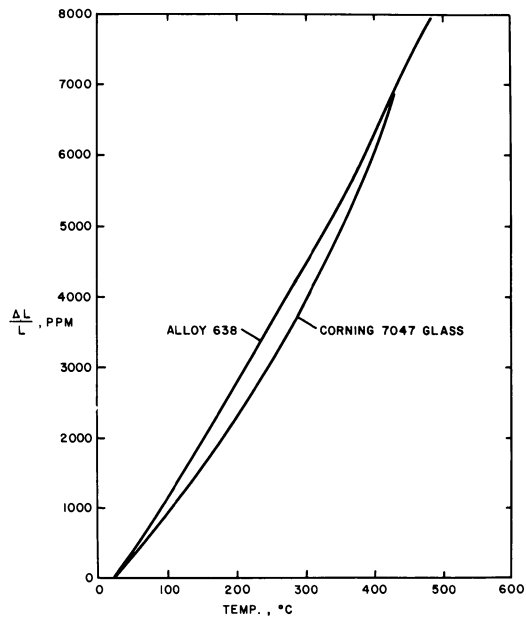

Figure 1

Thermal expansion ($\frac{\Delta L}{L}$) as a function of temperature for alloy 638 and Corning Glass 7047.

*Westinghouse Trade Mark

This oxide layer must have certain properties. Firstly, it must be well adhered to the metal to prevent metal-oxide interfacial failure after sealing. In addition, it must be reasonably strong itself to prevent fracture within the oxide layer and finally, it should chemically bond (either by a partial dissolution process or formation of a third phase) to the glass. Alloy F-15, in general, meets all these requirements. However, the oxide film (a complex mixture) is by nature brittle and unless very great care is taken to develop the optimum thickness[4] failure can result.

The only type of glass seal successfully used for copper alloys is the compression seal whereby the metal is put into tension by thermal contraction around the glass, thus placing the glass in compression. This type of seal can only be made with devices of specific design and, in general, is highly unsuitable for use in electronic devices. As previously mentioned, copper will not form a suitable metal-oxide-glass bond due to the nature of the oxides which form.

The advent of new high performance, high expansion glasses, such as Corning 7047 (expansion coefficient 140 x 10^{-7}/°C.) has considerably increased the interest in copper base materials. The expansion coefficient up to the softening point is reasonably closely matched with copper and, in particular, to the new copper alloy C.D.A. #638.* The expansion curves are shown in Figure 1. However, even though the expansion mismatch problem has been partially resolved, the greater problem of developing a suitable metal-glass bond has only recently been approached. A new family of copper alloys with special surface characteristics have been recently described.[5] Of these alloys, alloy 638, because of its unique oxidation characteristics can form an extremely strong bond with most glasses.

Data has been previously presented comparing alloy 638 with F-15 and C.D.A. #110 (E.T.P. copper) with a range of glasses of different expansion coefficient.[6] It was found that the alloy 638-glass system at higher thermal expansion mismatch was capable of producing bond strengths greater than equivalent seals made with alloy F-15. Moreover, fracture of the seal when it occurred was usually in the glass and not at the metal-glass interface as it was with alloy F-15. This indicated that an extremely strong bond could be obtained, in fact, a bond with greater strength than the glass, even with a relatively high degree of mismatch. In addition, a metal pre-treatment was found to be unnecessary to develop a suitable oxide for glass sealing.

Alloy 638 owes its glass sealing ability to the oxide film which forms at high temperatures. Above 300°C in air, a duplex oxide consisting of Al_2O_3 overlain with copper oxides is formed. In reducing atmospheres, the formation of copper oxides is prevented and only Al_2O_3 is formed. The temperature determines the kinetics of formation. It is this Al_2O_3 film, which is tightly adherent to the metal surface and elastic in nature due to its thickness ($\sim$200Å) which is responsible for the superior sealing characteristics of the material and its tolerance to relatively high degrees of mismatch.

The purpose of this paper is to describe some recent work actually performed under plant conditions. A single electronic device, a TO5

*Alloy 638 nominal composition Cu + 2.8% Al + 1.8% Si + 0.4% Co.

header assembly was selected for test purposes. Various sealing conditions and metal pre-treatments as previously determined in the laboratory were chosen. The results are discussed relative to optimum sealing parameters and the mechanism of bonding.

EXPERIMENTAL PROCEDURES

Materials

The standard TO5 header assembly (without the internal circuitry) consists of 8 lead pins, a suitably stamped header and can and a glass preform. The component parts and a semi-finished device are shown in Figure 2. A schematic diagram is also provided in Figure 3. The dimensions of the glass preform are such to be a loose fit around the pins and in the header can. During firing, the glass flows filling the whole cavity and forming an hermetic seal. The header cans were stamped out from .015″ gauge alloy 638 sheet employing tooling normally used for production of these components from F-15 alloy. The pins were cut from .018″ dia. annealed wire made in the laboratory (commercial material was not available at that time) by machining and subsequently drawing to finished gauge with one interanneal. All parts were subsequently degreased in benzene followed by a clean in 1N boiling NaOH soln. for 15 secs. followed by a 30 sec. dip in 12% H_2SO_4 at 150°C followed by a thorough wash and dry. The cleaning ensured an oxide and grease free metal surface.

The 7047 preforms were fabricated and supplied by Corning Glass Works, Corning, New York.

Seal Fabrication

The experimental sealing work was performed at the Seal-A-Metic Corporation, Division of Haledon Industries, Haledon, New. Jersey. Details of the sealing equipment employed are not particularly relevant and are not the subject of this paper. However, the techniques employed are typical of those used by the semi-conductor industry in general.

The header cans together with the glass preforms and pins were set up in carbon blocks. The whole assembly was then placed on the moving belt of a high temperature atmosphere controlled furnace and the firing cycle adjusted as necessary. Provision was made for both adjustments in firing parameters and glass annealing parameters (necessary to enable the required glass properties to be attained). Several individual runs based on previously obtained laboratory data were made varying firing temperature, belt speed (firing time) and atmosphere. In addition, in some cases the material was pre-oxidized in either air or water vapor saturated hydrogen. In one case, a special cycle was run involving stopping the belt at a specified temperature to allow metal pre-oxidation before raising the temperature for sealing. The glass annealing cycle was pre-determined and not changed throughout the tests. Glass annealing will not be discussed in this paper.

After fabrication, the TO5 headers were examined for obvious defects and then subjected to an hermeticity test. Following this, some

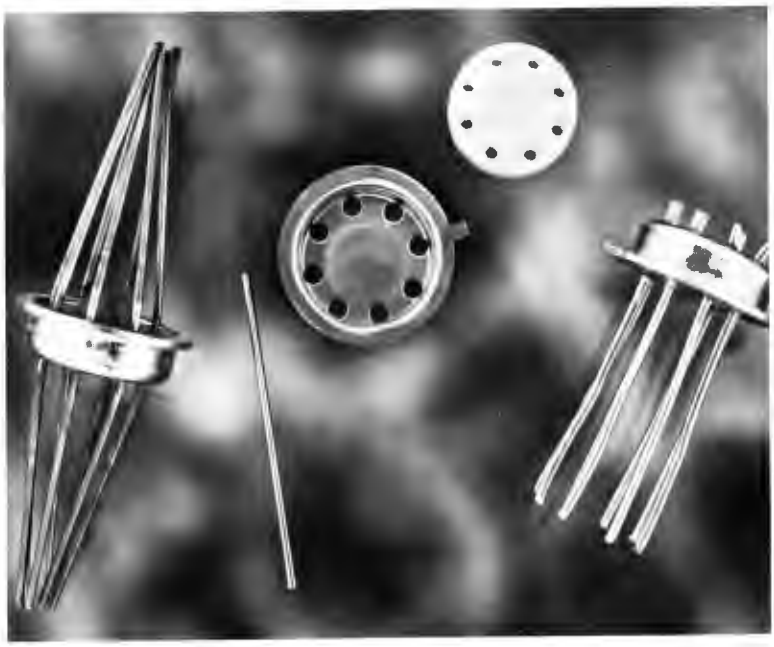

Figure 2
Component parts of a typical T05 header assembly.

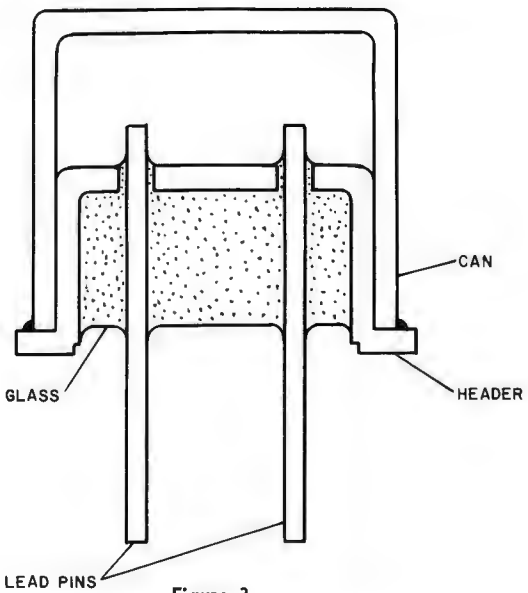

Figure 3
Schematic diagram of T05 header without internal circuitry.

samples were given a thermal shock test to simulate lead wire bonding conditions. Other samples were sectioned metallographically to observe the metal-glass interface.

Sealing Parameters

The furnace temperature was varied in 25°C increments from 700°C to 750°C. Belt speed was adjusted to either 3, 4 or 5 feet per minute. Adjustment of belt speed tended to change the heat up and cool down rate but not the actual sealing time at these low belt speeds. The faster the belt speed, the faster the heat up and cool down rates. The furnace atmosphere was adjusted to be either reducing, neutral or oxidizing by changing the gas mixture. A reducing atmosphere was mainly a mixture of approximately 6% CO_2, 4% CO balance methane whereas the neutral atmosphere consisted of approximately 10% CO_2 balance methane. Oxidizing atmospheres could be created by introduction of oxygen to the neutral atmosphere. Levels of ½% to 2% were tried.

Metal pre-oxidation was accomplished either in air (which develops an Al_2O_3-copper oxide mixture) or wet hydrogen (which develops only Al_2O_3) at 740°C. Headers and pins were heated prior to assembly in air for 1-5 mins. in 1 minute increments. Prior oxidation in wet hydrogen was done with a 15 minute or 60 minute heating. In some cases, the metal was etched in conc. HNO_3 for 30 secs. prior to assembly without subsequent oxidation.

In one case, after a suitable firing temperature and belt speed had been determined, a special cycle was employed. The headers were run through the normal cycle (belt speed 4 f.p.m.) until the temperature reached 540°C. The belt was then stopped for 3 minutes to pre-oxidize the metal before glass melting occurred and then the normal cycle was continued. A summary of all tests carried out is shown in Table I.

Evaluation Procedures

Hermeticity was tested at 10^{-6} torr pressure immediately after sealing. At least 6 samples were tested from each experimental run and acceptance was based 100% pass rate. Some samples were metallographically sectioned and examined.

A thermal shock test simulating die attach conditions was then performed on "good" samples. A standard heat column at 440°F was used. The header assembly was placed for 1 minute on the heat column and then removed and rapidly air cooled. The "shocked" samples were then tested for hermeticity. This kind of thermal shock test would not be required if die attach were by means other than Au-Si eutectic brazing. The new epoxy bonding techniques would alleviate the need for such a severe test.

RESULTS

The results of all the tests outlined in Table I indicate that the alloy 638-Corning Glass 7047 system is sensitive to certain sealing parameters. In particular, the furnace atmosphere and temperature were highly

Table I

Alloy 638 Condition	Furnace		Hermeticity Testing		
	Atmosphere	Temperature	As Sealed	Thermal Shock 440°C	470°C
As Received	All	700°C	F[1]	—	—
	All	725°C	F[1]	—	—
	Reducing	750°C	F	—	—
	Neutral	750°C	F	—	—
	½% oxidizing	750°C	F	—	—
	2% oxidizing	750°C	F[2]	—	—
Pre-Oxidized Wet Hydrogen (15 mins.) 740°C	All	700°C	F[1]	—	—
	All	725°C	F[1]	—	—
	Reducing	750°C	F	—	—
	Neutral	750°C	F	—	—
	½% oxidizing	750°C	F[2]	—	—
	2% oxidizing	750°C	F[2]	—	—
Pre-Oxidized Wet Hydrogen 1 hr. 740°C	2% oxidizing	750°C	P	P	F[2]
Pre-Oxidized in Air 740°C 2-4 minutes	All	700°C	F[1]	—	—
	All	725°C	F[1]	—	—
	Reducing	750°C	F	—	—
	Neutral	750°C	F	—	—
	½% oxidizing	750°C	P	P	F[2]
	2% oxidizing	750°C	P	P	P
Pre-Oxidized in Air 740°C 5 minutes	2% oxidizing	750°C	P	F	—
Special cycle—heat to 540°C in furnace, hold 3 mins.	2% oxidizing	750°C	P	P	F[2]

important. Within the range of belt speeds investigated (3-5 f.p.m.) no noticeable differences could be seen. However, no doubt significant deviations from these speeds may likely result in failure (see later). Briefly summarized the results would indicate the following optimum sealing parameters based on use of the Seal-A-Metic equipment. Hermetic seals which would pass a 440°C thermal shock test could be made within these parameters. The 470°C test is considered highly severe and is normally above that required to satisfy users.

1. Temperature of sealing — 750 ± 10°C. Lower temperatures may result in incomplete wetting of the metal parts due to high glass viscosity. Higher temperatures may result in a change in oxidation characteristics of the metal.
2. Belt speed (thermal cycle) — 4 ± 1 f.p.m. The significance of the thermal cycle is discussed subsequently.
3. Furnace Atmosphere — 2% oxidizing or above. No seals could be made in reducing or neutral atmospheres due to

either excessive bubbles developing in the glass ("blowing") or insufficient oxidation of the metal. A pre-oxidation treatment or change in thermal cycle, however, make possible the use of reducing atmospheres.

4. Metal pre-oxidation —
   a. 3 ± 1 mins. in air at 740°C.
   b. 1 hr. or greater in wet hydrogen at 740°C.
   c. Special cycling involving a "hold" time at an intermediate temperature during firing — 540°C for 3 mins. in this case.

These parameters are, of course, completely inter-related and changes to suit individual equipment can be made.

A metallographic cross section of a typical TO5 header is shown in Figure 4. It is difficult to detect any interfacial oxide layer due to its extreme thinness. In fact, it appears as though there is no interfacial layer. The oxide, however, is present and can be detected by other means. Thickness is less than 200Å.

DISCUSSION

The results quite clearly show that the alloy 638-Corning Glass 7047 composite is a viable system. Provided the sealing conditions are controlled within reasonable limits, hermeticity and thermal shock resistance can be obtained. The conditions of sealing are determined by the formation of the interfacial oxide layer. It is this layer that acts as an intermediary buffer between glass and metal.

Consideration of the mechanism responsible for the sealing characteristics explains in general the necessary sealing and processing conditions. To create a metal-glass bond, the properties of the metal, metal oxide and glass must be quite specific. In the present case, the expansion coefficients of metal and glass are closely matched which tends to reduce residual stresses along the metal-glass interface after sealing. In addition, the properties of metal and glass are fixed and, therefore, the intermediate oxide layer is controlling. If this layer does not have the required properties, the integrity of the bond will be destroyed.

Let us consider now the events occurring during seal fabrication. A typical temperature/time profile is shown in Figure 5. Assume first of all that no metal pre-treatment besides thorough degreasing has been performed. As the metal enters the furnace, in the necessary oxidizing atmosphere below 300°C copper oxides begin to form, Stage I. Copper oxides are somewhat undesirable since they do not contribute significantly to the bond. However, their presence may have a secondary effect (see later). Stage I should be as short as possible to avoid the formation of too thick a copper oxide film which can affect bond strength and in large quantities may change the properties of the glass adjacent to the interface. However, with very aggressive glasses (such as Corning 7047) the presence of some small amount of copper oxides may effectively reduce the dissolution rate of the subsequently formed Al_2O_3 which in some cases, particularly during long firing times may be an advantage.

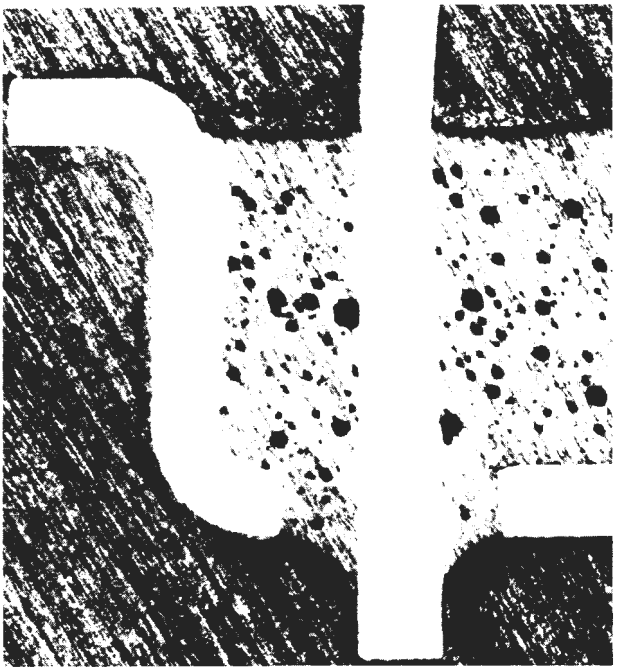

Figure 4
Section through a T05 header illustrating very thin oxide interfacial layer.

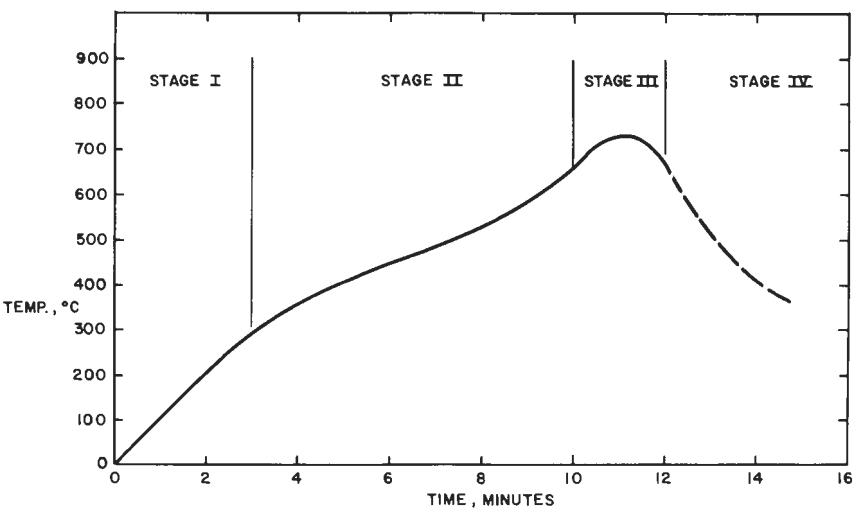

Figure 5
Temperature profile of sealing cycle for alloy 638 - Corning 7047 glass T05 headers.

Stage II which occurs above 300°C is where the Al_2O_3 film begins to form. The oxide forms slowly at first at the lower temperature until the whole metal surface is covered. Copper oxides form simultaneously until the whole surface is covered with Al_2O_3 when copper oxide formation stops. The formation kinetics of Al_2O_3 in air are shown in Figure 6. It can be seen that the kinetics are quite dependent on temperature. However, at 740°C a limiting thickness (~275Å) is reached in about 15 mins. Under the conditions of the present experiments (without any metal pre-oxidation) it is unlikely that the thickness of Al_2O_3 will be much above 100 Å. Apparently, this thickness of Al_2O_3 is insufficient to enable adequate bonding to occur (probably because the oxide is completely dissolved by the glass). Therefore, either a long heating cycle in this temperature range or a "hold" at below the sealing temperature may be required to generate a sufficiently thick Al_2O_3 film.

Stage III is the actual sealing range where the glass melts and begins to dissolve the oxide film. This stage must be sufficiently lengthy to enable complete glass melting and wetting of the metal surfaces, but sufficiently short to prevent complete dissolution of the metal oxide by the glass resulting in non-bonding. The optimum length of time is, of course, dependent on many factors. However, the Al_2O_3 film formed during Stage II should be sufficiently thick to avoid complete dissolution in Stage III.

Stage IV is the cooling cycle and is somewhat less critical than the other stages. However, some solid state diffusion of Al_2O_3 into the glass can occur when the glass is solidified and therefore, a fast cool down to the temperature of annealing should be accomplished.

A schematic illustration of the whole sealing cycle is shown in Figure 7.

Metal pre-oxidation is probably the most effective way of ensuring a sufficiently thick Al_2O_3 film. Pre-oxidation at 740°C in air for 3 mins. will develop around 100 Å of Al_2O_3 (see Figure 6). In addition, the normal heating cycle (Stage II) will form about 100Å more giving a total Al_2O_3 thickness of >200 Å. However, the results would indicate that too long a pre-oxidation treatment in air results in some degradation of the bond. This is unlikely to be due to the somewhat thicker Al_2O_3 film which forms but is more likely due to excessive copper oxide formation resulting in either degradation of the glass properties at the metal oxide-glass interface or non-dissolution of Al_2O_3 which is necessary for a bond to form.

Another more closely controlled pre-oxidation is by heating in water vapor saturated hydrogen (a wet reducing atmosphere). Figure 8 shows the oxidation kinetics at two temperatures. It is evident that ta 750°C, in 1 hour about 135Å of Al_2O_3 forms. Additional Al_2O_3 will also form during Stage II heating again with the development of >200Å total thickness of Al_2O_3. However, no copper oxides will be present somewhat accelerating the sealing process in Stage III.

The furnace atmosphere is only likely to affect Stages I and II with regard to the metal. A reducing or neutral atmosphere was found to be inadequate under the conditions of our experiments. This is no doubt due

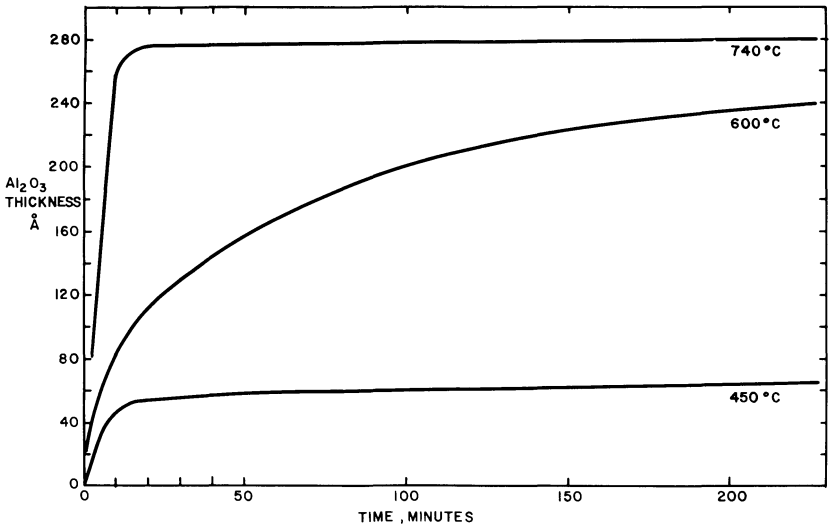

Figure 6

Thickness of Al_2O_3 formed on alloy 638 as a function of time heated in air at various temperatures.

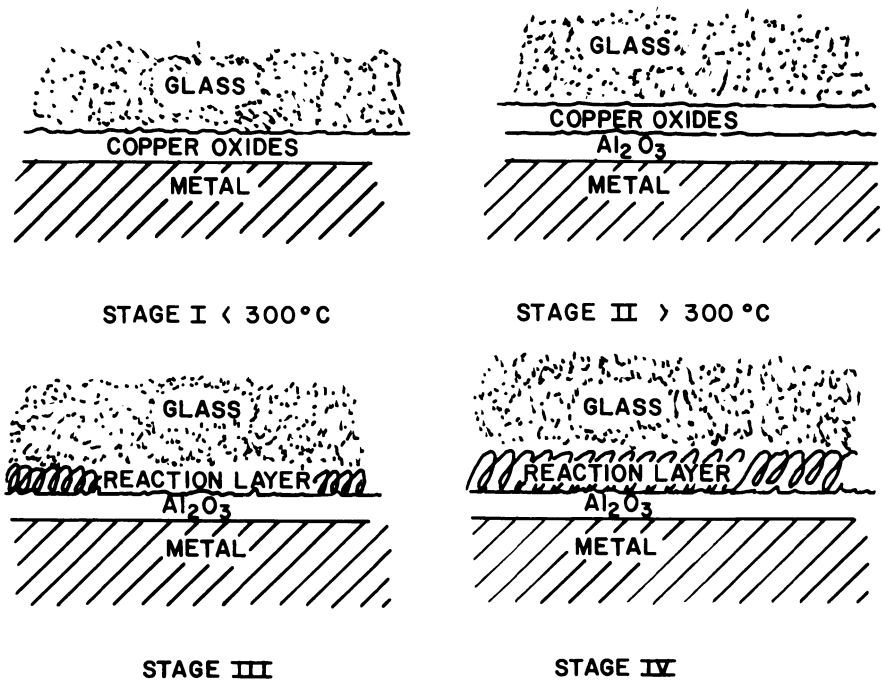

Figure 7

Diagramatic illustration of various stages in sealing process.

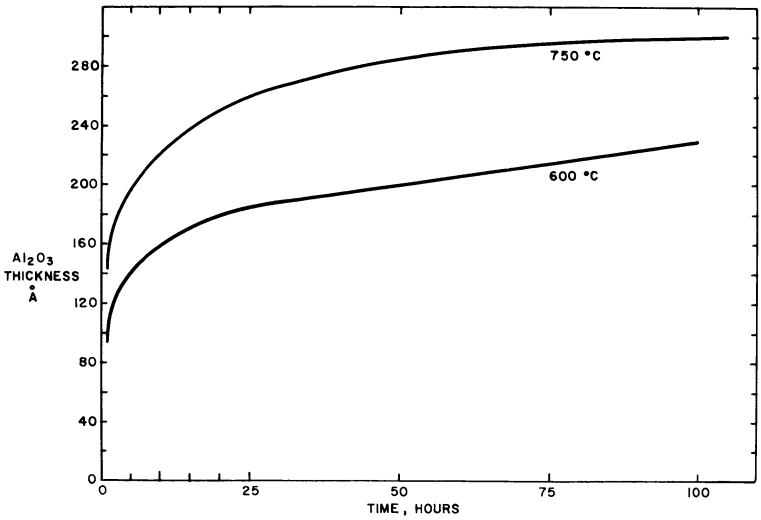

Figure 8

Thickness of Al_2O_3 formed on alloy 638 as a function of time heated in a wet reducing atmosphere.

to the slowing down of the Stage II oxidation kinetics. Apparently, a thick enough film of Al_2O_3 must be present to prevent complete dissolution in Stage III.

The controlling parameters in successfully sealing glass to alloy 638 are the thickness and solubility of the Al_2O_3 film in the glass. The properties of the glass, of course, determine the latter point. However, furnace temperature, heating time, sealing time, atmosphere and whether preoxidation has been carried out determine the success of the seal.

In summary, therefore, it would appear that under the sealing conditions used, the Al_2O_3 film must be in excess of 200Å to achieve an hermetic seal. Of course, changing sealing conditions may change this thickness. It is recommended that the following processing guidelines be used in making alloy 638-Corning 7047 glass seals. Deviation from these guidelines may result in non-bonding and the necessity to re-evaluate preoxidation or furnace cycle.

1. The heat up rate to 300°C should be as rapid as possible >80°C/min. is recommended. This assures that the amount of copper oxide formation is minimized.
2. The heat up rate from 300°C to the glass sealing temperature should be relatively slow to allow sufficient Al_2O_3 to form to achieve a good bond. A maximum rate of 40°C/min. is recommended.
3. The sealing cycle should be rapid to avoid excessive Al_2O_3 dissolution and consequent loss of bond. A total time above 650°C of 2 mins. is recommended.

4. Cool down rate after sealing should be initially rapid to avoid any excessive solid state diffusion. However, this stage is not critical.

The above points assume a sealing temperature of 750 ± 10°C and an oxidizing atmosphere (2% oxygen). In the particular equipment used in these studies, the appropriate cycle was achieved by adjusting belt speed to 4 f.p.m.

Pre-oxidation can be accomplished either in or outside of the furnace. Adjustment of the heating cycle (Stage II) to give either a "hold" at a given temperature or a slow heat up rate should be adequate. If pre-oxidation is carried out prior to sealing, oxidation in air at 740°C for 3 ± 1 mins. or at the same temperature in wet hydrogen for at least 1 hour is recommended. Particularly in the last case, because of the absence of copper oxides it may be advisable to heat for several hours. The hydrogen pre-oxidation is somewhat preferred for aesthetic reasons since the samples are bright and gold colored after sealing. In addition, they are somewhat easier to clean. However, pre-oxidation in air or during the actual sealing cycle can also be recommended as being highly effective.

As a final comment, these experiments describe only one glass, namely, Corning 7047. As other high expansion glasses become available, sealing parameters must be changed to account for the possible different chemical properties.

CONCLUSIONS

Alloy 638 metal/Corning 7047 glass seals can be effectively made under plant processing conditions. The quality of bond is somewhat dependent on sealing parameters. These sealing parameters (time, temperature and furnace atmosphere) are inter-related.

The bonding mechanism relies on the formation of a tightly adherent Al_2O_3 film on the metal surface which slowly dissolves in the glass creating a chemical bond.

The two important parameters governing bond formation are the kinetics of formation of the Al_2O_3 film (and its consequent thickness prior to bonding) and the dissolution rate in the glass. Sufficient Al_2O_3 must be present before sealing (>200 Å) to ensure incomplete dissolution in the glass at high temperatures. Complete removal of Al_2O_3 results in loss of bonding.

ACKNOWLEDGEMENTS

The author would like to thank the Brass Division of Olin Corporation for permission to publish this work. Thanks are also due to Mr. A. Dudas of Seal-A-Metic Corporation, who performed much of the experimental work and to Corning Glass Works for supplying the glass preforms. The helpful advice and discussions with many of my colleagues, in particular, Drs. E. J. Caule, J. A. Ford and M. J. Pryor are much appreciated.

Notes:

1. Failed due to incomplete wetting of metal by glass.
2. Not all samples failed.

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FABRICATION OF A TRAVELLING WAVE TUBE ENVELOPE

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INTRODUCTION

The travelling wave tube is an electronic amplifying device. It accepts a weak R.F. input signal and amplifies it many thousands of times. It performs the same function as its principal predecessors — the triode and klystron. It has one characteristic uncommon to the other devices: extremely wide bandwidth. The travelling wave tube is capable of performing this amplification at bandwidths up to several thousand megacycles.

The travelling wave tube was invented by Dr. Rudolph Kompfner during the World War II period of 1941 thru 1944. The importance of his invention was soon realized and intensive development work has been carried out in many laboratories to bring the tube to its present state.

HOW THE TWT OPERATES

Basically the travelling wave tube consists of a magnetic circuit and an electronic tube. For the purpose of this discussion we will concentrate on the electronic tube — in particular the glass envelope.

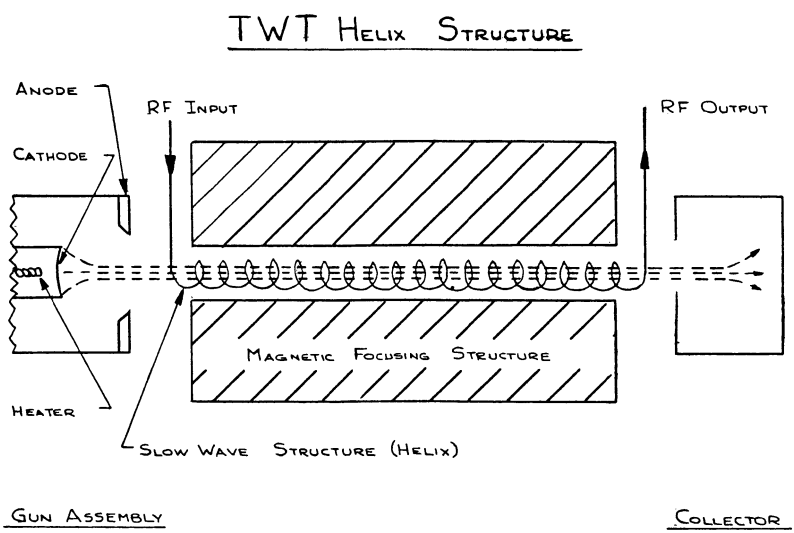


Figure 1

The first figure is a simplified sketch of a helix-type TWT; the original circuit invented by Dr. Kompfner. At the left of the diagram is the electron gun assembly. The cathode when heated emits a continuous stream of electrons. The magnetic field produced by the magnetic structures focuses the beam of electrons to travel the length of the tube. Without the magnetic field the electron beam would tend to disperse itself during its travel due to the combined influence of the mutual repulsion of electrons and the attraction resulting from the positive voltage on the helix. The electrons dissipate in the collector in the form of heat.

At the same time that the electron beam is moving through the tube, the desired RF signal is fed into the helix, which visually resembles a helical spring. This signal travels at the speed of light through the helix. However, because of the helical path, the forward motion along the axis of the helix is much slower — on the order of about one-tenth the speed of light. The electron beam is made to travel slightly faster than the signal wave. The result is that an interaction occurs between the electron stream and the RF signal. This interaction is such that some electrons in the beam are slowed by the RF field while others are accelerated.

As the "velocity modulated" electrons move down through the helix they form bunches (see Figure 2). The bunches, in turn, interact with the

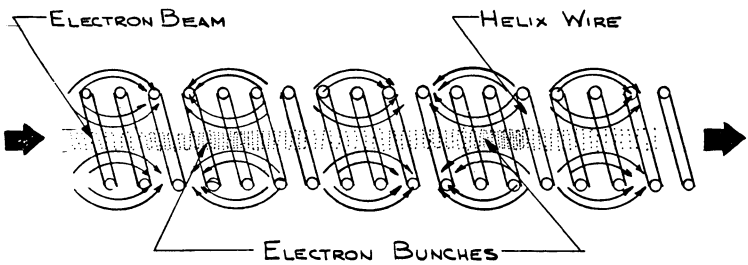


Figure 2

helix RF wave and surrender energy to it. This results in a great amplification of the RF signal. Signal TWT's have been built with power gains of more than 10,000,000 (70 decibels).

Since there are no tuned circuits in the path of the wave being amplified, the process is fairly insensitive to frequency changes, resulting in a tube capable of amplifying radio signals many thousands of times at bandwidths of up to several thousand megacycles. The significant feature of the travelling wave tube is its freedom from bandwidth limitations at microwave frequencies even up to 75,000 megacycles.

THE GLASS ENVELOPE

Figure 3 is a drawing of a representative glass envelope for a travelling wave tube. Certain specifications are extremely critical for the proper operation of the finished product.

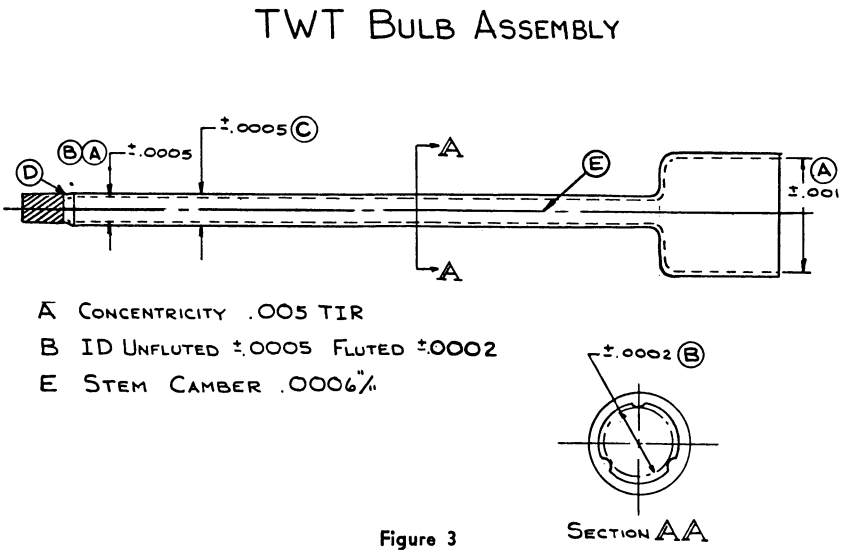


Figure 3

The concentricity (A) of the bulb I.D. to the stem I.D. is important. If the tube concentricity is maintained the alignment of the electron gun to the helix axis will be maintained.

If the electron beam is not properly directed to the axis of the helix, a portion of the beam might impinge on the helix, thereby causing a reduction in the efficiency of the finished TWT and reduce the tube life.

The inside diameter of the stem or barrel (B) must be closely held. The helix must be mechanically fixed to the stem at every turn. It must be tightly held, but it cannot be embedded along its entire circumference, else a portion of the energy will be dissipated through the glass wall. To accomplish this there must be flutes in the barrel section. This can be accomplished by fabricating integral flutes in the inside diameter or by inserting 3 rods during the assembly of the helix. The trend is toward the integral flutes. To insure the fixing of every turn of the helix the inside diameter of the flutes must be held to tolerances in the order of magnitude of ± 2 ten thousandths of an inch.

The outside diameter of the stem (C) must be held to tight tolerances for the fixturing required to "fix" the helix, and to position the magnets around the stem. It must also be concentric to the inside diameter of the stem for much the same reasons.

A collector (D) must be accurately mechanically sealed to a sleeve which is fusion-sealed to the stem with a minimum of dimensional distortion. The helix must reach extremely close to the collector, and as previously stated every turn of the helix must be captured by the stem.

The camber or "bow" of the stem (E) is critical since the electron beam must travel thru the inside diameter of the helix for its entire length without impinging upon the helix. When you consider that the inside diameter of the helix may be only .125" and the length of the helix may be 11" long, lack of camber is extremely important. The entire TWT may be flexed in operation to correct for any misalignment in assembly to insure proper direction of the electron beam, and to obtain optimum signal output.

Glass composition is an important consideration in the fabrication of a TWT glass envelope. If the tungsten helix is actually fusion-sealed to the barrel flutes, Corning 7720 glass (Nonex) must be used for the stem and barrel sections. To date Fischer & Porter has shown extraordinary capability with respect to Nonex construction. This will require the stem leads and the collector sleeve to also be made of tungsten, or composite materials which include tungsten in the sealing zones.

More often than not, helices are embedded into the flutes by a combination of mechanical fixturing and heat treatment, and since the metal is not fusion-sealed, a wider variety of metals and glass compositions is permissable (the helix itself must still be tungsten or molybdenum).

For several reasons, the use of Kovar-type alloys has distinct advantages and several helix-type TWT's are made using Kovar and either Corning's 7052 or 7056 glass (or their equivalents) throughout. Corning's 7056 glass has an advantage only if the heater coil is "pushed" so extensively that the emission coatings operate extremely hot and endanger sintering from elements released by the 7052 composition at these high temperatures. TWT tubes made from just one glass composition usually have commercial use only.

The more stringent military applications require a two-material construction. The most widely used glass components are Corning 7052 and 7070 glasses. The 7070 glass has extremely unusual electrical properties. These properties enable the TWT to have unusual characteristics. Such a structure, however, necessitates control of the graded seal required between the two glasses, and if the assembly includes a collector sleeve, of the graded seal at this location also. F&P's experience and capability with glass to metal sealing techniques and with graded seals has been highly instrumental in achieving a reputation for high quality workmanship in these type constructions.

MANUFACTURE OF THE MANDREL FOR THE GLASS ENVELOPE

Although all TWT envelopes consist of a bulb section and a stem section, the manufacture can be in one of two basic methods: one-piece or two-piece construction. The simpler commercial applications can be

made by one-piece construction, while military applications, and some commercial applications necessitate the two-piece construction.

In either method the key to the extreme accuracy of the finished product is precision in the fabrication of the "mandrel" for the vacuum forming of the glass envelope. Figure 4 shows examples of the mandrel design necessary for the vacuum reforming by the one-piece construction. As previously mentioned, the most critical portion of the TWT is the inside configuration of the stem.

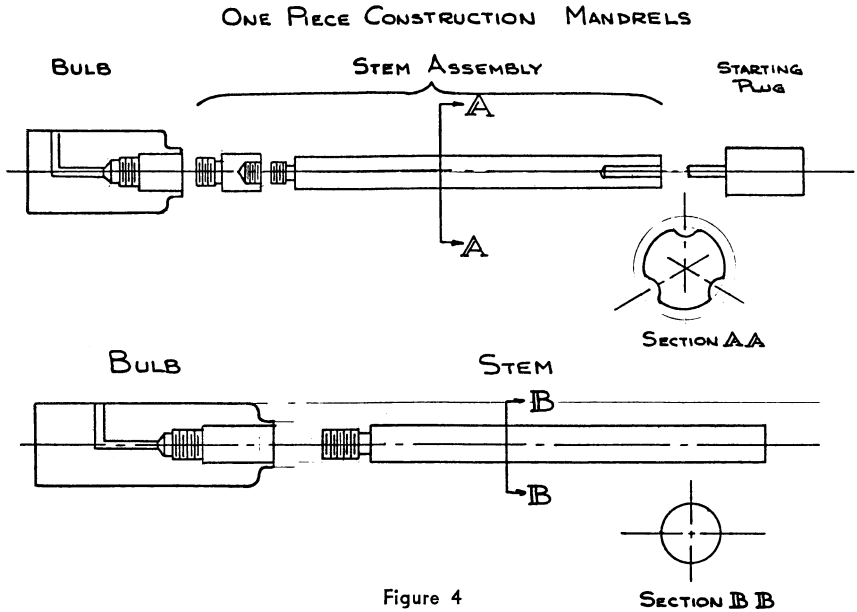
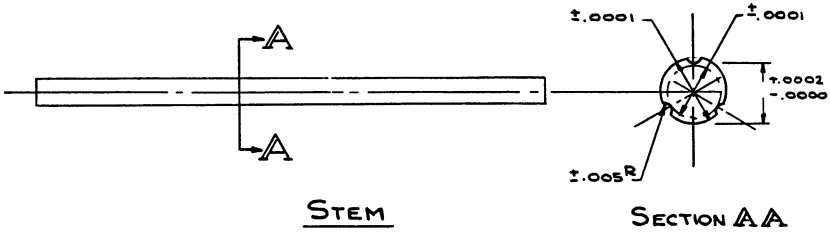


Figure 4

The one-piece construction is usually practical when the entire envelope is fabricated from one composition glass, and the outside diameter of the stem is not tightly controlled. The mandrel is generally precision ground in two or more parts and accurately joined to insure the precision alignment of the bulb and stem.

In many instances, the stem and the bulb must be separately fabricated and subsequently assembled by a skilled glassworker. Figure 5 shows an example of the mandrels fabricated for the two-piece construction with representative tolerances. As can be seen, the tolerances on the mandrel must be tighter than those of the finished glass envelope. The basic dimension of the mandrel must be calculated to allow for the differential expansion of the mandrel and the glass. Each of the glasses used in the TWT construction have different expansions, and these differences must be compensated for in the mandrel design.

MANDRELS FOR 2 PIECE CONSTRUCTION



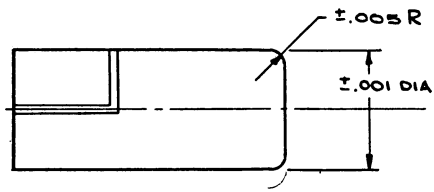


Figure 5

SPECIAL GLASS CONFIGURATION

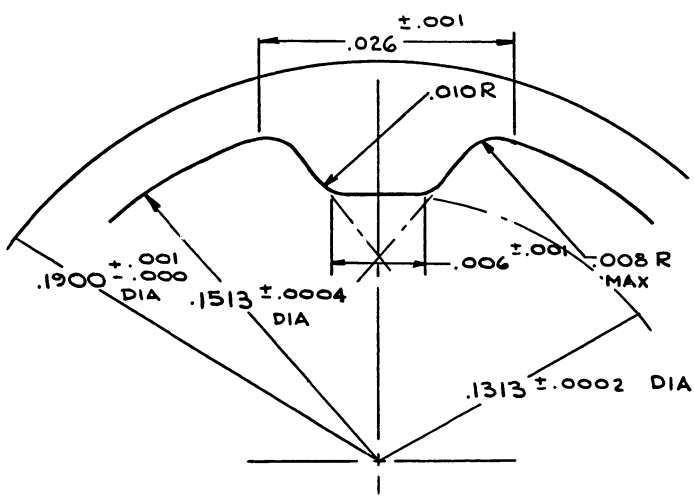


Figure 6

In some very critical applications special glass configurations are necessary. Figure 6 is an example of a critical cross-section with its respective tolerances. There are several methods available for the fabrication of the mandrel. By far the most accurate method is to precision grind every portion of the mandrel. Fischer & Porter has this capability and does very accurately precision grind all the mandrels for TWT applications.

MANUFACTURE OF THE GLASS ENVELOPE

Having a precision ground mandrel accurately sized to allow for the thermal expansion of the particular glass is one step toward a functional glass envelope. The mandrel and glass must be carefully prepared and heated to create the accurately formed glass component. Fischer & Porter has been a pioneer in the method of vacuum reforming glass components.

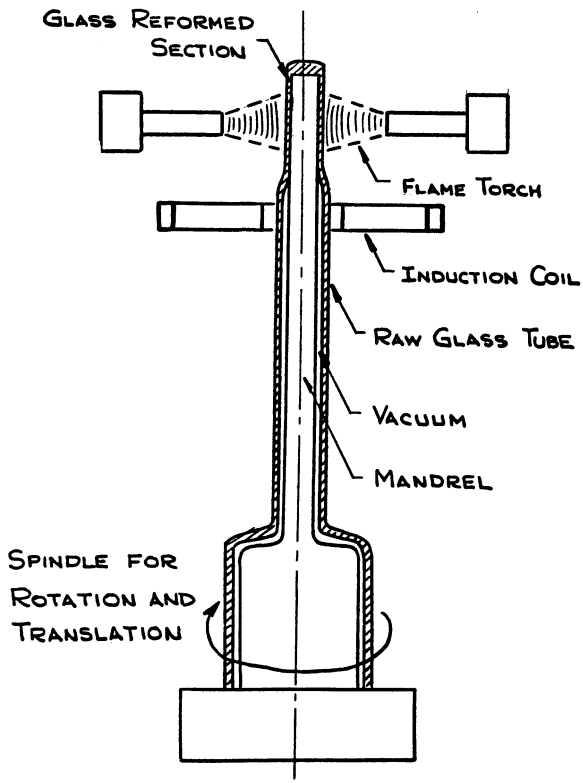


Figure 7

The vacuum forming of glass is simple in principle. A raw glass tube which is closed off is placed on an accurately precision ground mandrel and the area between the glass and mandrel is evacuated. The temperature of the glass is then increased to the region between the upper annealing temperature and softening point of the glass by means of a flame or other heat source. Atmospheric pressure causes the tube to collapse around the heated mandrel starting at the closed end where the heat is first applied and proceeding along the length until the desired portion has been shrunk on the mandrel. When applicable RF heating of the mandrel assists in the precision forming of the glass envelope, especially in preheating stages to minimize thermal cracking. As the heat is removed, the two materials cool. The greater contraction of the metal mandrel permits removal of the precision formed glass tube after cooling.

Accurate control of the heat source, tube rotation, and tube translation is necessary to maintain dimensional accuracy. Consistency of the finished tube requires accuracy in all three of the items involved in tube rundown, as well as in the chemistry of glass preparation and subsequent cleaning and the lubricants, coatings and/or parting agents used as an integral part of processing. The manufacture of precision glass components also requires an elaborate inspection and testing facility, and personnel to perform these operations who are accustomed to high-quality close-tolerance workmanship. F&P has a unique capability in this respect.

The manufacturing procedures differ materially between the one-piece, and two-piece constructions. Both procedures require the services of a skilled glass worker, controlled annealing and detailed quality control.

ONE PIECE CONSTRUCTION

Because of the significant size differential between the bulb and the stem in the one-piece construction it is necessary to accurately fabricate the glass envelope before the precision rundown. The prefabricated glass envelope must closely conform to the mandrel if the precision rundown is to create an acceptable envelope. While the inside diameter is extremely critical, the outside diameter must be closely controlled to accept the other components of the completed TWT assembly.

The precision forming operation leaves the completed envelope in a highly strained condition. This strain must be removed by a controlled annealing without distorting the high precision created in the vacuum forming process. Each different glass has its own annealing temperature, hence a different annealing cycle. The annealing must be accomplished in a vertical position to maintain straightness, and in some cases in individual, close-fitting quartz cells.

The critical dimensions must be checked on every TWT. Critical internal dimensions are checked with air gages or, in the case of fluted stems, accurately machined plug gages.

TWO PIECE CONSTRUCTION

The two-piece construction requires somewhat different techniques.

The stem can be fabricated in a conventional manner. If there is any camber in the precision formed stem, it must be removed by a straightening operation at this point. The bulb must be precision formed on a closed end mandrel. The glassblower must bottom off the glass with extreme care since the precision formed bulb is only as good as the preformed bulb. In many instances, the bulb will have a dimple accurately positioned in the center of the end to assist in the final assembly. This construction can also include an intermediate zone in the transition area to accommodate a "drift tube", or particle accelerator.

After the precision forming, the resultant strain again requires a controlled annealing cycle. This controlled annealing will allow the performing of the subsequent operations without undue breakage. Since annealing may result in small dimensional changes, the mandrels must be accurately precision ground to allow for these changes.

As previously mentioned, many TWT envelopes require a precision ground and polished outside diameter. The small sizes necessitate that the

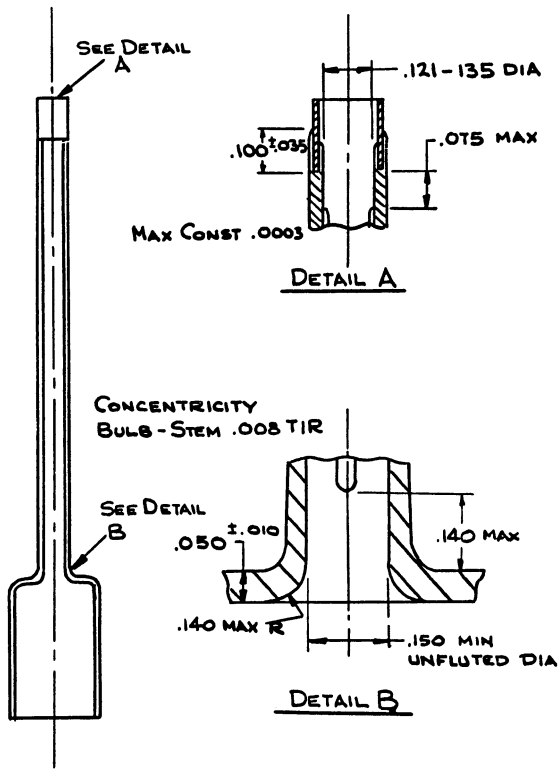


Figure 8

stems must be ground and polished on centerless grinding equipment. Centerless grinding equipment cannot improve the concentricity of the O.D. to the I.D. of the stem. The concentricity is a function of the wall uniformity of the raw glass, and the precision forming of the inside diameter.

The skill of the glassblower becomes the key to the fabrication of a completed TWT from the precision formed bulb and stem components. Machine tools can aid in the fabrication of the components but the steady hand of the skilled glassblower is required to complete the glass envelope. Wherever possible Quality Assurance is built into the construction, making Quality Control an automatic function.

Extreme care must be taken in the sealing operations in the two-piece construction. The concentricity of the inside diameters of the bulb and stem is held with the aid of proper fixturing. The configuration of the transition area must be tightly controlled, both inside and outside. If the inside diameter of the stem is fluted, the distortion must be held within a narrow band. Not only must the distortion be kept to a minimum, but the strain created in the sealing must be closely controlled. The subsequent annealing operation, in relieving the strain, may cause the size to change sufficient to create a rejected unit.

As in the one-piece construction, the completed assembly must be strain relieved. This annealing must be accomplished in a vertical position to minimize stem camber.

One principle advantage of the two-piece construction, is that the collector sleeve can be sealed to the stem as a sub-assembly, and the sub-assembly then sealed to the bulb component in final construction. This permits much tighter control over the critical geometry in this area with less cumbersome handling.

CONCLUSION

The fabrication of a TWT blends the mechanization of the precision forming operations, and the skill of the glassblower. The extremely tight tolerances created in the machine operations must be maintained by the glassblower by his or her skillful use of the torches and fixtures. Fischer & Porter has the blend of the sophisticated machinery and the skilled glass workers to fabricate TWT's and other equally critical glass components.

REFERENCE

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CHEMICALLY STRENGTHENED GLASS

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Corning, N. Y.

INTRODUCTION:

The subject of this paper will be a short commentary about Corning's chemically strengthened glasses. Hopefully this will give you a basic understanding of the glass — see how it differs from other glasses; what you can do with it, and what you can't do. We'll briefly run through the manufacturing process, look at some test results, and at some of the chemically tempered products now available.

DISCUSSION:

Chemically tempered glass has been around for a number of years. Most of you are familiar with our COREX® pipets and centrifuge tubes used in laboratories. We've also used this tempering process in auto windshields, laboratory bench tops, microwave ovens, and most recently, optical lens blanks. These are all areas where strength and resistance to abrasion are very important.

Let's take a closer look at the glass and how it's made.

By way of review, you are all familiar with standard methods of heat tempering — heating the glass and then chilling with air blasts at carefully controlled temperatures and time periods. This type of tempering can produce tensile strengths of up to 30,000 psi, depending on the materials and conditions used.

Now let's examine what happens in the chemtempering process. We start with a specific formula alumino-silicate glass. Note: that the glass has to be in its final finished form before the chemical tempering begins.

Basically, the procedure involves immersion of the glass to be strengthened in a molten bath containing potassium salts at 470°C, which is well below the point at which glass flows.

At this temperature then, the requirements for chemical equilibrium between the sodium ions in the glass and the potassium ions in the molten bath, are such that ion exchange will occur between the bath and the surface of the glass. In other words, potassium ions will replace sodium ions and diffuse into the glass.

As this ion exchange process continues, the large potassium ions, in displacing the smaller sodium ions, produces high surface compression in the glass. As the high surface compression increases, we steadily increase the internal tension in the piece. Any applied force must produce a tension higher than the built-in compression in order to put the glass surface in tension before breakage can occur. The surface compression of about 60,000 P.S.I. developed during the ion exchange procedure results in a glass which is more than twice as strong as a heat tempered glass.

To show rather dramatically what this process can do, let's take a look at the testing procedures used by lens manufacturers so their eye glass lenses will meet mandatory FDA regulations. Every eye glass lens must now undergo the famous "Ball-drop" test, where a 5/8" steel ball must be dropped from a height of 50" directly onto each lens manufactured. To illustrate the comparative strengths of different lenses, we increased the size of the weight (five times) and the height as well. Using a 1" (five ounce) steel ball, we first dropped it on standard heat tempered lenses. They broke at a height of 54". At 60", the plastic lenses broke. We started the chemically strengthened lenses at this height, steadily increasing the level upwards. Finally at 126", the chemtempered lens gave up.

SUMMARY:

Chemical tempering obviously sets an entirely new strength level for glass. At the present time, we're actively investigating a number of market possibilities where we can best utilize this unique process.

Unfortunately, applications at present are limited in the glass blowing community. The glass must be in its final form before going through the chemical tempering process. After tempering, it cannot be lampworked. Silkscreening, autoclaving, etc., is OK but heating to the softening point destroys the high surface compression and its strength. Actually, the glass itself is rather difficult to lampwork and will not seal to 7740 or soft glass. Further developments could possibly change this, but not in the near future.

Hopefully, this presentation will give you a better understanding of what chemical tempering is, and what it can do should anyone inquire or need information at your place of work.

METAL TO CERAMIC SEALS
HIGH CURRENT CAPACITY GLASS TO METAL SEALS
QUARTZ SEALING WITH R. T. V.

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General Electric Company
Corporate Research & Development Center
Schenectady, New York

This paper will first concern the building of metal ceramic seals for vacuum devices.

Because there have been many books, papers and reports written on metal-to-glass sealing technology there is nothing new in this paper. Its sole intent is to give to my fellow glassblowers an understanding of a technology that has in a great many cases replaced the glassworker and hopefully to whet your appetite enough so that you will look further into the subject and learn more about it so that if you are called upon to make a ceramic to metal seal you will be prepared to do the job.

Why do we use ceramics instead of glass? - some of the advantages are higher mechanical strength and generally better thermal shock; lower dielectric loss especially at elevated temperatures; higher processing and operating temperatures. There are also disadvantages. They are more costly to make and losing the ability to visually see what is going on inside a vacuum device are a couple of the disadvantages.

There are various types of ceramics available and many companies that sell ceramics. Ceramics can be purchased with the metalized ends or plain. A few companies selling such ceramics are Coors, McManel Corp. and General Electric Co., Microwave Tube Division, Schenectady, New York.

The term metalizing is used to define a process where a dense, predominately metallic coating is applied in such a way that it will be tightly adherent and vacuum tight. The metallic coating will differ with different type ceramics. If one applies powdered molybdenum to the surface of a pure alumina ceramic and heats it to a very high temperature in a non oxidizing atmosphere no bonding will be effected since there is no appreciable reaction between these materials below the melting point of alumina. If however, the same powdered molybdenum were coated on a high alumina, (94% alumina) with the balance silican dioxide (SiO_2) magnesium oxide (MgO) calcium oxide (CaO) and heated to 1500°C, some bonding would be obtained. Microscopic examination would show a sintered but porous molybdenum layer which had become attached to the ceramic by virtue of a penetration of the glass phase of the ceramic into the pores of the molybdenum coating. The extent of penetration depends on the composition of the ceramic, the porosity of the molybdenum layer and the furnace atmosphere. If, however, manganese is added to the molybdenum, reaction takes place at a lower temperature. However, the manganese must be oxidized to manganese oxide (MnO). This is

achieved during sintering by maintaining enough moisture in the hydrogen sintering atmosphere to drive the reaction in that direction. Thus, when molybdenum-manganese mixture is applied to 94% alumina and heated in a wet hydrogen atmosphere to 1500°C, the manganese oxide reacts with the crystalline and glassy phase of the ceramic at the interface, forming a liquid which can easily penetrate the pores of the molybdenum layer. Thus, the molybdenum layer will be attached to the ceramic by means of a partly glassy, partly crystalline "reaction product" which interpenetrates the molybdenum coating and the ceramic. The moly-manganese coating can be painted on or silk screened. Vitta Corporation of Wilton, Conn. also produces a tape type material which can be used, but caution must be exercised so the right tape to match the ceramic is used.

Since the molybdenum is a porous coating, and is not readily wet by the commonly used hard solders (silver, silver-copper, gold-copper) it is desirable to apply a thin layer of nickel and or copper by electro-deposition to enhance flow of the braze material over the surface. Thus, at the brazing temperature, the braze material and electroplated metal will melt and flow, filling all the pores and produce a vacuum tight seal. After the nickel (we prefer nickel) is plated on the moly-manganese, it is sintered in dry hydrogen at 1200°C. If the molybdenum layer is thin or the pores are large and interconnected, the electroplating and braze material will be able to penetrate and seal off all the channels and a good vacuum tight seal will result. If, on the other hand, the molybdenum is thick or the pores small, the braze material will penetrate only a short distance thus leaving a seal that will probably leak.

There are various types of ceramic metal seals that can be made. The type we make for vacuum tube envelopes are flat seals to the end of the ceramic called stack seals. The wall thickness of the ceramic is ¼" thick and the ceramic pieces are cut and ground flat, perpendicular to the axis of the tube to about .001. The end pieces of metal that are brazed to the ceramic are made of .020 ceramvar which are spun to shape and coined. Coining is a process where the parts are put between flat plates in a hydraulic press and pressed flat and smooth.

A back up ring of ceramic is made about ¼" long. This ground to the same tolerances as the ceramics for the body and prepared the same way. The back up ring is used as the end ceramic to add strength to the metal ceramic seal. In language familiar to the glass blower it would serve the same purpose as sealing glass to both sides of a metal to glass seal. It prevents the metal from tearing away from the ceramic.

Figure 1 shows a 6" diam. 2" long piece of ceramic plus the back up ring.

Figure 2 shows the sequence in setting up the tubes for brazing, the ceramic tube is set up on a flat plate of moly and in the following sequence: the back up ring, a washer of brazing material .005 thick, ceramvar metal spinning, ceramic. This would be one end of the tube. The opposite end would be set up the same and any subsequent sections in the tube body would be the same. A weight of one quarter pound per square

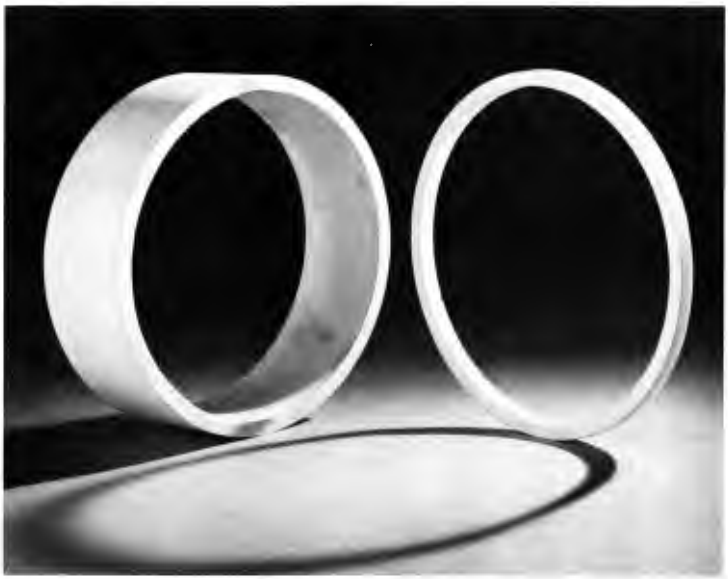

Figure 1

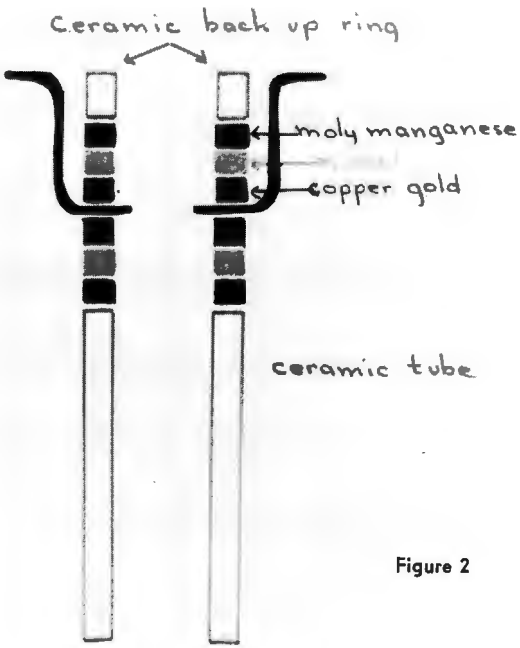

Figure 2

Figure 3

inch is placed on top of the assembly and the furnace is now brought up to the brazing temperature.

Figure 3 shows a completed tube body using this technique. The metal ends will accept flanges that are welded in place. The center metal seal in this case is provided for shields to be welded inside the tube. Actually this tube could have many sections to it. We are at present working on seals that are 9″ in diameter and have six sections which means we are making seven simultaneous brazes.

The choice of a metal to seal ceramic is dictated by the thermal expansion of the materials. Kovar and ceramvar, both nickel, cobalt, iron alloys are found most useful for high alumina ceramics. In this tube body we are using .020 ceramvar purchased from Wilbur Driver Co., Newark, N.J. The brazing material being used is a silver-copper alloy having a liquidus point of 780°C (Cusil made by Western Gold and Platinum); copper (65%) gold (35%) alloy having a liquidus of 1040°C can also be used.

Figure 4 is a schematic of another type metal ceramic seal used for electrical feedthrough: the ceramics are prepared by grinding a 7° taper on the end and metallized as explained previously. The metal part to be brazed also has a 7° taper machined or spun so that the ceramic fits inside the metal. These parts are set up and brazed as previously explained. In this case we electroplate copper on both the metal taper and ceramic before brazing. References for this part of the paper are from "Glass to

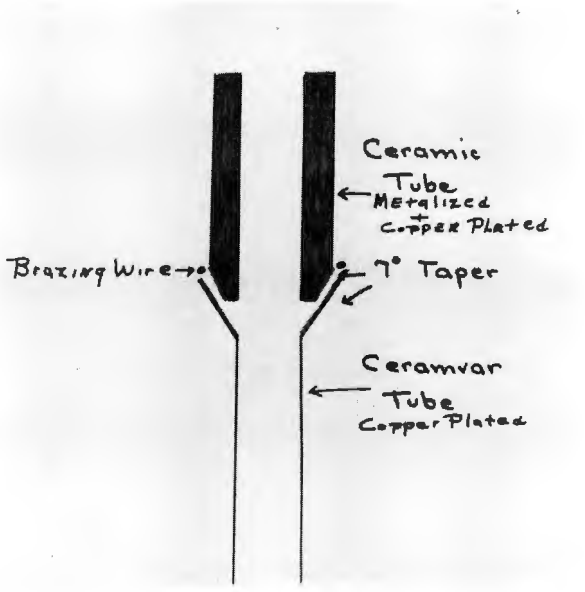

Figure 4

Ceramic Seal Technology" by R. H. Bristow, "Materials and Techniques for Electron Tubes" by Walter H. Kohl and Vitta Corporation.

The second part of my paper concerns electrical feed through into glass or metal vacuum systems.

When it becomes necessary to insert electrical feed throughs in glass envelopes where the power requirements make it impossible or unwise to go directly from the metal lead through glass, we have been using a technique where we seal the glass to a piece of Kovar tubing and proceed in the following maner: (Fig. 5) as an example, we use a piece of ½" dia. Kovar tubing, approximately ¾" long. The glass is sealed to the Kovar tubing and prepared to seal to the tube body. We make a plug out of stainless steel flat stock. The O.D. of the plug will be a tight fit inside the Kovar tubing. The plug will be machined so that a ¼" rod can be brazed into the plug. The one side of the plug is prepared to weld into the Kovar tubing. The plug with the rod can be heli-arc welded into the Kovar tube. (Fig. 6 shows the assembled parts) now the glassed end of the Kovar tubing can be sealed into a glass envelope. The ¼" rod that is brazed into the plug can be made of a variety of metals such as stainless steel, molybdenum, tungsten, copper or Kovar. The diameter of the rod can be increased and also the diameter of the Kovar tubing. In fact, there is no reasonable limit to the size. Glass seals to metal rods are governed by the amount of current being applied through the conductor. If a great amount of current is being applied the leads will heat up and crack. With this design we have stronger construction without worry of seal failure. (Fig.7)

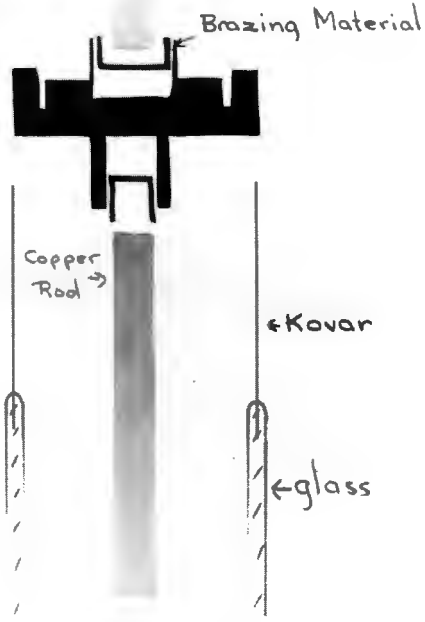

Figure 5

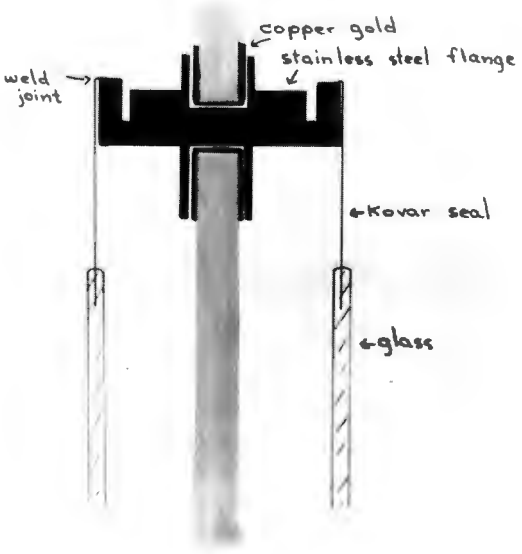

Figure 6

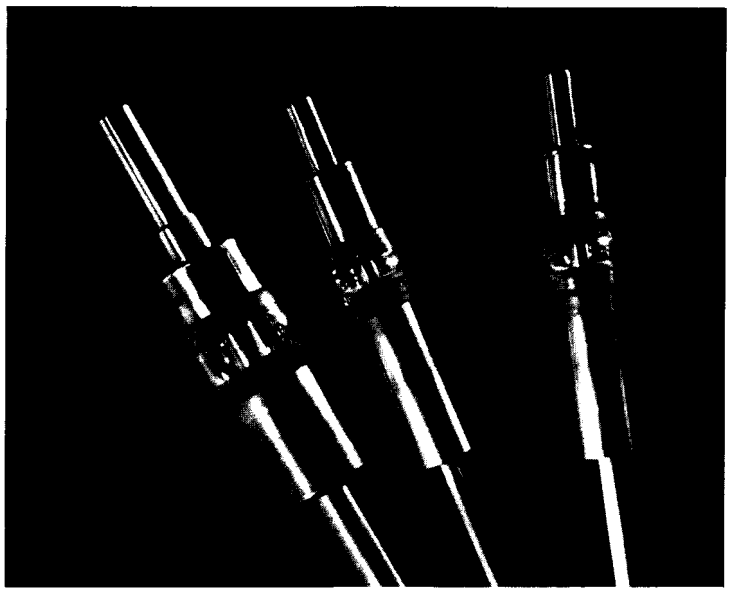

Figure 7

this same scheme can be used to make feed throughs through metal tube bodies or shear seal flanges. In this case we seal Kovar tubing to both ends of a glass tube. The length of the glass tubing being equal to the diameter. Then the one Kovar tube has the plug with the conductor. The opposite Kovar seal is welded to the metal sheer seal flange or tube body. In some cases these seals are more reliable than ceramic metal feed through, especially where the seals are repeatedly baked out around 400°C. In most cases they are cheaper to make than metal ceramic feed throughs.

Figure 8 shows a metal flange with 4 small seals. These have approximately $\frac{1}{8}$" stainless steel conductors. It also has two $\frac{1}{2}$" diameter conductors plus 2 glass seals used in this case to introduce gas into the system.

The third part of this paper concerns sealing quartz tubing into stainless steel or any metal using General Electric R.T.V. 60 compound where temperature requirements are not over 250°Cent.

Here we are not limited by the size of the tube such as would be required if we were making a graded seal from quartz to metal. (Fig. 9) in the following manner we have sealed up to 6" diameter quartz tubing into stainless steel flanges. First, the metal flange is machined about .020 larger inside than the outside diameter of the quartz tube. Then all the parts to be sealed are primed with S4004 silicone primer — after allowing at least an hour to dry — the R.T.V. 60 is mixed with a catalyst and applied to the sealing surfaces. The parts are put together and the cavity around the quartz tube and the metal flange are filled with the rubber compound and allowed to air dry overnight. As I previously stated, these seals are good for about 250°C. We have also used this technique for many more

Figure 8

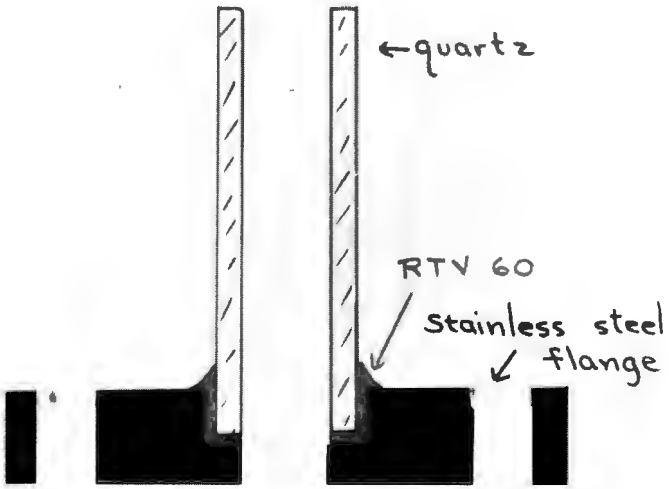

Figure 9

applications. (Fig. 10) a few examples are odd shape cells where heat would destroy the optical flatness of the windows. (Fig. 11) quartz con-

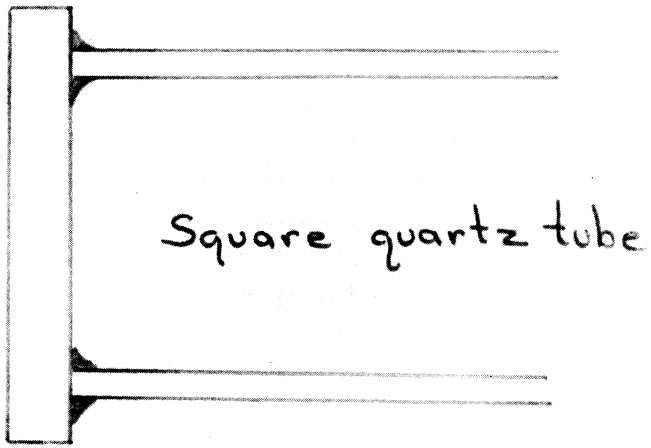

Figure 10

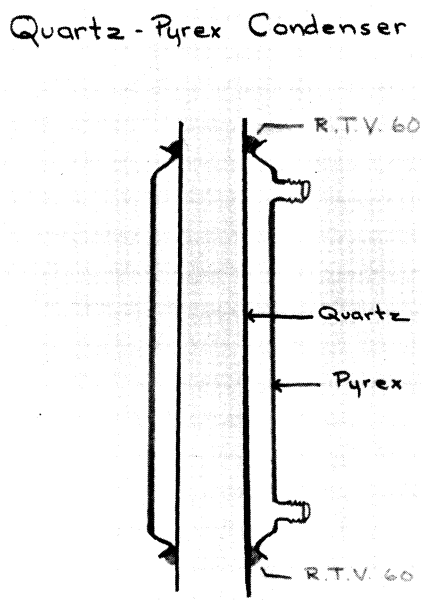

Figure 11

densers of large sizes where the criteria may be quartz inside but the outside could be made of Pyrex. Here the area where the ring seal is normally made is joined together with R.T.V. 60. (Fig. 12) sealing rectangular windows to metal systems has been accomplished in this manner. (Fig. 13) shows a piece of stainless steel machined to a rectangular di-

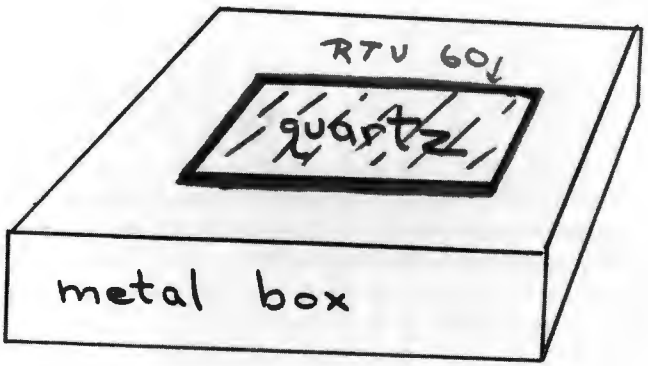

Figure 12

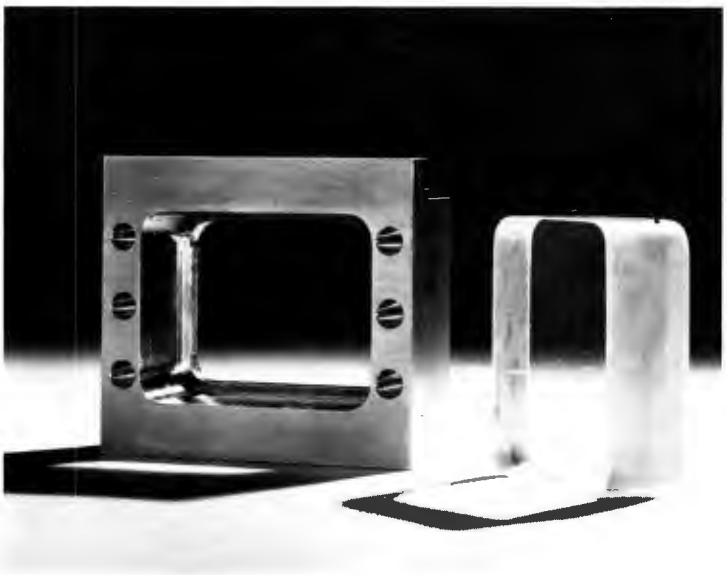

Figure 13

mension. A Pyrex window ¾" thick is to be sealed in using R.T.V. 60. At this point the parts have been primed. Next the outer perimeter of the glass was painted with the R.T.V. and allowed to dry. R.T.V. 60 is only one of a family of silicone rubber adhesive sealants available from General Electric Company, Silicone Products Department, Waterford, New York. These room temperature vulcanizing products have many uses in the laboratory. We have used them to repair leaks or breaks in metal and glass systems.

Gaskets of unusual geometry, gaskets for bell jars, form in place gaskets, shock and vibration dampers and as a mold material for short run epoxy or expanded foam parts are but a few of the uses we have found for R.T.V. in our laboratory. R.T.V. is a product with so many uses that I cannot imagine a laboratory being without it.

AN ALL-GLASS DEVICE FOR FABRICATING GEL ELECTROPHORESIS SLABS

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Gel slab electrophoresis is an especially demanding biochemical analysis technique. Chemical constituents in a solution are separated from one another on the basis of their individual patterns of migration in a gel base that is precisely oriented in a strong electrical field. Research on the identification and structure of proteins requires the production and manipulation of substantial numbers of gel slabs according to exceedingly rigorous specifications.

It is critical that the cells which contain the fragile gel slabs be geometrically precise, transparent, structurally rugged, chemically inert, free from contaminants, and adherent to gel at all surfaces. Also, they must be resistant to strong cleaning solutions and very stable in the presence of drastic thermal and electrical gradients. Development and production of such a device poses a number of difficult technical challenges which can be met with very careful fabrication methods.

Figures 1 and 2 show a cell bank that meets the requirements. It is constructed completely of borosilicate glass with all partitions merged into an integrated all-fused assembly.

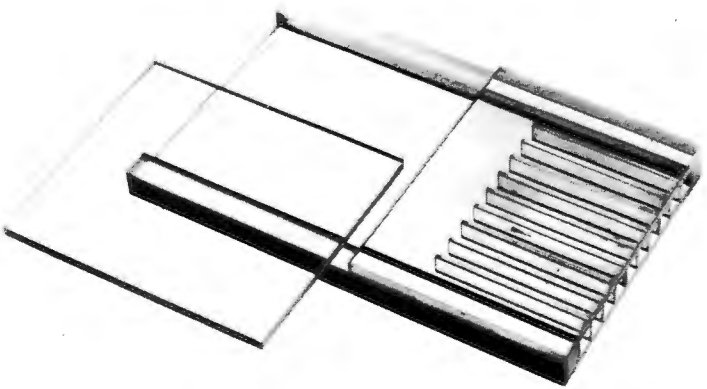

Figure 1
Gel Slab Manufacturing Device-Overall View

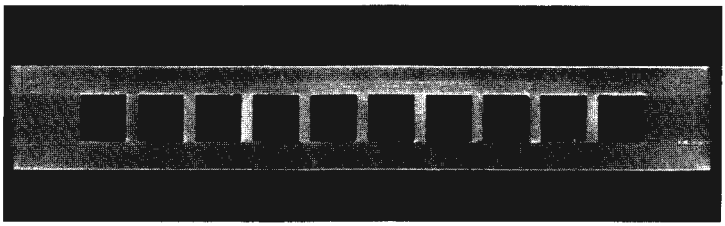

Figure 2

Gel Slab Manufacturing Device-End View of Fused Cell Bank

Cells made from borosilicate glass with fused joints are preferred over the alternatives of plastic devices, glass members with glued joints, or solder glass assemblies. Glass is preferable to plastic because of its superior chemical properties. Glued joints are undesirable because of weakness, lack of resistance to cleaning solutions and presence of a fillet of glue along each joint which violates the gel-to-glass interface integrity. Borosilicate is preferable to other types of glass because of its satisfactory adherence to the gel, its strength, thermal resistance, and chemical purity, and the ease with which it can be cleaned with strong reagents. Furthermore, a solder for borosilicate is not available; contact tape is unacceptable.

For the particular apparatus described here, it was necessary to use partitions 1 mm in thickness with individual chambers as close to 4 mm square as possible. Also, a method was needed which would be relatively inexpensive, since the small numbers required did not warrant the development of an expensive molded or pressed glass construction. Hand sealing might have been possible, but would have been unreliable and expensive because of the difficulties in working with 1 mm partitions and avoiding excessive distortion of the chambers.

The approach which proved successful was to seal the entire unit at once in a high temperature oven, with all components positioned precisely by a graphite jig. This process achieves the desired construction in one simple, inexpensive operation.

The proper type of graphite used is of the utmost importance. The material must have a coefficient of thermal expansion slightly greater than that of borosilicate glass to allow easy separation of the graphite from the cell assembly after cooling. A suitable graphite is 9326 fine grain, furnished by the Airco-Speer, Carbon Products Division, St. Mary's, Pennsylvania 15857.

Component parts are prepared using optical procedures to maintain close tolerances and thus assure precise fitting. Polishing of all the surfaces to be sealed allows for homogeneous fusion with a minimum amount of heat; ground surfaces tend to result in weaker seals.

The method for achieving the polished surfaces requires only a routine knowledge of optical techniques. A pitch polisher can be inexpensively made with materials commercially available from Universal Shellac, New York, New York.

After all parts are mounted on a suitable flat and a surface using 95 aluminum oxide is obtained, the pieces are then ready for polishing. The polishing compound is a mixture of cerium oxide and barnesite, which when used with the pitch polisher, will develop a superior finish of optical quality. Special care must be taken to clean all parts prior to the fusing process.

A block of graphite 6 mm thick covers the cell assembly during sealing to maintain a slight pressure on the sealing surfaces. Chamber orientations are maintained by the positioning of graphite spacers between the partitions (Fig. 3). Dimensions of the spacers allow for their expansion during sealing to assure contact of the parts.

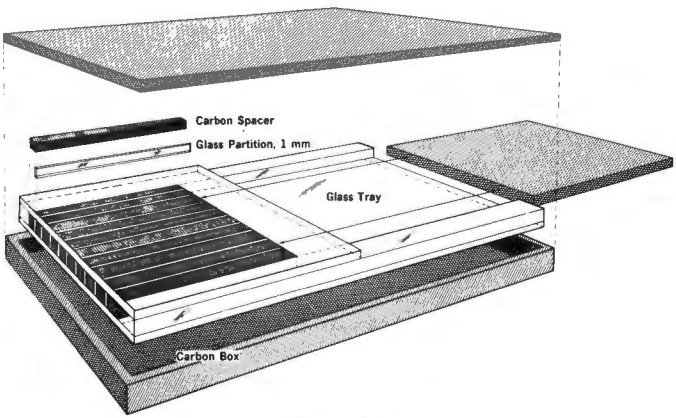

Figure 3
Cell Assembly

The entire cell assembly is placed in an oven with high temperature capabilities. Fusion is accomplished by raising the temperature of the oven to 800°C, holding the cell at this temperature for 15 minutes, then allowing the oven and cell to cool at a rate requiring about four hours to reach room temperature.

The success of this method depends upon scrupulous attention to several details. Excessive temperature or time of fusion results in devitrification, or frosting, of the glass and excessive distortion of the shape. It is essential that all joints be precisely matched to fit perfectly before sealing so they will fuse cleanly with minimum heat. All polished surfaces must be free of dirt or foreign material.

The finished cell, free of distortion with all joints clearly fused, is now in regular use at the National Institutes of Health.

The techniques described here are applicable to a wide variety of

special purpose scientific glass apparatus where technical demands are especially rigorous.

The special oven (Fig. 4) was designed and built by our Instrument Fabrication Section for optical sealing of borosilicate glass cells. The basic unit is a Dynakiln ceramic oven, Model H1300.

Figure 4
Special Oven for Optical Sealing of Borosilicate Glass Cells

Three one-inch-thick layers of marinite with a six-inch-sqare Vycor window in the center replace the original cover (Fig. 5). Positioned above the window is a Sherr Tumico monochromatic light for viewing the interference fringes during sealing. A port in the center layer of the marinite is removable for inserting the torch for fusing.

A stainless steel turntable is mounted through the bottom of the oven. It is driven by a 1/50 H.P. Bodine electric motor having 7.2 RPM and a 240:1 ratio gear box. Raising or lowering the turntable from below readily enables positioning of the work to the proper height. A Minarik variable

Figure 5
Top View of Special Oven

speed control provides for proper rotation and adds the advantages of forward, reverse, and jogging motions.

For ease of operation, the control box is mounted on the left side of the oven.

Excellent quality optical sealing can be achieved with the use of this oven.

GROWTH OF SHAPED SAPPHIRE SINGLE CRYSTALS

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ABSTRACT

The principles of a new crystal growth technique, the Edge-Defined, Film-Fed Growth process, are reviewed. It is shown that the method depends upon the wetting of one solid by the liquid of another, in the absence of harmful interactions. The application of the method to the growth of shaped single crystals and, in particular, to the growth of single crystal sapphire filament, tubing, and ribbon is described.

INTRODUCTION

The Edge-Defined, Film-Fed Growth process (EFG) is a new method of single crystal growth by use of which it is possible to produce intricately shaped single crystals at low cost.[1-3] Our earliest growth experiments which led to the development of this method were aimed at the development of a process to grow single crystal sapphire filaments. These experiments were successful and resulted in the production of high strength[4-5] sapphire filaments more than 400 feet in length with diameters, normally, of about 10 mils.

One of the important tasks in this work was the development of a system of size and shape control so that the filaments would be regularly and precisely shaped. Success in meeting this requirement was followed by recognition that the method we used to control shape had far more general applicability and could in fact be used to produce much more complicated shapes. In the following, we will describe the basic way in which the process operates and then give several examples of shaped sapphire crystals which have been grown.

APPARATUS

EFG is carried out in a standard pulling frame which supports the growth furnace shown in Fig. 1. In this apparatus, the growth setup is contained in a flowing argon atmosphere within a double-wall quartz, water-cooled furnace chamber which has been described in the literature.[6] Sight ports are provided in the form of quartz tubes passing through both furnace walls, with a flat quartz window on the outer end. Heating is accomplished by means of an rf coil placed around the chamber, suscepting either to an insulated carbon susceptor or to the metal crucible itself. Growth is carried out by dipping a seed into the melt at the top of a die as described in the following section. This seed is then withdrawn, in controlled fashion, through an opening at the top of the furnace.

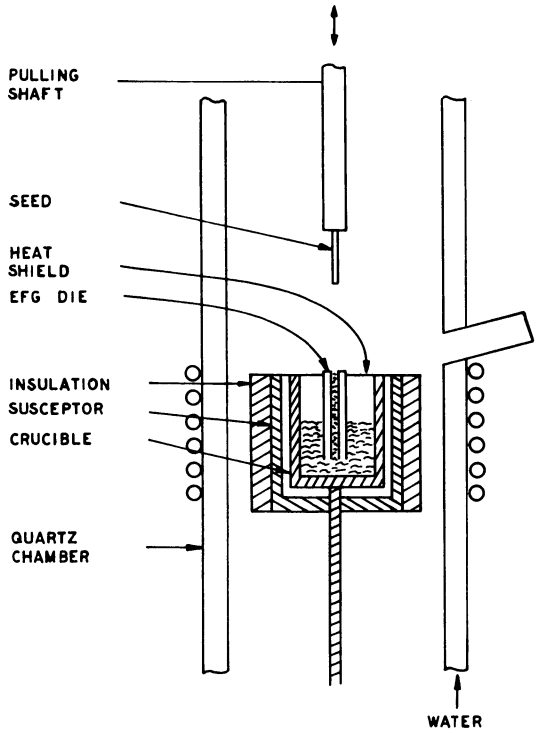

Figure 1

Typical growth furnace with pulling frame not shown

BASIC PROCESS OPERATION

The operation of the EFG process depends in two ways on the behavior of liquid films on solids which they wet. First, capillary rise is used to transport liquid from the main reservoir to the top of a die; then the surface tension is used in another way to hold the liquid film at the edges of the die while crystal growth is carried out.

Consider a solid rod with a fine, axial capillary, placed vertically in a pot of a liquid which wets the solid. Under these conditions, the liquid will rise in the capillary a distance determined by the surface tension of the liquid, its contact angle on the solid, and its density. In the case of liquid alumina, the distance that the liquid will rise, when the solid is molybdenum, is of the order of 1 in. or greater when the capillary is of the order of a few tens of mils diameter. These relationships are shown in Fig. 2.

If the solid rod rises a distance above the liquid in the pot which is less than the distance that the liquid could rise, then the situation becomes as shown in Fig. 3. In Fig. 3a, the liquid is shown having just risen to the

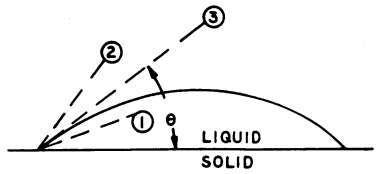

Figure 2(a)

Liquid droplet on a solid which it wets. No. 3 shows equilibrium contact angle (liquid stationary); No. 1, liquid retreats; No. 2, liquid advances

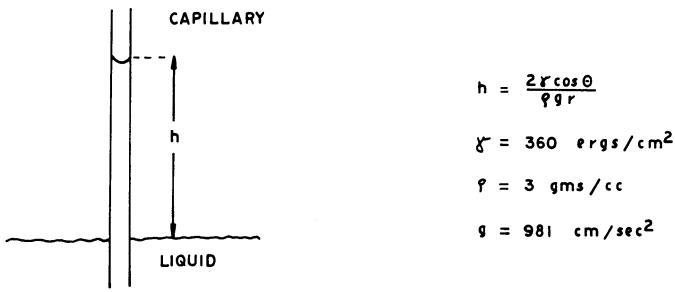

Figure 2(b)

Relationship between capillary rise, surface tension, and contact angle

top of the capillary in the rod. By itself, the liquid cannot flow over the edge of the capillary onto the top surface of the die, since the contact angle above the top surface would determine spreading (see Fig. 2a).

In Figs. 3b-e ,the necessary operations to cause the liquid to spread are shown. A solid seed of the same material as the liquid is dipped into the liquid. This seed is then withdrawn and temperature is adjusted so that the liquid begins to freeze on the end of the seed. As the seed is withdrawn, the liquid is drawn upwards, as shown, so that the equilibrium contact angle on the top surface is exceeded. Now the liquid begins to spread, and as it does so, the solid growing from it also becomes wider. The liquid is ultimately bounded by the extreme edges of the die, since, once the liquid has spread that far, it cannot flow around the corner for the same reasons that it could not flow over the top edge of the capillary.

Thus, the size and shape of the growing crystal are controlled by the external dimensions of the top of the die. But additional controls are also possible. Consider the die shown in Fig. 4, for example. Here a capillary in a solid rod is again used to conduct liquid to the top die surface. In this

case, however, the top die surface has been modified by a blind hole drilled into it. During seeding, the liquid will spread to this hole, but will stop at its edge since it cannot distinguish a blind hole from an outside edge. Thus, the crystal, which grows only above the liquid film, will grow with a hollow cavity above the blind hole, mirroring its cross-sectional shape.

GROWTH OF SHAPED SINGLE CRYSTALS

Consideration of the mechanism described in the last section shows that the basic requirements for crystal growth by the EFG method are that the liquid must wet the die material without reacting with it. In a great many cases, these conditions are readily met, and we have applied this

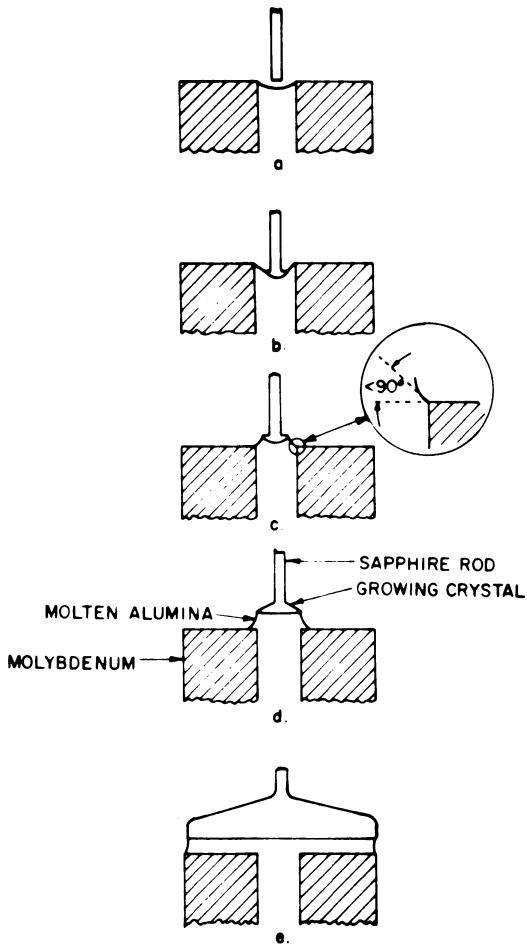

Figure 3
Sequence of events during seeding

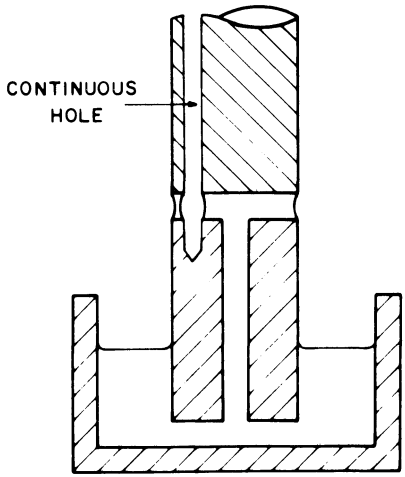

Figure 4
Method of growing a cavity in a solid rod

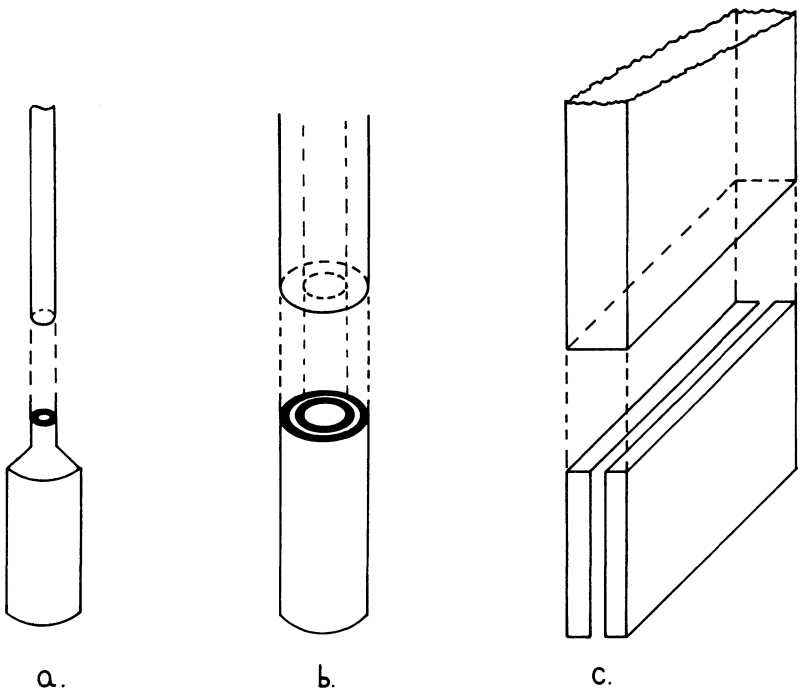

Figure 5
Die configurations, respectively, for filament, tube, and ribbon growth

method of crystal growth to a large number of crystalline metals, alloys, ceramics, ceramic alloys, and semi-conductors. The method seems particularly well adapted to the growth of shaped sapphire crystals which, in turn, seem especially ubiquitous, having a wide variety of commercial applications.

Sapphire shapes are grown in numbers up to 25 simultaneously per setup using molybdenum setup components. These crystals range in complexity from very simple ones, such as 0.010-in diameter filament, to relatively complicated ones, such as the multibore tubing or twisted, rectangular bourdon tubes.

Fig. 5 illustrates the dies which would be used to produce filament, tubing, and ribbon. The filament die is a molybdenum tube which tapers to a small diameter tip. A fine hole is drilled through the tip to reach the liquid supply. This basic type of die is used to grow filament as fine as 0.004 in. diameter, with a standard size of 0.008 to 0.010 in. diameter.

The second part of Fig. 5 illustrates the die which would be used to grow tubing. This die is comprised of two axially concentric molybdenum tubes, the annular space between forming the capillary. We have manufactured sapphire tubing larger than 1 in. diameter and as small as 0.01 in. inner diameter with 0.005 in. wall thickness. An alternative to this die configuration would be use of a solid annular section with holes drilled full length to supply liquid.

Figure 6

Illustration of several shaped sapphire crystals grown from the melt

The last section of Fig. 5 illustrates a die from which sapphire ribbon can be grown. This die is simply two parallel plates which can be spaced up to 0.050 in. apart to form the capillary. At the present time, ribbon occupies the center of our production activities and is grown in quantity up to 2.1 in. wide. Very thin ribbon has been produced (0.004 in.) as well as plates having thickness of the order of 0.1 to 0.5 in. and widths of the order of 25 cm.

Fig. 6 is an illustration of several sapphire shapes which have been grown by EFG. The choice of possibilities is much greater since virtually any shape for which a die can be machined can be grown. Of those shapes illustrated, all but the filament are too stiff to be spooled and therefore have lengths up to 5 ft., the stroke of the machine. Filament is spooled as it is grown and is commonly grown in lengths greater than 100 ft.

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6. H. E. LaBelle, Jr., and J. T. A. Pollock, Rev. Sci. Inst., **42**, 160 (1971).

CONSTRUCTION TECHNIQUES USED FOR MICRO HIGH PURITY SILICA CELLS

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INTRODUCTION

Having found our chosen profession personally satisfying, it is with a great deal of pleasure that I present this paper. Glassblowing for me has been like having a hobby, where all the materials are furnished, someone is always drawing up new plans, a place is provided for the work to be performed, and on top of all this, they pay you to do it. "The secret of life is not to do what you like, but to like what you do," Au.

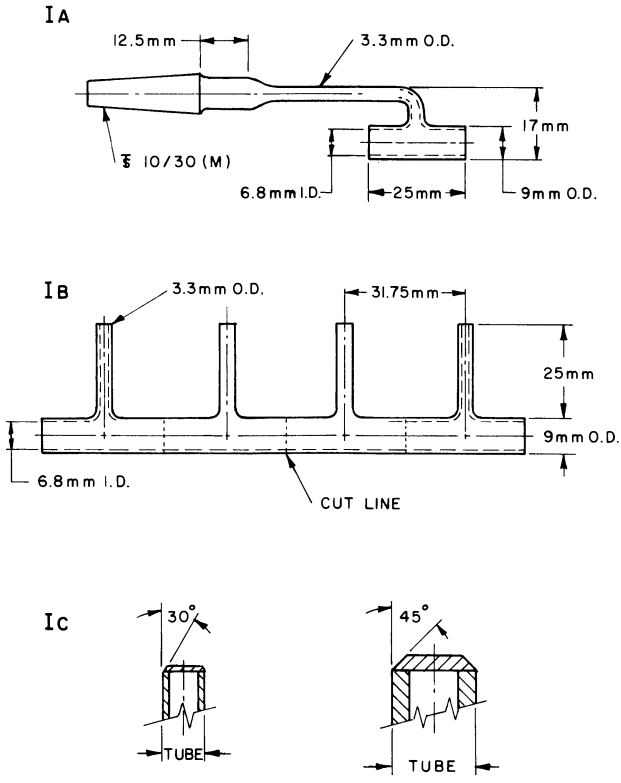
The four cells mentioned in this paper are only a small fraction of the types constructed by the Chemisty and Chemical Engineering glassblowers.

The first cell is of standard wall thickness with an I.D. of 6.8 mm and an O.D. of 9 mm, as shown in Figure 1A. The tubing is first cleaned with "Joy" and hot water and rinsed with distilled. Care is taken to leave no fingerprints on the tubing. Before sealing of the sidearms, methyl alcohol is used to remove dust from the outside surface. The end of the sidearm tube is sealed off and blown out in preparation to sealing. A Dual Differential Valve developed by Werner F. John, of Dayton, Ohio, is used for all sealing operations. This valve allows an inert atmosphere inside the cells and prevents any silica from reaching the glassblower from the cell. Sidearms are sealed to the tubing on a row (see Figure 1B), and then the tube is sent to the Optics Shop to be cut, ground, and polished with the ends of the tube parallel to each other and optically flat.

The Optics Shop also prepares the windows so that they are approximately 0.5 mm larger in diameter than the O.D. of the tubing they are to be sealed to. The top edge of the window is beveled at a 45-60° angle, so that the bottom edge will be easier to seal to the cell wall.

The angle of the top bevel is determined by the wall thickness of the body tubing (see Figure 1C). These cells are chucked in a vertical altitude for sealing. It is convenient to use a variable speed motor working through a gear reduction box so that there is a slow even turning of the cell. If the cell bodies have been properly ground and polished flat, nothing more is required but to select the proper size torch tip and flame type to make the seal. In this case a No. 2 National torch tip was used, keeping the top of the flame below the top of the window. See Figure IIA. Care is taken to just touch the junction of the cell wall and lower knife edge of the window. Sealing can be visually followed by looking through the edge of the window as it clears from the torch flame. See Figure IIB. After one end is sealed the cell is rotated 180° and the second window is sealed, followed by the sealing of the sidearm extension. After the final seal is

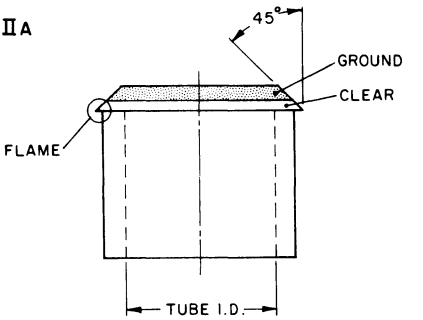
*Work performed under the auspices of the United States Atomic Energy Commission.

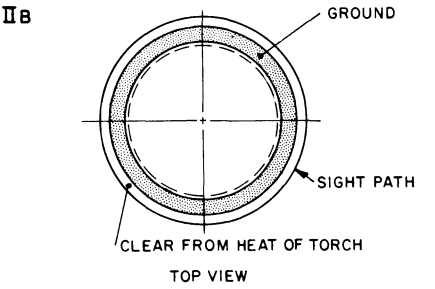


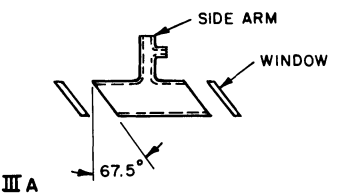
made, any remaining silica dust is flamed from the cell body using brushing strokes.

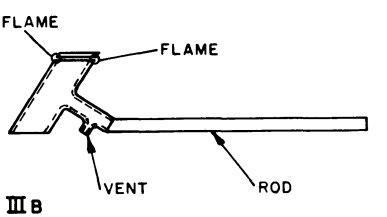
The second group of cells is shown in Figure IIIA. You will note in the breakdown that the edges of the windows are parallel to the O. D. of the tubing. The cell bodies were prepared as with the first group as pertains to cleaning, preparation of sidearms, and sealing of sidearms before being sent to Optics. Elwin C. Yoder, of Argonne's Optical Department, and his men prepared these cells by grinding and polishing the cell body ends, so that the windows would be parallel to each other, but at 67.5° to the cell body. These windows were sealed using a ring stand and clamp holding a quartz rod temporarily sealed to the longer than necessary sidearm. See Figure IIIB. A small vent hole was left in the extended sidearm tubing, so that the second window could be sealed. Split "Pyrex/quartz" glasses were used in this sealing operation, because the initial contact of flame to quartz should be kept to a minimum of white light. Again it is most important that the cell ends and windows are both optically flat, within 2-3 wavelengths. Tangent flame contact and flame height below the top of the window is maintained throughout the sealing time.

The sealing operation of the third group of cells, which has 6 mm O.D. thin wall tubing, has much in common with that used for the second

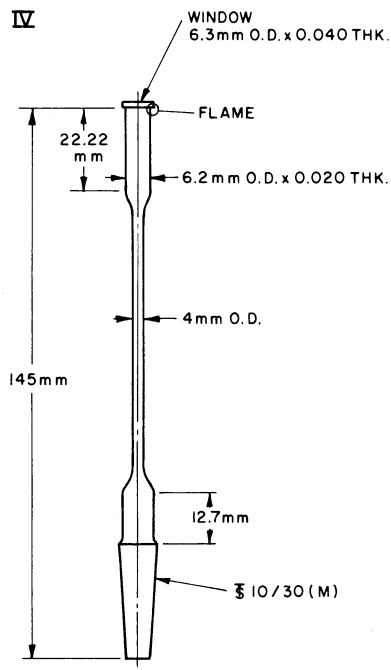

IIA
45°
GROUND
CLEAR
FLAME
TUBE I.D.


IIB
GROUND
SIGHT PATH
CLEAR FROM HEAT OF TORCH
TOP VIEW


SIDE ARM
WINDOW
67.5°
IIIA

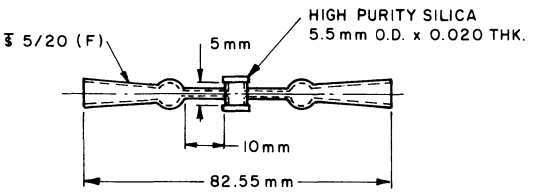

FLAME
FLAME
VENT
ROD
IIIB

group. Note in Figure IV that the windows have no bevel and are greater in thickness than the wall of the cell body. Contact of flame to junction of window and cell wall should be kept below that level of white light which would be uncomfortable using "Pyrex" or didymium glasses.

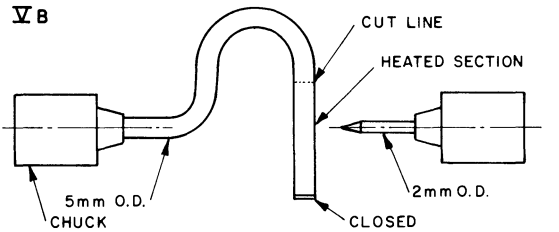


The last group of cells in this paper, shown in Figure VA, is the smallest and most demanding. Note that the cell body is of 5.5 mm O.D. thin wall and that the two 0.5 mm capillary sidearms are on the same axis. No intrusion on the I.D. of the cell body was permitted in the sealing of the sidearms. This was accomplished by chucking the cell body tubing in the headstock chuck of the lathe and then bending the tube as shown in Figure VB so that it ends up perpendicular to the lathe bed. A pointed quartz rod was chucked in the tail stock and inserted into the heated cell body wall by a turn of the wrist. The resultant thin cone of cell body quartz material was scratched using a sharp tungsten carbide knife and then broken off. See Figure VC. The capillary sidearms were prepared for sealing by closing one end and then blowing it out. After some trial and error, the proper shape was arrived at so that when the sidearm was sealed to the thin cone of the cell body (see Figure VIA) the I.D. and O.D. of the sidearm were parallel again (see Figure VIB). After the first sidearm was sealed, the cell body tubing was cut from the bent tube in the headstock and the capillary sidearm was chucked in the headstock. The second

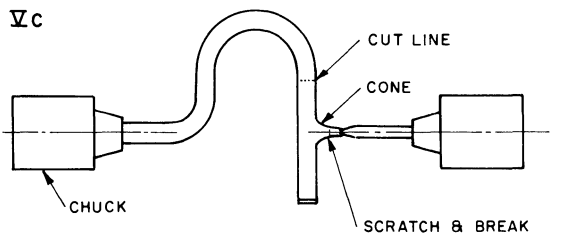
V A



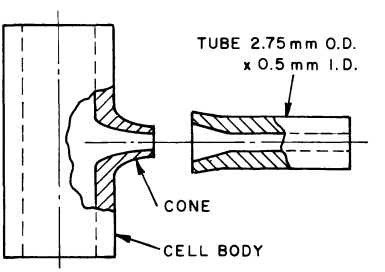
V B



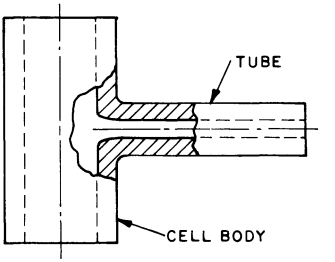
V C



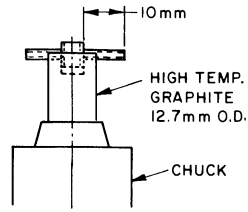
VI A



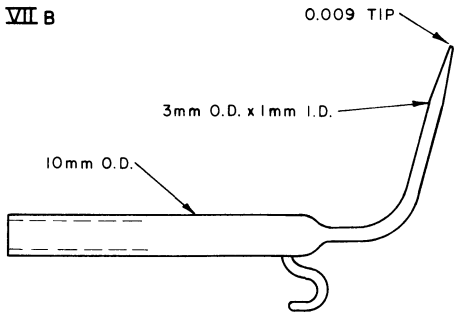
VI B



VII A



VII B



TORCH

sidearm was sealed just like the first and then both were cut off to a length of 10 mm from the cell body. The cells were then sent to Optics where they were cut, ground, and polished to a distance of 5 mm between parallel windows. The cell ends and windows were optically flat to within two wavelengths. The windows were 0.020″ thick high purity silica and unbeveled due to their thinness and small size.

A high temperature graphite holder (see Figure VIIA) was used to hold the cells during the window sealing operation. This holder was chucked on the top of a gear-reduced variable speed motor turntable so the rate of rotation could be controlled and the cell could be stopped at will, if necessary.

The torch used was made by sealing 3 mm O.D. x 1 mm I.D. quartz tubing to a 10 mm O. D. handle (see Figure VIIB). The tip of the torch was drawn down to an I.D. of 0.009″ giving a visible hydrogen/oxygen flame length of about 18 mm. This torch was then connected via rubber tubing to a National hand torch where the mixture adjustments were made. There are many micro hand torches available commercially that may also be used.

The final seals were the joining of the 5/20T outer joints to each end of the sidearm tubing and the removing of any silica dust with brushing strokes of the torch.

OUR MERCURY DIFFUSION VACUUM PUMP AND ITS BACKING PRESSURE REQUIREMENTS FROM THE MECHANICAL PUMP

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ABSTRACT

Tests were conducted on three mercury diffusion vacuum pumps of the design that is used in the Chemistry department, University of California Berkeley. The tests were to determine the effect of varying the jet clearance of the diffusion pump, the fore-pump pressure, and the heat supplied to the mercury reservoir. The jet clearance's were one, two, and three millimeters. Best performance was achieved with the two diffusion pumps having larger jet clearance. They maintained a steady vacuum of at least 3×10^{-6} torr even with a fore-pump pressure as high as 500 microns. Varying the heat input to the mercury reservoir caused the internal pressure of the mercury pumps to change. An internal pressure of twenty to twenty-five millimeters of mercury gave very satisfactory results.

This paper will describe the experimental data collected from our mercury diffusion vacuum pump. The purpose of the experiments was to find how high the fore-pump pressure could be as a function of the jet clearance before the mercury diffusion pump would become inoperable.

Mercury diffusion pumps usually operate in conjunction with a mechanical fore-pump. The mechanical or roughing pump should provide a suitable vacuum for the steady operation of the diffusion pump. Performance characteristics of mercury vapor pumps are influenced by the temperature of the cooling water, the power input to the heater, and the jet annulus, or clearance. Our standard laboratory diffusion pump has two jets, is water cooled, and requires about 160 watts of power to the heater (Figure 1). In order to find our diffusion pumps limiting operating conditions three pumps with different jet clearances were constructed. The three diffusion pumps were tested one at a time on the same vacuum rack, using the same liquid nitrogen cold trap, vacuum measuring instruments and mechanical fore-pump. One stopcock and a thermocouple vacuum gauge (Norton NR 801) were located at the fore-pump side of the diffusion pump. A Phillips ion gauge was located on the high vacuum line (Figure 2). The stopcock was used to admit air into the system through a very fine capillary that was one foot long and had an inside diameter of 20 microns. The capillary was drawn on the end of a 8 mm tube. The 8 mm end of the tube was attached to the stopcock by a one inch length of suitable rubber tubing. The stopcock was opened and very small pieces of the capillary were broken off of the end of the fine capillary to slowly admit in increments increasing amounts of leakage. A few minutes were allowed between increments so that the mercury pump and mechanical pump

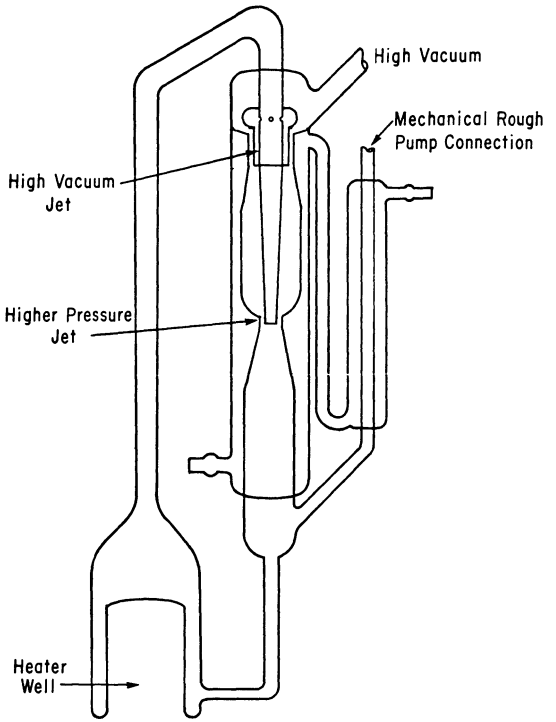


Figure 1. Our Standard Mercury Diffusion Pump.

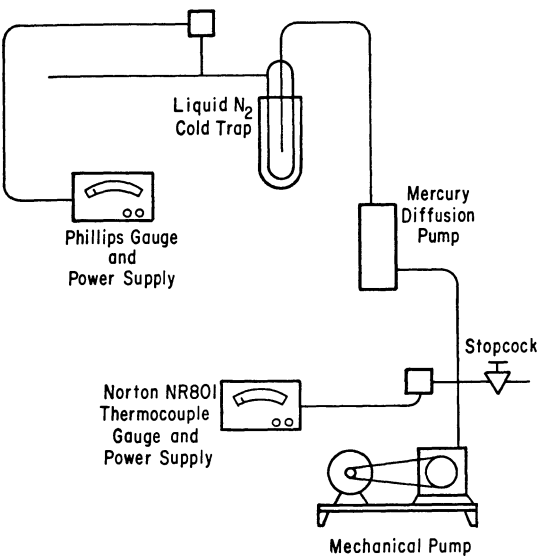


Figure 2

could recover. Observations were made and recorded from both vacuum gauges.

The amount of heat applied was also varied. Raising or lowering the electrical 160 watt heater (Figure 3) or using a powerstat to change the amount of electrical power to the heater was used to vary the heat applied

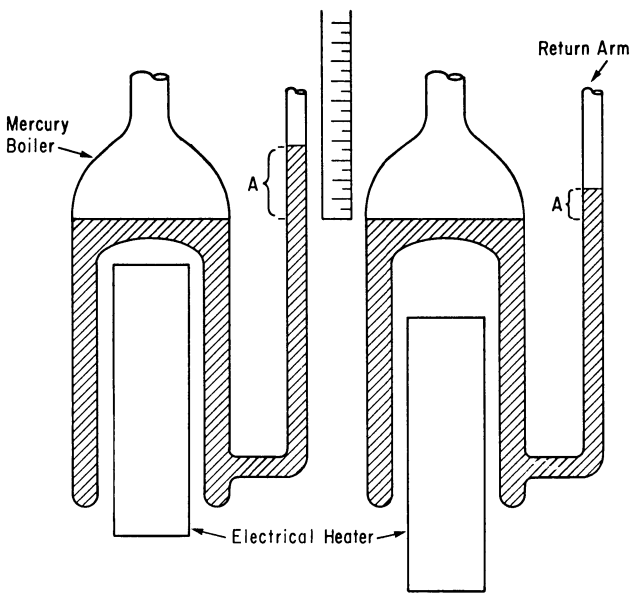


Figure 3

to the mercury, which would in turn effect the internal pressure caused by the hot mercury vapor squirting through the two jets of the diffusion pump (Figure 4). This change was easily seen by the amount the mercury would rise in the return arm, as measured from a cold start to hot operating condition.

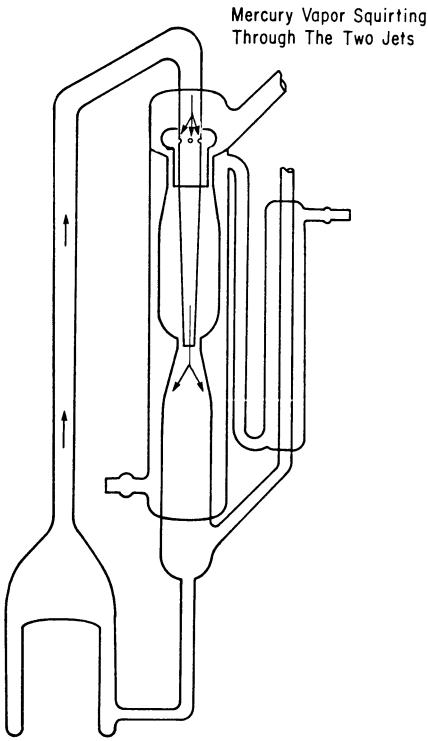
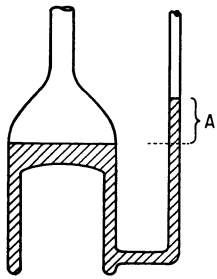


Figure 4

The first diffusion pump tested had a one millimeter annulus clearance jet system. The heater was employed at three different heights, with increasing air leakage to the mechanical pump (Figure 5). This diffusion pump stopped pumping after one day of testing because the condensed mercury formed a ring or pool of mercury around the one millimeter annulus of the lower jet causing the upper jet to be inoperative. A one millimeter jet diffusion pump is therefore not recommended in our design. The second diffusion pump tested had a two millimeter annulus clearance jet system (our standard). The mercury heating was also at three different heights and with an increasing air leak to the mechanical pump (Figure 6). With an internal pressure of 27 millimeter and over 600 micron pressure at the fore-pump side this pump maintained a steady vacuum of 6×10^{-7} torr. This pump design has been in use for many years. The third diffusion pump tested had three millimeter clearance jet system. The mercury heating and the air leak was conducted in the same manner as before (Figure 7).

MERCURY DIFFUSION PUMP WITH 1 MM. ANNULUS JET SYSTEM



A = Internal back pressure of the diffusion pump. Height in mm.

B = Mechanical pump roughing pressure.

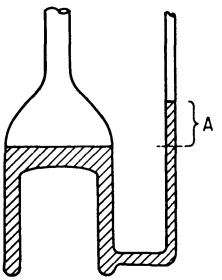
C = High vacuum, measured with Phillips ion gauge.

A	B	C†
22 mm	20 microns	3×10^{-6} torr
22 mm	160 microns	3×10^{-6} torr
22 mm	825 microns	Not Stable
15 mm	20 microns	3×10^{-6} torr
15 mm	260 microns	3×10^{-6} torr
15 mm	550 microns	Not Stable
7 mm	20 microns	3×10^{-6} torr
7 mm	150 microns	3×10^{-6} torr
7 mm	320 microns	Not Stable

†"Not stable" means that the pressure began to rise and change erratically.

Fig. 5.

OUR STANDARD MERCURY DIFFUSION PUMP WITH 2 MM. ANNULUS JET SYSTEM



A = Internal back pressure of the diffusion pump height in mm.

B = Mechanical pump roughing pressure

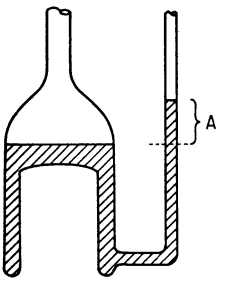
C = High vacuum, measured with Phillips ion gauge

A	B	C†
27 mm	25 microns	6×10^{-7} torr
27 mm	120 microns	6×10^{-7} torr
27 mm	620 microns	6×10^{-7} torr
27 mm	750 microns	Not Stable
13 mm	26 microns	6×10^{-7} torr
13 mm	120 microns	6×10^{-7} torr
13 mm	210 microns	Not Stable
4 mm	28 microns	7×10^{-7} torr
4 mm	34 microns	Not Stable

†"Not stable" means that the pressure began to rise and change erratically.

Fig. 6.

MERCURY DIFFUSION PUMP WITH 3 MM. ANNULUS JET SYSTEM



A = Internal back pressure of the diffusion pump height in mm.

B = Mechanical pump roughing pressure. measured by thermocouple vacuum gauge Norton NR 801

C = High vacuum, measured with Phillips ion gauge

A	B	C†
26 mm	16 microns	8×10^{-7} torr
26 mm	325 microns	8×10^{-7} torr
26 mm	505 microns	8×10^{-7} torr
26 mm	805 microns	Not Stable
20 mm	17 microns	8×10^{-7} torr
20 mm	320 microns	8×10^{-7} torr
20 mm	550 microns	Not Stable
15 mm	15 microns	8×10^{-7} torr
15 mm	30 microns	Not Stable

†"Not stable" means that the pressure began to rise and change erratically.

Fig. 7.

The first pump with one millimeter clearance worked well until its close fitting jet became choked with a mercury ring. The second pump (our standard) tolerates a wide range of roughing pump pressure when the internal pressure is at least 15 millimeters or more up to 26 millimeters. Additional pressure will cause the mercury to scatter and to be collected much faster in the liquid nitrogen trap. The third pump with three millimeters clearance performed well, which shows that in the selection of glass tubing for construction, slightly larger clearances can be tolerated rather than smaller.

SAFETY IN THE GLASS SHOP

Although it was intended that the Safety in the Glass Shop session should be a panel discussion, it turned out to be a group discussion with over thirty delegates participating.

The following report is a summary of the topics discussed, compiled by R. H. Searle, charter member and former chairman of the ASGS Safety and Hazards committee.

To set the stage, a brief report on the supervisor's role[1] for implementing the details of the Occupational Safety and Health Act (OSHA)[2] was given by the moderator. The success of this role depends, in large measure, on management's awareness of a good safety attitude. But, a good safety attitude doesn't just happen. It is the result of a thorough knowledge of the hazards involved with the assigned job or operations and the control or elimination of these hazards.

The ASGS Safety and Hazards manual has already gone a long way toward recognizing the hazards and unsafe conditions associated with day-to-day glassblowing work. Although OSHA inspectors are concerned with the compliance of the Federal Regulations for the protection of workers and the public, cooperation and safety awareness can go a long way toward making their task a simple one.

Many items were touched upon in this discussion, but primary interest centered on oxides of nitrogen, asbestos, metal fumes, safety equipment, and burner hoses.

In many small glassblowing shops (one or two persons tucked away in neglected basement rooms), the build-up of oxides of nitrogen pose a serious respiratory problem.[3] Ventilating systems consisting of a minimum of one complete air change every three minutes should be standard. Because of the toxic gases released when glassblowing operations are in progress, exhaust air should be directed to the atmosphere.

Handling loose fluffy asbestos in the glassblowing shop can be extremely hazardous.[4] Any glassblower who anneals his glassware by plunging it into a can of fluffy asbestos risks maximum exposure to the microscopic asbestos fibers.[5] When it is necessary to fabricate products containing asbestos, a suitable respirator must be worn or substitute materials[6] should be found. Many glassblowers use asbestos paper which contains a binder.[7] This material can be used safely provided it is unburned (binder not burned out). When the binder is burned out, then the product can produce a fibrous dust too fine to be observed but easily transpired into the lungs. Some individuals are more susceptible to asbestos dust than others. Regular physical checkups with full chest X-rays may help to detect the onset of future trouble.

It is a known fact[8] that regular smokers exposed to asbestos dust are 92 times more prone to lung cancer compared to non-smokers exposed to no asbestos. Whether or not you become a statistic depends on your safety attitude and your regard for your own long life.

Of the several asbestos substitutes, fiberglas narrow fabrics (ribbon)[9] and ceramic papers[10] have been tried with marginal success. None seem to have the sticking quality of wet asbestos paper. Perhaps the use of more clay in the binder of the ceramic papers would help to alleviate this problem. Anyone knowing of a good asbestos substitute should communicate with the editor of this publication.

Metal fumes, particularly mercury and cadmium, have been known and guarded against for a long time. Careful handling and cleanup when working with mercury[11] will certainly limit one's exposure. Mercury should be handled in a facility with seamless floors and good ventilation.[12] Care should be taken, when cleaning up mercury spills with a vacuum cleaner, that the mercury fumes are not discharged into the breathing air.

Many silver solders contain cadmium[13] and when heated tend to discharge cadmium vapor into the surrounding air. Here is an example of a toxic material that can be eliminated without destroying the effectiveness of the solder. Various suppliers[14] now make cadmium free silver solders and these should be selected when the job calls for silver soldered joints.

A convenient form of flip type[15] colored glassblower filter was demonstrated. For those who must wear prescription spectacles the flip type filters are quite convenient. These filters can be supplied in didymium or #6 welder's green.

In the matter of hose specifications for hand torches and bench burners, little information is available. OSHA Rules and Regulations mention hoses used on oxyacetylene torches and in welding work.[16] As a general glass shop rule, hoses should be inspected at least twice a year, at which time they can be tested to at least twice their working pressure. Care should be taken to use either an inert gas or water in making these tests. Shop working pressures vary, but common pressures from the reducing valves are as follows: burner gas ¾ to 1 psig, oxygen 8 to 20 psig, hydrogen 5 to 10 psig, and air 5 to 20 psig.

Literature on sources of supply for safety equipment was given.[17] If your local library does not have these sources, then contact with the larger university or city libraries might prove useful.

In summary, it appears that further study and updating of safety concepts must continue and that the annual symposia are a good place for the exchange of glass shop safety information and ideas. In addition, the most successful safety programs are those which emphasize employee participation in their formulation and administration. It makes good sense to work safely!

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IN ATTENDANCE

The following are on record as having attended the Eighteenth Symposium on the Art of Glassblowing held at the Denver Hilton Hotel, Denver, Colorado, June 12-15, 1973. As a fully paid registered participant, these persons are entitled to a copy of the Proceedings.

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