Proceedings

THE TWENTY-THIRD SYMPOSIUM

ON THE

ART OF GLASSBLOWING

1978

THE

AMERICAN SCIENTIFIC GLASSBLOWERS SOCIETY

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MOULDING, SHAPING AND WORKING OF GLASS WITH GRAPHITE

by

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For many years in the glass industry graphite has been a common material for making tools for the pressing, shaping, working and moulding of glass. It has drawbacks due to its very rapid oxidation and because it is a 'dirty' material. At the end of this talk I will show how we have partially overcome these disadvantages by the use of a protective coating.

For many years now we have used graphite to make moulds for small development runs of valve bases, for producing calibrated and shaped tubing and also for the tooling of glass on the lathes and on the benches. We use graphite because, for small numbers, graphite moulds and mandrels are easier and cheaper to produce than metal moulds. To mould tube bases we take two graphite half-moulds in which we pour a measured quantity of glass granules. We then place the mould into an electric or gas furnace in a metal box which has nitrogen passing through it. It is heated for a given time at a given temperature and pressure is applied from a weight on the top. On my first two slides you will see a base that we have been producing in this way for many years. Also on the slide is another tube base which would be very difficult to produce on a conventional base-making machine. It has a standard glass on the top surface and a black glass on the bottom surface. This base is for a photomultiplier tube. It must be opaque and the top surface must be polished. We could not find a black glass with the right matching expansion coefficient that would give a polished finish so we made the composite structure shown here. This technique for producing bases is slow when compared with the conventional method which uses a base-making machine and metal moulds but this is not so important when quantities are small. When the glass and the mould are heated to the same temperature problems due to sticking can arise from metal moulds but can be avoided by using graphite.

An example is shown on my next slide which shows an alpha-numeric gas discharge tube which has very deep cavities moulded into the glass. This requires a very long moulding time and quite a high temperature. A Kovar lead frame is placed at the bottom of the mould. We place on top of that a pre-form made from black glass and then the other part of the mould. A weight is then placed on top of the mould as before and the whole is placed in a furnace for 2 hours at 950°C (or 1750°F). This is a true moulding rather than just a pressing, and there are no problems due to sticking.

My next slide shows another part for which the carbon moulding technology is used. This is a separate glass spacer for a gas discharge tube and it is very difficult to produce this part in any other way. It was made initially by drilling pieces of glass but this is not a satisfactory long term solution, besides which it results in a circular hole whereas a square hole is more efficient. By carefully choosing the amount of glass and controlling the shape of the mould the holes can be moulded into the component.

My next slide shows an experimental tube body which we have made by moulding. Because the amount of material which is used is quite small, precise dimensions can be achieved which would have been difficult to obtain from pressing using a metal tool. The technique thus permits us to realise components more simply and more accurately than alternative technologies and is very appropriate for a research laboratory where the number of parts required is comparatively small.

We now turn to an altogether different application which is illustrated in the next slide. This shows a range of shaped and calibrated glass tubing made by a method which has been in use in our laboratories since the early 1950's. The method consists of passing a heated glass tube over a graphite mandrel so that the glass is stretched to take up the shape and dimensions of the mandrel.

The mandrels can be very precisely machined so that the dimensions of the tubing which is produced can be equally precise. The glass to be shaped is chosen to be slightly smaller than the furnished tube and a lead piece of slightly larger size to pass over the mandrel is attached to it. A weight is attached to the glass, and the glass and mandrel are mounted vertically in an electric furnace. At the appropriate temperature the gravitational force due to the weight slowly draws the tube over the mandrel. The lengths of tube that can be drawn this way are limited only by the height of the laboratory. For tubing of 1/2 inch to 1 inch diameter drawing speeds of about one foot per minute are used. This technique is extremely simple to use, the equipment required is minimal and the range of tubing that can be produced is quite large. The accuracy and straightness is better than we could obtain from any other commercially available source. The accuracy and uniformity of the bore is illustrated in the next slide which shows how a ball bearing will give almost a vacuum-tight fit all along the length of this precision-bore tube.

The most common use of carbon tools in glass-working is in the form of paddles for shaping softened glass on the lathe or on the bench and for reaming glass and quartz tubing. For this purpose carbon is the most useful material but because of the granular nature of the material the particles get on and in the glass and all over the hands of the operator. As well as being unpleasand it can be a serious problem in cases, for example, where electrical insulation is important.

This leads me to the last part of my talk which is concerned with the coating of graphite to protect it from oxidation and to prevent particles becoming detached from the surface. This technique was devised by Dr. Lersmacher of our Aachen laboratories, originally for coating filaments but we have applied it with very encouraging results to glass working carbon tools such as those which I have described. The coating is pyrolytic graphite and it is obtained by cracking carbon containing vapours, such as hydrocarbons, usually at temperatures above 2000°C. This results in deposits of synthesised graphite in which the crystallites are highly oriented. Such layers have high densities, are metallic in appearance, completely impervious to gases and much stronger than polycrystalline graphites.

The next slide shows some moulds used for making bases in one of our factories. Here, of course, mould life is very important because with the larger number of moulds in use, costs can become significant. These components are made on a belt furnace so that a number of moulds are in use at any one time. Uncoated moulds can be used for about 60 passes in the furnace at 900°C whereas coated moulds have been used for up to 400 passes so far and they are still in use. The slide shows one of the uncoated moulds which has been through the furnace 60 times and it is so badly worn as to be finished for production. The other mould has been coated and has been through the furnace 200 times. There is still extensive life in it. Glass blowing tools also benefit from this form of coating and there are some available for inspection at the end of the talk.

Coating graphite in this way thus extends tool life and gives a hard surface finish. This gives rise to some difficulties and there are some rules to follow if they are to be avoided. The coating closes the pores in the carbon, giving a smooth surface finish not unlike a metallic surface. Consequently sticking will occur in moulds if the whole mould is coated. We have found that all the advantages can be gained by leaving the surfaces which contact the glass uncoated and coated the remainder of the mould. The glass which is being moulded prevents oxidation and breakdown of the contact surfaces whilst the pyrolytic graphite protects the remainder of the mould. Sticking occurs when the molten glass is in static contact with the coated carbon surface. In the manufacture of shaped glass, the glass which is drawn over the mandrel is not molten but only softened and there is always some relative movement between the glass and the mandrel. These two factors ensure that sticking does not occur and mandrels for this application can be coated without affecting the process.

A similar situation arises in one of the processes which occurs in the manufacture of fibre optic plates. Briefly, a fiber-optic window consists of a large number of fine glass fibres which are stacked parallel to one another and then fused. The making of the fine fibres is a two stage process. Glass rod is drawn to about 0.7 mm diameter and a few hundred of these fibres are stacked into a bundle before being drawn yet again to their final size. These multifibre bundles are fused together to form the finished plate. After the first drawing stage the stack of fibres is put in a jig and heated so that the fibres are just stuck together. Because this is a softening process and not a melting process it is once again possible to use a pyrolytic graphite coating on the jig without the risk of sticking. There is a further major advantage of coating in this instance. Because the fibres are so fine, it is essential to avoid foreign particles and the whole operation is carried out in a clean room. It is clear from what has been said that conventional carbon mould can give rise to great problems from loose particles which the coating prevents.

Thus we find carbon a very useful material from which to make a wide variety of glass working tools, particularly moulds. Because of the porous surface of the material the glass does not stick to the mould in the same way as it does to a metal mould. We are thus able to mould parts in carbon which we would find impossible using a metal mould.

At the same time we have been able to greatly extend the life of such tools and to reduce the problem of loose material by applying a protective coating of pyrolytic graphite. By avoiding having molten glass in static contact with the coated surfaces, sticking can be avoided.

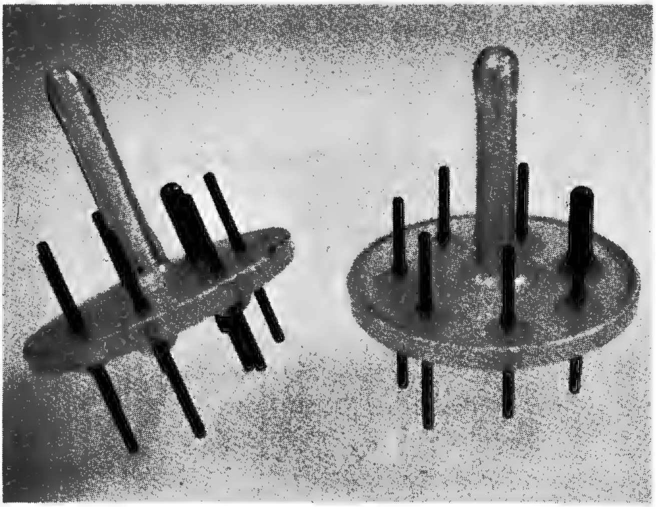
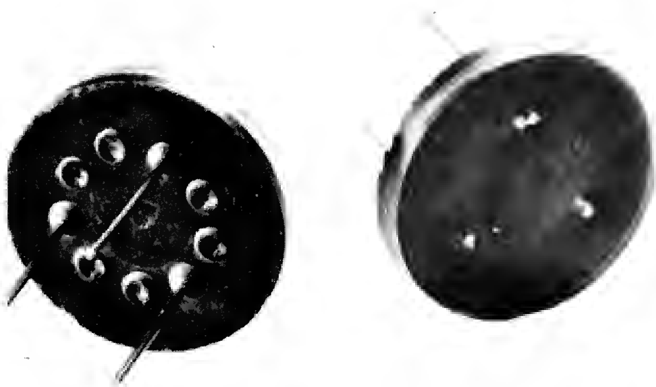


Figure 1.



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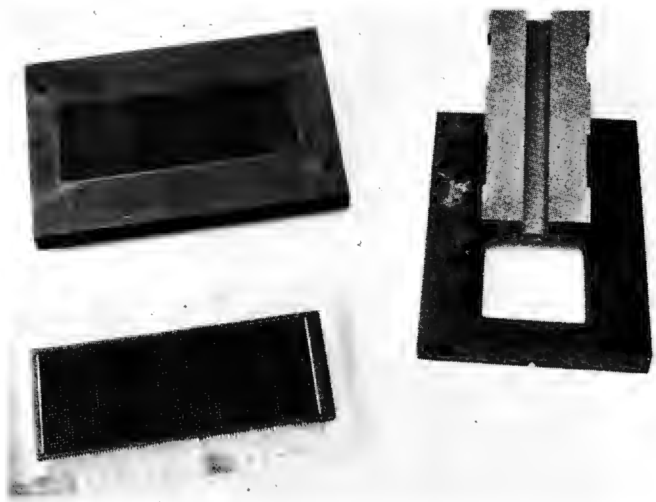
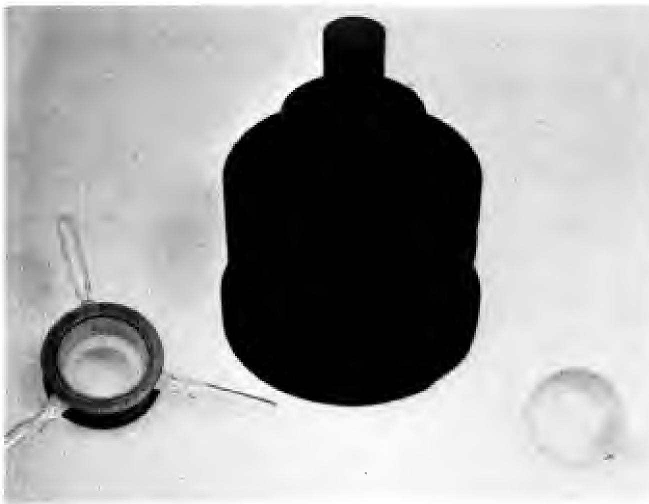


Figure 3.

Figure 4.



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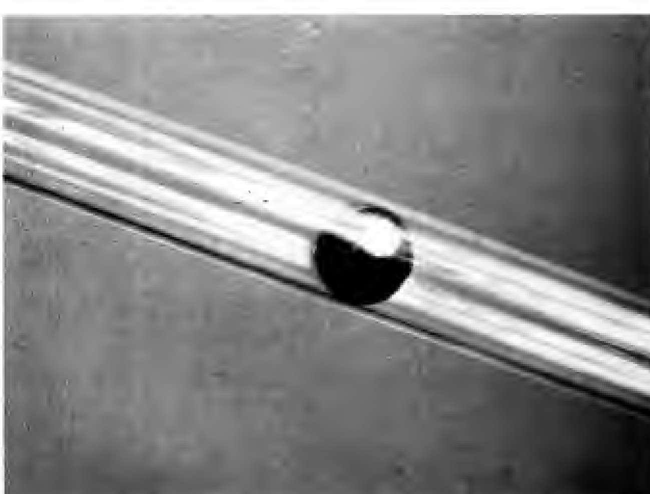


Figure 6.

Figure 7.

Figure 8.

Figure 9.

PRECISION TUBING REDRAW 0120 GLASS

by

Carl L. Nyman
Cathode Ray Department
Tektronix, Inc.
Beaverton, Oregon

Our requirements are I.D. .960" ±.002" length 8" straightness. .957" X 8" steel mandrels must pass through entire length.

The first tubing used on this project was purchased vacuum shrunk tubing. This tubing was satisfactory but expensive. Also, during further glassing operations, there was a certain amount of losses at each step. This tended to make the vacuum shrunk tubing much more expensive on a ship tube basis.

I had seen some glass redraw operations demonstrated and we designed a set up of our own.

Our first set up was to have a carbon mandrel from an eye bolt in the ceiling. The glass with a 12" starter piece of tubing attached was hung inside a wound mullite oven. The bottom end of the starter piece was weighted and the 110 volt winding variac control was turned on.

At this point, our trial and error education began.

Carbon, when heated in air to a necessary temperature to soften 0120 glass (approximately 950° Centigrade) becomes porous and begins to break down on the surface. Also the cheaper grades of carbon with large grain size deteriorate even more quickly.

The hot zone of the oven and the leading edge of the mandrel must be in the correct relation -- if it is too high, the glass begins to gather at the top and seals to the sides of the oven. If it is too low, the glass thins at the bottom and drops off.

Centering of the glass both on top and bottom is important if you want to draw straight tubing.

The winding on the oven itself is important. In our final design, we have three separate zones in a single winding. (Figure 1)

The top is spaced wider to provide a pre-heat. The "hot" section is more closely wound, the bottom anneal section is again more widely spaced. This is controlled by a single variac.

One of the most important parts of the process is to keep your carbon from oxidizing. We found a nitrogen bleed tube placed inside the bottom of the tubing to be most effective. The carbon mandrel itself has two 1/8" bleed holes that go through to keep a flow of nitrogen passing over and through the carbon during the entire redraw.

This carbon has been used for many redraws (Figure 2). The most damage occurs to the carbon, however, if it is allowed to hang in the hot zone after the glass has dropped off (Figure 3).

This was solved by designing a counter-balanced carbon holding rod. This rod also has centering washers. When the glass falls from the mandrel, it strikes a switch which turns off the oven and turns on a nitrogen cooling coil at the top of the oven. The counter-balance weight pulls the carbon out of the hot zone where it is cooled in a nitrogen atmosphere. The nitrogen blows for a pre-set amount of time and turns off. The bottom bleed tube supports the tubing until it can be removed (Figure 4).

This feature allows the operator more freedom as the redraw is automatic and does not require constant supervision.

CENTERING OF GLASS

The carbon mandrel assembly with the glass loaded is (Figure 5) hoisted with a cable into position and secured. The glass also passes through two centering irises. These are closed on the tubing when it is in place. This keeps the glass from swinging within the oven.

The next step is to fasten on the five-pound weight. The weight is held by a hose clamp on the end of the starter piece. A cushion of masking tape is placed under the hose clamp.

Now the nitrogen bleed tube is placed into the bottom holder and the trip switch is put in place under the foam cushion. The variac and bottom nitrogen bleed is turned on and the master control switch is set. On a routine redraw that is all the operator has to do until the glass has dropped off onto the cushion.

With a new set up, it is wise to mark the glass at the top and at the bottom of the oven and watch when the glass starts moving. If the top moves and the bottom does not -- your mandrel is too low. If the bottom moves and the top doesn't -- your mandrel is too high or the glass has stuck to the sides of the oven. If either of these occur, turn off the oven and allow to cool.

DRAW SPEED

We find that the glass starts moving in approximately 15 minutes and draws at the rate of one to two inches per minute. The glass tubing drawn is approximately 44 inches long with a 12 inch starter piece attached during the draw. It elongates approximately one inch. Our mandrel size is .963" and is highly polished to a crocus cloth finish.

We find that Kost Kutter 14 carbon is the best for our purposes. Another good carbon is POCO E.D.M. 3 -- both of these carbons have a small grain size.

WASHING GLASS

When the glass has the starter pieces attached, it is important to wash the glass to remove any chips or dust particles that will scratch the carbon or make striations in the tubing being redrawn.

We have designed a special wash and rinse tank with brushes and an internal spray (Figure 6).

Our redraw process has proved to be a valuable tool for experimental tube types. When a glass size is not readily available, we have been able to supply resized tubing for pilot runs.

The smallest we have redrawn has been .750" I.D. and the largest is 3" I.D. On the precision redraw, the best results are obtained when you only have to move the tubing .010" to .015". Our vendor specifications on our .960" raw tubing is .945" to .960"

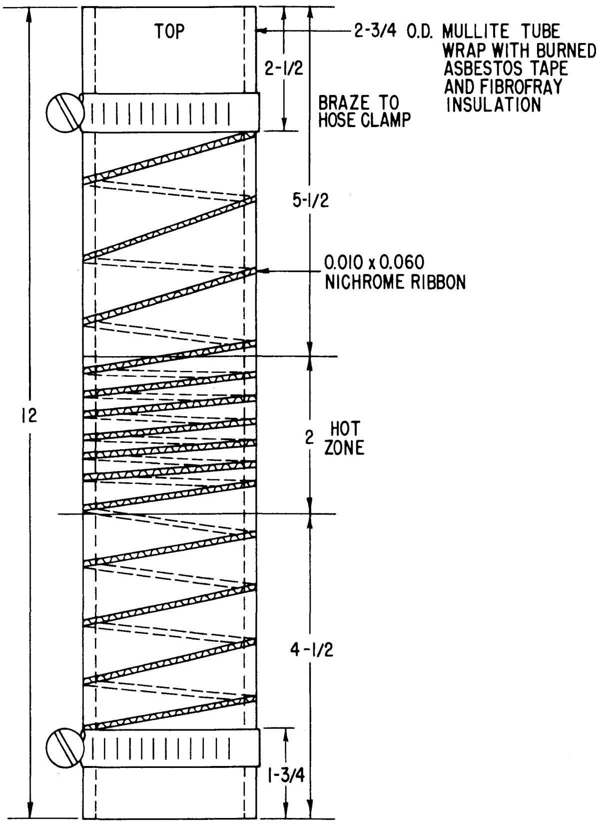

TOP
2-3/4 O.D. MULLITE TUBE
WRAP WITH BURNED
ASBESTOS TAPE
AND FIBROFRAY
INSULATION
2-1/2
BRAZE TO
HOSE CLAMP
5-1/2
0.010 x 0.060
NICHROME RIBBON
12
2
HOT
ZONE
4-1/2
1-3/4

Figure 1.

Figure 2.

Figure 3.

CARBON MANDREL ASSEMBLY

1. HOOK
2. CENTERING WASHER
3. LINE
4. COUNTER BALANCE
5. CARBON MANDREL
6. ADJUSTABLE STOP RING

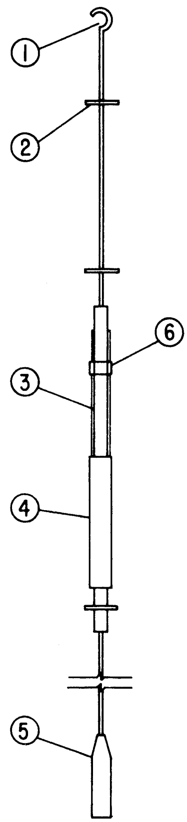
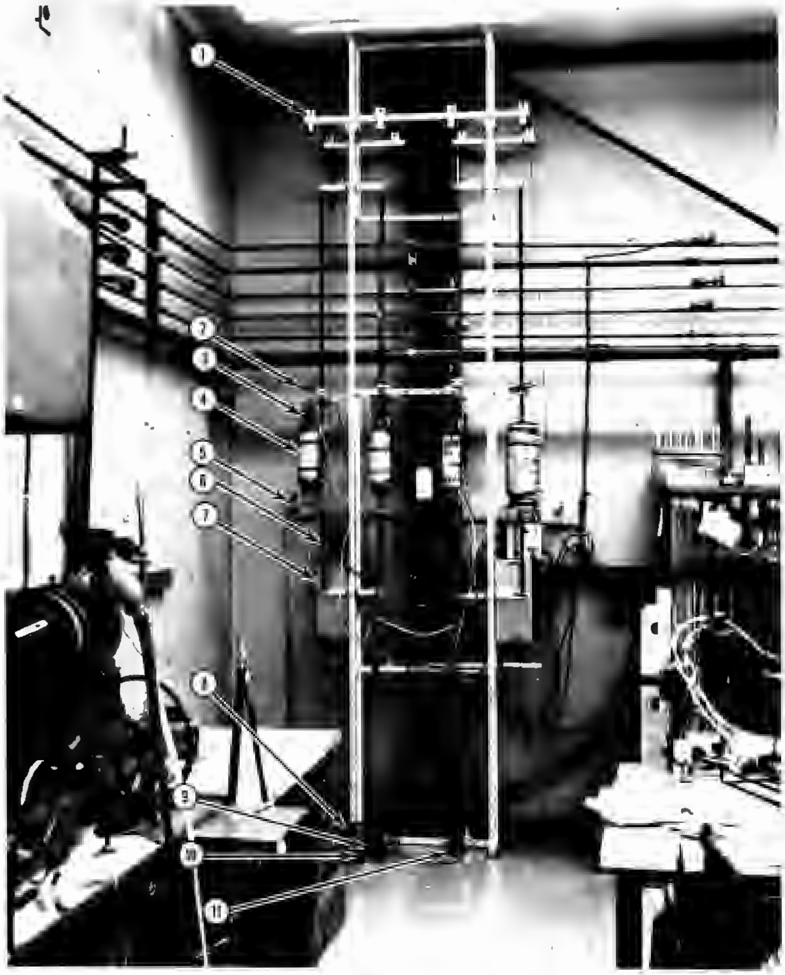


Figure 4.



1. Top pulley and taper socket
2. Centering iris, top and bottom
3. Nitrogen cooling coil
4. Mullite oven
5. Nitrogen flow meter
6. Master control box
7. Powerstat control
8. Foam cushion
9. Master control activator floor switch
10. Nitrogen bleed assembly
11. 5 lb. weight

Figure 5.

1. Water control wash tank
2. Wash tank
3. Rinse tank
4. Tergitol bottle
5. Water control rinse tank

Figure 6.

ACTIVE METAL BRAZED CERAMIC-TO-METAL SEALS

by

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INTRODUCTION

Hermetic ceramic-to-metal seals are required in an increasing variety of technical applications ranging from electron tubes, where they were first employed, to exotic energy conversion devices. One method for making such seals is active metal brazing. In this process the brazing alloy wets and bonds to the ceramic without need for premetallization. Active metal brazed seals are often used in applications where high service temperatures and/or corrosive environments preclude use of more conventional metallized ceramic seals or glass-to-metal seals.

In this paper the basic mechanisms involved in active metal brazing are briefly explained, three variations of the process described, and several practical examples of such seals given.

BASIC MECHANISMS

The key characteristic of active metal brazing is that the molten brazing filler metal or alloy wets and forms a chemically-bonded interface directly with the ceramic. This is due to the presence in the brazing filler of "active" metals such as titanium, zirconium, or the like which have a strong affinity for oxygen. When the brazing alloy is melted, active metal atoms come into close proximity with oxygen ions that comprise the outer boundary layer of the ceramic and, at a minimum, form chemical bonds with these oxygen ions. More commonly, a more extensive chemical reaction occurs at the interface between the active metal and the ceramic, forming a transition oxide compound (e.g., titanium aluminate in the Ti-Al_2O_3 case) and/or an active metal oxide at the expense of the original ceramic oxide. An attempt to illustrate the formation of interfacial bonding is shown in Figure 1.

When the ceramic/metal assembly is cooled, the brazing liquid of course solidifies but the chemical bonding is retained. In the absence of excessive stresses in the seal arising from thermal expansion (or, more precisely, contraction) mismatches between the ceramic and metal components (including the brazing material), or mechanically weak laminar phases in the solidified brazing filler, a strong, hermetic brazed joint results.

TYPES OF ACTIVE METAL BRAZED SEALS

There are at least three variations of the active metal brazing process. In the hydride method the active metal constituent is introduced in the form of a hydride powder, e.g., TiH_2, ZrH_2, suspended in an organic lacquer and painted onto the ceramic seal surface. When the assembly to be brazed is heated to a few hundred degrees centigrade the hydride decomposes. The active metal is then free to alloy with the metal member in the seal assembly or with additional brazing metals to produce a molten filler (rarely is brazing done with a pure active metal owing to their inconveniently high melting points).

In the prealloyed method, the filler is an alloy consisting of a combination of one or more active metals and, commonly, a nonactive metal. The composition of the alloy is chosen to produce a convenient melting temperature. The alloy is usually made by arc melting to form ingots which are then comminuted into powder under an inert cover gas. The powder is applied to the seal in an organic lacquer vehicle.

In the alloyed in-situ process, one or more active metals are placed in the seal as pure metals, usually in the form of washer preforms. Upon heating to the necessary temperature (typically a eutectic point), the metals alloy with each other and/or the metal member in the seal assembly to form a liquid phase.

EXAMPLES OF APPLICATIONS

Solder Seal Arc Lamp

The endseals on some fused quartz envelope arc lamps are made by the hydride method of active metal brazing. In these seals

(Figure 2), titanium hydride is applied to the fused quartz. Invar sleeves and lead-indium solder preforms are positioned appropriately and the assembly is then heated to over 500°C under inert gas. Decomposition of the TiH_2 occurs and the free titanium dissolves into the already molten solder to "activate" it and promote strong bonding to the fused quartz.

Cardiac Pacemaker Feedthroughs

In this application compatibility of the seal components with body fluids is of primary concern. The silicate glassy phase present in most thick film metallizings is not biocompatible thus precluding use of conventional metallized seals. In one successful pacemaker seal design (Figure 3) titanium hydride and gold are used to braze the stainless steel body to a Al_2O_3 ceramic insulator. The assembly is brazed under dry hydrogen or in vacuum at the melting temperature of the gold which, upon melting, is "activated" by the titanium. The gold, titanium, and stainless steel components are all compatible with body fluids. Note the "knife edge" butt seal configuration which is used to counteract effects of the thermal expansion mismatch between Al_2O_3 and stainless steel.

Alkali Metal Vapor Turbo-Alternators

Rankine cycle energy convertors were at one time being seriously developed for spacecraft use. In these closed loop systems (see Figure 4), a nuclear reactor is used to heat alkali metal vapor which in turn drives a turboalternator. A ceramic liner is deployed between the rotor and stator to contain the alkali metal vapor (Figure 5), in this particular case, potassium. The ceramic-to-metal seals at the ends of the liner, so-called "bore seals", must have long term resistance to attack by the hot potassium vapor. Here again the glassy phase in conventional metallized seals precluded their use because of its vulnerability to potassium attack.

An active metal brazed seal was successfully developed for the bore seal application. A prealloyed powder of the composition

60%Zr-25%V-15%Nb was used in the final evaluation assemblies to hermetically join a high purity BeO ceramic liner to Nb-1%Zr metal seal flanges in the "sandwich" configuration shown in Figure 6. Brazing was performed in vacuum at approximately 1300°C. In long term potassium exposure testing at 840°C no seal degradation was observed. Other slightly lower melting brazing alloys were also found to be effective including 56%Zr-28%V-16%Ti (m.p. 1250°C), 48%Zr-48%Ti-4%Be (1050°C), and 75%Zr-19%Cb-6%Be (1050°C).

Alkali Metal Vapor Arc Lamps

Various companies have employed active metal brazed seals in special purpose alkali metal vapor arc discharge lamps. One example is a potassium arc lamp (Figure 7) developed by the author's company for use as an optical pump source in a Nd:YAG space communications laser. In this application, the need for high temperature compatibility with potassium vapor is again a key factor controlling seal material selection. The end seals in this lamp are brazed with zirconium using the in-situ alloying process. The arc tube is polished synthetic sapphire (single crystal Al_2O_3) and the endcaps are Kovar. These seals exhibit unusually good oxidation resistance as well as potassium compatibility and good thermal cycling durability. Lamps have been operated in air for over 2000 hours with the seals exposed and at temperatures of over 600°C.

Battery Cell Feedthroughs

Active metal brazed seals have been employed for some time in insulated terminal feedthroughs on aerospace nickel-cadmium cells (Figure 8). In this application, the corrosive potassium hydroxide electrolyte environment within the cell causes rapid degradation of conventional metallized seals due to KOH attack of the glassy phase. Metallic constituents in such seals including the molybdenum or tungsten in the metallizing and silver or copper in the brazing filler are also susceptible to electrochemical attack that can result in insulator shorting.

Terminal seals brazed with titanium or zirconium (alloyed in situ with the nickel or Kovar metal member) have proven to be extremely reliable and are now used exclusively in aerospace Ni-Cd cells. Here again the "sandwich" configuration is employed (Figure 9), primarily to allow brazing preforms to be preplaced between the Al_2O_3 ceramic and metal member. This facilitates the in situ alloying process and avoids the need for the molten filler alloy to flow into the seal (most active metal fillers have sluggish flow characteristics).

As shown in Figure 9, the seal assembly can be mechanically buttressed for use in nickel-hydrogen cells where a hydrogen gas pressure of several hundred psi must be contained when the cells are in the fully charged state.

New high energy density batteries presently under development for potential use in electric automobiles and for "peak shaving" energy storage at power plants are likely to require active metal brazed seals. Two leading battery concepts are the lithium-metal sulfide battery and the sodium-sulfur battery. Both operate at elevated temperature (>400°C) and have extremely corrosive internal environments. In the lithium-metal sulfide system, cell components, including the feedthroughs, are exposed to a LiCl-KCl fused salt electrolyte and probably molten potassium and lithium. Corrosion reactions are enhanced during charging when voltage is applied to the cells. High purity BeO or Y_2O_3 ceramics are used in the feedthroughs. Molybdenum is presently the preferred electrical conductor material. Various active metal brazed seals are presently being evaluated but success to date has been limited. In the sodium-sulfur cell (Figure 10), a solid electrolyte (beta alumina) is used. Sodium ions diffuse through the electrolyte to form sodium polysulfide at the sulfur side during discharge. The primary seal problem in these cells is the reactivity of metal seal components with the sulfur polysulfide. "Conventional" seal metals such as niobium, nickel, and Kovar are not resistant to these materials. Similarly, common active metal brazing constituents (e.g., Ti, Zr) may be incompatible with the sulfur/polysulfide environment.

Until acceptable brazed seals are developed, mechanical compression seals can be used in the terminal feedthroughs for lithium and sodium batteries. Unfortunately, these tend to be relatively bulky and are only marginally hermetic.

CONCLUSION

Active metal brazed ceramic-to-metal seals can be effectively employed in applications where conventional seals are unsuitable. As reflected in most of examples given above, such applications are often associated with severe environmental conditions. As new and exotic ceramic/metal devices are conceived, particularly in the energy field, this joining technique will undoubtedly find further utility.

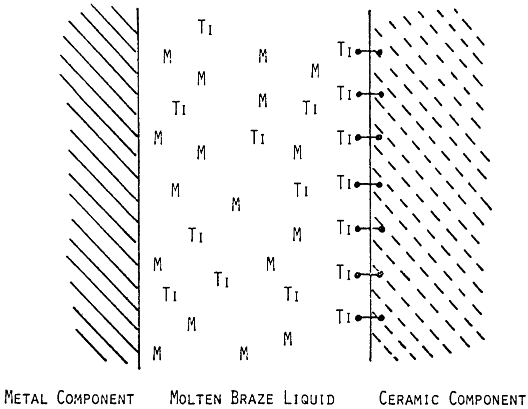

FIGURE 1. Basic Mechanisms of Active Metal Brazing

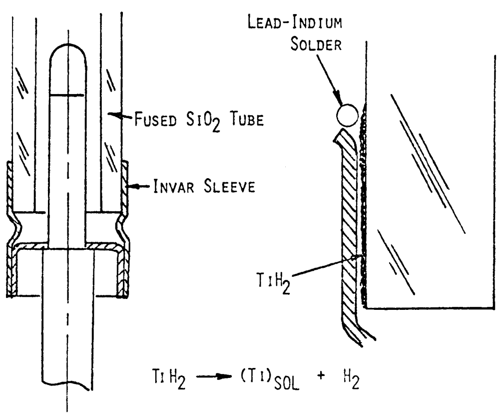

FIGURE 2. Arc Lamp with Soldered Seal made by Hydride Active Metal Process

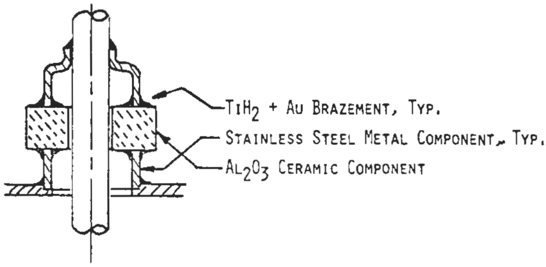

FIGURE 3. Cardiac Pacemaker Feedthrough Brazed by Hydride Active Metal Process

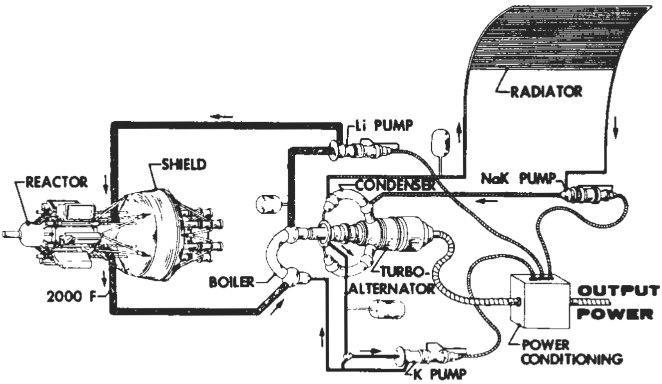

FIGURE 4. Rankine Cycle Alkali Metal Vapor Energy Convertor for Spacecraft

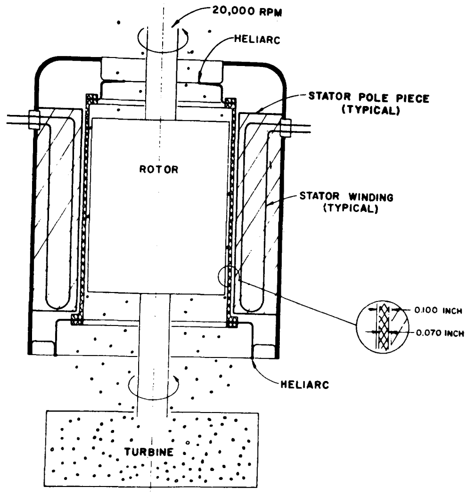

FIGURE 5. Detail of Turboalternator with Ceramic Liner (Double Cross-Hatched)

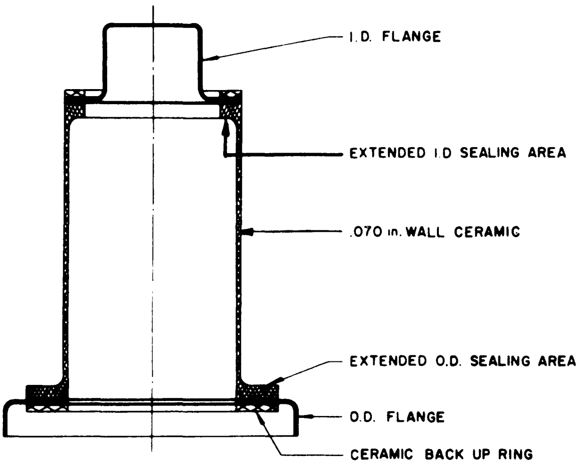

FIGURE 6. Brazed Ceramic/Metal Assembly for Turboalternator Liner

FIGURE 7. Potassium Arc Lamp

FIGURE 8. Cell Covers with Brazed Ceramic/Metal Feedthroughs for Aerospace Ni-Cd Batteries

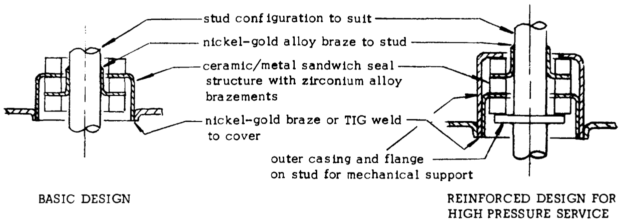

FIGURE 9. Ni-Cd Cell Feedthrough Detail

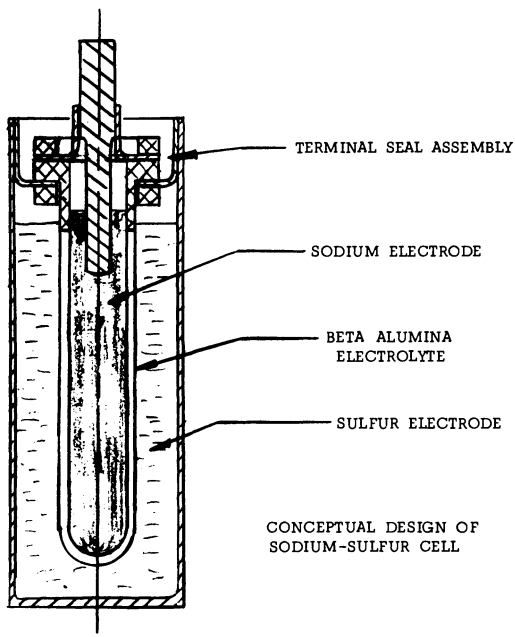

FIGURE 10. Proposed Sodium/Sulfur Cell with Brazed Ceramic/Metal Terminal Feedthrough

MACHINING OF GLASS WITH A CO_2-LASER

by

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Eindhoven, The Netherlands

SUMMARY

An 80 watt CO_2-laser is used in the Philips laboratory glass workshop. It has proved to be a helpful tool in shaping special glass products.

A short introduction to the physics of a laser system is given and some results are presented. During the meeting a film about its performance was shown and some laboratory workshop products were exhibited.

INTRODUCTION

For some years we have been using a CO_2-laser for the cutting and drilling of glass in our laboratory glass workshop. Although the laser is not a simple tool for general applications and needs both a skilled operator and special safety arrangements, it has proved extremely useful. In some cases no other tool could have done the job. In this paper we shall consider a number of practical applications. For a general understanding we start with a short introduction on the physical background of laser action.

PRINCIPLE OF A LASER SYSTEM AND LASER MECHANISM

Our CO_2-laser system consists of a sealed-off glass tube i.e. the actual laser tube 1 in Figure 1, about 150 cm long and about 2 cm wide. Two parallel mirrors (2 and 3) are mounted vacuum tight at both ends of this tube. A concentric tube (4) enables the first tube to be cooled by tap water flow. Tube 1 is filled with a suitable gas mixture, mainly CO_2- and N_2-gas and some water vapour, their total pressure being about 6 torr (5 in Fig. 1). By applying a high voltage (about 25 kV) to both annular electrodes 6 and 7 a gas discharge current up to 25 mA can be maintained.

The mechanism of the CO_2-laser action can be regarded as being a chain of molecular energy steps, i.e. excitation, transfer and de-excitation as shown in Figure 2 and these are the most probable steps in this typical gas mixture:

1. electrons in the gas discharge easily excite N_2 molecules into vibration,
2. the vibrational energy is transferred very quickly from N_2 to CO_2 molecules by mutual collisions,
3. emission of light quanta with laser wavelength $\lambda = 10.6\ \mu m$ brings the CO_2 molecules to a lower vibrational state and,
4. the vibrational energy is dissipated very quickly via collisions with H_2O molecules.

In this process the transitions from the upper to the lower CO_2-level (i.e. step 3, giving rise to the emission of light quanta with $\lambda = 10.6\ \mu m$) do not take place spontaneously but under the influence of electromagnetic radiation of the same wavelength already present in the tube and reflecting to and fro between the two parallel mirrors 2 and 3, to form a standing wave in the resonant cavity between these mirrors. Radiation generated later has - due to this interaction - the same phase and the same direction as the radiation already present. Thus a coherent beam is obtained which continues to grow in intensity (because the gas discharge continues to pump energy into the resonant cavity) until the losses have become equal to the gain. An important part of the losses is comprised by our laser beam, i.e. the radiation which is allowed to escape from the resonant cavity. This is accomplished by making one of the mirrors semi-transparent.

A laser beam hitting the surface of a material will be partially reflected, the remainder being transmitted and absorbed (Fig. 3). The absorbed energy is

$$I_0 \cdot (1-R) . e^{-kd}$$

where I_0 = the light intensity leaving the laser
R = coefficient of reflection, thus (1-R) is the energy fraction available for machining

k = absorption coefficient

d = depth the beam travels into the solid.

The absorption coefficient k depends primarily on the material and the wavelength. It is shown in Figure 4 for fused silica and germanium at room temperature. As can be seen, germanium is transparent to CO_2-laser light and it can be used therefore as a material for windows and lenses. Fused silica, on the contrary, has a relatively high coefficient of absorption, so this material can be cut and drilled with a CO_2-laser if the energy density can be made high enough (which is achieved easily).

In general oxide materials can be machined quite efficiently with CO_2 lasers as the energy loss due to reflections is relatively low, see Figure 5. Metals, on the other hand, reflect CO_2-laser light strongly and thus can be worked better with another type of laser.

To increase the laser energy density at the surface of a workpiece the beam is focused on this workpiece with a lens (Fig. 6) made, for example, of germanium. Under favourable circumstances (i.e. when the laser is operating in the so-called TEM_{00} mode) the focussed beam or "spot diameter" is given theoretically by

$$d = \frac{4\lambda f}{\pi D}$$

where λ = laser wavelength = 10.6 μm for a CO_2-laser

f = focal length of the lens

D = diameter of the laser beam entering the lens.

In our case D = 5 mm, f = 50 mm, and thus the spot diameter d = 130 μm. With a laser beam of 50 watt the average power density in the spot will be about 5.10^5 watt/cm^2. However, the real power density in the centre of the spot will be higher and the power density distribution in the cross-section of the spot is roughly Gaussian (Fig. 7). Because of this typical power distribution it is possible, for example, to cut fused silica of 2 mm thickness with a minimum width of 50 μm. Due to this power distribution and the finite velocity of drilling or cutting the cross-section of such a hole or cut is tapered. During this drilling or cutting action the material heats up and evaporates. A cross-section of a cut is shown in Figure 8. The depth-to-width ratio of the holes and cuts is increased by a natural phenomenon: the laser light is reflected internally in the hole or slit.

PRACTICAL WORKSHOP EXPERIENCE

In the Figures 9, 10 and 11 we show a number of workshop products made of fused silica, borosilicate and some synthetic workshop materials.

Some of the results are summarized in Table I.

Table I

DRILLING OF HOLES WITH CO_2-LASER

Material	Power	Maximum Depth of the Hole	Minimum Diameter of the Hole
Fused Silica	80 W	6 mm	40-50 μm
Borosilicate	80 W	2 mm	150-200 μm
Araldite	65 W	15 mm	40-50 μm
Perspex	65 W	25 mm	1000 μm

CONCLUSION

The CO_2-laser has proved to be a useful tool in a laboratory workshop.

It offers new shaping possibilities, not obtainable with conventional workshop rechniques.

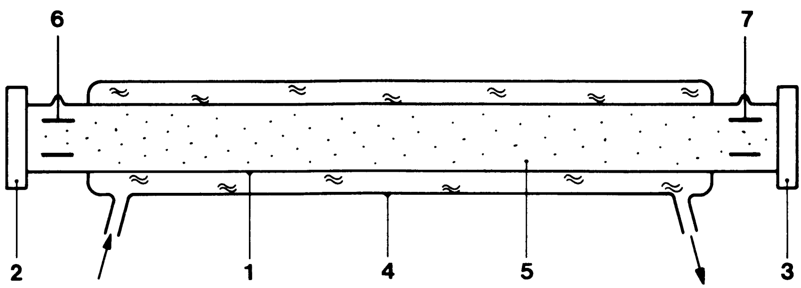


Figure 1. Diagrammatic Representation of a CO_2-Laser

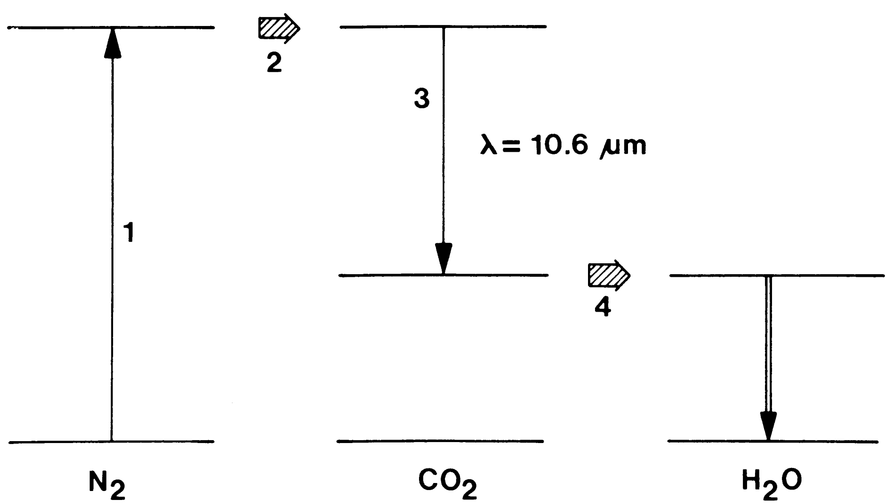


Figure 2. Diagrammatic Representation of the CO_2-Laser Operation. Heavy arrows denote processes that occur relatively fast.

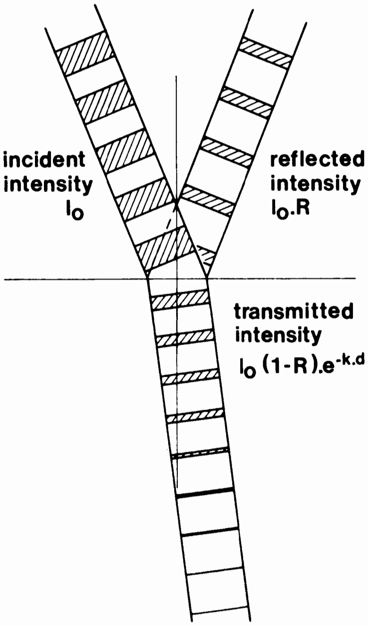


Figure 3. Reflection, Transmission and Absorption of a Light Ray

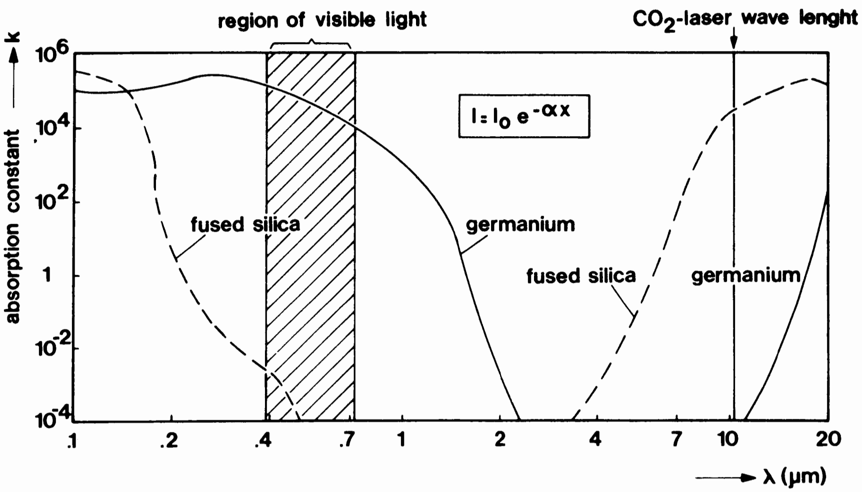


Figure 4. Absorption Constant k of Fused Silica and Germanium at Room Temperature as a Function of Wavelength

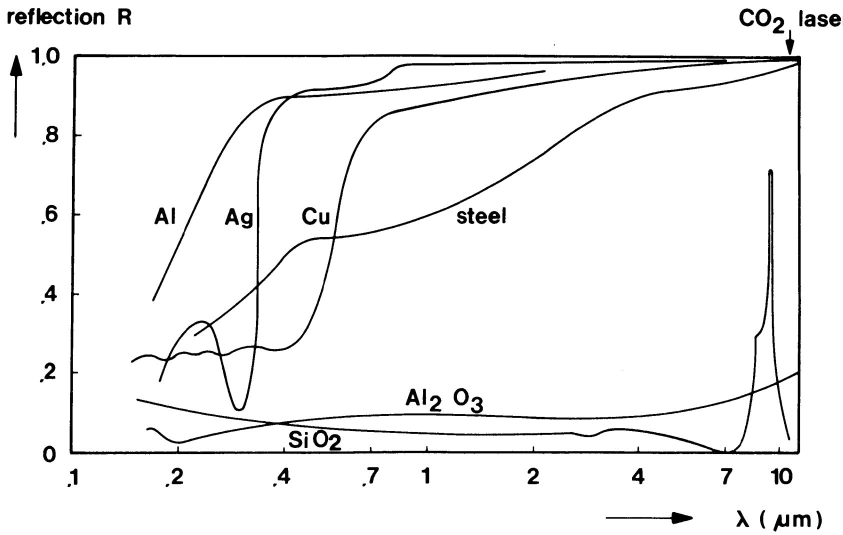
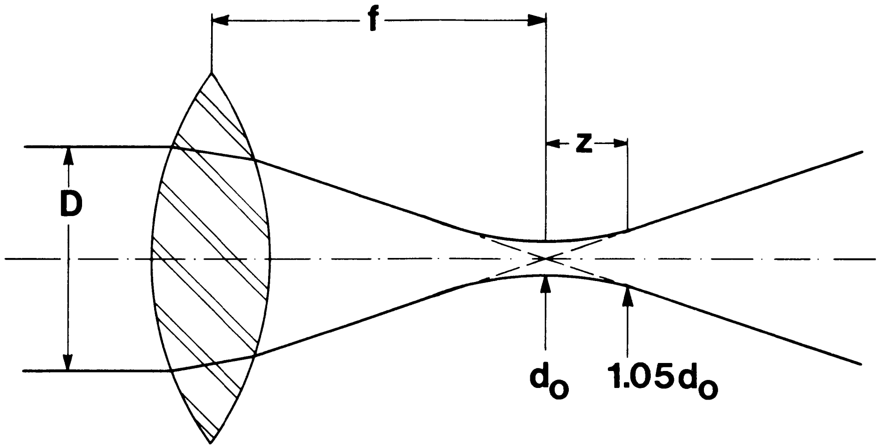


Figure 5. Coefficient of Reflection of Some Metals and Some Oxides at Room Temperature



$$d_o = \frac{4\lambda f}{\pi D}$$

Figure 6. The Focusing of a Laser Beam by a Germanium Lens. The distance 2z is defined as the focal depth.

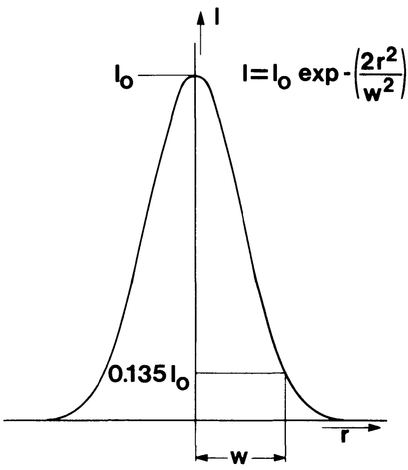


Figure 7. The Power Distribution in the Laser Beam Spot

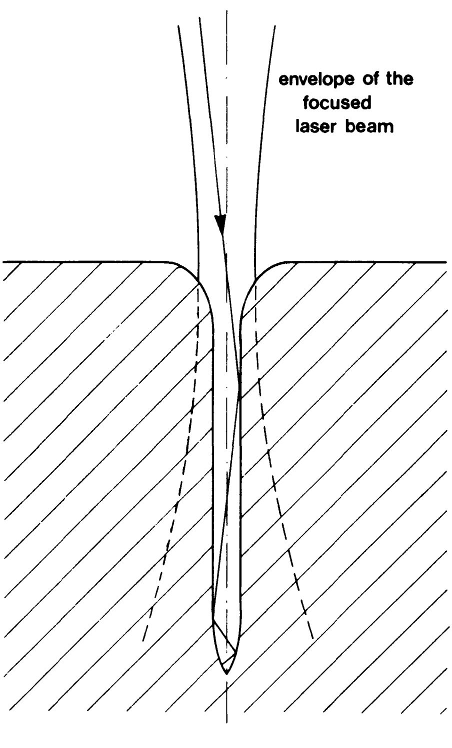


Figure 8. A Focused Laser Beam During Drilling or Cutting. Owing to successive reflections of a light ray its drilling or cutting efficiency is greatly increased.

(a)

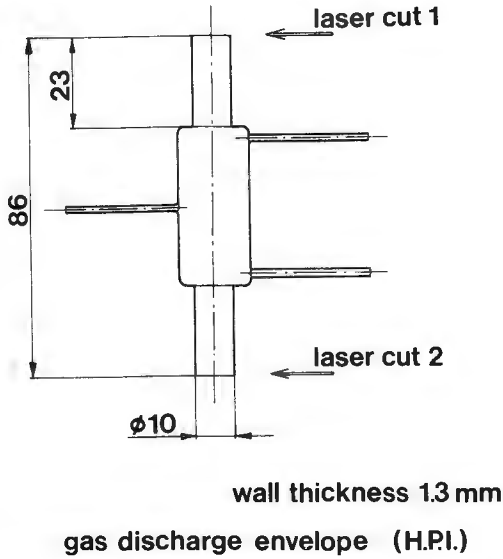


Figure 9. Clean Cutting of a Fused Silica Gas Discharge Envelope. The laser parameters were: 65 watt, spott diameter 135 µm. The glass wall was 1.3 mm thick, its outer diameter was 10 mm. It was cut in 18 seconds (2 revolutions). (Photo 9b)

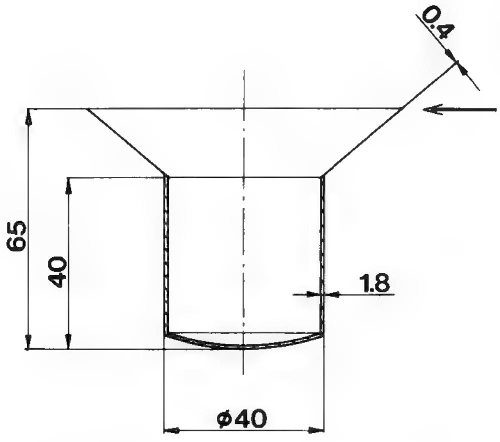
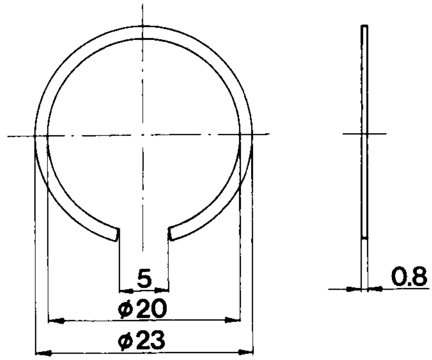
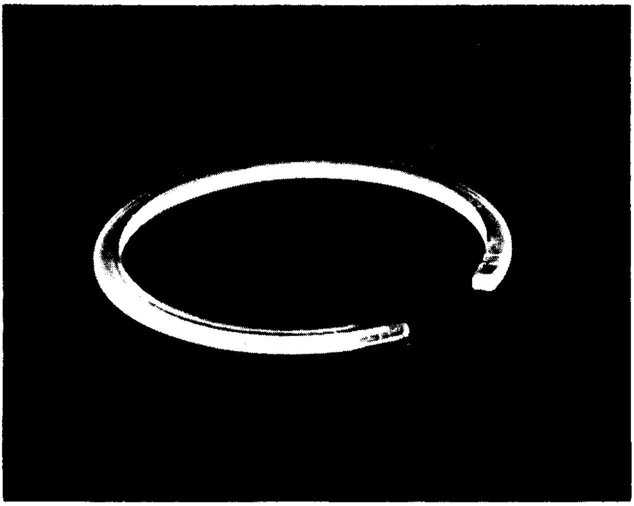


Figure 10. Cutting of Some Fragile Structures of Fused Silica.

Figure 10a. Cup with a Rim. (Photo 10ab). 65 watt, 25 sec., 0.4 mm thick wall.



(b)



(bb)

Figure 10b. Elastic Spring Ring. (Photo 10bb).
65 watt, 30 sec., 1.5 mm thick.

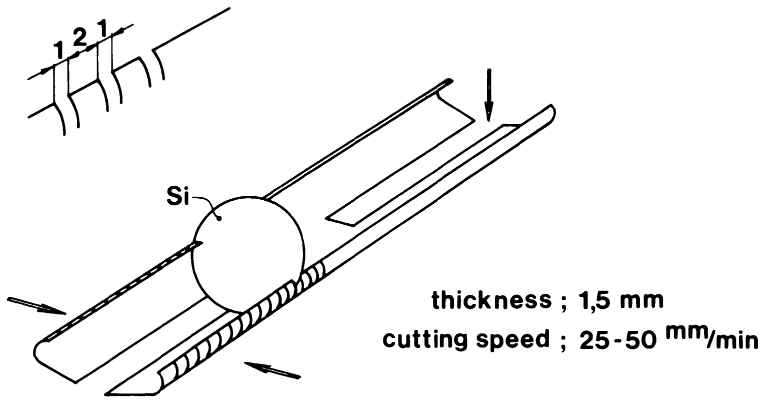


Figure 10c. Rack for Etching Silicon Slices.
65 watt, cutting speed 25-50 mm/min
1.5 mm thick.

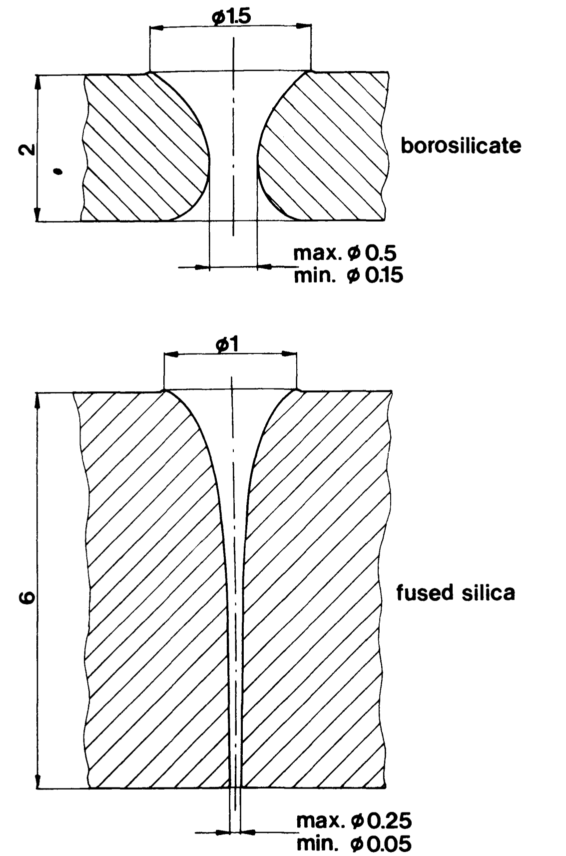
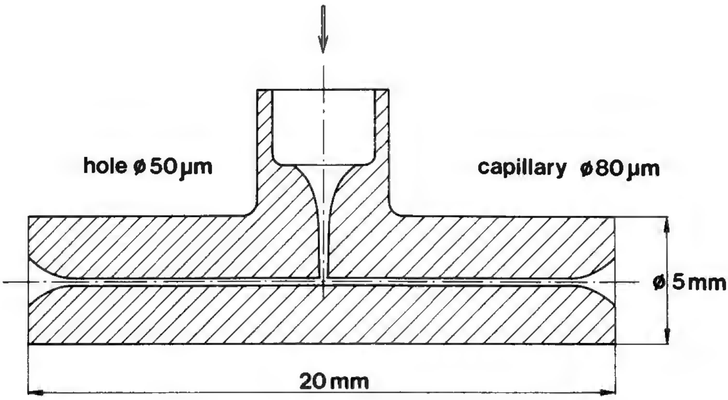
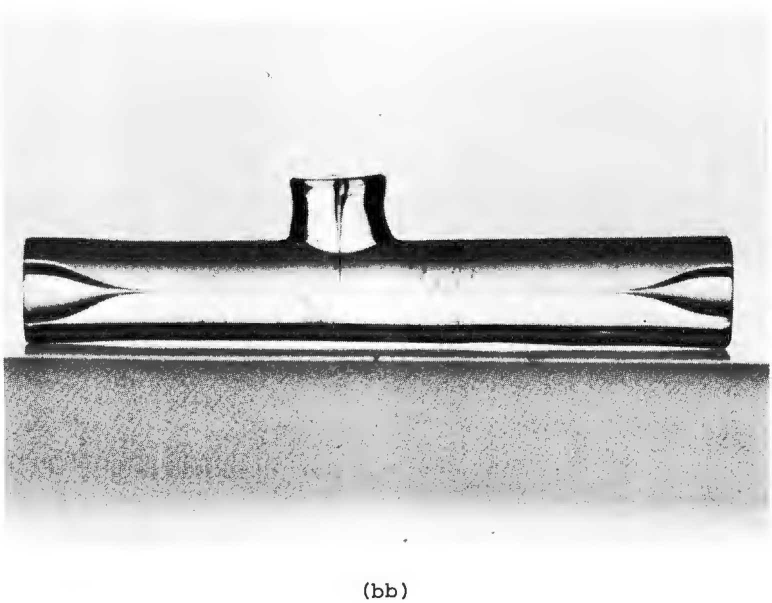


Figure 11. Drilled Holes in Fused Silica and Some Other Materials

(a). Holes in Borosilicate, Fused Silicate, Araldite and Perspex. (The shape of the holes in araldite and perspex are similar to those in fused silica.)

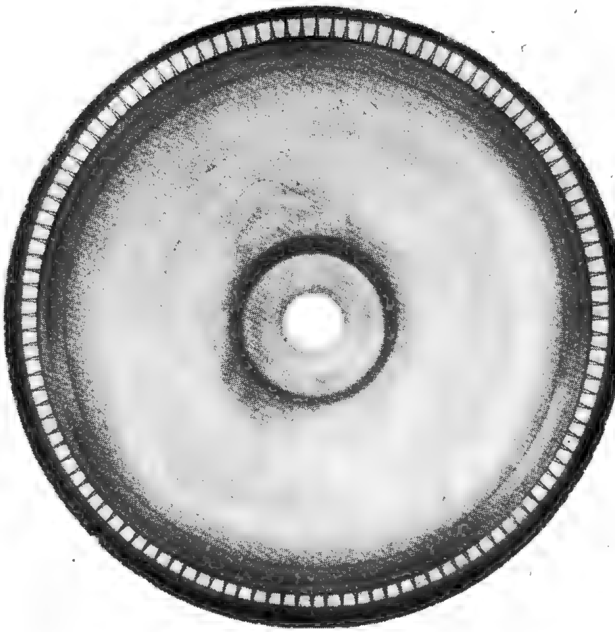


(b)



(bb)

Figure 11b. Side-Hole to a Capillary in Fused Silica Ø 50 µm, 1 sec. (Photo 11bb)



(c)

(cc)

Figure 11c. Series of Holes in a Wheel of Fused Silica 120 Holes, Ø 80 µm, 0.9 sec./hole. (Photo 11c en cc)

FINNED ALUMINUM SPOOLS FOR USE ON SUN AND PLANET CHUCKS

by

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ABSTRACT

A device has been invented that consists of three finned spools, machined from aluminum, which when installed on the fingers of a sun and planet chuck, allows the chuck to be closed to a zero clearance. This device also increases the accuracy of the chuck. In practice, fine wire may be chucked up for sealing without other support, while the full use of the chuck is not impeded. Other benefits include the ability to chuck up and hold accurately, flanged tubes, Erlenmyer flasks, cylinders with hexagonal bases, etc., all without the use of asbestos.

EXPERIMENTAL

The lathe which I use in the Department of Chemical Engineering is a Heatway with a 6" bore and is equipped with a sun and planet chuck on the head stock and a scroll chuck on the tail stock. With the wide variety of work that is required to be done using my lathe, I have found two situations involving the s and p chuck that I felt warranted some study.

The first situation has been the inability to be able to grip very small tubing in the 2 to 7 mm range and because of the unevenness in the braided asbestos tubing covering the chuck fingers, I have had difficulty in being able to grip accurately tubing in the 7 to 10 mm diameter range without first wrapping a piece of asbestos tape around the glass tubing.

To overcome this situation I used a standard small scroll chuck with a tubular shank mounted in the s and p chuck with a hose clamp around the fingers to maintain alignment; however, this setup limited the maximum capacity of the headstock to 30 mm diameter tubing.

The second situation has been the inability to be able to grip certain shapes of apparatus such as right angle bends, "U" tubes and cold traps where the fingers of the chuck grip the side tube before being able to close around the tubular body.

In this situation I used 3 aluminum spools which were installed on the chuck fingers after removing the braided asbestos. These worked well but they had to be removed and the asbestos reinstalled in order to work with larger tubing which the spools were unable to support. As well the initial problem of gripping small tubing was worsened when using the spools.

It was at this point that I wondered if some type of device could be designed which could:

(a) be permanently installed on the chuck.
(b) be able to handle small tubing without limiting the capacity of the chuck.
(c) be able to handle problem apparatus.
(d) eliminate the use of asbestos.

RESULTS

The results of my efforts took the form of 3 machined aluminum spools equipped with fins which allow the spools to intermesh to the point where the clearance is zero.

The spools installed on the fingers of the s and p chuck can accurately hold a piece of wire as small as 0.6 mm in diameter.

Aluminum was chosen as a starting material to make the spools because of its ease of machining but has proven to have been a good choice. The length and diameter of the spools were chosen arbitrarily. The hole through the centre of the spool is such that it allows the spool to slide freely along the chuck finger without being sloppy, and is held in place by means of an allen screw. The distance between the fins is twice the width of each fin to permit intermeshing with the other two spools.

The spools have proven to be very versatile in a variety of applications.

Four foot lengths of small diameter tubing may be easily joined and no problem with crushing of the glass has been experienced.

For larger apparatus 3 normal spools of the same diameter as the finned spools are positioned at the other end of the fingers for additional support. Although designed to intermesh the spools may be aligned to accurately hold a variety of glass shapes.

Erlenmeyer flasks are easily supported even with a capacity of 4 litres. Gripping by the neck is no problem either, while at the same time using no asbestos.

Glass cylinders with hexagonal bases are easily and quickly gripped by the base and the cylindrical portion will rotate accurately.

Round bottom flasks over a capacity of 1 litre because their contour can be a slight problem. I have had to resort to using a pad of asbestos for extra gripping force.

QVF pipe flanges as large as 9 inches in diameter may be held without slipping. No damage is done either to the finned spools or the precision ground surface of the flange.

CONCLUSIONS

For a period of two years a device in the form of 3 finned spools machined from aluminum has been used daily on a s and p chuck which has greatly increased its versatility by:

1. being permanently installed on the chuck.
2. being able to close down to zero, while not limiting its full capacity.
3. being able to hold a variety of glass shapes quickly and accurately thereby reducing set-up time.
4. being able to virtually eliminate the use of asbestos and in so doing, the accuracy of some work involving small diameter tubing has increased as well as reducing the amount of exposure of the glassblower to asbestos.

ACKNOWLEDGEMENT

I would like to thank Gordon Kearns of our Departmental machine shop for his accurate workmanship in the making of these spools, the Professor Michael Charles, chairman of our Department, whose support has made it possible for me to attend this 23rd symposium and to present this paper which I hope has been of interest to you.

Figure 1.

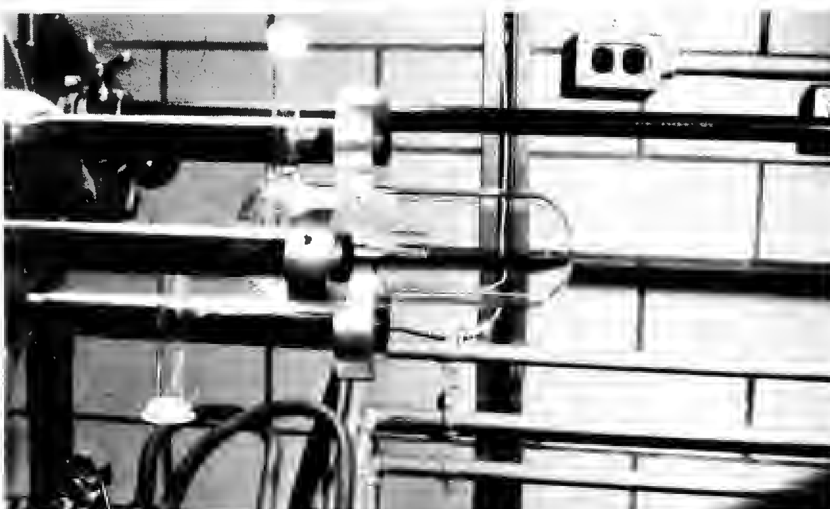

Figure 2.

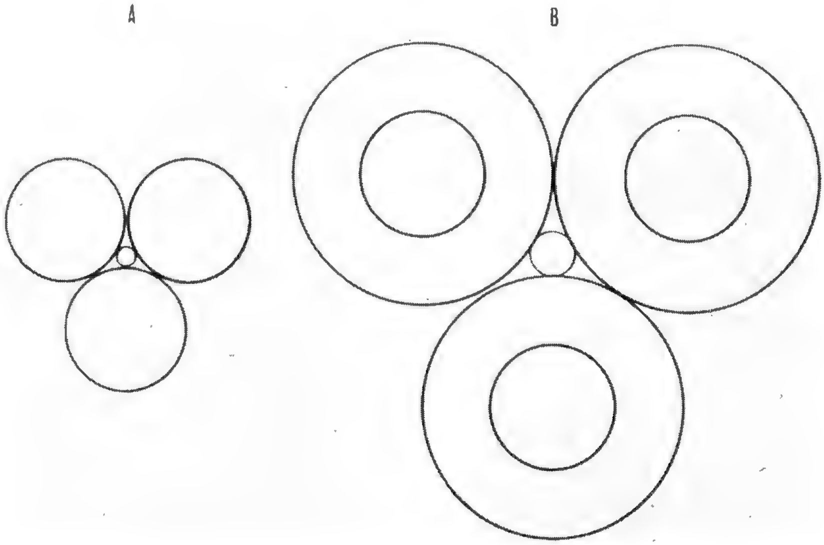

Figure 3.

Figure 4.

Figure 5.

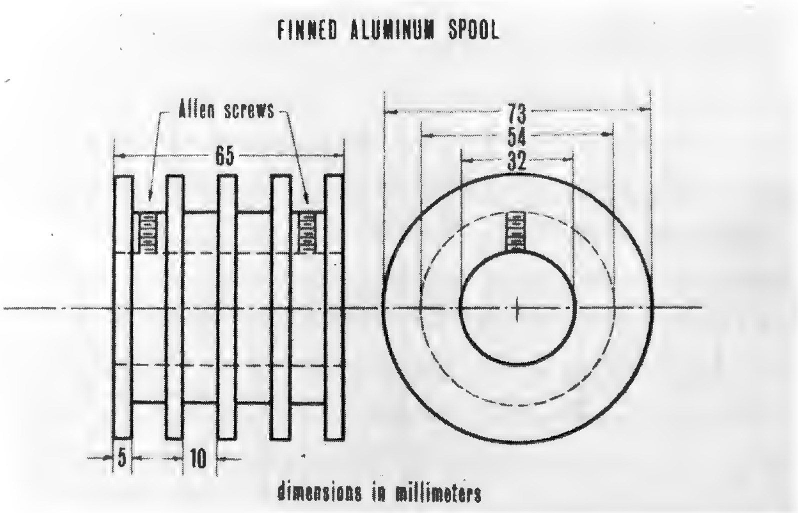

Figure 6.

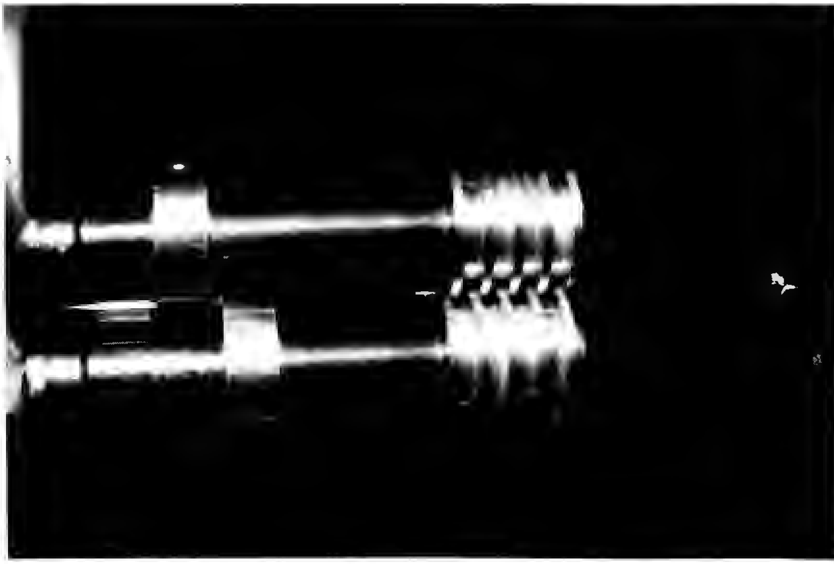

Figure 7.

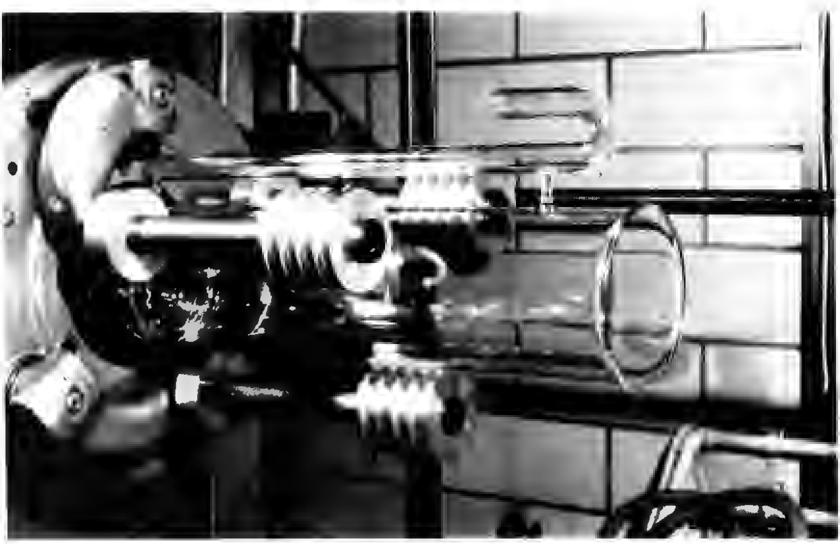

Figure 8.

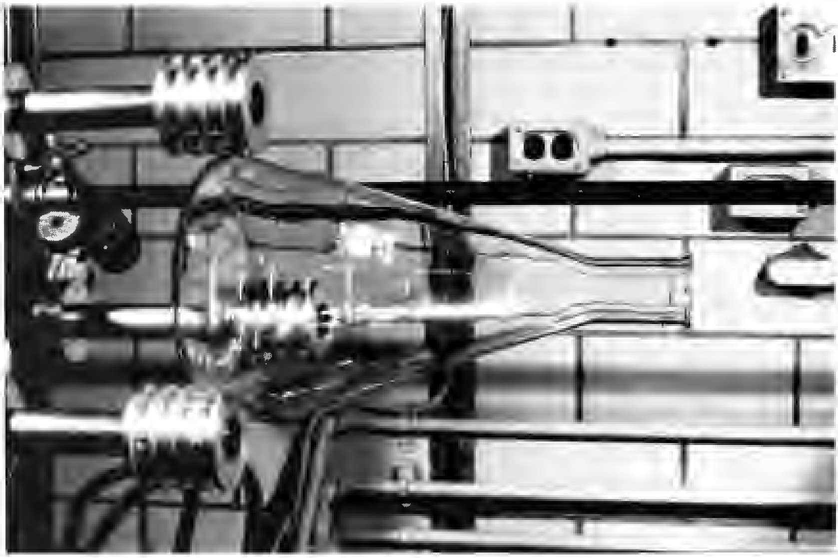

Figure 9.

Figure 10.

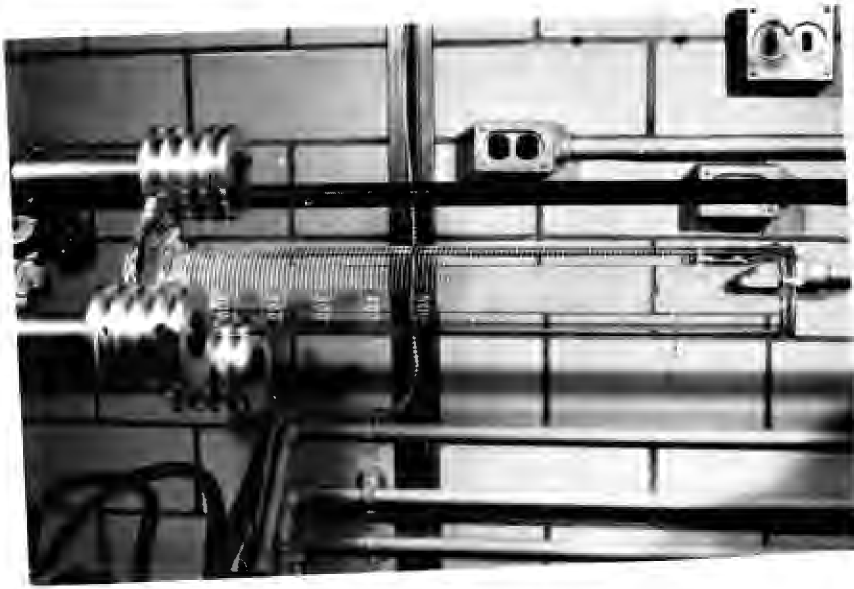

Figure 11.

Figure 12.

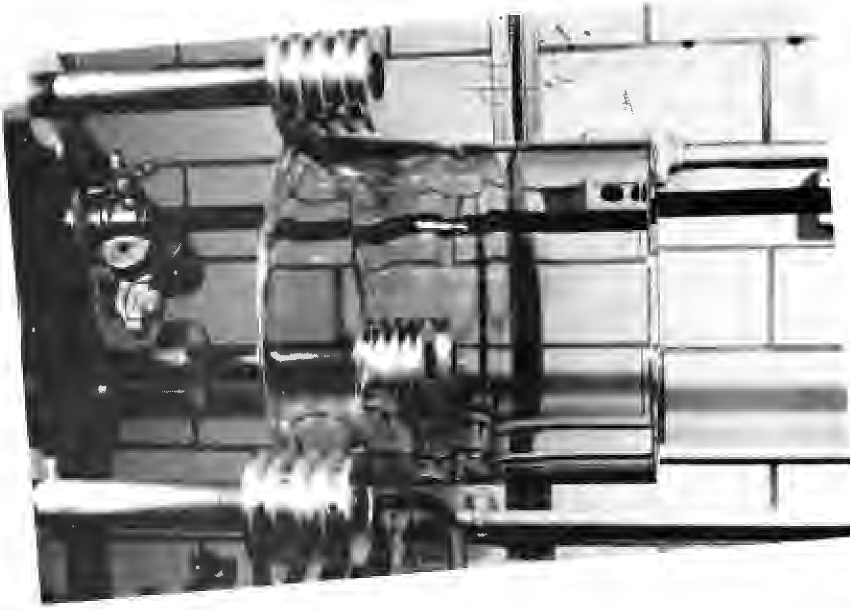

Figure 13.

BONDING OF FUSED SILICA FOR HIGH TEMPERATURE TRANSDUCER APPLICATIONS

by

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Seattle, Washington 98124

ABSTRACT

There is a need in Aerospace for high temperature instruments such as extensometers, accelerometers and pressure transducers. Good elastic properties make fused silica an attractive material for use in those instruments.

The paper describes the attempts of various sealing methods which were investigated for bonding the fused silica parts of the instrument together which would be compatible with the high operating temperature and the thin film metal electrodes.

The method investigated includes the direct fusing, bonding with borosilicate and aluminosilicate glasses and a low expansion devitryfying frit.

The development of the transducer was done by Boeing Instrument Development Laboratories, supported by NASA Lewis Research Center Contract NAS3-19556.

This paper limits itself only to development of fused silica bonding techniques.

The full report No. NASA CR-135282 is available from N.T.I.S., Springfield, Virginia 22161.

INTRODUCTION

There is a requirement to measure the pressure inside aircraft engine around 650°C over the pressure difference of 69KPa (10 psi) and dynamic response of 2000 H2. The transducer has to operate without external cooling and has to be as small as feasible so not to interfere with the engine performance.

The concept is using a fused silica diaphragm bonded between fused silica recessed discs to form the pressure sensing element. The deflection of the diaphragm is measured by the capacitance changes between metal films deposited on both sides of the diaphragm and inside the recesses of the discs.

The physical properties of fused silica makes it the best material for transducer construction. Its high elasticity at 650°C limits the hysteresis. Its high purity guarantees the high electrical volume resistivity. The low coefficient of thermal expansion gives the instrument the dimensional stability and thermal shock resistance.

The material used for the transducer development was Dynasil 4000 and 1000 supplied by Dynasil Corporation.

THE BOND REQUIREMENTS

The method fo bonding the diaphragm to the recessed discs must preserve the advantages of fused silica properties and also protect the properties of metal film electrodes and interconnectors.

The bond must withstand the forces of 207 KPa (30 psi) at 650°C. No creep of the joint can be allowed for continuous operation. The bond line must have consistent thickness for dimensional control. The joint must have high electrical resistance at 650°C. The bonding temperature must be below the temperature at which metal film electrodes would degrade.

DIRECT FUSING

Initial attempt was to fuse 25 mm dia. fused silica discs, 1.6 mm thick by diffusion bonding in an inert atmosphere. Graphite supports were used and contact pressure in range of 200 KPa (29 psi). No acceptable bonds could be made below 1200°C, much too high for preserving the integrity of the metal films.

BOROSILICATE GLASS

The Vitta tape method of fusing Quartz by means of borosilicate glass Corning Code 7070 was described on 15th ASGS Symposium by Mrs. Kitty Ettre. The application of transfer tape gives a bond line of very consistent thickness, a very important consideration in the transducer design. The sensor, fused silica component of the pressure transducer, consisted of two recessed discs 25.4 mm dia. with metal films deposited by sputtering inside the recesses and .25 mm thick diaphragm with metalized surfaces on both sides.

The G-1015 transfer tape was very easy to apply to the discs and made a very good bond at 900°C. The bonds were consistent and the instrument operated very well below 500°C. But at 650°C the bond yielded under air pressure exerted on the diaphragm, distorting its shape - the can effect. This bonding method was not acceptable for our temperature requirement but would be most preferable for similar instruments operating at lower temperatures.

GLASS-CERAMIC

The only hope was now in a low expansion glass-ceramic seal which would crystalize below 1000°C. Such a material designated "E-Frit sample No. 1" was supplied to us at no cost by Corning Glass Works. This devitryfying frit is Corning proprietary composition, has no code designation and is not sold in Corning line of products, but can be obtained from Corning Technical Products Division as a special order.

The frit was supplied in a fine powder form. It was applied by silk screening technique sued for thick film microelectronic technology. The slurry for screen printer coating was done by mixing by hand with standard silk screen printing oil Emflow 43 and thinner Reagent 16 (products of Electro Materials Corporation). This slurry or ink, as it is called by silk screening trade, is easier to apply with screen printer than a conventional Amyl Acetate and Nitrocelulose.

Several experiments were conducted in finding the best way to apply the frit, single or double coat, matting the surface dry or wet. The frit was even applied with toothpick under the microscope. The temperature cycle for firing the frit consisted of heating to 200°C with 30 minutes period to drive the solvent off and another 30 minutes period at 600°C to burn off the binder residue. The final curing was done at 970°C for one hour. The support frame was made also from fused silica, and consisted of flat base with guide rods fused to it. The guide rods were keeping the components in alignment, which was critical due to complex interconnecting metal film strips. The dead weight in a form of high density zirconia block supplied the contact pressure of 50KPa (8 psi). Over 20 fused silica sensors were constructed. The final series of sensors were 1 cm dia. (.4"). The diaphragm thickness was 254 mm (.010"). The parts were manufactured by Mindrum Precision Company.

The following illustrations are describing the pressure transducer:

Figure 1. Sensor fused Silica Components
"A" is the view of the top of the upper recessed disc. The holes are for the gas access to the diaphragm, the metal film is on the opposite side.
"C" is the diaphragm which has identical metalization on both sides.
"E" is the inside view of lower disc. It has also metalized strip on the outside surface for interconnectors.

Figure 2. The Sensor Assembly - top view
The view shows the film interconnectors and lead wires attached to them.

Figure 3. The Side View of the Sensor
The diaphragm is clearly visible and the film interconnectors.

Figure 4. The Parts of the High Temperature Transducer
Below the fused silica sensor is the ceramic washer (without gold o-ring).

Figure 5. Sensor in the Test Fixture
It shows the lead wires attachments and the spring load of the seal.

Figure 6. Transducer Assembly
The upper disc of fused silica sensor is visible with four gas access holes.

ACKNOWLEDGMENT

The author is indebted in this work to the following persons:

Mr. Herbert L. Minkin, Project Manager, NASA Lewis Research Centre, Cleveland, Ohio

Mr. Dick Egger and Mr. Chuck Sikorra, Boeing Instrumentation engineers responsible for transducer development

Dr. Francis Martin and Mr. William C. Lewis of Corning Glass Works responsible for supplying us with devitryfying frit.

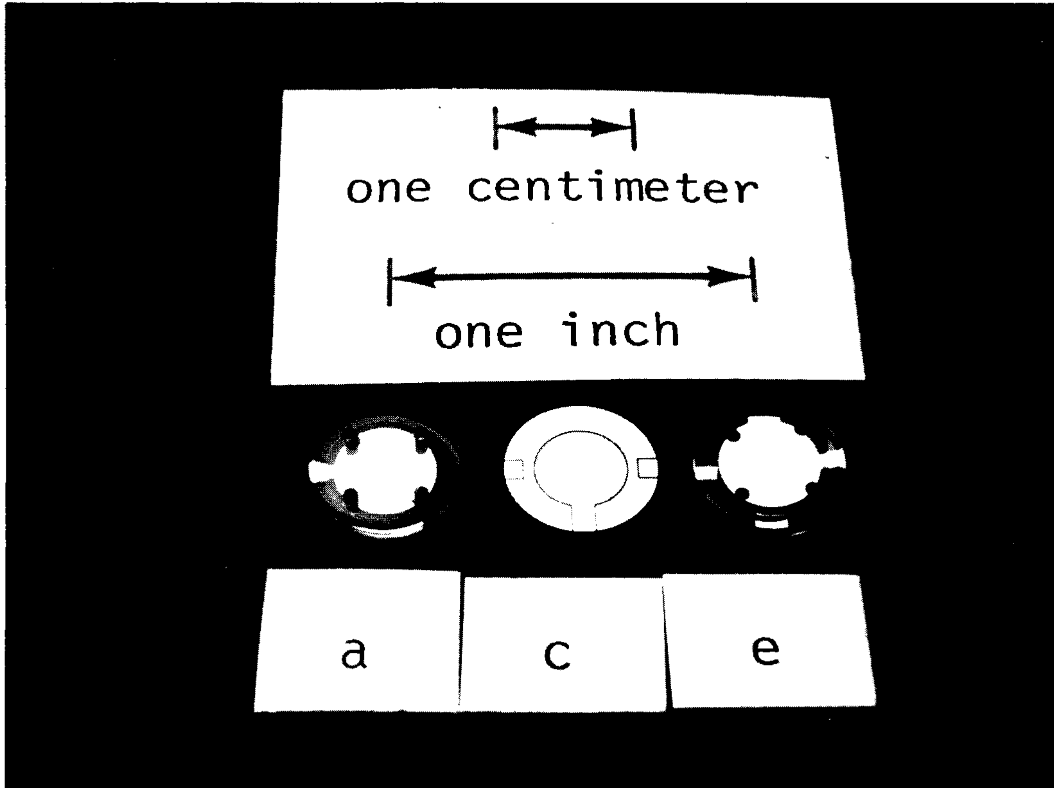

Figure 1. Sensor Fused Silica Components

Figure 2. The Sensor Assembly - Top View

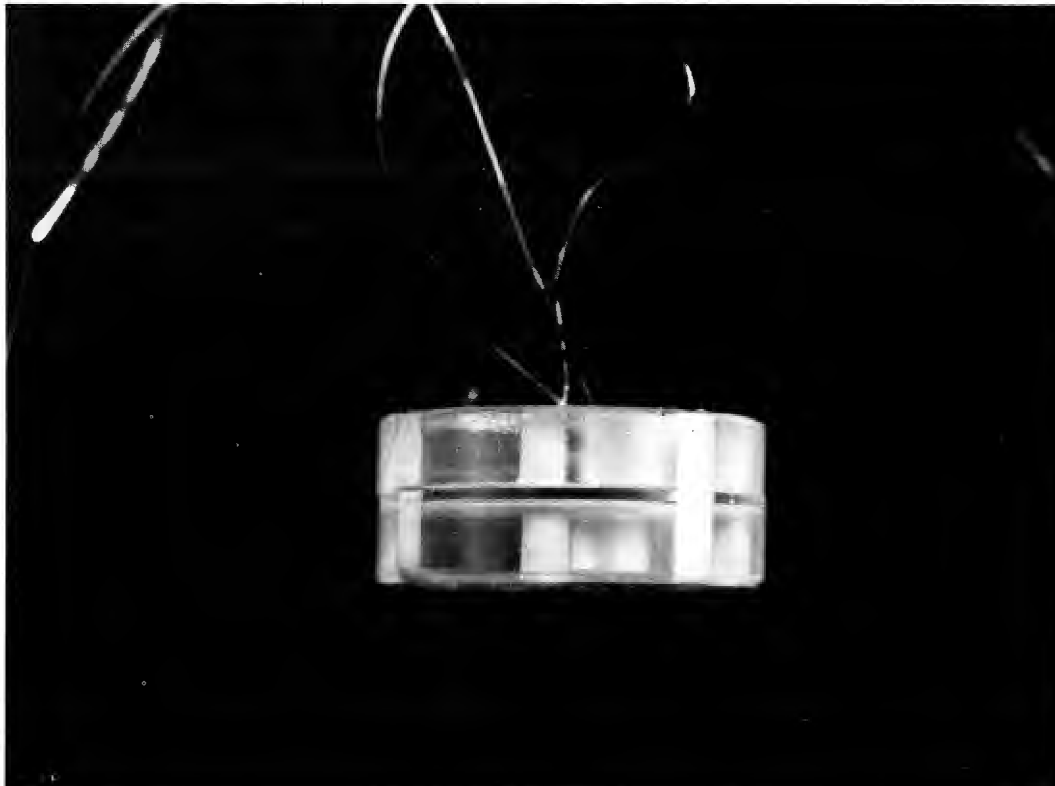

Figure 3. The Side View of the Sensor

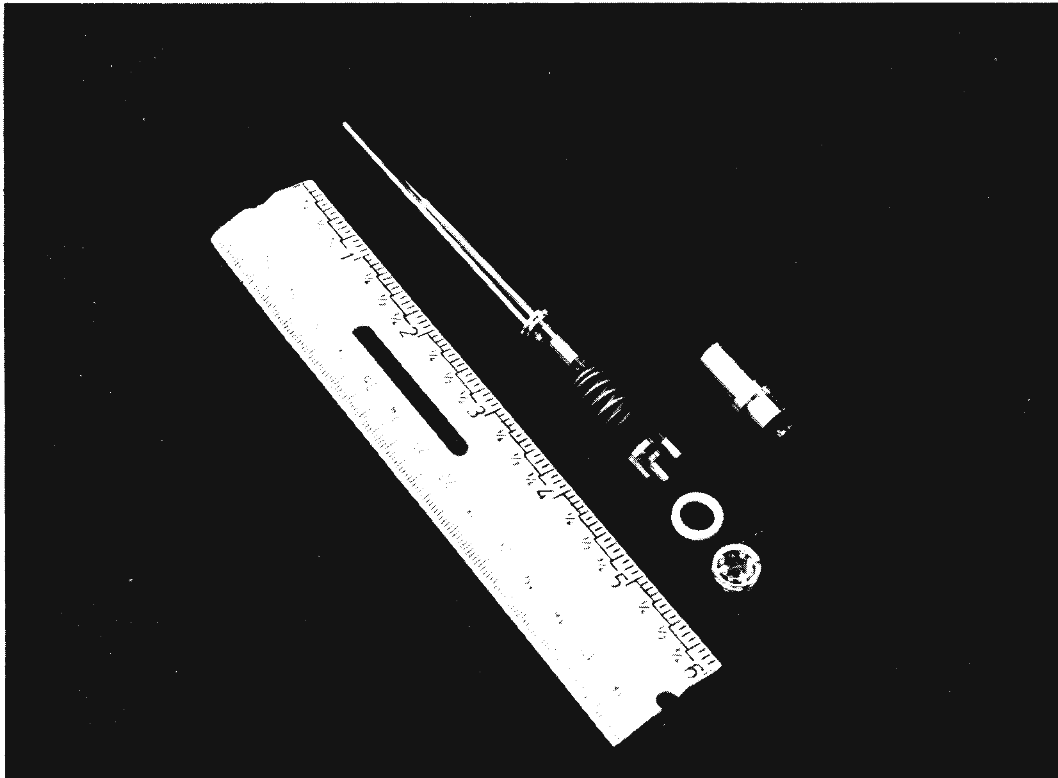

Figure 4. Parts of High Temperature Transducer

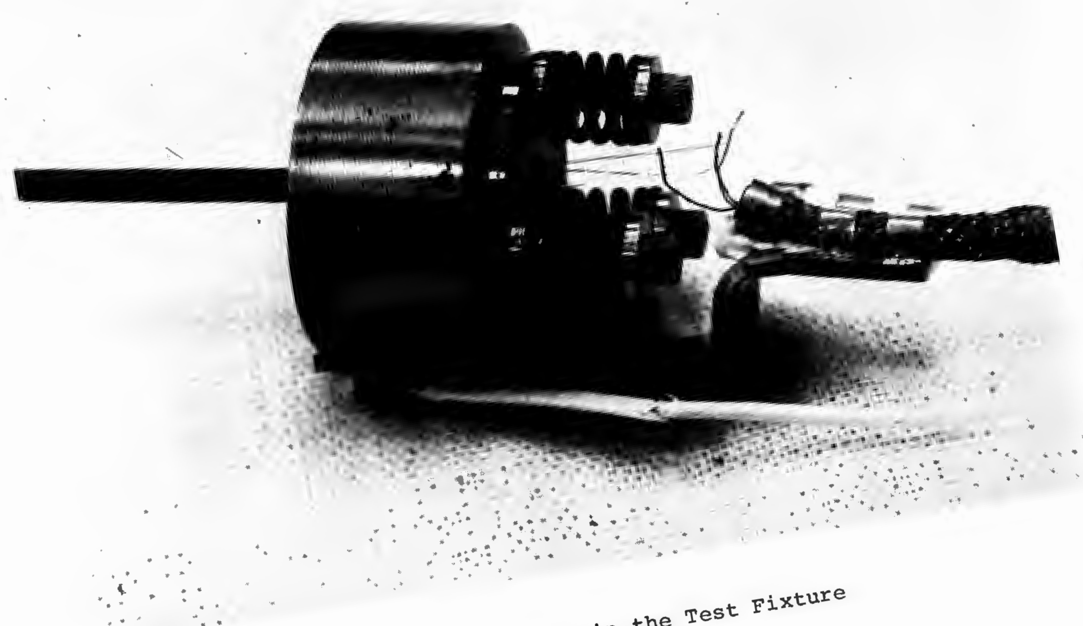

Figure 5. Sensor in the Test Fixture

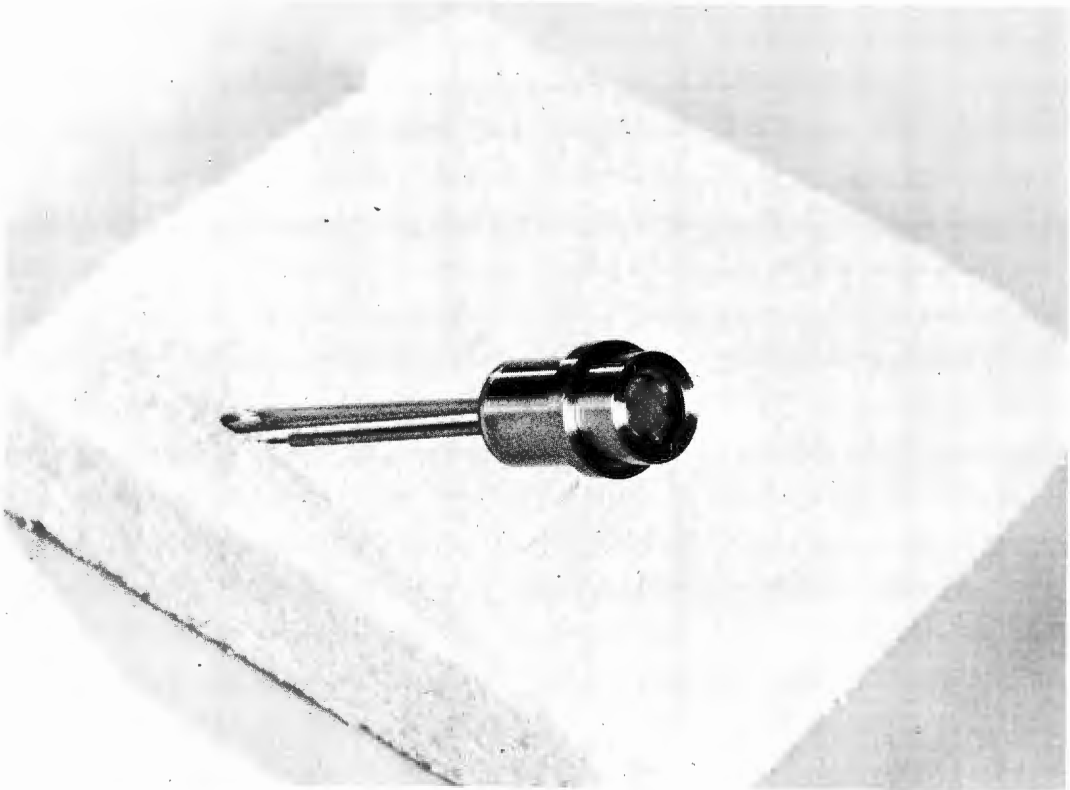

Figure 6. Transducer Assembly

SAFETY IN THE GLASS SHOP

by

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In our day-to-day work environment, safety should be our most important commitment.

A good safety record is by no means an accidental achievement. Rather it is an accomplishment, attained by the positive thinking attitude of the recipient. Each person is responsible for knowing the hazards of their job. Each supervisor must be responsible for the proper safety instruction of all new personnel. Likewise, older personnel, when moved to a new work location, must also be properly re-instructed.

When handling dangerous or unfamiliar materials, we must acquaint ourselves with the hazards involved and the consequences of overexposure. Many excellent safety handbooks, etc., are available for consultation as to the proper methods necessary for the save handling of these items.

As new fields of interest are entered, one must evaluate the potential hazards to be encountered. Draw upon your memory banks for past experiences covering similar operations. Your many options should be investigated and only after a complete survey has been made, a safe work procedure, covering that entire operation, should be written. The procedure must then be filed in your safe work procedure manual where it will be available for departmental reference.

In regard to safe work procedures, I am reminded of a personal experience back in the days when safety procedures were a mouth-to-ear operation. I had been involved in a column silvering operation using the Brashear Method. Following the instructions from a handbook, everything went along fine. The silver deposited out in bright shining film and was I pleased. After rinsing the column with distilled water I dried it and fused the evacuation tube to the vacuum connection in the annealing over. At that time the whistle blew, and I had to close up the shop and catch my ride home.

By the way this all occurred on a Friday afternoon. On Sunday my mind flashed back to the silvering operation and I momentarily reviewed what had transpired during Friday afternoon. I now recalled that the instructions cautioned one to dispose of all of the spent silvering solution. Had I done this? I quickly made a trip back to the plant and looked at the silvering area. Sure enough a beaker in the sink had a light metallic film floating on its surface of water. I was perplexed as to what action to take. I hastily reviewed the silvering process and made a special note on silver fulminate and its explosive qualities. I threw some protective gear over my head and body and reached over and slightly opened the faucet of the sink which was located directly above the beaker. When the first drop of water hit the beaker, it caused a very sharp detonation which broke it and splashed the spent silvering solution in all directions. I was plenty shook up but fortunately not hurt. I learned a great lesson in a hurry and no matter what goes on, as a silvering deposition is completed, the first order of business is to flush all materials down the drain with plenty of water. Then, and only then, is the column flushed and completed. I might also add that this near-miss resulted in a new method being developed to make high reflecting heat-shield columns. A column that proved superior to silvered columns but that again is another story.

I do not profess that one has to establish a safe work procedure for every little thing that goes on in your shop. To do so could lead up to an untenable situation. A better approach would be to accept the concept that "all injuries are preventable."

With this positive statement as our watchword, I am positive that injuries to ourselves will all but be eliminated.

I am reminded of the visit that was made to the Stuart & Son glass plant during the 1976 First International Scientific Glass-bower Symposium in England. In groups of at least 10 persons we were invited to tour the entire plant and to walk the operation floor. To observe at firsthand the ancient art using blowpipes to produce beautiful lead crystalware. Many of us were given the opportunity to handle the molten glass, to blow a bubble and to try out their grinding operation. At distances of no greater than 8 feet we watched this fascinating operation which transformed a molten red hot glob of glass into a thing of beauty. Young men rushing everywhere, with pontel sticks of molten glass, to supply the finish craftsmen with their raw material. Items being re-turned for a reheat, to the glory hole. Others gathering the completed crystal object in their forked sticks and rushing them to the annealing lear. It seemed to me a miracle that no one was burned or scorched by the close proximity of our groups in relation to the operations going on. During a tea break I discussed this most unusual safety attitude with a supervisor. I was assured that all employees were made aware of the hazards involved and that the department had not suffered any type of serious incident in several years. These young fellows demonstrated that it does not pay to take chances. They knew the hazards involved within their operations and refused to take chances which would force them into a situation whereby one of their visitors would get injured.

Wouldn't it be wonderful if we too could program ourselves to be aware of impending disaster. With this type of attitude we could enjoy life to its fullest.

I am a fool's advocate of "Murphy's Law." The law implies: (1) nothing is as easy as it looks; (2) everything takes longer than you think; and (3) if anything can go wrong, it will.

A good example of this is James J. Kilpartick's Law of Peanut Butter and Jelly Bread which is covered by Rule No. 3.

> "Given a piece of bread slathered with peanut butter and jelly, the bread, when dropped, always will land jelly side down."

We have much to learn from "Murphy's Law" and other basic rules of conduct in respect to safety. One great lesson to be learned is that if the project seems to be getting off poorly, "you know" on the left foot, why don't we stop for a minute and give some more thought as to what is going on. We could save ourselves a heap of trouble that may be heading our way.

For those of you who have never run afoul of Murphy's Law let me enlighten you with some highlights that come to mind during my 42-plus years of apprenticeship.

> A heavy cardboard box that is overwrapped at the seams with gummed paper. Cutting the seams with a knife first does not produce a clean cut. You then insert your hand in an available opening and subsequently you tear a couple of fingers open from the large staples hidden beneath the tape.
>
> An old pocket knife that seems to have a loose hinge pin. During a tough cutting operation the hinge pin fails, the folding knife blade cuts your hand.
>
> Removing frozen standard taper joint without protective hand gear. The glass disintegrates and you suffer a cut hand.
>
> Pushing a glass tube into a stopper or tubing without protective gear. The tube breaks and you push the glass thru your hand.
>
> Bending a cold piece of glass and the glass particles fly up into your face.

Drilling a glass plate with a tube drill. The glass is not secure, the drill grabs, a flying piece of glass strikes your chest.

How about a good whiff of ammonia hydroxide or hydrochloric acid. You know one of that kind of peals the back of your head off. It can really shake you up.

I have seen HF acid spilled on one's hands and an attempt was made to wipe the acid off with a towel instead of flushing the area with copious amounts of water. This fellow had sore fingernails for several weeks.

I would like to mention the time when a Variac was used as the power source for a hot wire tube cutter. One day the polarity was changed. You have no idea what the feeling is to be grounded at about 50 volts. That kind of experience could kill you. One should use a 32 volt isolated transformer for this operation.

Then there is the story about the glass blowers working late at night on a 4" glass pipe. The late hour was necessary to get away from some of the daytime heat. While sitting down during a rest period, a rattlesnake crawls through the open door and starts to engage in conversation with you.

There was also the time when someone threw hot glass into a wastepaper basket. A fire extinguisher was brought into play -- only the darned thing was empty. Next a beaker which happened to be full of liquid was thrown onto the fire. Do you know what -- the beaker contained a water-acetone mixture. Things really got quite warm in a hurry. Needless to say, fire extinguishers to this day are inspected on a weekly basis.

Oxygen and other large fuel cylinders present a constant danger while being handled and stored. While in use the cylinder must be securely fastened so as to prevent it tipping over and falling. A falling cylinder can have its valve broken off at which time we have a rocket on our hands. This rocket has been known to blast through a concrete wall, then become airborne for some 1200 feet.

Adapter fittings used between a cylinder and a regulator is a positive no-no. Their use is a violation of all good safety practices. I recall when a mix-up occurred and oil pumped nitrogen was substituted for oxygen. The explosion killed four engineers and destroyed an entire building.

Let me stress that Murphy's Law may not have been involved in all of my examples but you can be assured that the warning sign of "trouble ahead" was there and was being ignored.

How many times during your life have you ignored a warning sign and how many times have you suffered because of your own neglect. Say for instance a speed zone sign. I realize that things are not as simple as a sign -- yet we learn while making mistakes. And while we make mistakes we are prone to get hurt and when we get hurt we lose time off from work, our income suffers and we have placed a big burden on our families. In general we have established an economic crunch upon our lifestyle: one that may take us many months to recover from.

Wouldn't it be much better if we were to build within our brain cells a yellow caution flag which would alert us to impending danger. We are involved with and use many dangerous materials. When used improperly, disaster awaits us. Therefore, let's wave that yellow caution flag and remember Murphy's Law. It pays to play it safe.

Let our watchword be "We will do it safely or we won't do it."

FABRICATION OF GLASS LASER FUSION TARGETS

by

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"Work performed under the auspices of the U.S. Department of Energy by the Lawrence Livermore Laboratory under contract number W-7405-ENG-48."

ABSTRACT

Fusion research is being vigorously pursued at Lawrence Livermore Laboratory and other laboratories throughout the world with the ultimate goal of generating power from controlled thermonuclear reactions.

We will present two approaches to the problem of constructing glass targets with diameters of 100-500 μ and wall thicknesses from 1-20 μ.

INTRODUCTION

There are many research programs trying to alleviate the energy shortage. One of these programs is the laser fusion effort at the Lawrence Livermore Laboratory. Figure 1. This paper discusses our efforts to supply the program with high quality, deuterium tritium gas filled microspheres for Laser Fusion Targets.

The range of specifications we have to meet are: Figure 2

1- diameters from 50μ-1000μ
2- wall thicknesses from 1μ-20μ
3- sphericity better than 5%
4- concentricity and wall uniformity better than 5% between inner and outer surfaces.
5- surface finish 1000 Å or better
6- formed of a glass composition which is permeable to DT gas at 400°C, and which will hold DT gas up to 100 atmospheres at room temperature.

Commercially produced spheres do not meet the sizes tight tolerances required of target spheres for this program. We can see in Figure 3 that the concentricity and wall uniformity of the commercial spheres are quite inferior to those showing our production. Figure 4.

In the production of these target spheres we have used the liquid droplet technique, to give precisely controlled mass from sphere to sphere. This technique was first published by C.D. Hendricks and S. Babil in a process of manufacturing uniform solid glass balls from aqueous solutions of glass-forming oxides.

The ball production system we have developed (Figure 5) consists of four basic sections.

1. Droplet generation
2. The drying column (comprised of the encapsulation region and the dehydration region)
3. High temperature oven (comprising the transition region and the refining region)
4. The collection region.

The complete system is shown in Figure 6.

Droplet Generation

The glass forming compounds used are generally sodium silicate, potassium hydroxide, lithium hydroxide, boric acid and water. An aqueous solution of the glass forming oxides is fed under pressure through an orifice. As the solution passes through the orifice it breaks up into drops. This drop forming effect is known as the Rayleigh Effect shown here in Figure 7.

We control the drop formation by vibrating the droplet assembly. We accomplish this with a piezoelectric transducer coupled mechanically to the droplet generator. By using an oscillator to drive the piezoelectric crystal at a frequency of 4000-8000 KHZ we produce very uniform 150 micron diameter drops. Figure 8.

Each drop, after leaving the orifice, passes through a charge ring which inductively charges the droplet to approximately 200 volts. During normal operation 1 droplet of every 32 is left uncharged and is allowed to pass undisturbed into the oven. The other 31 are deflected electrostatically and recycled. We can regulate the spacing between droplets and keep them from colliding with one another. If these droplets were allowed to collide we would lose control of the droplet mass. A typical droplet generator is shown in Figure 9.

Drying Column

Figure 10. The drying column consists of a 9' length of 3" diameter stainless steel tubing. This tubing is wrapped with 6, 1000 watt heating tapes evenly spaced along its length. Each heating tape has its own thermocouple and controller. As the 150 micron diameter droplet falls through the drying section of the oven it first passes through a temperature zone of 300°C to 400°C. In the upper portion of this section, surface water is rapidly vaporized and a gel ball is formed (Figure 11). The gel ball at this point has an outer semi-dryed membrane with a liquid center. The remainder of the drying column temperature profile is designed to vaporize the internal water at a rate equal to the

rate of diffusion of this water vapor through the highly permeable outer membrane. If the temperature is too high the balls will explode from excess pressure, and if it is too low they will collapse to a solid bead. As water is being forced through the outer skin of the ball the glass forming oxides from the trapped solution are collected on the inner walls, and become part of the gel membrane.

The gell ball is now expanding in diameter due to a slight internal pressure. At the end of the 9' long drying section the ball has a diameter of about 1.5 mm and a wall thickness of approximately 1000Å.

High Temperature Fusing Section

The fusing section is a 1500°C oven 4 feet long with 3 individually controllable sections. The tube for this section is usually 3" diameter quartz, but we have been experimenting with 99% alumina tubing and it has surpassed the quartz in performance.

The ball now enters the transition region which is the upper 12" of the high temperature oven. The transition region's temperature is held at 1100°C to 1200°C. It is here in the transition region that the glass forming oxides are fused into glass. As this transformation begins the gel wall turns to liquid, its viscosity drops, and the microsphere begins to collapse under the influence of surface tension forces. Since there is little or no excess pressure within, the collapse is rapid but is partly offset by a buildup of internal pressure due to decreasing inner volume, and to the release of new water and possibly other occluded gases generated during the glass formation process. We have determined that to produce highly concentric balls this glass forming chemical reaction should take place as rapidly as possible, in order to ensure uniform collapse. Balls emerge from this section of the oven with a diameter of about 200-300 microns.

Refining Section

The ball next enters a section called the refining zone which is the bottom 2 feet of the high temperature oven. This section is run typically at about 1200°C. In this section the chemical reactions are completed and any pockets of incompletely reacted material are turned to glass. Any small bubbles trapped within the walls diffuse out. The ball shrinks somewhat in diameter in this section due to the permeability of the glass, and if the viscosity of the glass is low enough any defects in sphericity will be eliminated due to surface tension.

Collector Section

This section is constructed of 3" diameter stainless steel tubing 2 feet long. This tube is long enough to allow the balls emerging from the high temperature oven to cool below their strain point before coming to rest. At the bottom of this section is a line connected to an exhaust blower through a flow meter. The blower overcomes the convection current of the oven and ensures a net downward movement of the balls.

Chemical Treatment

The glass spheres are taken from the oven and sieve cut to a specific size range. They are next put into a pressure vessel and subjected to a high pressure to implode the imperfect or defective spheres. Next they are washed with dilute acids and solvents in a procedure that we have developed until it is determined that they are clean, Figure 12, and at this point they are ready for drying, final characterization, and storage in alcohol. The wash technique that we use results in microspheres that have high surface smoothness (better than 1000Å) and good resistance to weathering in air. The spheres now can be filled with DT gas as the need arises.

SUMMARY

We have developed systems which have the capability of producing large numbers of high quality laser fusion targets. By controlling both temperature and transit time through the oven we can produce large quantities of highly concentric balls with varying wall thicknesses and diameters.

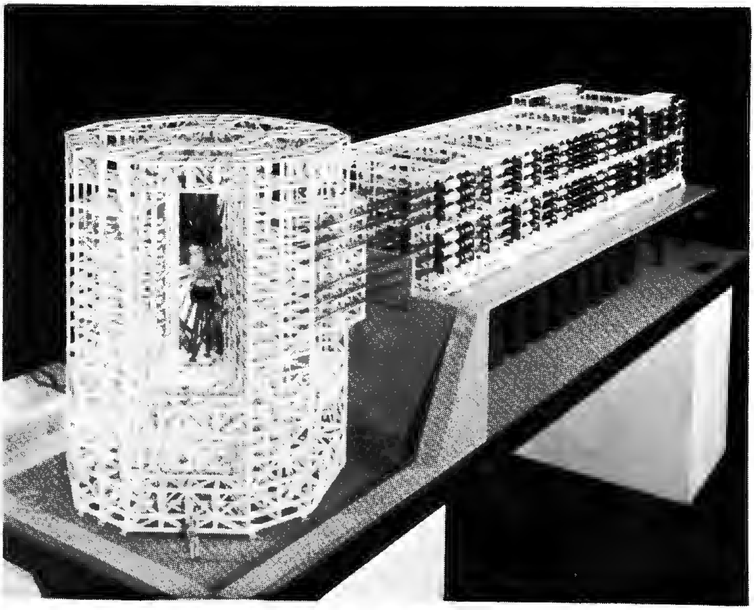

Figure 1. 20 Trillion Watt "Shiva" Experimental Laser Fusion Facility

1) Diameter range 50 μ – 1000 μ
2) Thickness range 1 μ – 20 μ
3) Sphericity better than 5%
4) Concentricity and wall uniformity better than 5%
5) Surface finish better than 2000 Å
6) Strength – able to contain up to 100 atmospheres of DT
7) Composition – low Z material

Commercial glass microspheres are unable to meet these criteria

Figure 2. Criteria for Laser Fusion Targets

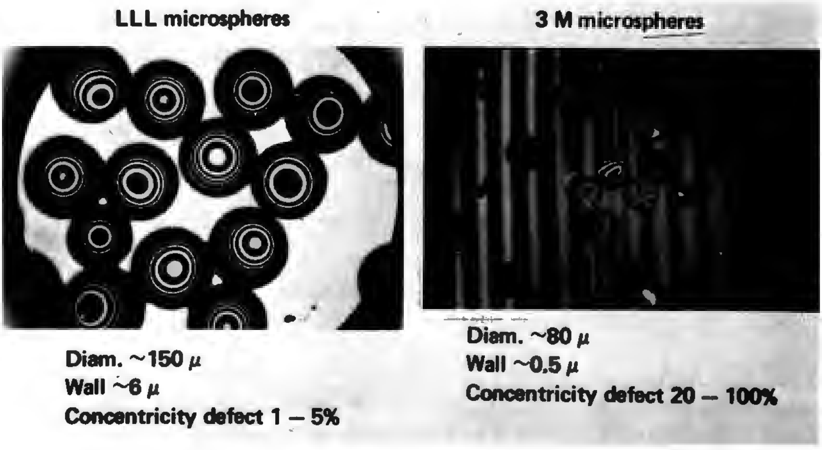

Figure 3. Laser Fusion Glass Microspheres

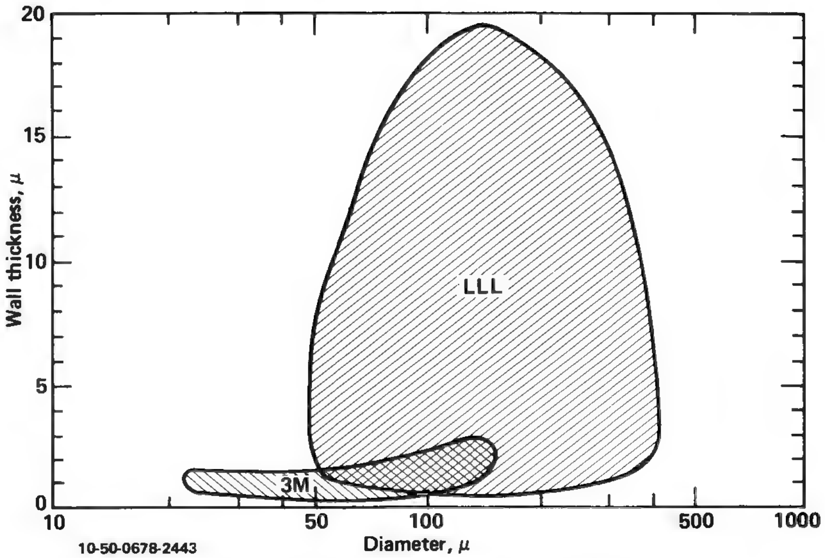

Figure 4. Glass Microsphere Production

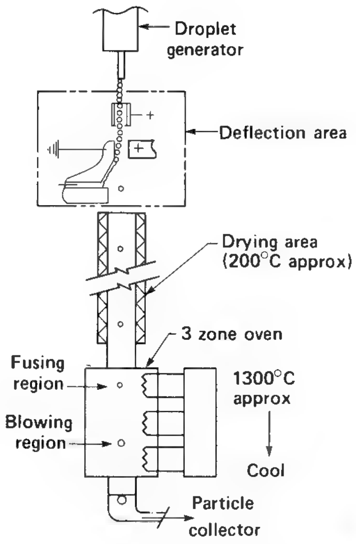

Figure 5. Schematic of Droplet System

Figure 6. Target Sphere Production System

Figure 7. Droplet Stream Showing Rayleigh Effect Stimulated and Stabilized

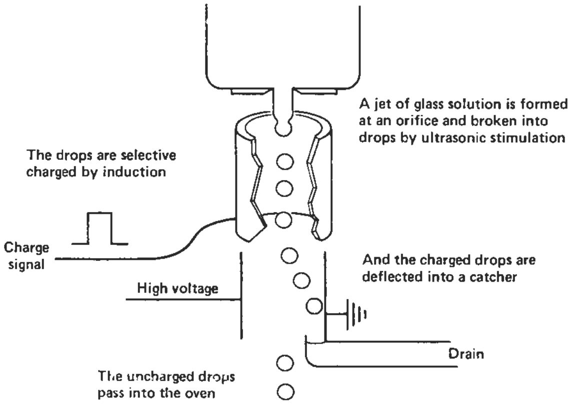

Figure 8. The Droplet Generator Is a Microdispenser

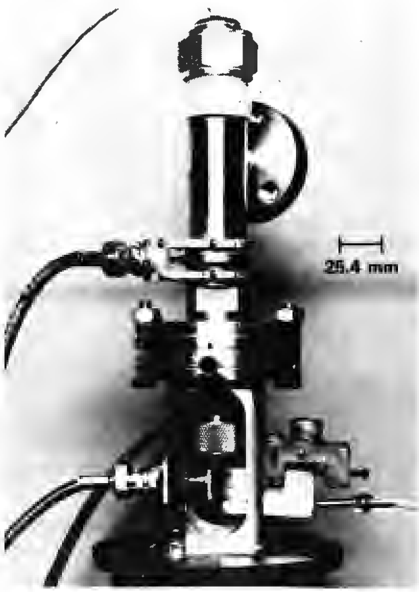

Figure 9. Droplet Generator

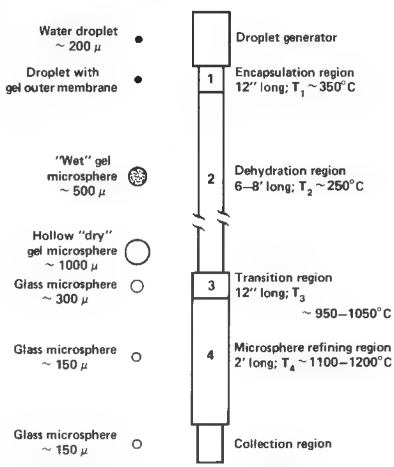

Figure 10. Liquid-Droplet System

Figure 11. Gel Microsphere

Figure 12. Microspheres After Correct Wash

GLASS-TIPPED PROBE FOR A MASS SPECTROMETER

by

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This paper discusses the design and problems encountered during the construction of a modified glass-tipped probe which is inserted into a Mass Spectrometer.

A Mass Spectrometer is an instrument which produces a beam of ions from a substance being investigated, sorts these ions into a spectrum according to their mass to charge ratios, and records the relative abundance of each type of ion present. The ionizer, analyzer and ion detection stage of all Mass Spectrometers operate in a vacuum. The background pressure is usually in the order of 10^{-7} to 10^{-10} torr. The ion source consists of a hot filament, an ion chanber and an ion lens. In this case a glass-tipped probe is used to insert the sample into the ion source. The original Mass Spectrometer probe was purchased from the manufacturer with a glass end built of Corning Glass No. 7052 sealed directly to Kovar. Due to the high voltage (approximately 8,000 volts) the glass tips were having holes burned through them frequently, resulting in loss of vacuum in the ion chamber.

The replacement cost of a probe is approximately $2,000.00.

I was asked if a repair job or replacement could be done on a short notice because the Chemistry Department had only one probe. In a discussion with the Chemistry Department it was decided that two new probes would be made in their entirety.

Repairing the old probe was not too difficult because the glass was all Corning No. 7052; however, the dimensions were tight. In the end of the probe there is a 2 mm i.d. well that holds a 1 mm o.d. capillary which in turn holds the sample to be analyzed.

First I changed the sample capillary from soft glass to Corning No. 7052 but that did not help. The next stage was to change it to Corning No. 7740 which did help. Then I changed the glass probe from Corning No. 7052 to 7740.

In the construction stage the probe was striped clean and new Corning No. 7052 was added, next Corning No. 3320 and then Corning No. 7740 to finish the probe. This worked well and lasted about six times longer than the original Corning 7052 tipped probe. However, this was still not good enough.

While the stainless steel probes were being made in our machine shop, I continued to experiment with the one we had. It was found that a quartz capillary worked very well which then led to the manufacture of a quartz-tipped probe.

I began looking for a quartz to metal seal with a maximum o.d. of 12 mm with a 1/2 mm wall. Bomco was the only company that was even close, but not close enough. I ordered some Kovar then welded it to the stainless steel probes. I decided to make the grades approximately 5 mm long and 12 mm o.d. I fired the probe in a wet hydrogen oven and then sealed on a piece of Corning No. 7052 approximately 5 mm long, then a piece of Corning No. 3320 approximately 5 mm long, then a piece of Corning No. 7740 approximately 5 mm long, then a piece of Corning No. 7240 approximately 5 mm long, then a piece of Corning No. 7230 approximately 5 mm long that ended in a round bottom. Next I took a piece of quartz 4 mm o.d. and pushed up a maria and made a ring seal in the end of the Corning No. 7230 to finish the grading from Kovar to quartz in a total of five different grades. The other two probes were finished the same way so the Chemistry Department had three usable probes. The only time I get one back is when they drop one and I now have time to repair it because of the two spares.

In the scientific glassblowing field we constantly are being challenged by new and unique problems; through perseverance and the application of new techniques, solutions can ultimately be found.

IN ATTENDANCE

The following are on record as having attended the Twenty-third Symposium on the Art of Glassblowing held at the San Jose Hyatt House, San Jose, California, June 19-23, 1978. As fully paid registered participants, these persons are entitled to a copy of the Proceedings.

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