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THE TWENTY-FIFTH SYMPOSIUM
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THIN FILM INSULATORS AND CONDUCTORS

David W. Skelly

The field of thin film deposition is extremely broad and has been well reported with respect to both materials and technology in several comprehensive texts.[1-4] The purpose of this discussion is to place the recent growth of thin film technology in perspective and to describe several unusual applications of thin films.

Thin films defined here are those which are less than 10 microns (0.4 mil) thick and are usually not self-supporting.

There are four basic types of thin film deposition:

1. Chemical deposition — wet plating
2. Evaporation
3. Sputtering (r.f., d.c., ion plating, neutral ion milling)
4. Chemical vapor deposition

Each technique has its advantages and disadvantages; however, it is the dry, vacuum processes of evaporation and sputtering which have grown so remarkably.

This expanding interest in thin film deposition arises from three forces:

1. Ecology
2. Economy
3. Special properties

Ecological forces are usually more legislative than altruistic. Environmental standards in many countries including the U.S. are becoming more severe and are approaching the limit of zero-ecological-impact for metal treatment processes. Japanese manufacturing, for example, now excludes wet plating processes because of the environmental impact of the waste solutions. As a result, there has been a major expansion of the use of the dry methods in Japan.

Economic factors are also forcing thin film deposition expansion. Cladding of inexpensive base metals on a large scale has been reported by S. Shiller et al. of E. Germany[5] for sheet steel (with aluminum and copper on both sides). These

materials are continuous sheet stock 60" × 0.020" using 1/2 megawatt evaporator and 1 megawatt preheater. Zinc coated steel, on only one side, is used for auto body parts to prevent rust out. Government regulations are beginning to demand extended useful life of consumer products because of the scarcity of materials within many countries. Steel, copper, chromium, etc. cannot be indiscriminantly scraped; the cost of reclaim and re-fabrication is too high and the impact on energy resources is too great.

Although the United States has not been burdened in this respect in the past, external and internal demands on our natural resources will certainly bring us to that stage in the near future.

Inexpensive components made from materials such as glass and plastic can be subsequently coated with thin films of costly elements such as gold, platinum or chromium for electrical or decorative purposes – these are further examples of cost effective thin films. The inside of plastic wristwatch cases are often vapor coated with a thin copper coating for part of the digital watch switch circuit. Rapid, adherent, uniform and low temperature magnetron-sputtered copper has been used in this application.

Finally, the special properties of materials deposited in thin films may be more important than the ecological or economic factors; sometimes thin films offer a unique answer to a particular problem. The following examples illustrate the special properties of some thin film materials as well as several important aspects of certain deposition methods.

Liquid Crystal Cells

Many of the early twisted nematic liquid displays used in digital wristwatches used several thin films on the surface of the glass display cell to align the liquid crystal and turn on the display. Figure 1 shows an activated liquid crystal display cell. Figure 2 shows a cross section of a liquid crystal cell. The liquid crystal molecules are long rod-like molecules. On one surface of the cell all the molecules in contact with the glass are aligned along one cell axis, on the other surface they are aligned along a

perpendicular cell axis. The molecules in the bulk of the liquid align along these layers resulting in a "liquid-crystal" where the molecules are twisted 90 degrees. This layer has the ability to rotate light 90°. Placed between crossed polarizers the "twisted" layer will make the polarizers appear uncrossed. This is, in fact, how the display works. A "twisted" cell is placed between crossed polarizers and it appears clear or uncrossed. A second thin film layer in the cell is a transparent conductor which, although nearly invisible to the eye, is used to place a small electric field across the liquid. The molecules in this region align with the field, lose their twist, and cause the crossed polarizers to appear truly crossed and opaque.

There are two points to this thin film example: first, the molecules of liquid crystal require some surface force to cause them to align along the cell axis. Silicon monoxide is evaporated at a grazing incidence to the surface causing the growth of a "dragon's scale" like surface where the lifted ends of the scale point toward the evaporation source. Figure 3 shows transmission electron micrographs along with an artist's rendition of the surface structure. The liquid crystal molecules align in an energetically favored direction along the scale, parallel to the direction of evaporation. Each plate is aligned differently in the evaporator to achieve the twisted effect. The second point of this example is the transparent conductor. For many years, tin oxide, produced by spraying alcohol solutions of tin chloride onto hot parts was the transparent electrode of choice (a chemical vapor deposition). Pure tin oxide is an oxygen deficient semi-conductor, unstable in air at high temperature, it is often doped with antimony for increased stability. The glass warps during deposition of the material since the surface temperature is close to the annealing temperature of the glass. Although float glass is available, flat to within a micron per radial centimeter, conductive glass of the tin oxide type seldom is flat to within 10 microns per radial centimeter. The more recent sputtered coatings of indium oxide (another oxygen-deficient semiconductor in the pure state) are doped with tin for stability and are coated at less than 200°C and contribute very little to glass warping. Their film thickness is less than 10^{-5} cm. This material can be patterned by etching in warm dilute hydro-

chloric acid. The sputtered materials are used in the liquid crystal cells where flatness is critical to cell uniformity because the liquid thickness is only 10 microns.

Vidicon Faceplates

The vidicon is a T.V. camera pickup tube used for black and white applications such as security or surveillance. Figure 4 shows a photograph of a pickup tube. The light image is projected by a lens system onto the flat faceplate of the tube. It is converted to an electronic "image" and transmitted to the receiver for reconstruction. Thin films, coated onto the inside of the faceplate act as the converter from the light image to the electronic image. Figure 5 shows a schematic of a cross section of the tube. On the vacuum side of the faceplate a transparent conductive layer (like indium-tin oxide) acts as a conductive ground plane. A layer of antimony sulfide acts as a photoconductor. An electron beam charges the inside surface to a fixed negative potential by scanning over it, depositing electrons. The antimony-sulfide, conductive only where light strikes it and insulating everywhere else, allows the electron charge to escape to ground in the light regions. This creates an electron charge image from the light image. The electron beam, returning to a light spot after scanning the rest of the raster (picture area), deposits additional electrons when they have been conducted away. These additional electron deposits are detected as electron signals and transmitted to indicate that that region of the raster is bright. The grey scale or brightness of an area is determined by the number of electrons required to restore the original electron charge.

The sharpness of the image will depend in part on the electrons from a dark region not diffusing across the surface to a light region, smearing the image edges. This is accomplished in the thin film deposition. Figure 6 shows a top and cross-sectional view of the antimony sulfide layer. Notice the elongated, nodular structure. Contact between adjacent nodules is minimal, while it is quite continuous between the vacuum surface and the transparent conductor. Antimony sulfide evaporates at less than 600°C, and although it breaks up during

evaporation into antimony, sulphur and various other combinations, it results in a deposited stoichiometric layer of Sb_2S_3. The nodular form arises from evaporation in 150 microns of argon. The evaporated atoms collide often, with argon and with each other, on their way to the substrate. They grow to be spherical, the size depending in part on the argon pressure. Elongation of the nodules takes place in a subsequent high vacuum evaporation on top of the spherical particles.

Energy Efficient Light Bulbs

The conventional incandescent light bulb only emits about 7% of the input energy as visible light. The other 93% is lost as heat. Most of this heat loss is in the form of infrared light which is transmitted from the filament through the glass bulb. If this energy could be reflected back to the filament by a coating inside the bulb it could be used to heat the filament, thus reducing the input energy required to maintain the filament temperature. Some researchers have reported a 50% reduction in power.[6] That is, the light output is equivalent to a 100 watt bulb with only 50 watts input.

Figure 7 shows the desired film structure. Thorington et al.[7] have shown that a three layer coating of titanium dioxide/silver/titanium dioxide will produce the desired reflection of infrared light while not interferring with the transmission of visible light. Although evaporation could be used to coat inside a glass envelope, the slow temperature rise and decay from any evaporator boat make thickness control difficult when layers are only 200 angstroms thick (about one millionth of an inch). The spherical geometry of the bulb is ideal for sputtering. The sputtering process is shown schematically in Figure 8. An r.f. or d.c. discharge is used to produce ions of an inert gas in a partially evacuated chamber. These ions, positively charged, are attracted to the cathode, collide with it and knock off neutral atoms of the material which is to be deposited. The cathode is made of or coated with the coating material. The object to be coated, or substrate, is usually located between the cathode and anode (in some cases the object to be coated is used as the anode). When the discharge

is turned off the deposition ceases instantly to provide good film thickness control. Adhesion is usually superior in sputtered films because ion bombardment cleans surfaces well. In addition, some of the deposited atoms are charged due to passing through the plasma and these produced strong chemical bonds with the substrate. A cathode, as source of materials, placed inside the bulb while an anode (a screen in this case) is placed outside, as in Figure 9. By using radio frequency (r.f.) rather than direct current (d.c.), the glass envelope does not significantly affect the plasma discharge. R.f. sputter coating can be carried out inside of an insulating vessel with one electrode on the outside. The source of material can be either a metal or an insulator coated on metal. For titanium dioxide deposition the source consisted of titanium dioxide coated onto a copper sphere. In r.f. sputtering this insulating layer on the cathode does not significantly affect the discharge and the material which is sputtered off is that which is exposed to the plasma, in this case titanium dioxide. The temperature rise of the glass bulb during deposition will only be a few hundred degrees centigrade. Thermal evaporation of insulators requires a very hot source, often greater than 2500°C which may overheat the substrate. A major advantage of r.f. sputtering is the capability of depositing refractory insulators at rather low temperature.

Thin Film Strain Gauges

Strain gauges are simply resistors.[8] When the resistor is stretched or compressed, it changes resistance value. The change in resistance usually follows the equation:

$$\frac{\frac{\Delta R}{R}}{\frac{\Delta L}{L}} \approx 2$$

where ΔR is the change in resistance, R is the resistance, ΔL is the change of length due to the stretching or compressing and L is the length. A 100 ohm resistor, one inch long, when length is changed by 0.001 inch will have a resistance change:

$$\Delta R = \frac{\Delta L \times R \times 2}{L}$$

$$= \frac{0.001 \times 100 \times 2}{1} = 0.2\Omega$$

When the strain gauge or resistor is bound tightly to another surface, the change in resistance can be related to the strain (elongation or compression) on that surface.

Usually strain gauges are made of wire or foil bonded to the surface of interest. In some applications the environment is too severe for the glue to hold and the bonded gauges are so large that they affect the results. In these cases thin film gauges can be fabricated directly on the test components. Sputtering is used to coat the various layers and simple photofabrication techniques, borrowed from microcircuit technology, are used to define the resistors. Figure 10 shows a schematic of the gauge and its four layers.

Jet engine components are thoroughly tested during design stages to assure that none of the components are being strained beyond the safe limits. The test environment in the compressor can be 450 psi, 650°C, more than 100,000 g's of acceleration, and with strains approaching the limits of the strongest metals. Under these conditions, the thin film gauge, being less than 0.0003 inch thick, becomes the surface of the blade, does not affect the aerodynamics of the gas flow[9] (and therefore does not affect the actual strain measurement). Gauge failure is only achieved when the surface itself fails by cracking. All other types of gauges, prefabricated and bonded to the surface, deteriorate under the severe test conditions.

In this example, r.f. sputtering was used to deposit aluminum oxide as well as the gauge alloys.

The atoms which are sputtered from the cathode collide many times with inert gas molecules before reaching the substrate because the pressure of inert gas is relatively great ($\sim 10\ \mu$) in the sputtering process. The atoms arriving at the substrate can arrive from many angles; therefore, it is not strictly a line-of-sight process, and can be used to coat around three-dimensional objects such as jet engine blades or even wires. The coating thickness change is significant on the back

sides of large objects, and it is necessary to rotate parts for uniform coverage.

Alloys are particularly different to evaporate with composition intact because the different components have different vapor pressures. Sputtering removes the surface of the cathode by bombardment, a few atom layers at a time, so that all the alloy atoms are sputtered with no composition change. This is a major advantage of r.f. and d.c. sputtering.

Summary

The evaporation of materials in high vacuum or at intermediate pressures of inert gas can be used to produce coatings with unusual but useful surface structure as in liquid crystal alignment coatings and photoconductor layers.

Sputtering can be used to deposit films in confined spaces where the object to be coated is the vacuum chamber. Sputtering is not a line-of-sight deposition and can be used to coat three-dimensional objects like jet engine blades. Refractory materials can be deposited at low temperatures, and alloys are easily reproduced in thin film form.

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9. P.M. Niskode, J.H. Foster, C.B. Jones, VI Interamerican Conference on Materials Technology, San Francisco, California, August 12, 1980.

Figure 1. Liquid crystal watch—display and electronics

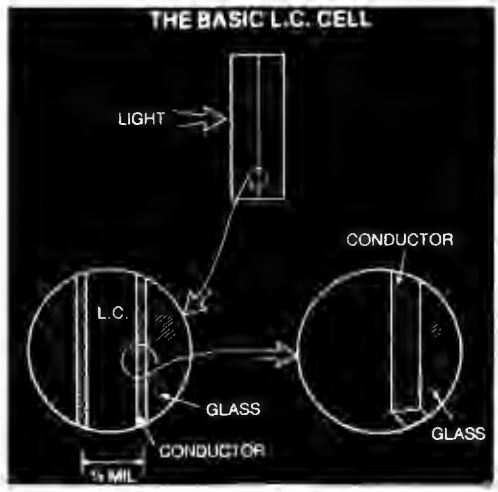


Figure 2. Cross section of a twisted nematic liquid crystal cell

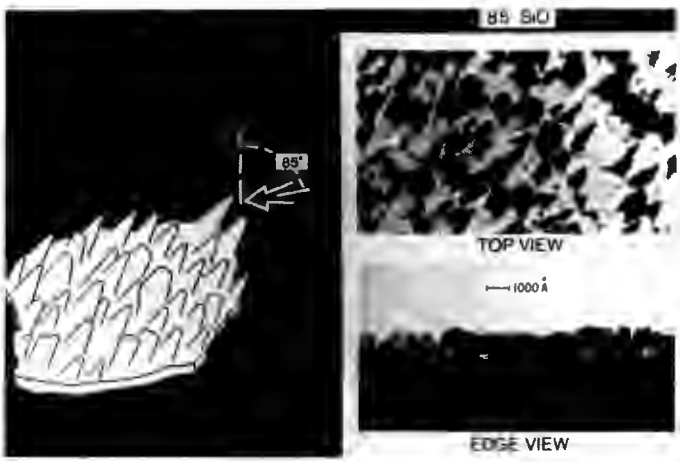


Figure 3. "Dragon scales" of silicon monoxide formed by grazing incidence. Left—artist's conception; right—transmission electron micrographs of the actual films.

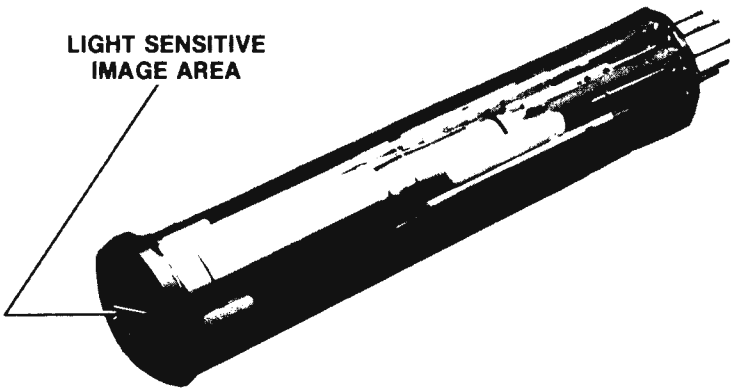


Figure 4. Black and white TV camera pick-up tube—light sensitive area is vapor deposited antimony sulphide on the inside of the face plate.

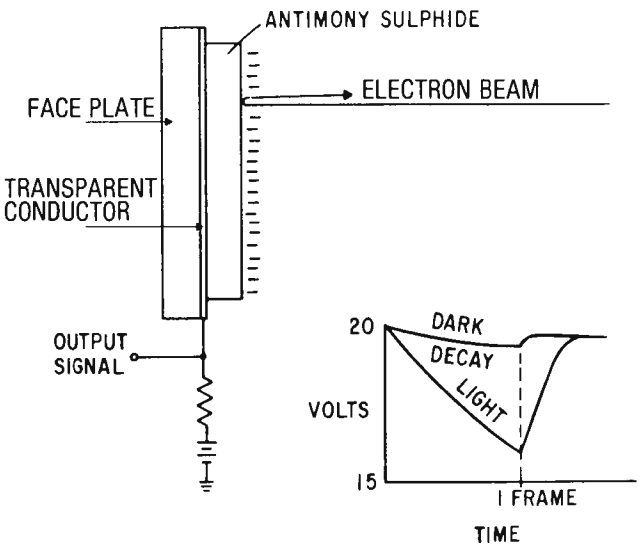


Figure 5. Schematic of the operation of a vidicon tube

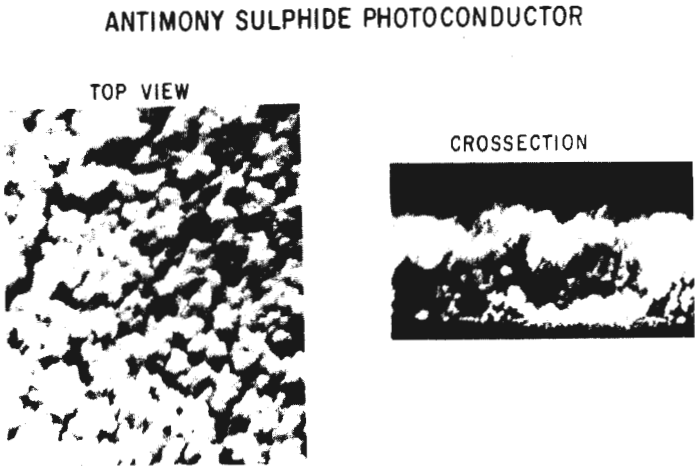


Figure 6. Electron photomicrograph of an antimony sulfide layer—nodules are about one micron in diameter (left—top view, right—cross section).

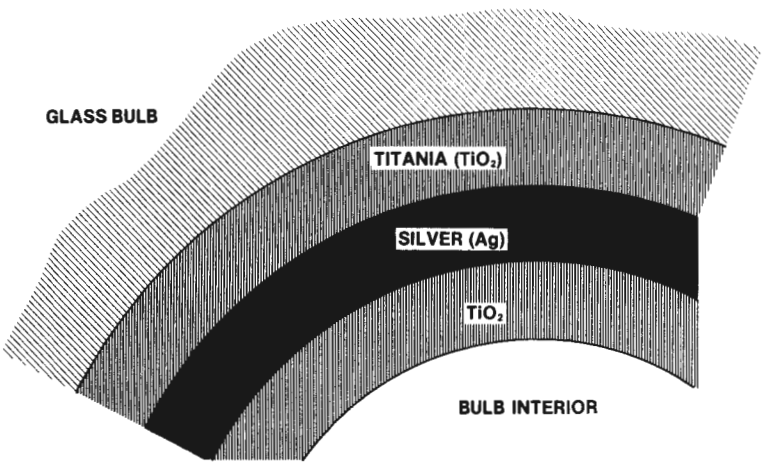


Figure 7. Thin film structure for improved lamp efficiency

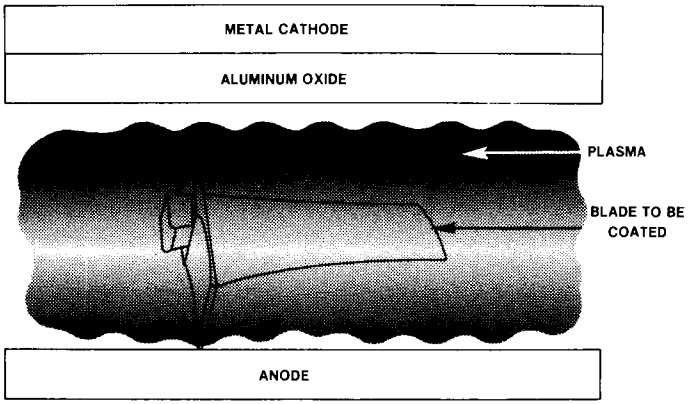


Figure 8. R.f. sputtering of aluminum oxide

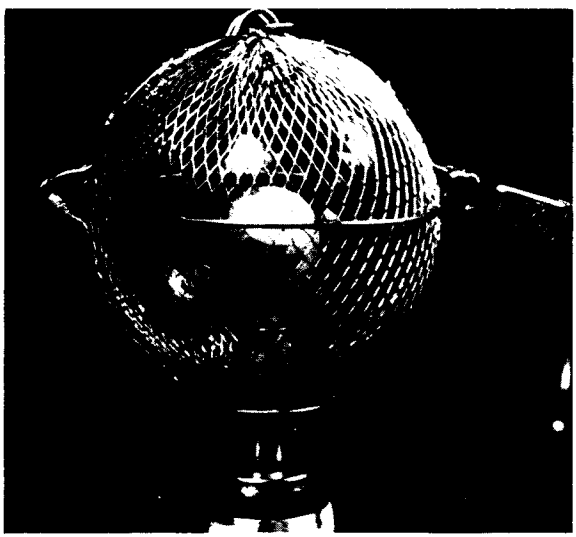


Figure 9. Sputtering inside a light bulb—the silver cathode is inside the bulb; the anode screen is on the outside.

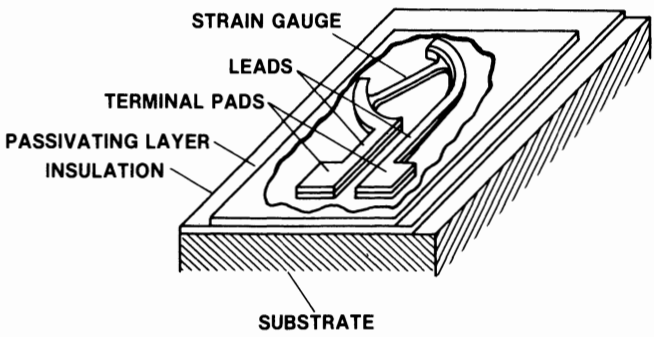


Figure 10. Artist's conception of a thin film strain gauge

TOOLS: A FEW MODIFICATIONS AND INNOVATIONS

Raymond L. Carew

This paper introduces several tools designed to make a few tasks simpler and safer. With the aid of drawings and pictures I hope to show how we have made use of these helpful items.

The tools that I will be showing you will be:

1. A tool for controlling or reshaping the outer diameter of glass tubing.
2. A beveling tool for reducing the diameter of windows and fritted discs.
3. A tool for holding a window or fritted disc in place inside a glass envelope for effective sealing.
4. An adaptation of a small dry belt-grinder to a wet-to-dry, special purpose unit.
5. A breath isolator; a unit used primarily where safety and system contamination are a possible problem.

The first tool which I will show you I will refer to as an "outer diameter control device" (Figure 1). I must first note that variations of this same tool have been talked about and written about before; however, the one which we have been using is different in a couple of ways (Figures 2 and 3). Ours mounts on one lathe, our Litton ESA. Our machine shop aided us in making the mount. Four bolts attach it firmly to the burner carriage on the lathe, making it quickly and easily attachable and detachable. The roller is hollow 304 stainless steel and rotates on bearings placed in the ends of two cantilevered arms. These cantilevered arms make it possible to use the tool on very small diameters, as well as very large diameters. The O.D. control device is mounted on a machine lathe tool holder, such that the center line of the glass tubing is on the back side. The machine lathe tool holder gives us fine adjustments and precisely repeatable diameters, as well as angles. We coat the stainless steel roller very lightly with boron nitride. We use both spinning and blowing methods to expand the glass as it heats to a point of touching the roller. For the internal pressure, instead of using our lungs we have found that

the use of dry nitrogen together with a high and low pressure regulator coupled with a flowmeter, gives good consistent results and fine control (Figures 4 and 5). One of the major reasons for developing this tool and technique was to produce conical sections for our Department of Physics (Figures 6, 7, and 8). These large conical sections were scored and thermally shocked at a nominal length to remove them from the end pieces. They were then ground to precise length dimensions, and given a reflective coating by evaporation. We produced forty cones, twenty each of two different sizes and angles. We worked closely with Dr. Michael Medinnis of our Department of Physics who will be using them in the study of high energy physics.

The high energy physicist seeks to understand the fundamental structure of matter. The experiment which will use the cones produced in our glass shop is concerned with the structure of the proton. To study this structure, protons are accelerated to very high energies and made to collide with other protons. The results of these collisions are sprays of many different types of particles. Most of these particles are extremely short-lived; and though they are traveling at velocities approaching that of light, they travel much less than one micron before decaying into more stable particles. The only charged particles which survive long enough to travel 10 or more meters are protons and 2 types of lighter particles called "pions" and "kaons."

It is important to distinguish between these three types of particles. And this can be accomplished by making use of the so-called Cerenkov-effect. It is well known that no particles can travel faster than the speed of light in a vacuum. However, when light travels through matter (such as air or glass) it slows down so that particles can in fact travel faster than the speed of light in a medium. In glass, light travels 2/3's as fast as in vacuum; in air light travels about .01% slower than in vacuum. When a particle travels faster than the speed of light, it produces light in ways analogous to the production of sound by an airplane traveling faster than the speed of sound. By measuring this light it is possible to distinguish between the three particles mentioned (i.e., protons, pions and kaons). The piece of

equipment that is used to measure this light is called a Cerenkov counter.

In general, the Cerenkov counter consists of a radiator in which particles produce light (in this case a volume 1 meter long by 1.5 meters by 3 meters in cross section is used), elliptically configured mirrors to focus the light (in this case, the mirror area is about 1.4m × 4m per mirror, with sixteen mirrors in the counter) and photomultiplier tubes to convert this light into an electric pulse.

As with sound produced by an airplane traveling faster than the speed of sound, the light produced in a Cerenkov counter is in the form of a shock wave. The elliptically figured mirrors focus this light to a ring (annulus) whose diameter can be large compared to the diameter of the available photomultiplier tubes. In our case the diameter of the Cerenkov ring is about nine inches whereas the largest available phototube which will suit our needs is only eight inches. In order to enlarge the useful area of the phototube, which acts as a funnel, it reflects the light from the focused Cerenknov ring into the surface of the phototube.

Because much of the light produced is in the near ultraviolet, care must be taken to produce a reflecting surface on the cone which reflects as far as possible into the ultraviolet. This is done by vacuum aluminizing the interior surface of the cones. One of the best substrates for the vacuum aluminization process is glass.

Another application of our O.D. control device is in the making of the tail sections of liquid helium dewars. As most of you are aware, there is a need many times to produce four concentric walls which must not touch each other within a confined space and yet retain strength enough to withstand the forces of static and dynamic vacuum. In these instances we need to know precisely what the outside diameter and wall thickness will be before we start (Figure 9). We have worked out a formula which makes trial and error obsolete.

The next tool which we will take a closer look at is the "Breath insolator, inert gas, blowing device." This device serves a number of useful functions. I must stop and give credit to an article in *R&D Magazine,* June 1967; our device is a

variation on that same theme (Figures 10 and 11). We have fixed up a very portable unit which can be used in labs where we make on-the-spot repairs to vacuum systems. The purposes which it serves are many. By using dry nitrogen, which is inexpensive and inert, we do not contaminate the system with our breath, which is probably very important to the researcher; but what is even more important to us is that anything which the system may have in it does not contaminate us. It keeps unhealthy vapors such as mercury and other undesirable contaminants from vaporizing and intermixing with our breath. When accurately adjusted the neoprene rubber diaphragms will flex and allow both positive and negative pressure to be applied to the system being prepared. We also use the "breath isolator device" when we are sealing dewars and other items into our own pumping system. It greatly reduces the pump-down time. Figure 12 is a diagram of the heart of our portable system.

My next subject deals with the conversion of a simple dry belt grinder device converted to wet use. It is inexpensive to make and yet it is a fast hard worker. We will disassemble and reassemble it piece by piece (Figures 13-19). As it sits now stripped of all of its PVC splash guards, it is easy to see that it is a simple one inch belt grinder found in many machine shops and hobby shops. Since we are interested in using it wet, we will quickly reassemble it and tell you that we hide our recirculating pump inside the cabinet below and use a water soluble oil to help keep the metal parts from corroding and rusting (Figure 20).

Now I would like for you to see our "disc-holding device" (Figure 21). We have a line drawing showing the basic breakdown of this tool. Its basic use is to hold, by means of a moderate vacuum, a glass disc or sintered glass disc inside a tube (Figures 22-27). With the use of various length extensions we can reach points very deep inside the cylinders. Another application, which I haven't attempted to show, is that one tube can be held concentrically inside another for that type of sealing operation.

The last tool which I will introduce you to I call a "beveling tool, or trimming tool" (Figure 28). We have a line drawing illustrating the basic parts of this tool. This is the third

generation of this tool, each incorporating some improvements on the previous ones. This beveling tool is the smaller of two sizes that we have made and used over the years. As shown on the line drawing, we use doublesealed bearings for longer life. Incidentally, since the bearings are the real effective force in this tool's efficiency, it has been made so that they are replaceable (Figures 29 and 30). In Figures 31-33, I have prepared a beveled edge on both sides of a glass disc. One thing I failed to show, but which I should mention, is that I used a very thin piece of rubber as an interface on both sides of the glass to prevent steel against glass contact. This tool serves the purpose of changing the diameter of an existing disc; you may change a disc to a square, or vice-versa; one may alter the size of a fritted, or sintered disc, or chamfer the edge of a piece of glass or metal.

It should be noted that I haven't spent an undue amount of time giving very detailed and specific dimensions. It is my contention that they are not complicated enough to necessitate explanation of specific dimensional characteristics; therefore I have chosen to emphasize their function rather than clutter the drawings with dimensions; they are not extremely important.

I would like to acknowledge Mr. Ed Wheeler, whom many of you may known; it was because of him that I made my start in scientific glassblowing and his encouragement that kept me in it during the very beginning; the late Vic Thomson with whom I worked for approximately five years; the people in our machine shop in the Department of Chemistry at UCLA; my colleagues who do the bulk of the work in our glass shop; the Department of Chemistry at UCLA; Dr. Christopher Foote, and Dr. Kyle Bayes, Chairman and Vice-Chairman respectively; and Mr. A. Barry Hoelsher, our laboratory manager, for their support which helped make this paper and this trip possible; and my lovely wife who, with patience and cooperation, made the preparation of this paper a lot easier. As I close I would like to share just one more thought with you, a thought which captures a feeling that a beginner may have in scientific glassblowing: "But when the heat has brought the glass into soft state, it is by no means easy so exactly to turn the tube at both ends alike, and so lightly yet equally hold them, that the

soft part shall retain its cylindrical shape; being neither twisted nor bent, nor elongated, nor thrust up." — Michael Faraday

Figure 1.

Figure 2.

Figure 3.

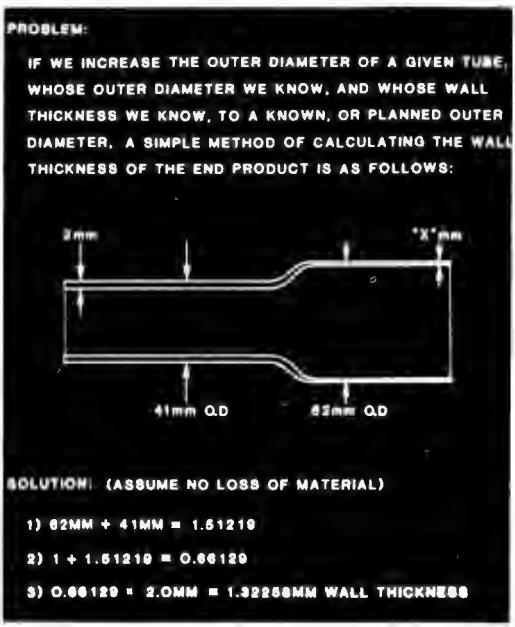
Figure 4.

Figure 5.

Figure 6.

Figure 7.

Figure 8.



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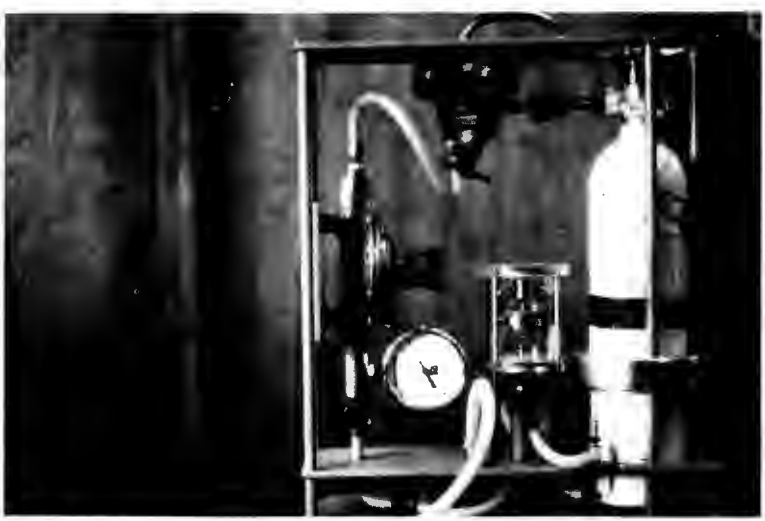

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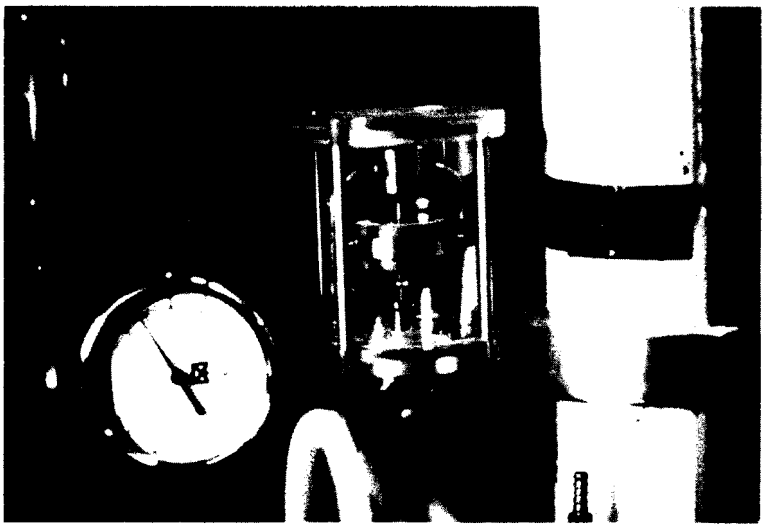

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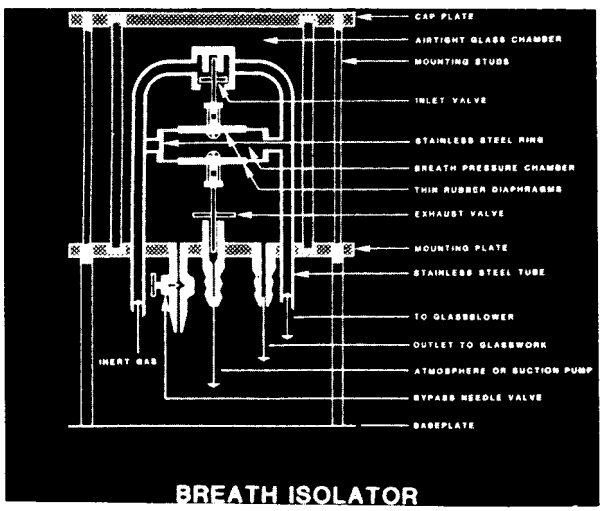

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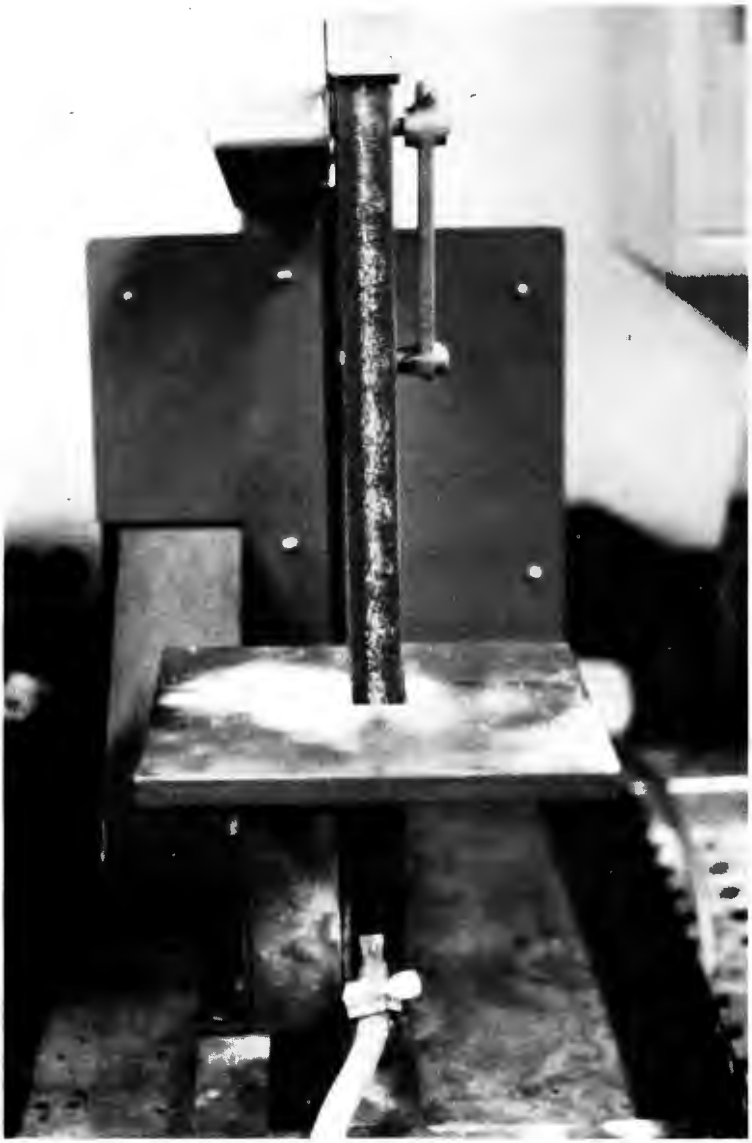

Figure 13.

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Figure 16.

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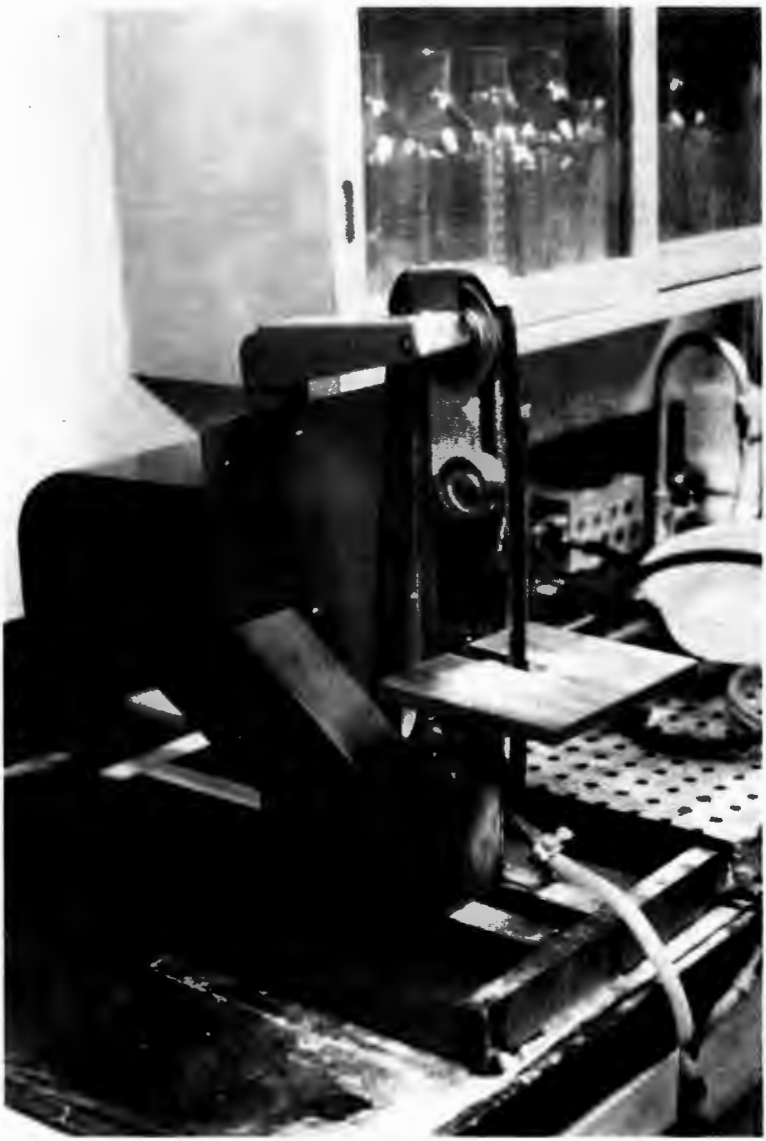
Figure 19.



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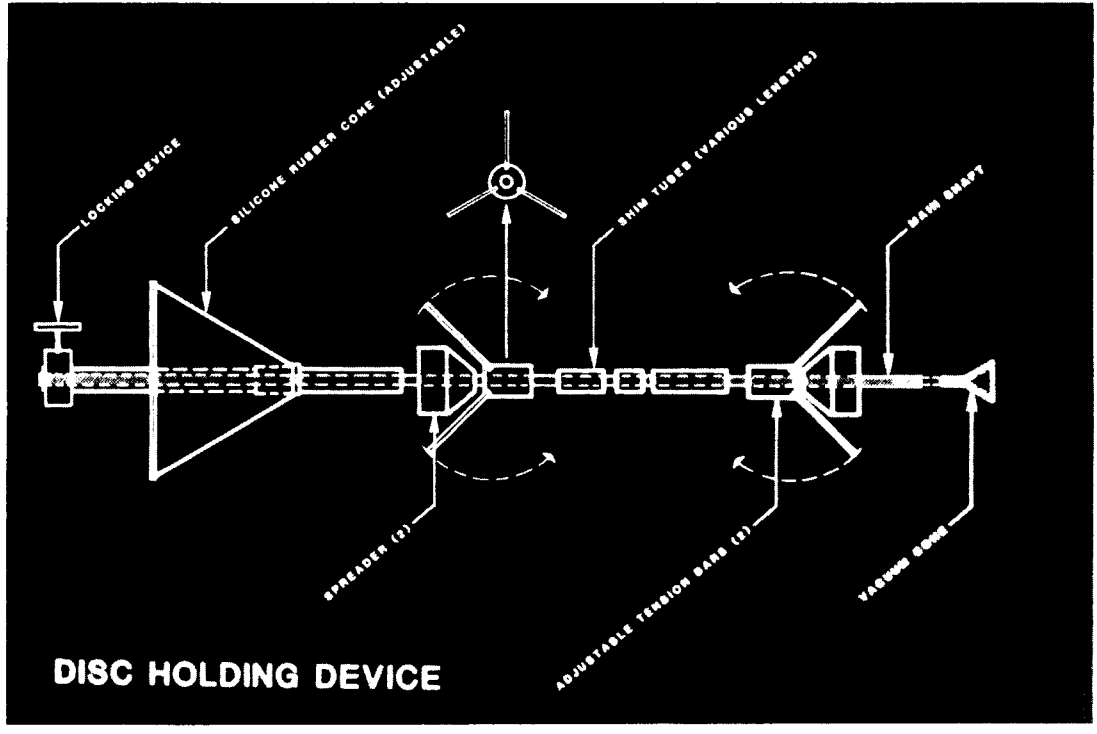
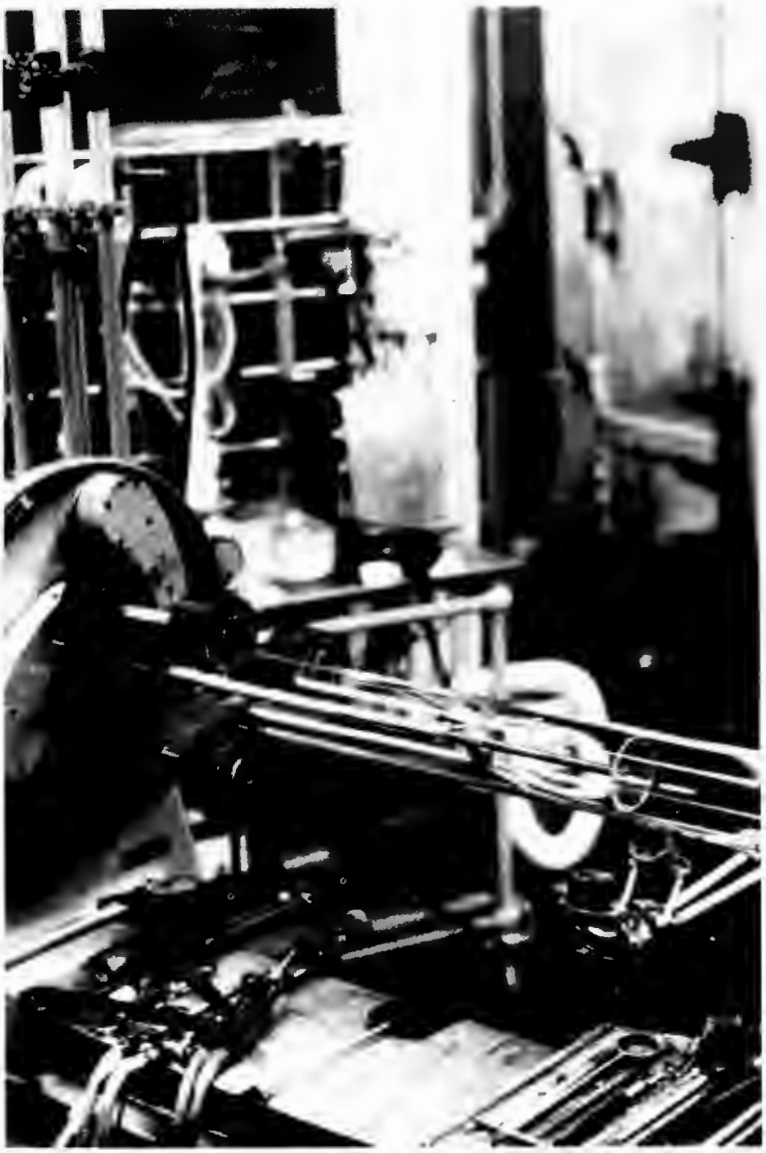

LOCKING DEVICE
SILICONE RUBBER CONE (ADJUSTABLE)
SHIM TUBES (VARIOUS LENGTHS)
MAIN SHAFT
SPREADER (2)
ADJUSTABLE TENSION BARS (2)
VACUUM CONE
DISC HOLDING DEVICE

Figure 21.

Figure 22.

Figure 23.



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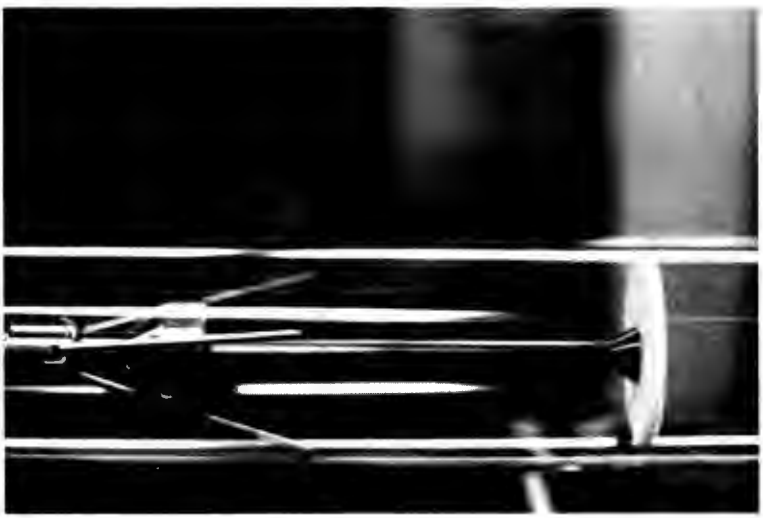

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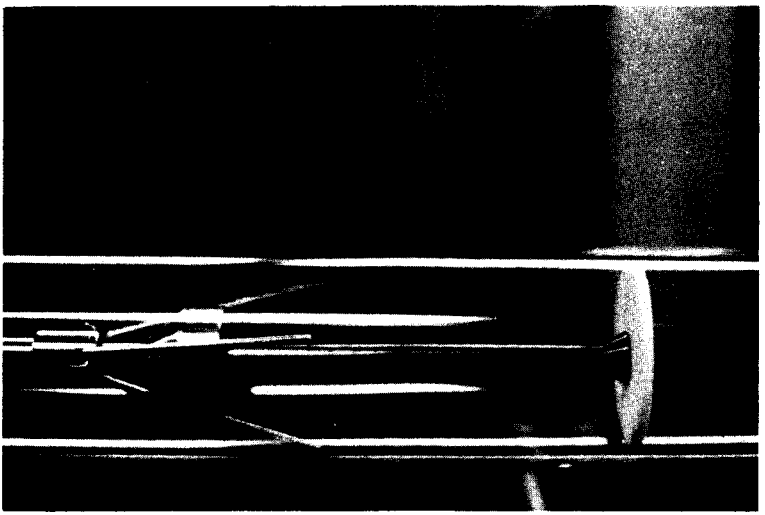

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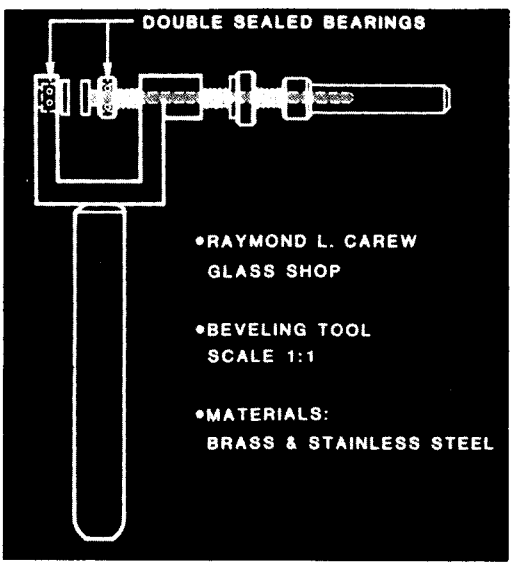

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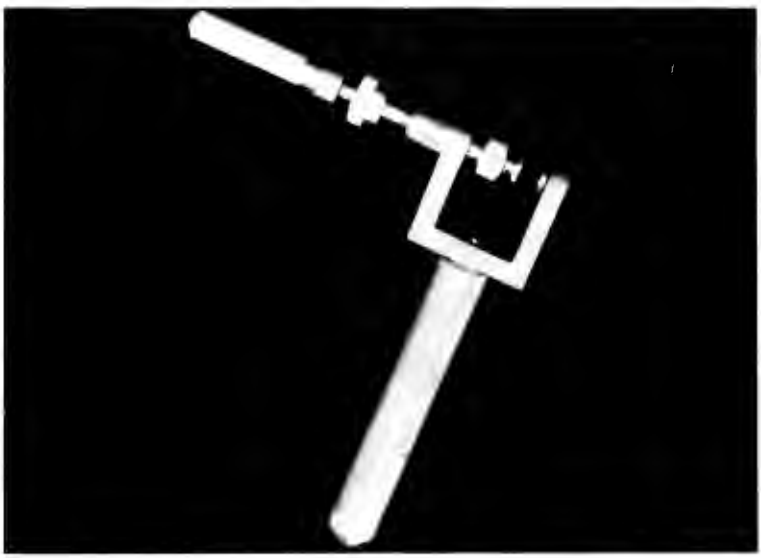


Figure 30.

Figure 31.

Figure 32.

Figure 33.

THE CHANGING ROLE OF THE PROFESSIONAL GLASSBLOWER

Randolph H. Searle

Why does the scientific glassblower need to be concerned with anything beyond his or her ability and acquired skills as a manipulator of glass? To be sure the scientific glassblower has made complex apparatuses involving dissimilar material seals and glasses of various compositions. So, after five or ten years of applied principles what more is required for professional performance?

Before these questions can be answered it is necessary to have a clear understanding of what scientific glassblowing is. The U.S. Department of Labor's Dictionary of Occupational Titles[1] defines a laboratory apparatus glassblower under the heading of Bench Work Occupations and a scientific glassblower under Ceramic Engineering Occupations.

Briefly, the function of the laboratory apparatus glassblower is to manipulate and blow glass by itself and in association with other materials and to make apparatus for specific needs. To do this the laboratory apparatus glassblower will develop customer's sketches into a working plan with the assistance of various available tools and, when finished, will inspect and test the product for adherence to specifications.

The scientific glassblower, on the other hand and in addition to the above functions, consults with scientists and engineers on the proposed operation, properties and design of apparatus using his knowledge of glass technology, effects of diverse environments and the various manipulative arts. He may be called upon to train laboratory apparatus glassblowers, prepare cost estimates and supply specifications for support services such as machine, welding, carpenter and electrical shops.

Now that the terms have been defined it is obvious that as the acknowledged "glass expert" in his or her respective institution or company, the scientific glassblower is expected to be knowledgeable in glass technology as well as a wide range of associated disciplines (Figure 1). This degree of

professionalism can only be achieved and maintained by considerable personal effort.

All too often individuals who have attained some pinnacle of success feel that there is nothing more to be done to change their position. This attitude could not be farther from the truth. As a service person it is your obligation to meet the needs of scientific research and development which is constantly changing as the thrust of business and society changes. Analyzing and developing new approaches for solving problems calls for wide experience and specialized training.

New insight can be acquired through extension courses, seminars, symposia, and libraries. Seminars such as those offered in conjunction with the Society[2] have proved to be effective in updating the glassblower's proficiency. One can keep up with current technology by scanning scientific journals or trade magazines or reading abstracts. Many scientific articles contain new apparatus or modifications and sources of materials that are useful in solving a variety of apparatus construction jobs. When you find good ideas, write them in a notebook and list the references so that you can find them again when they are needed. Make a habit of spending an hour or more per week at your library and, in addition, build a personal library of references such as those listed in Figure 2. A partial list of journals is given in Figure 3. An up-to-date collection of apparatus and supply catalogs is extremely useful when designing new equipment. Figure 4 contains an abbreviated list of catalogs. The time and money you spend acquiring these helps is an investment in your *own* future. The better informed you become the more valuable you will be to your employers.

The scientific glassblower has to be constantly aware of creeping obsolescence or change in the needs for his skills. These conditions can be anticipated and partially offset by becoming more diversified. Look for new ways for doing old jobs more efficiently. Investigate the use of alternate materials which eliminate some of the drawbacks of previous applications. Consider ways to make certain operations or products more safe and economical. Develop techniques that extend the basic manipulations.

Here I would like to cite an example of the use of an alternate material that helped solve a laboratory's problem. Several years ago a highly radioactive chemical process necessitated the use of a remote shielded facility and master-slave manipulators. As with many laboratory scale apparatus, altering the arrangement was frequently required. Because of the high radiation, the apparatus had to be made from stainless steel and glass. Most elastomers and greases could not be used to seal the joints because the materials broke down under the intense radiation. Even the glassware turned brown and had to be replaced when visibility was completely obscured.

Several solutions for disconnects were tried. Shielded "O" ring metal disconnects had much favor but were too awkward and fragile when connected between service lines and the glass pots. Low density, virgin, polyethylene plastic ball and socket joints suggested by the scientific glassblowers were accepted because of their flexibility and no need for grease. This plastic could also withstand the various laboratory reagents as well as the intense radiation. As tubing, this plastic could be formed much the same way as glass except at a much lower temperature (120°C). A hot air torch using nitrogen or argon gas serves as a very good heat source and keeps the melted plastic from charring.

Suitable spherical joints were compression, hand formed using polished aluminum molds. Later they were made in a bench model injection molding machine. Joints, thus formed, could be attached to polyethylene service lines using a suitable glass sleeve and a metal mandrel and fusing while compressing. On cooling, the exterior mold could be either slid off or crushed. *All* molds, of course, had to be related with a mold release so that the plastic would not stick. For the joints to make vacuum tight seals (10^{-2} torr) it was necessary to seal a polished socket joint on the glass parts. Glass socket joints that were tooled with a fire glazed finish and not ground were satisfactory for this service. Through experience, it was found that the addition of a short guide on the ball joints allowed them to be remotely placed in the corresponding glass socket joints without falling out until a clamp could be put on.

Standard spherical joint clamps were modified to aid in pickup by the manipulator fingers. There were then coated using a fluidized bed and powdered, virgin, low density polyethylene.

Another example where diversification helped solve a problem was when a request was made for a more rugged 45° mirror on a 0.060" diameter borescope. The front silvered mirrors were very susceptible to impact, scratches, and changes in temperature.

Having worked with lapidary equipment in a gem and mineral hobby it was decided to try a polished metal mirror since a front surfaced mirror is an aluminum coating on an optically polished glass substrate. In this case nichrome metal was chosen because this material was available and under most atmospheric conditions acquires only a very thin oxide film similar to aluminum.

Because of their small diameter the nichrome wire rods were bundled together and inserted into a suitable stainless steel tube holder that in turn would fit into the head of a faceting machine. Green lapidary wax was used to hold the rods in the tube holder. Later the finished mirrors were loosened by heat and the residual wax removed by methanol. Great care had to be exercised to prevent cross contamination of grinding and polishing compounds. Each time a finer grit or polish was used the bundle of rods still in the faceting had was placed in a beaker of clean water in an ultrasonic cleaner to remove all traces of grit or polish. Final polish was made with 1/4 micron alumina specially prepared for this purpose. Diamond was used for the rough grinding.

The finished mirrors were flat, without rounded edges because they were bundled during the grinding and polishing process. Each mirror was assembled in its borescope tube and held in place by a laser spot weld suitably located so as not to distort or discolor the mirror surface.

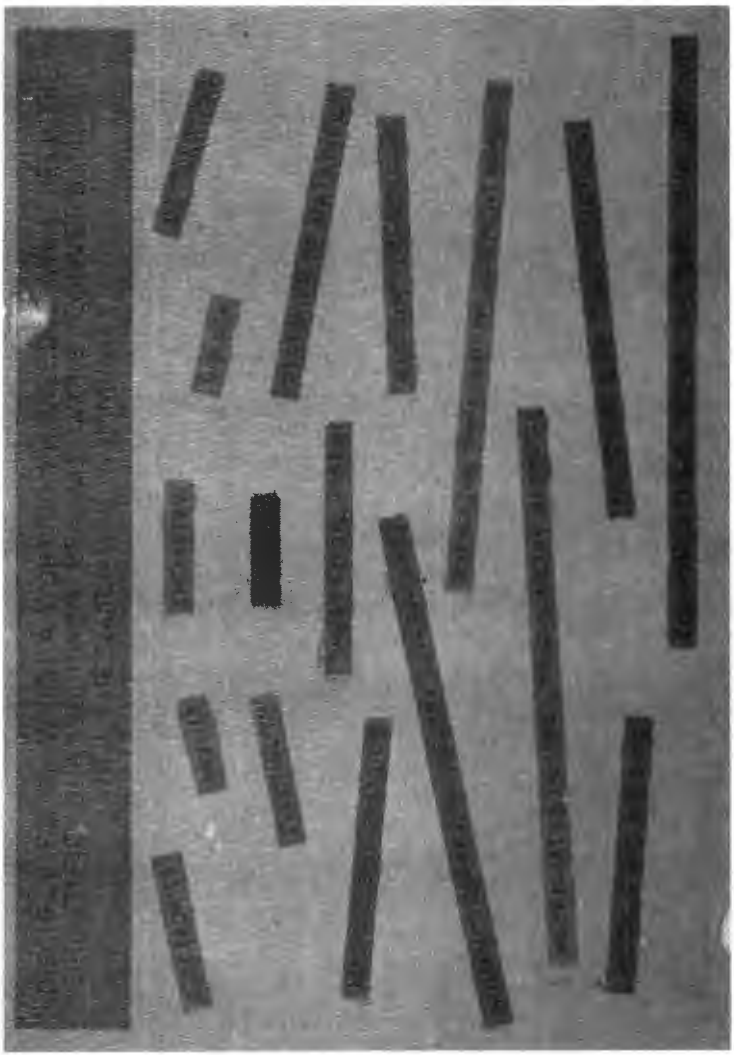
In conclusion scientific research is constantly probing the unknown for answers. Products and processes, unthought of today, may become readily available and routine in less than a generation. It is not enough for the scientific glassblower to become a skillful glass manipulator, he must expand his

horizon to include a good working knowledge of a variety of disciplines. As a professional he should be able to build on the past, organize materials and ideas, and pioneer new approaches for solving assigned proposals. Now is the opportunity to put meaning into the term "glass expert." Become a contributor to the team that is pushing back the frontiers of science and industry! Be innovative and flexible!

References

1. Published by the Dept. of Labor and available at the U.S. Government Printing Office.
2. American Scientific Glassblowers Society.

June 1980



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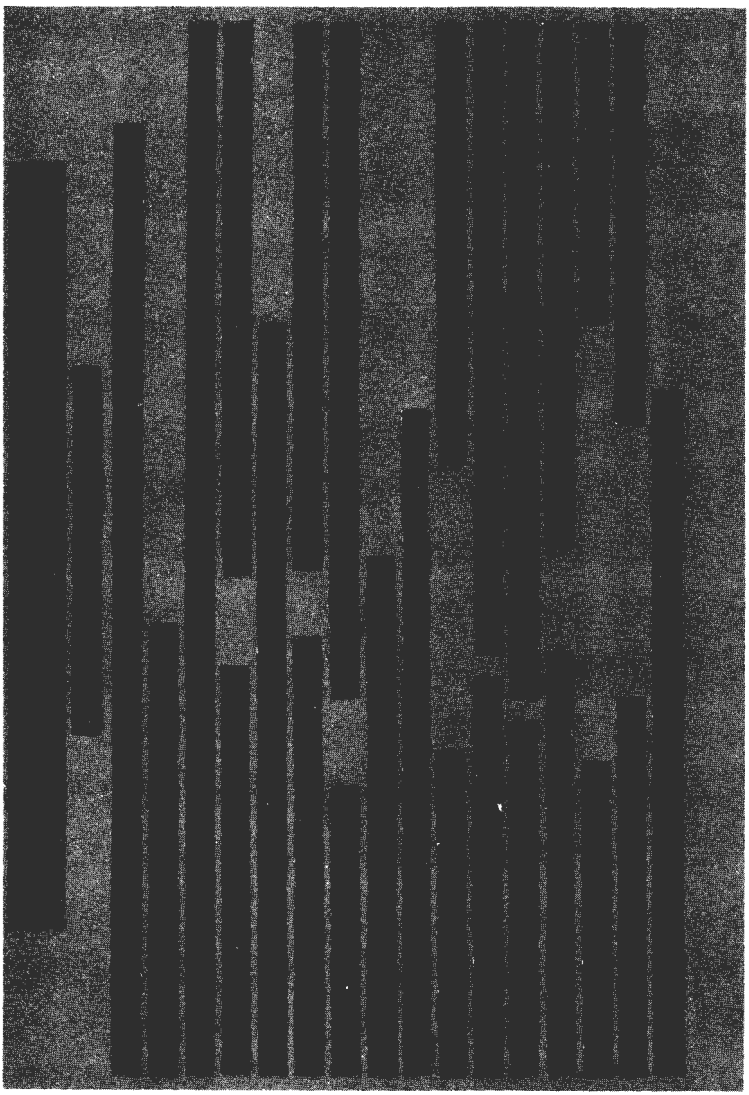

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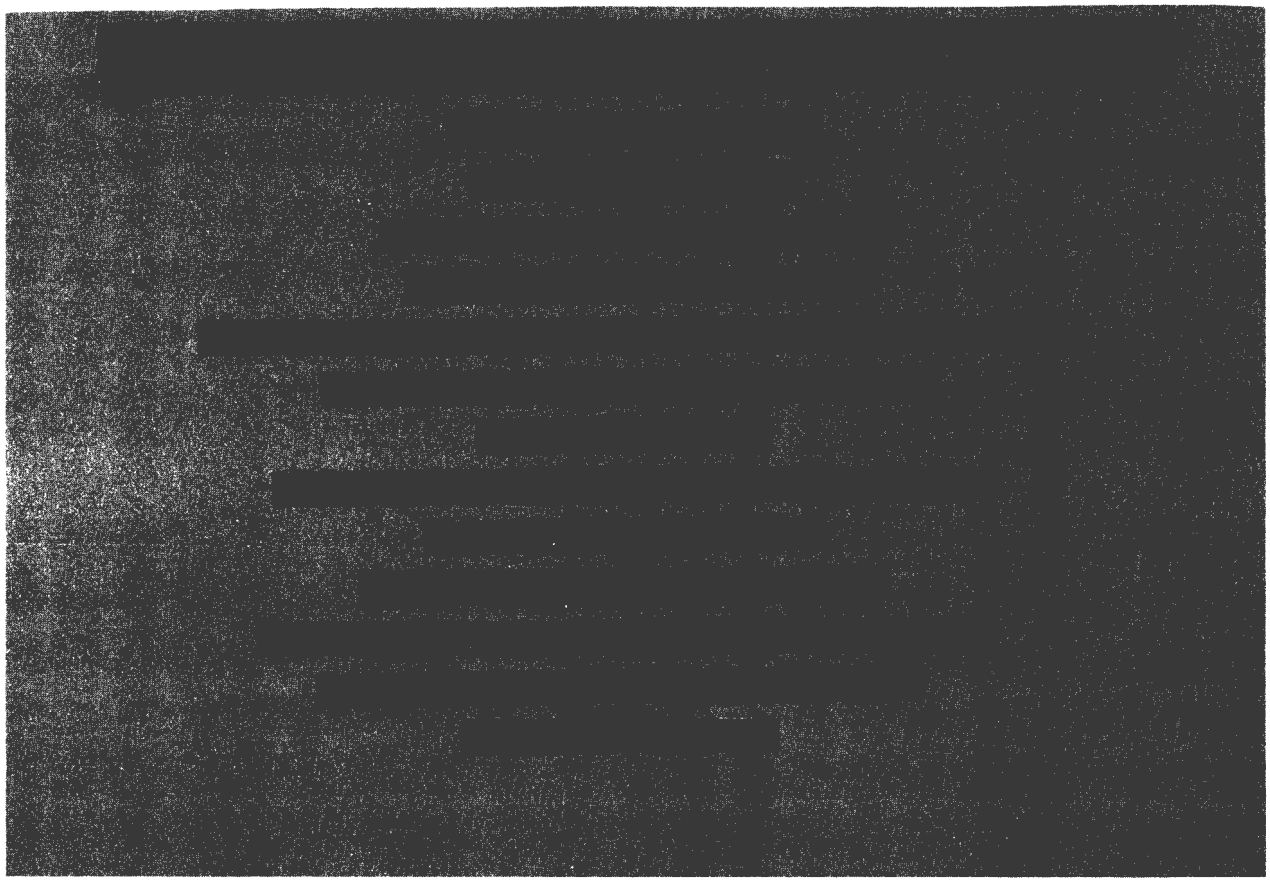

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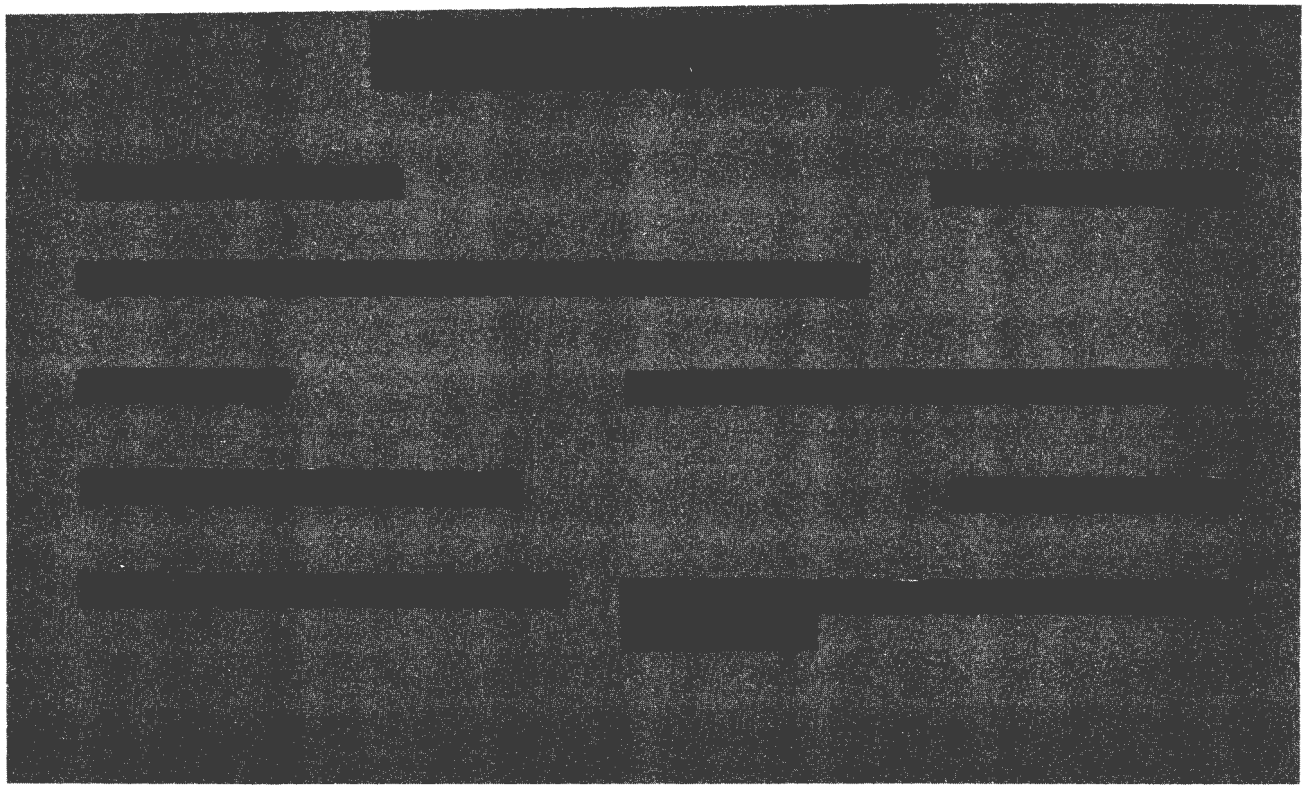


Figure 4.

ULTRASONIC MACHINING HAS BECOME AN INVALUABLE ADDITION TO THE GLASSBLOWING SERVICES AT LAWRENCE LIVERMORE LABORATORY

Hartford L. Rutan

Lawrence Livermore Laboratory

My name is Hartford Rutan, known to most as "Root" (Figure 1). I am a Tech Specialist at the Lawrence Livermore Laboratory, a part of the University of California. My main job responsibilities are Glassblowing. I have 32 years experience in Glassblowing, with the last 19 years at Livermore. Over the past 3-1/2 years, a great deal of my time has been involved with ultrasonic machining.

This is what I would like to speak about today (Figure 2). Ultrasonic machining: An invaluable addition to the services rendered by the Lawrence Livermore Laboratory Glass Shop.

I am sure that two words that I have used arouse some question in your mind: Glassblowing and Machining.

For a brief moment, each of you scan your daily routine and you will find that you are not only involved in blowing glass, but are involved in the design, cutting, grinding, vacuum technology and may other phases too numerous to mention. The term Glassblower is archaic, for the customer today makes demands on us far above just blowing glass.

This talk will be divided into three parts (Figure 3). I will answer the questions, Part 1. What is it? How does it work? and relate to you in Part 2 some of the applications at Livermore, and in Part 3 show some of the benefits derived.

Part 1. What Is It? How Does It Work?

Ultrasonic machining (Figure 4) could be compared to a pneumatic drill. A shaped tool is attached to the driver and the up and down motion chips away the material to form the desired cavity.

In reality, ultrasonic machining is the use of ultrasonic vibrations for the purpose of machining hard and brittle materials (Figure 5). This is done by coupling an electronic generator, a magnetostrictive package, a transmitting cone and a toolholder-tool combination.

The electronic generator converts 110 or 220 volt, 3 phase, 60 cycle A.C. into high frequency power. This power is delivered to the magnetostrictive package by means of an exciting coil. The package is solidly connected to the transmitting cone and the two together are called the transducer. The transducer converts the high frequency power into mechanical energy, oscillating linearly 20,000 times per second. A tool is solidly connected to the toolholder which is mechanically connected to the transducer. The tool holder-tool combination receives the mechanical energy from the transducer.

The length of the stroke at the tool face is determined by the type of toolholder selected (Figure 6). There are two types: Non-amplifier and amplifier.

The non-amplifier toolholder is merely an extension of the transducer (Figure 7), varying in size from 1" to 3" in diameter and producing a stroke the same as the transducer between 0.0008 and 0.001 of an inch.

The amplifier toolholder (Figure 8) ranges from 1/4" to 3 1/2" in diameter and uses three basic shapes: the double cylinder, the tapered cone and the chisel. This toolholder expands and contracts within its own length, thus amplifying the stroke produced by the transducer to a maximum of 0.0025".

The simple ABC's (Figure 9) of ultrasonic machining are:

A. Abrasive
B. Bombardment
C. Cavitation

A. Abrasive. The most important item of the whole process of ultrasonic machining is the abrasive. The abrasive, in a slurry, is fed to the workpiece by a recirculating system at approximately 7-1/2 gallons per minute. This quantity also acts as a coolant for the tool and workpiece. The abrasive grains are the cutting edge of the tool, therefore the abrasive controls the

size, the finish and the cutting rate. The most universally used abrasives are boron carbide, silicon carbide and aluminum oxide.

B. Bombardment. A tool stroke of a few thousandths of an inch and oscillating linearly approximately 20,000 times per second, drives the abrasive grains. The shape of the tool determines the precise pattern of the abrasive against the workpiece.

C. Cavitation. The extremely fast motion of the tool face produces cavitation of the abrasive liquid. This turbulent action of the liquid acts as a pump to feed the abrasive to the workpiece and to remove the minutely cut particles from the cutting area. This is also where the machine derives the name "Cavitron."

Figure 10 is a picture of a typical 1000 watt Cavitron and the one in use at Livermore today. The ultrasonics are affixed to a standard Bridgeport mill. There are smaller as well as larger machines available. This machine was manufactured by Sheffield Corp. and is now made by Bullen Ultrasonics in Ohio. There are two other machines that I know of: Rayathon and Branson; although they differ in shape and size, they use the ultrasonic process for machining.

Notice the slurry recirculating system; also we have added a rotary table for special applications, travel dial indicators, frequency counter and vacuum assist. The vacuum assist allows us to move the slurry into the cavity and remove the cut particles faster.

We have also added a micrometer feed and limit control and the Sonitrol (Figure 11). The Sonitrol is an automatic feed control.

Here the face plate is removed (Figure 12) so that you can see the cooling jacket which houses the magnetostrictive package. We use a recirculating system for this also. If the machine is put into continuous operation it might be necessary to add a refrigerant.

Part 2. Applications at Livermore

A. Slotting
B. Precision holes
C. Small holes and blind holes
D. Other applications (Figure 13)

A. Slotting. A request came to us to make a quartz cell. The requirements were: Starting with a piece of quartz 1" by 4" and 0.060" thick. This piece was to have two slots 0.118" wide and 0.118" apart and 0.032" deep. The two slots were to be parallel and 3-1/4" long. All tolerances were plus or minus 0.0005". The bottom of the slot was to be flat and parallel to the surface. The corners formed at the bottom were to be square. A very important factor was that a number of these cells would be needed and therefore must be easily reproducible. The job was accomplished by making a stainless steel tool with precision accuracy to the necessary shape, affixing it to a toolholder and with micrometer feed control, ultrasonically machining the cell in one operation. The customer requirements were such that no conventional method could be used.

We have also machined a 0.010" slot across the face of a micropipet for a needed application (Figure 15). The slot is meant to be off center; I wouldn't want you to think that we missed that far.

Figure 16 shows a curved slot 1/8" wide, approximately 1" long on a 14 mm radius. This was machined through a concave, convex reflector 0.120" thick. The highly polished aluminum surface was not disturbed. Note the reflection of the tool on the part.

B. Precision holes. Our laser group asked if we could put some small holes through 2" quartz discs, 1/2" thick at a 45 degree angle (Figure 17). One surface of the discs were optically polished to a flatness of lambda over 20, or approximately one millionth of an inch. This flatness could not be disturbed. By design, it was necessary to machine these holes starting from the back surface. First a 3/8" diameter hole was machined to within 0.062" of the polished surface, then the small hole was machined through the disc. There could be no chipping where the small hole came through the polished

surface. A number of hole sizes were machined from 0.022" to 0.108" in diameter, with a tolerance of plus or minus 0.0005".

These holes (Figure 18) could have been done by conventional methods, but it was found that the conventional method disrupted the surface flatness which meant that the surface would have to be repolished.

Ultrasonic machining causes no disturbance to the polished surface, which meant that the customer was able to purchase commercially produced discs and have the Glass Shop put the holes through at a fraction of the cost of conventional methods.

Figure 19 is an interferometer picture of the disc before and after machining, showing no optical change.

We also machined elliptical holes through like substrates in the same manner (Figure 20). The X and Y dimensions of the ellipse were 0.039" by 0.159". There is no other method for producing this type of hole.

The machining of scraper mirrors, the name of these parts, is a routine operation at Livermore today.

*C. **Small holes and blind holes.*** Routinely we machine holes of less than 0.030" in glass quartz, ceramic and thin metal pieces for our own use and to assist other groups (Figure 21). Small holes are probably our most challenging and most requested service of the Cavitron. We have not had the time to investigate any smaller size, but I understand that others have machined holes of 0.005" diameter.

A special job for Sandia of Livermore requires the machining of three blind holes and one 0.010" hole through a 0.010" face at a 45 degree angle. The hole part was machined ultrasonically except the OD. A piece of heavy wall quartz was selected and sealed off at one end. The ID was machined to the shape shown in one operation with a tolerance of plus or minus 0.01". The sealed end was then machined to leave a face of 0.010". Two 0.030" holes were machined at the outer perimeter on the center line, to a depth of 0.050" with a flat bottom. A 0.010" hole was then machined through the 0.010" face at a 45 degree angle, on the same center line. The edges of the hole were to be chip free on both the entry and exit surface. I

know of no other way that this total result could have been accomplished.

Figure 22 is an end view of the actual part magnified many times. Note the small hole and remember that it is only 0.010" in diameter.

D. Other applications. The machining of multiple holes in a precise pattern at the same time is one of the unique capabilities of ultrasonic machining (Figure 23). Here we machined six 0.060" and six 0.040" holes through a 1/4" disc in 75% less time than it would have taken to drill the holes one at a time.

The precision of the hole pattern does not vary from part to part due to the fact that all holes were machined at the same time, making all the parts interchangeable.

These are some examples produced to show the versatility of ultrasonic machining (Figure 24). In the foreground is a shape used for electrode holders in a special laser. Not only does the shape hold the electrodes but it also establishes precise spacing between them.

Note the square hole and the hex hole an observe the sharpness of the corners. The winged shape was used to make impeller blades. The five slots were machined with a single tool. A unique pattern was made by soldering a number of copper tubes together and then attaching them to a toolholder. Lastly, to show that the machine is not limited to glass, we have machined a 1/4" hole through a piece of pure tungsten carbide. This was done in less than 20 minutes.

Part 3. Benefits

A. Tolerance. The precision and finish of ultrasonic machining depends on the size and finish of the tools, fineness of the abrasive, and the material being machined (Figure 25). A 25 micro-inch finish may be obtained with 240 grit with a fast cutting rate. A 10 micro-inch finish requires up to 800 grit and is much slower. Tolerances of plus or minus 0.001" are generally obtained with 240 grit and tolerances of plus or minus 0.0005" or better may be obtained with finer abrasives as secondary operations, all based on the substrate being machined.

B. Materials. Materials readily machined ultrasonically are those which were both hard and brittle and those which combine hardness with high density (Figure 26). Typical are carbides, ferrites, silicone, silicon nitrite, aluminum oxide, glass and quartz. Ultrasonic machining is a non-thermal, non-chemical, non-electrical process. Consequently, there can be no change in the metallurgical, chemical or physical properties of the material being machined.

C. Shapes and sizes. As you have heard and seen here today, ultrasonic machining is only limited in size and shape by the size of the available toolholder and the ability of producing the necessary tool (Figure 27).

D. Time. I could bore you with a number of charts and graphs of time studies done by myself and many others now using ultrasonic machining. Suffice it to say that all studies prove that ultrasonic machining has been found to be 40 to 80% faster than conventional methods of machining glass and quartz (Figure 28). Percentages vary for other materials. Some examples: One 0.040" hole through 1/4" glass took 15 minutes using 600 grit. Twelve holes were put through at the same time in less than 40 minutes. 5/8" holes were machined through 0.600" glass in less than 3 minutes. 3/8" counterbores to a depth of 0.200" were done in 30 seconds. These are just a few examples but will give you some idea of the value of the time-saving capabilities of ultrasonic machining.

E. Safety. The ultrasonic machining process is one of the safest machining techniques known (Figure 29). Electrically: The generator has a series of interlocks to prevent the operator from contacting the high voltage. Mechanically: The only moving part during operation is the downward movement of the transducer unit. This movement is controlled either by counterbalance or automatic feed. The counterbalance exerts no undue pressure and the automatic feed is a pressure sensing unit which retracts when it feels resistance.

The only danger point is the vibrating toolholder-tool combination: Excessive physical contact at this point can result in tissue damage. However, once the machine is operating, there is ro reason that the operator should come in contact with the toolholder-tool combination. Airborne noise can cause discomfort if the machine is not properly tuned.

F. No other way. The routine machining of various hole and disc sizes can be done fast and accurate, but he use of ultrasonics for this purpose is impractical unless there are large quantities (Figure 30). The value of ultrasonic machining is doing the jobs that cannot be done any other way. In the machining of small holed, blind holes, multiple hole patterns, unique shapes in non-conductive materials with high precision and a rapid cutting rate, there is no other way.

Concluding my presentation today, let me say that I do not represent ay manufacturer of ultrasonic equipment, but I must acknowledge Bullen Ultrasonics of Eaton, Ohio, for the training they gave me, their cooperation on problems that I have had and the use of some of the information contained in this report (Figure 31).

I do not expect, nor do I suggest that you return to your job and proceed to purchase ultrasonic equipment. My intention for giving this talk is to familiarize you with ultrasonic machining in general.

Hopefully I have answered the questions (Figure 32):

1. What is it? How does it work? by relating to you the
2. Applications at Livermore and explaining the
3. Benefits derived.

Above all, it has become an invaluable addition to the services rendered by the Lawrence Livermore Laboratory Glass Shop (Figure 33).

Figure 1.

Figure 2.

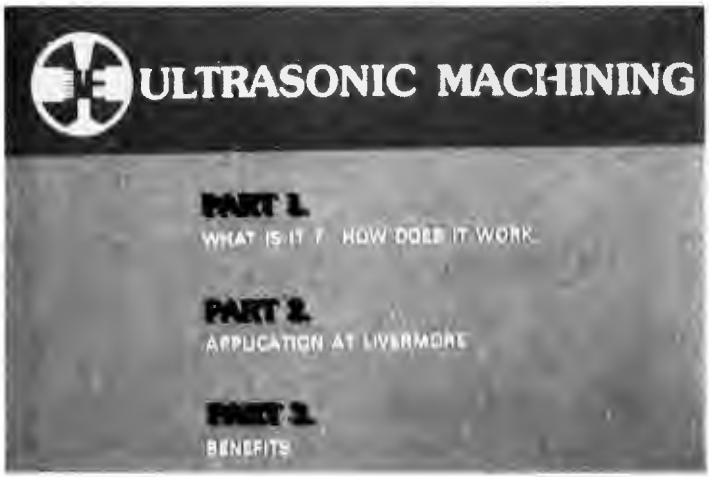


Figure 3.

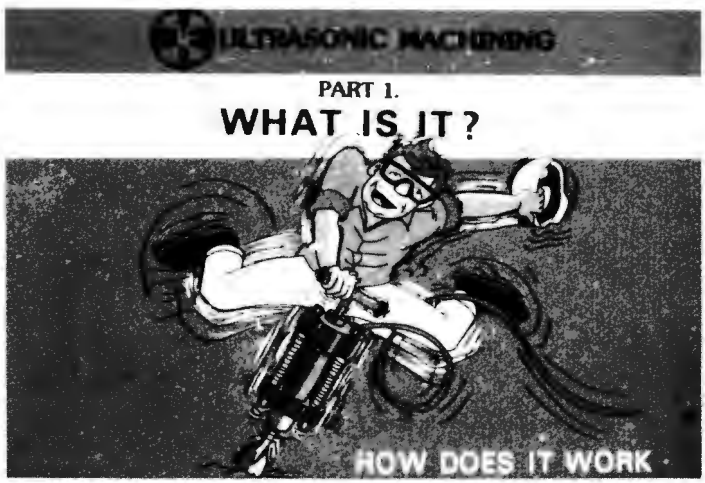
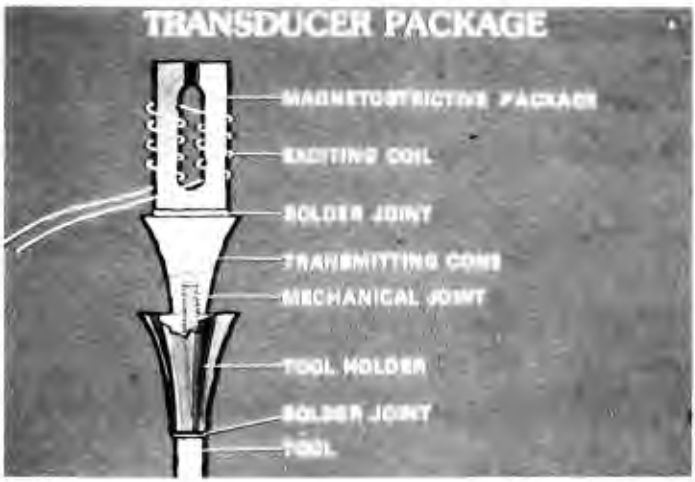


Figure 4.



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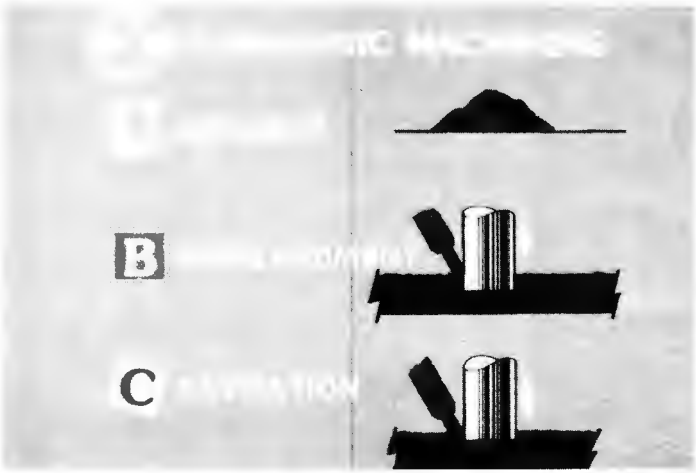

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Figure 10.

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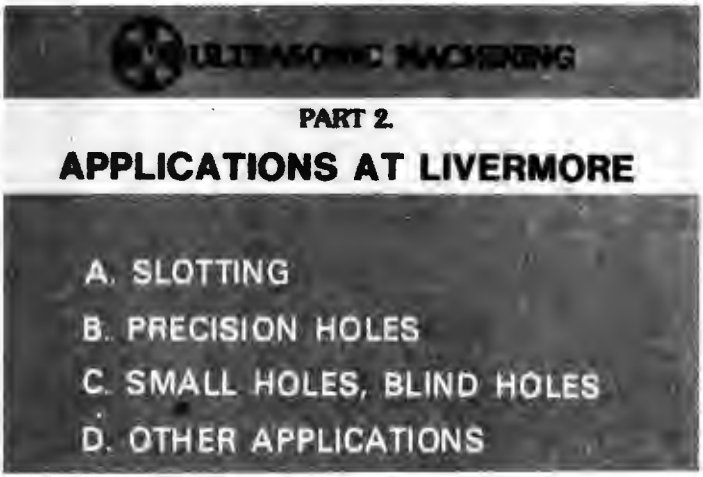

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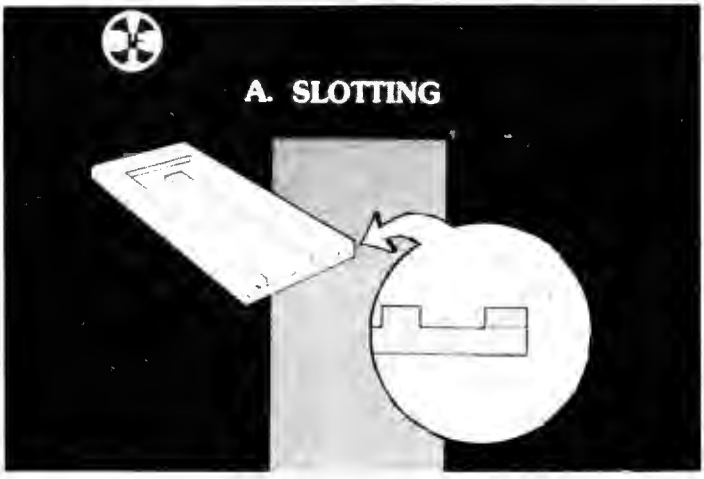

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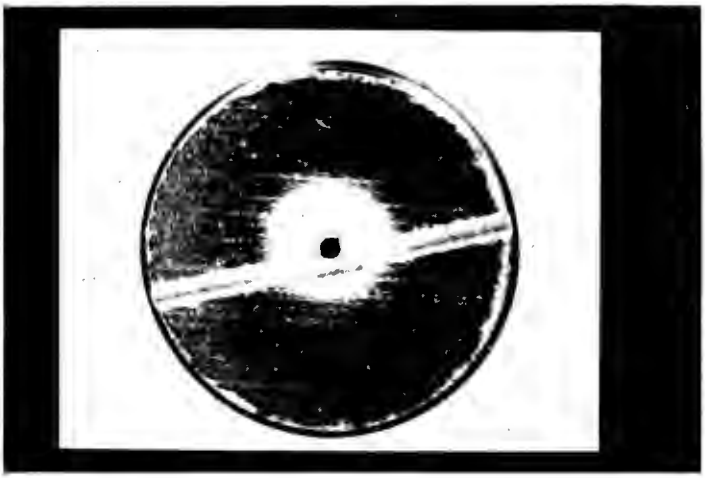

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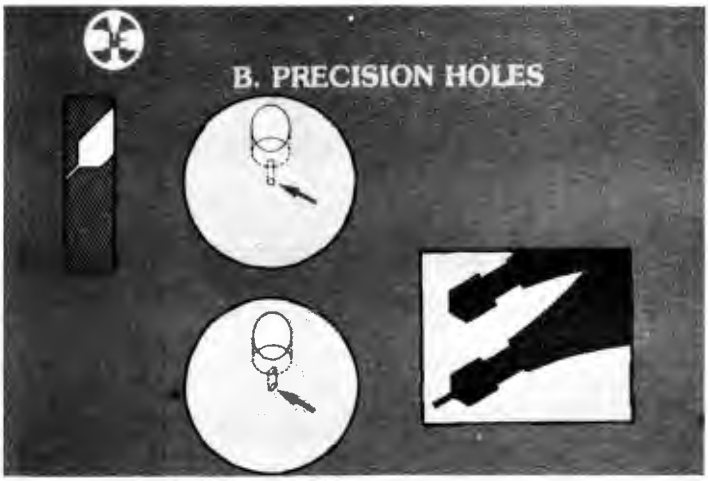

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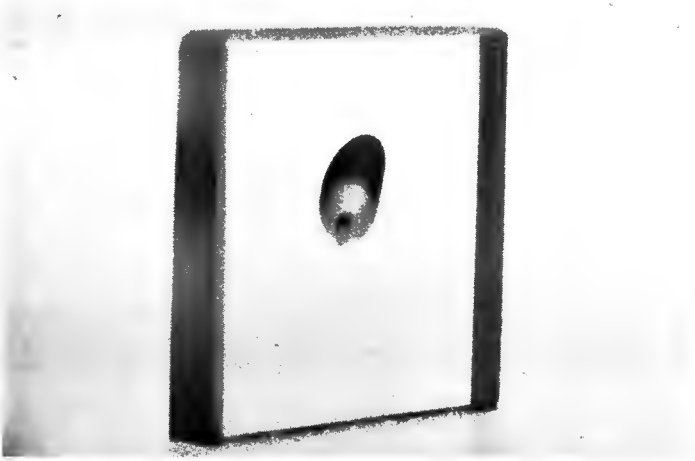

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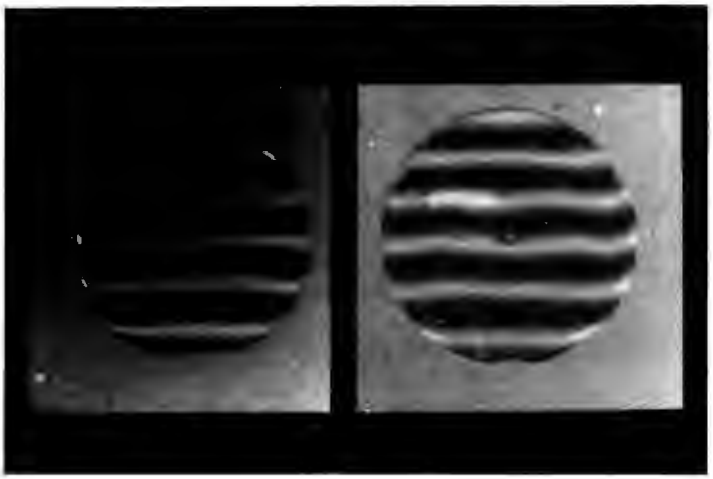

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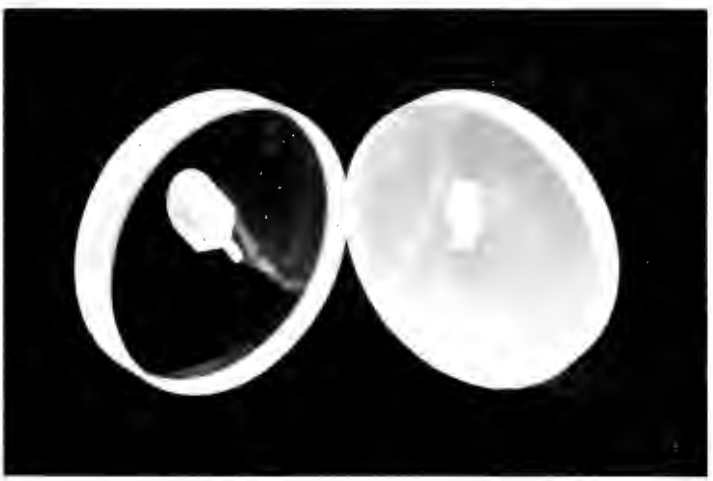

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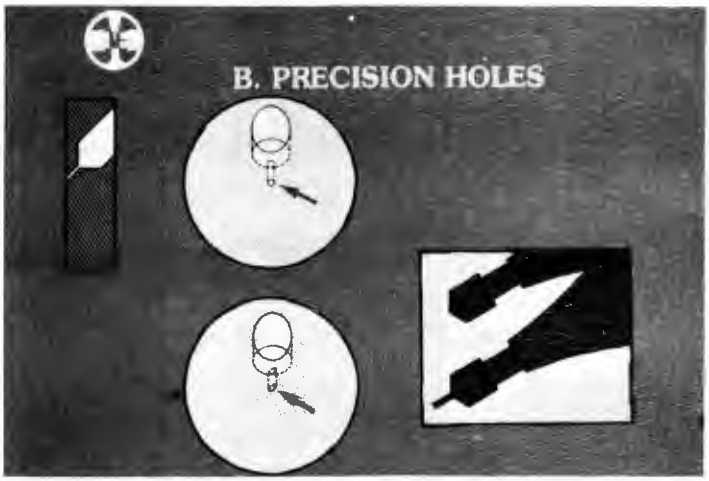

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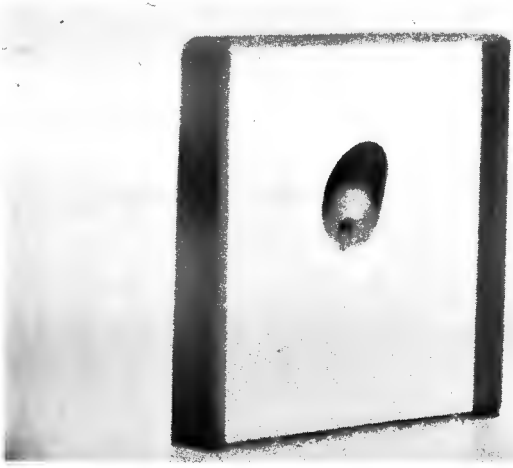

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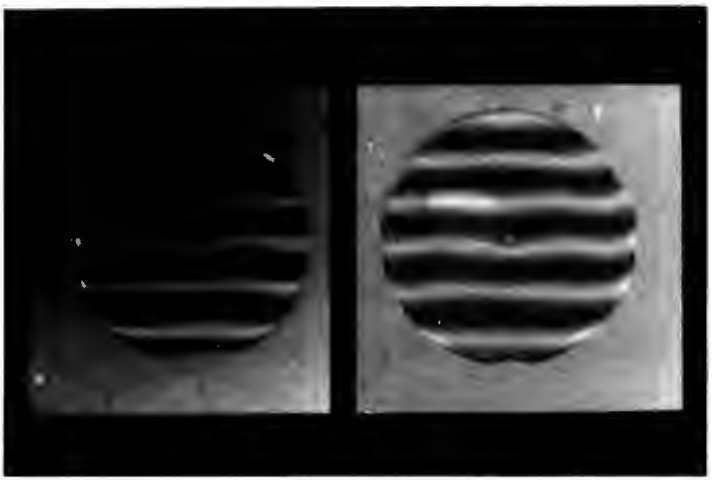

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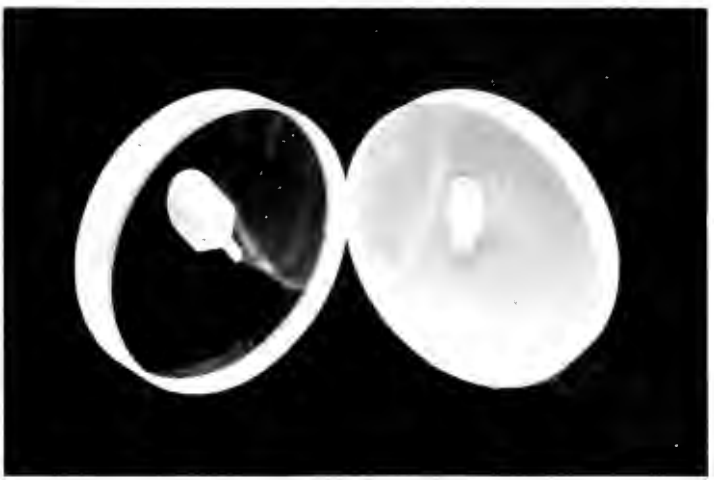

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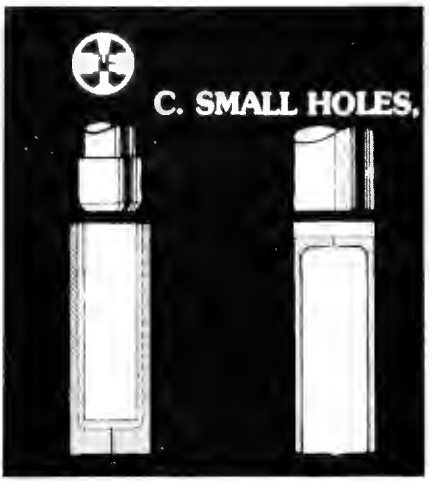

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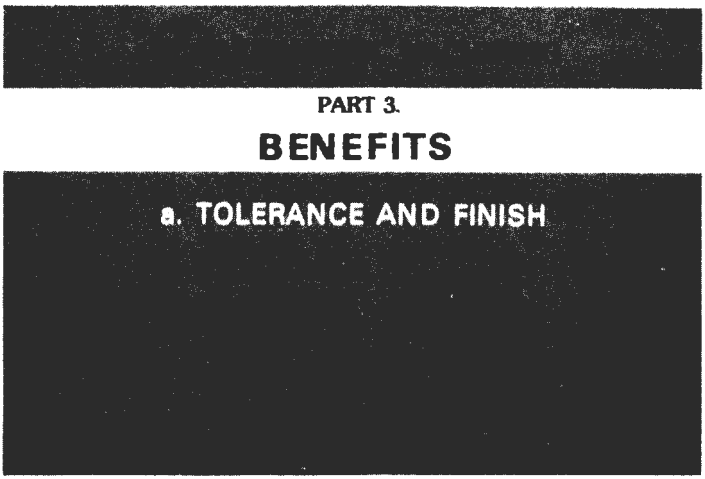

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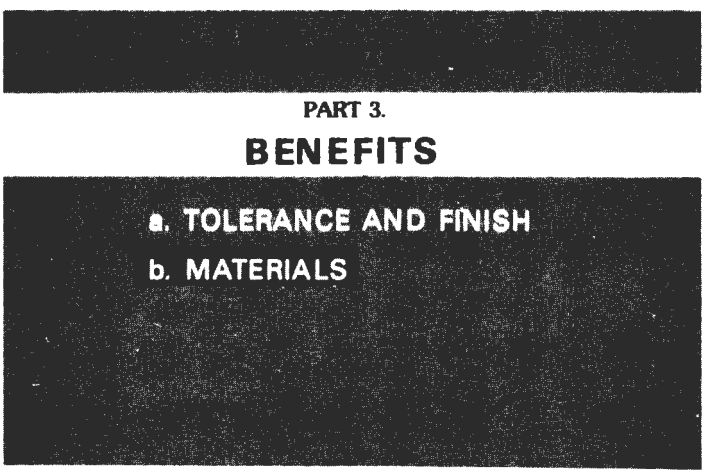

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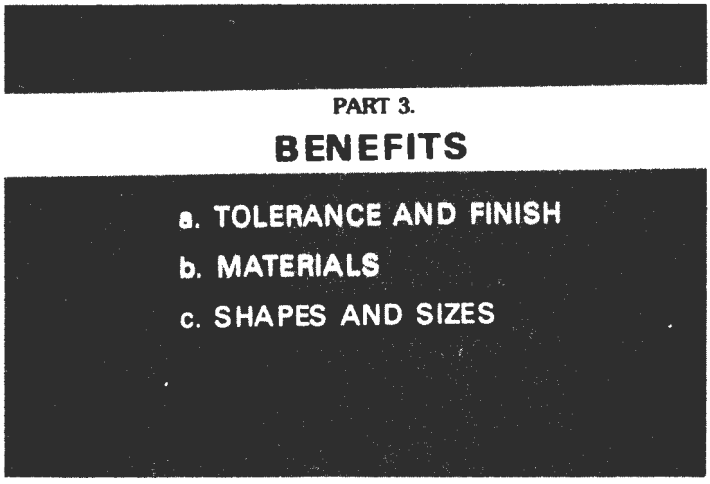

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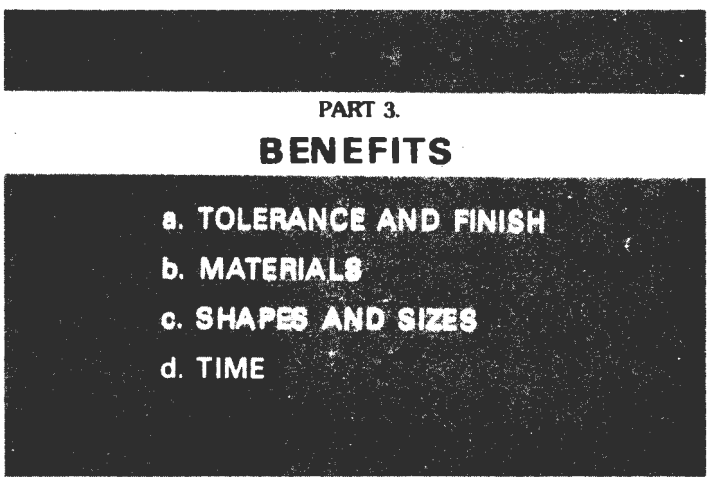

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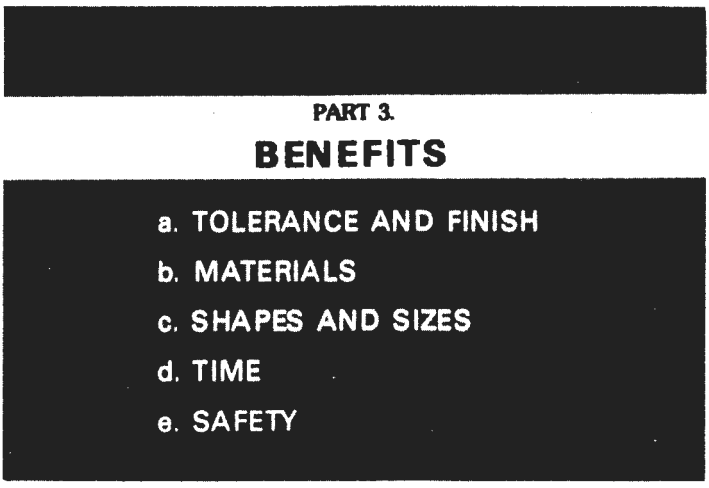

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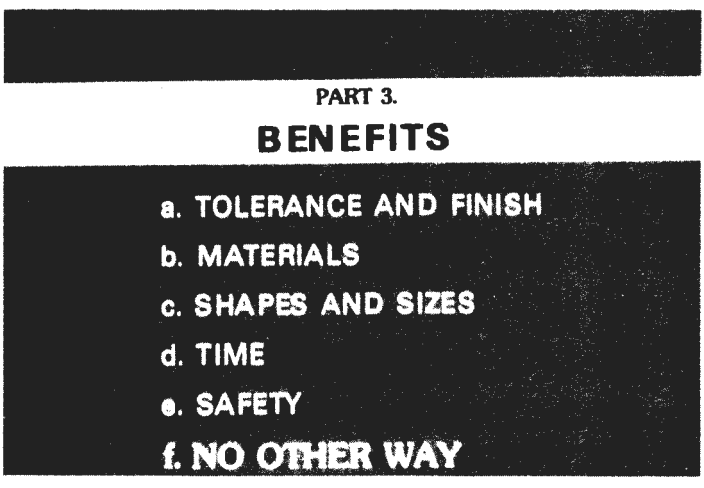

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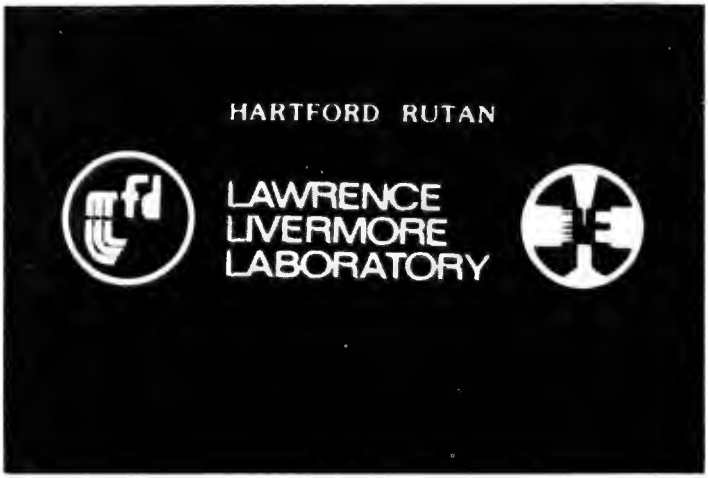

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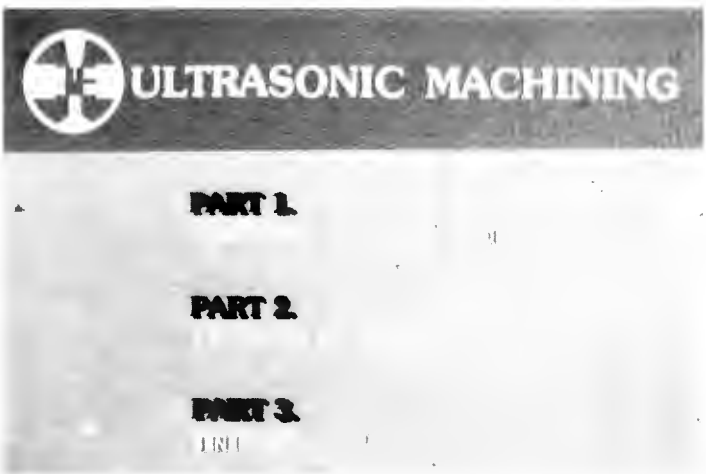

Figure 32.


ULTRASONIC MACHINING
(IMPACT GRINDING)
AN INVALUABLE
ADDITION TO THE SERVICES
RENDERED BY THE
LAWRENCE LIVERMORE LABORATORY
GLASS SHOP

Figure 33.

VENTILATING A SCIENTIFIC GLASSBLOWING LAB (Basic Principles)

Frans Van Damme

Introduction

In order to maintain proper working conditions, a good functional ventilation system is essential in the glassblowing lab. Over the years, architects have been among the pioneers in the use of exhaust hoods as safety devices in laboratories and have been successful, to a degree, in bringing the technology of this means of environmental control to the present state of development. Nevertheless, a lot still needs to be done to achieve a wider understanding of the basic principles of hood design and ventilation systems in general.

As you know, various gases and particles are developed when working glass. Some are silicon oxide, boron oxide, nitrogen dioxide and others. Such gases and particles have a definite effect on the health of humans and particularly the respiratory organs can easily be attacked and damaged when such contaminants are inhaled. With this, we have determined the presence of the hazard that we try to eliminate and, consequently, that's the first step in designing a ventilation system.

In this particular case, we deal mainly with gases (NO_2 and N_2O) that are slightly heavier than air combined with a mixture of extremely small particles (SiO_2 and B_2O_3). We can, therefore, assume that a number of normal air exchanges in the glassblowing lab would be sufficient to eliminate the problem. Thus, once the source of contamination is determined, we can design a system for the removal of the contaminants.

Ventilation or Exhaust System

A ventilation or exhaust system for a glassblowing lab may consist of three main parts.

1. Canopy hoods at the sources of air contamination. In this case, hoods need to be placed over lathes and benches.

2. The branch and main ducts through which an air stream transports the contaminated air to the atmosphere.
3. Air moving equipment to produce the required air flow into the canopy hoods.

Of course, ventilation system designs may vary, and in some cases it is advisable to seek professional help. There are a number of circumstances that can contribute to real problems in installing ventilation. When moving into a new building, it is generally accepted that the architect has provided the glassblowing lab with proper ventilation. Most of the time, however, we deal with an existing building where the glass lab needs to be relocated or expanded. Some rooms may have existing ventilation where the air-intake is variable; others may have a nonchangeable intake or no intake at all.

So we may conclude that not every room in a building is suitable for a glass blowing lab when it comes to proper ventilation.

Canopy Hoods

The most effective hood is one that will require the minimum exhaust volume for effective ventilation control. Therefore, the design needs to respond to the actual operation for which control must be obtained.

In the case of glassblowing, according to the experts, the ideal situation would be to develop an airflow barrier between the glassblower and the torch. But that's ruled out because of the many impracticalities.

The size of the hoods is based on the approximate working area over benches and lathes. A hood does not necessarily cover the entire bench but only part of it as the flame is angled away from the glassworkers. The distance between the bench top and the bottom of the hood is important, however. The shorter that distance, the more efficient the hood will be. Approximately 3 ft is a convenient and functional height. There are various canopy hood-designs but the type shown in Figure 1 is typical and generally acceptable for the glassblowing lab.

Calculations of Hood Size, Face Velocity and Number of Air-Exchanges

In such calculations are two variable factors:

1. The hood size
2. The face velocity

The face velocity is the air flow in CFM or cubic feet per minute at the hood face area. This velocity can be between 4 and 100 CFM. The minimum reasonable number of air exchanges is between 5 and 10 per hour.

Example I. A glassblowing lab with a room size of 25 ft in length, 15 ft in width and a ceiling height of 10 ft needs to be ventilated. The room has two canopy hoods of 2 × 2 ft (variable). Assume that a 60 CFM (variable) is acceptable.

Volume of room: L × W × H or 25 × 15 × 10 = 3750 ft^3

Surface area of canopy hood: 2 × 2 = 4 ft^2

Total surface area of canopy hoods: 4 ft^2 × 2 = 8 ft^2

Total face velocity per minute: 8 × 60 CFM = 480 CFM

Total face velocity per hour: 480 CFM × 60 = 28.800 CFH

Number of air exchanges per hour: $\frac{28.800}{3750} = 7.68$

The air exhaust can by ±90% of air supply to create a slight pressurization in the room.

In this example:

Air exhaust is 28.800 CFH

Air intake should be:

28.800 + 2880 (1/10 of exhaust) = 31.680 CFH

Example II. How do the two variables (hood size and face velocity) affect the calculations:

- Same room size of 3750 ft^3 (constant)
- Same 60 CFM (constant)
- Hood sizes: 3 × 3 ft (variable)

Surface area of canopy hoods: 3 × 3 × 2 or 18 ft^2

Face velocity per minute: 18 × 60 CFM = 1080 CFM

Face velocity per hour: 1080 CFM × 60 = 64.800 CFH

Number of air exchanges per hour: $\frac{64.800}{3750} = 17.28$

Example III.

- Same room size of 3750 ft^3 (constant)
- Same hood size as in Example I (2 × 2 ft) (constant)
- Face velocity of 40 CFM (variable)

Surface area of canopy hoods: $2 \times 2 \times 2\ ft^2 = 8\ ft^2$
Face velocity per minute: $8 \times 40\ CFM = 320\ CFM$
Face velocity per hour: $320\ CFM \times 60 = 19200\ CFH$
Number of air exchanges per hour: $\frac{19.200}{3750} = 5.12$

While the room size remains unchanged, the hood area and face velocity can change the rate of air exchanges drastically. This means that the two variables have to be brought in balance.

Example IV. Rooms with existing ventilation (air intake and exhaust).

A room of L 36 ft, W 34 ft and height of 11 ft has an existing supply air rate of 2500 CFM.

A total of four hoods need to be installed while still maintaining a good air exchange rate.

After manipulating hood sizes and face velocity numbers a few times, a reasonable successful result was as follows. Using a 100 CFM.

Hood	Hood face area	Hood face velocity
I	5.69 ft^2	569 CFM
II	2.78 ft^2	278 CFM
III	8.00 ft^2	800 CFM
IV	8.00 ft^2	800 CFM
TOTALS:	24.47 ft^2	2447 CFM

$$\frac{Exhaust\ rate}{Supply\ air\ rate} = \frac{2447\ CFM}{2500\ CFM} = 0.98 \rightarrow 98\%$$

The air pressurization is 2%.
Volume of room: L × W × H or $36 \times 34 \times 11 = 13.464\ ft^3$
Air intake per minute: 2500 CFM
Air intake per hour: $2500\ CFM \times 60 = 150.000\ CFH$
Air exchanges per hour: $\frac{150.000}{13.464} = 11.14$

Ducts

As in the hood design, we try to eliminate as many obstacles as possible so that air can flow freely and with little resistance. Aerodynamics of flow patterns can determine the success of a ventilation system. Therefore, we should avoid (besides a lot of other details) the use of square ducts as much as possible. The velocity losses can easily be kept to a minimum if round ductwork is installed. For toxic gases, a flow velocity of 2000 CFM is generally acceptable.

The diameter of the ducts depends on the CFM of air that pass through, and many publications in the field of ventilation will illustrate tables or charts where one can read the desired duct diameters. There are, however, a number of construction details that can affect the flow losses considerably.

a. A duct enlargement or duct contraction (Figure 2) should gradually taper from one diameter to another. A general rule of 1 unit change in diameter for every 5 units changes in length seems to deliver good results.
   Example: A 5" diameter ductpipe needs to be connected on a 7" ductpipe. The diameter difference between the two pipes is 2". This means that the tapered connection should be 2 × 5 or minimum 10" in length.
b. The elbow radius in ductwork (Figure 2) is of importance as well. The radius should be kept as large as possible. About 2-1/2 times the diameter of the ductpipe is a good radius. Figure 3 indicates velocity losses at various radius lengths.
c. Branch entries (Figure 2) also can cause air flow resistance if not properly constructed. The angle of entry should be kept as small as possible. Figure 3 explains the velocity losses at various angles.

Exhausts

The hood and ductwork combination can be tied in with an existing system. If no central ventilation is present, a separate

exhaust fan can be mounted on the roof or a side wall of the building. An up-blast centrifugal exhaust fan is most recommendable.

Air Intake

To avoid drafts and assure a good circulation, the air intake should be placed as far as possible away from the exhaust sources (hoods). Preferably, air-intake should be placed in the floor or transported through ducts that stop at about 1 ft above the floor and placed in strategic positions.

Over-Ventilating

Over-ventilating occurs when the number of air exchanges is too high. Besides being ineffective, such a situation can also be expensive when we either preheat or precool the intake air flow. Figure 4 illustrates that the person working in front of a hood can develop an airstream obstacle and, therefore, can create a low pressure or vacuum in front of him/her. This would be a very undesirable aerodynamic flow because the purpose of the ventilation in this case is to remove gases and small particles out of the workers' area of breathing.

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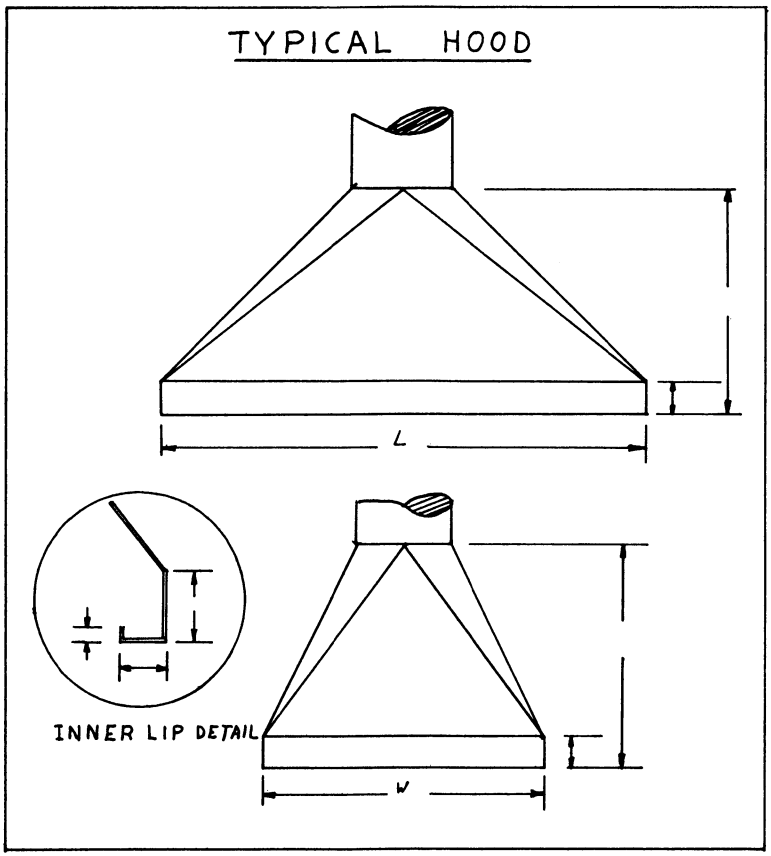


Figure 1.

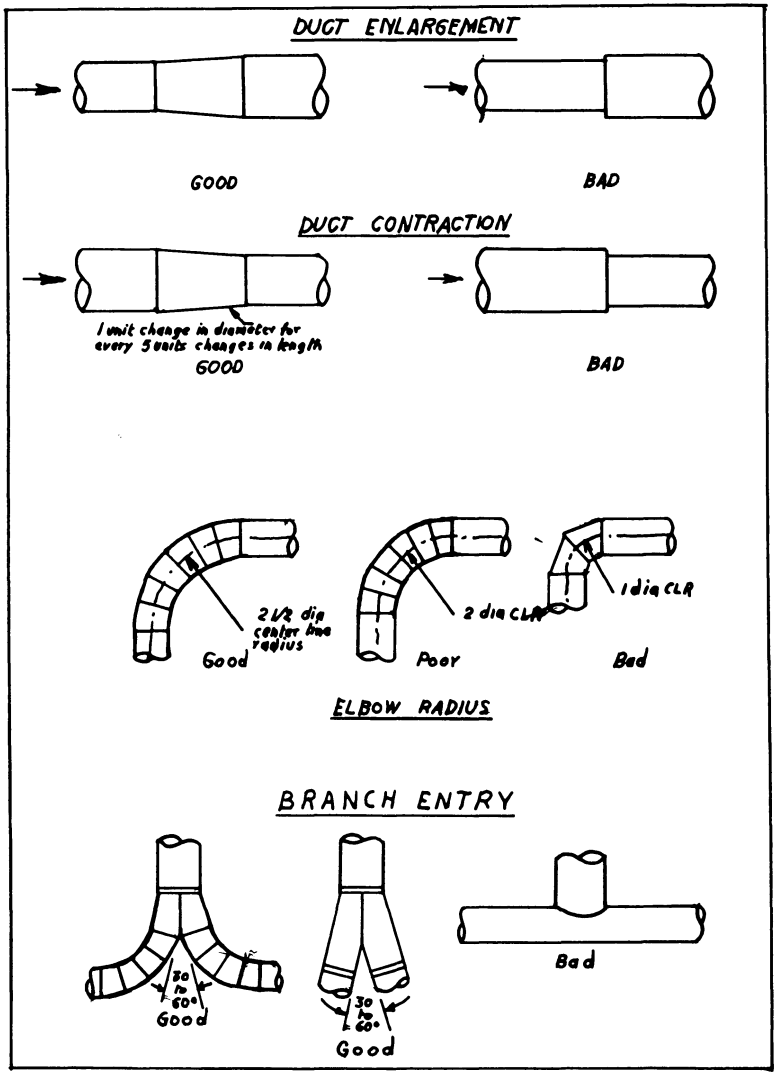
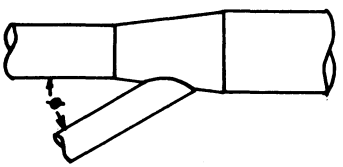
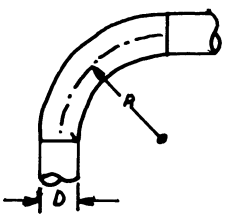

DUCT ENLARGEMENT
GOOD
BAD
DUCT CONTRACTION
1 unit change in diameter for every 5 units changes in length
GOOD
BAD
2 1/2 dia center line radius
Good
2 dia CLR
Poor
1 dia CLR
Bad
ELBOW RADIUS
BRANCH ENTRY
30 to 60°
Good
30 to 60°
Good
Bad

Figure 2.



Angle θ Degrees	Loss of Velocity In Branch
10	0,06
15	0.09
20	0.12
25	0,15
30	0,18
35	0,21
40	0,25
45	0,28
50	0.32
60	0.44
90	1,00

BRANCH ENTRY LOSSES



R, No. of Diameters	Loss of Velocity
2.75	0.26
2,50	0.22
2.25	0,26
2.00	0.27
1.75	0.32
1.50	0.39
1.25	0.55

ELBOW LOSSES

Figure 3.

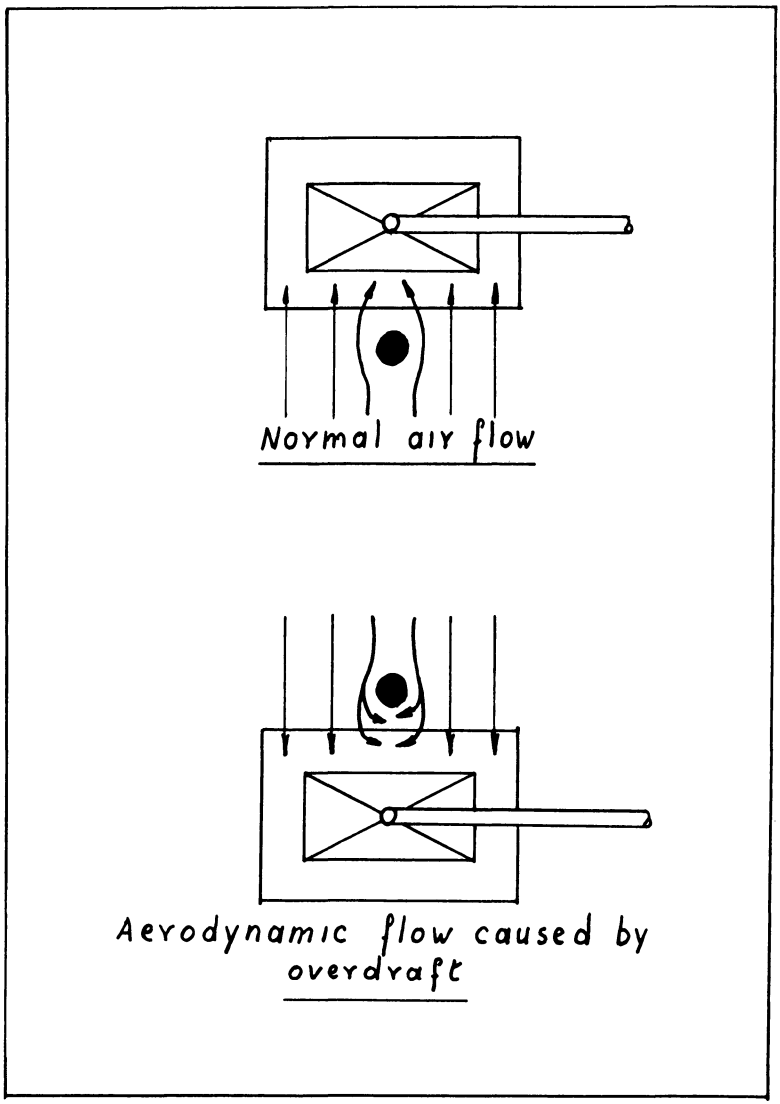


Figure 4.

GLASS-TO-METAL SEAL QUALITY DETERMINATION BY METALLOGRAPHY

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Introduction

Silicon integrated circuits presently require the use of hermetic packaging with glass-to-metal seals[1] for reliable operation. The packages must be capable of maintaining a clean and dry internal environment over a long period of time. Some typical electronic component packages are shown in Figure 1. Packages such as those shown are most often tested by helium leak detection to 10^{-8} torr or by dye penetrant through a 4-12h exposure to the dye. Recent work[2] has shown that a package may pass the requirements of this kind of testing but that the same package will not remain hermetically tight after torque loading or after fabrication processes such as cover welding. Therefore, other means of evaluation seem necessary if package quality is to be assured throughout assembly processes.

Metallography

Standard procedures are used for the metallographic preparation of glass-to-metal seals. A sealed component can be sectioned either transversely or longitudinally. At the seal interface the glass provides a hard surface which helps to prevent rounding or other artifacts. The inspection criteria that are of most interest include effects on the glass, the glass-metal interface and the metal members.

After sealing, the glass can be studied for the volume fraction of voids due to gas evolution, also for the size and distribution of gas-formed voids. The glass-metal interface can be inspected for oxide precipitates in the glass, foreign material at the interface, and residual intergranular oxide in the metal at the interface. The metal member outside the seal area can be evaluated for the degree of intergranular degradation due to

prior processing.

The metals involved in hard glass seal making are high-nickel alloys which normally require active etchants. In the case of glass-metal seals, the surface of the metals has been thermally affected by pre-oxidation and other heat treatments. Therefore, metallographic etching for the purpose of accentuating the glass-metal interface area is relatively simple. One of the most effective etchants is a 5% nitric acid in alcohol (Nital) mixture, although other, more severe etchants can be used.

Glass-Metal Microstructure

As a result of processing treatments, the metal in a sealed component is fully annealed, and a coarse, equiaxed grain is evident in the bulk material. Pre-oxidation for sealing results in the formation of an oxide scale and an intergranular oxide on all metal surfaces. When glass is bonded to a pre-oxidized metal surface, several characteristic microstructural features are formed, and by visual inspection, their effect on seal quality can be determined.

Glass. During sealing, gasses are evolved in differing amounts as a function of glass composition, makeup, cleanliness and so forth. The resultant bubble quantity, size and distribution are readily discernible in the metallographic section as shown in Figure 2.

When hermeticity tests are made on representative samples, the following criteria appear to offer acceptable quality in terms of the condition of the glass:

1. gas volume	33%
2. bubble size	0.75 mm
3. bubble stringers	20% of seal length

Interface. When glass is flowed on a pre-oxidized metal surface, the glass dissolves the oxide scale and bonds to the metal under it. As a result of wetting and bonding action, a

characteristic product can be detected in a metallographic section, as shown in Figure 3. The oxide scale appears as a precipitate in the glass, and the metal side of the interface contains the remnants of the total oxide that was seen prior to sealing. The extent of this residual intergranular oxide is proportional to the degree of prior oxidation. This relationship can be used for the specification of a required oxide quantity. During the present study, a residual oxide in the range of 2.0-6.5 μm was determined to be acceptable.

Conclusions

Standard metallographic procedures are effective for the evaluation of glass-to-metal sealed components. The formation of a glass-metal seal results in several characteristic features for which acceptance criteria have been developed.

Inspection for residual intergranular oxide, for oxide precipitates and for gassiness is suggested as a viable procedure for process control to assure hermeticity in high reliability electronic components.

Acknowledgment

The author is grateful to John McCormick and the Rome Air Development Center for their support of this work.

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2. J. McCormick and L. Zakraysek, "A Metallographic Test for Glass-to-Metal Seals," RADC Contract F30602-78-C-0086, Dec. 1978.

June 23, 1980

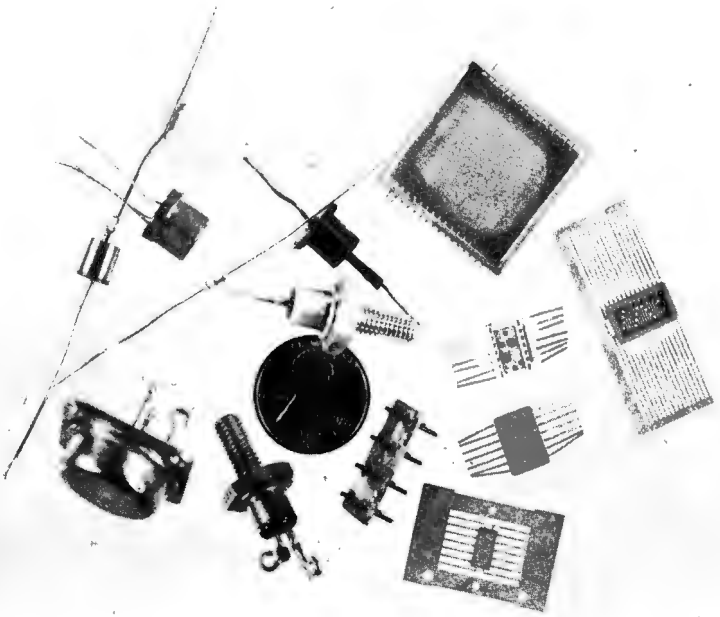

Figure 1. High reliability packaging

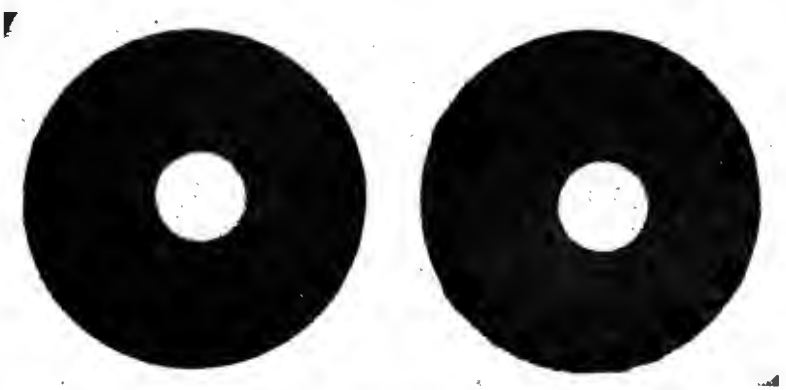

Figure 2. Effect of gas bubble formation

Figure 3. Glass-metal interface effects

THE DEVELOPMENT OF HIGH DENSITY PbO MACROCHANNEL PLATES FOR HIGH ENERGY GAMMA-RAY DETECTION

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Introduction

Honeycombed arrays to improve the detection of high energy gamma rays in gas-filled detectors have been developed by groups at the Lawrence Berkeley Laboratory (LBL)[1,2] and the European Center for Nuclear Research (CERN).[3] In order to obtain a high absorption for photons whose energies lie above that of x-rays in the electromagnetic radiative spectrum, the arrays or converters are generally constructed from a heavy element, such as lead, bismuth or tungsten. These arrays are specially designed to have a large surface area-to-volume ratio to provide a high collection probability for electrons generated by gammas in the material. Also the arrays have the capability to efficiently transport these electrons to a detector by means of suitably applied electric fields.

At LBL we are developing thick macrochannel arrays by fusing thin wall glass capillaries of high lead content and investigating the improvements in the absorption and detection of gamma rays for gas-filled detectors, called multiwire proportional chambers (MWPC). Figure 1 shows a cross-sectional view of an MWPC sandwiched between a pair of macrochannel arrays. The entire assembly is enclosed in a gas-filled container. One application for these detectors in which we have been involved is the construction of a medical-positron-imaging camera which detects paired gammas from the annihilation of matter with antimatter, that is, electrons with positrons. By investigating the use of small capillaries [less than 1 mm in inner diameter (I.D.)] we hope to improve the detection

efficiency and spatial resolution of the camera.

Lead Glass Tubings

Our gamma converters were constructed from glass of the highest lead content obtainable, which yet was sufficiently ductile for the drawing of thin wall tubings. This turned out to be lead glass with a concentration of 79% PbO by weight and with an overall density of 6.2 gm per cm^3. The first drawings of tubings of Garner Glass Co.[4] from the 79% PbO glass had an I.D. of 1.33 mm and 0.129 mm wall thickness. Table 1 lists the various lead glass compositions which have been tested. The dimensions of the tubes drawn from these are shown in Table 2.

The 79% PbO (Hi-D) glass was obtained in the form of 5 kg slugs from Nuclear Pacific, Incorporated.[5] The first two drawings of the 79% PbO glass by Garner produced only a 10% yield, primarily due to early devitrification in the glass. The yields of later drawings were significantly improved by polishing the slugs and slightly reducing the draw temperature. We now obtain an overall 50% yield of which approximately 15% exhibits slight devitrification. For our purposes, those tubes that exhibit slight devitrification are still usable. The tolerances of the inner diameter, outer diameter and roundness are within 4% while the concentricity holds to 1%. Tubes are broken into approximately 0.7 m lengths.

Construction

Due to the large amount of tubes necessary to assemble arrays that were 5 by 5 cm^2 or 10 by 10 cm^2 in cross section, a simple handling procedure was developed. An amount of tubes which covered an area of 10 to 15 cm by 0.7 m were placed adjacent to each other and made flush at one end. Masking tapes fastened the tubes at 13 cm intervals temporarily while nicks were made with a carbide blade. The tubes could then be easily broken into batches of approximately 30 to 50 which were stacked in a graphite mold covered by a weighted top lid as shown in Figure 2. This mold which has a 15 cm by 10 cm by

10 cm volume can be reduced to smaller volumes by substituting graphite spacers in the mold.

A number of glass tubings were placed within the mold so that the lid was raised above the rim of the mold by a gap, as shown in Figure 2. The height of the gap was selected to yield a desired compression for the tubes. For example, we allowed a 2% compression on 0.91 mm I.D. tubes which amounted to a 1 mm clearance for a 5 cm by 5 cm by 13 cm boule. An additional 1 mm was added to the gap to compensate for some settling of the tubes before the softening temperature of the glass was reached. Actually, of the arrays fused, we noticed that the top and bottom rows of tubes were compressed the most.

The mold filled with glass tubings was placed in a furnace with a temperature rise of 100°C per hr. Our runs required two working days. The oven was set to reach approximately 50°C below the softening temperature by the end of the first day at which time a temperature difference of approximately 40°C existed between the mold and the oven. The temperatures of the glass, mold and oven were allowed to reach equilibrium slowly overnight. The tubes were fused on the second day, then the boule was annealed for 8 hr before being cooled over a weekend.

On the second day, after the temperature of the entire assembly had reached 50°C below the softening point (softening points for 79% and 59% PbO glass are 445° and 538°C, respectively), the temperature was then raised 10°C per hr. The weight of the lid compressed the tubes so at the softening temperature they fused to form a boule. After the lid was completely closed the tubes were allowed to tack firmly by their own weight for 1/2 hr. Then the furnace was opened to allow the temperature of the glass to drop 30°C. The furnace was then closed and allowed to cool slowly to 370°C for the annealing process.

Because of possible large thermal gradients within the high density lead glass the boule was allowed to cool to room temperature slowly over two days before removing it from the furnace. Also we restricted the tubings to shorter lengths, e.g., 13 cm, since we had found difficulty in the cooling and handling of a boule of 26 cm in length.

The raw boule was sawed into slabs of 0.5, 1, 2, and 4 cm thicknesses with a circular carborundum blade, rather than a diamond blade which had the tendency to damage the edges of the slabs. The slabs were polished flat on both ends with the side of a carborundum saw to provide smooth surfaces.

Conductivity Development

In order to drift electrons efficiently (which are generated by the absorption of photons) through the tubes of a converter, the inner surface of the tubes must have a uniform resistive layer. In our surface conductivity investigations, we attempted to develop surface conductivity with stannous chloride ($SnCl_2$), with high resistive carbon point and by the hydrogen reduction method. The three methods are briefly described below. We found the hydrogen treatment to be the best.

The $SnCl_2$ technique was performed by immersing arrays in various concentrations of $SnCl_2$ solutions. The arrays were then baked in air in order to oxidize the $SnCl_2$ into conductive stannous oxide (SnO). In the resistive carbon method the arrays were simply immersed in a dilute solution of high resistive carbon paint for a period of time and then dried. Electrical contacts on the ends of the converters were made by a thin conductive epoxy[6] layer dabbed on with a rubber stopper. We found that by dabbing the surface as compared to rolling or brushing we avoided clogging the tubes with epoxy. Table 3 shows the resistances obtained for various converters by the $SnCl_2$ technique.

Tests were made to determine uniform conductivity of the tubes processed by each of the three methods. A voltage was applied to the converter and a probe was inserted into a tube to measure the voltage along the surface. We found the conductivity to be nonuniform for the majority of the tubes treated by the $SnCl_2$ method. Although uniform conductivity was found for the carbon resistance method, poor gamma detection efficiency was obtained.

Hydrogen Reduction Preparation

In the hydrogen reduction treatment, PbO in the glass is reduced to lead according to the chemical reaction $PbO + H_2 \rightleftarrows Pb + H_2O$. The glass is blackened 1000 to 5000 nanometer (nm) deep due to the reduction of PbO to Pb. However, according to Blodgett[7] only the top 100 nm is conductive. Blodgett developed a silicate layer (SiO_2) 100 to 400 nm thick to cover the Pb layer from moisture. This silicate film was developed prior to the hydrogen treatment.

With this SiO_2 layer on our arrays no deterioration in the resistances of our H_2 treated converters has been encountered over periods of months. However, for our purposes, we found that a SiO_2 layer was probably unnecessary since the converters were placed in desiccators immediately after the H_2 treatment and then into our MWPC which was usually filled with a gas mixture of 30% CH_4 and 70% Ar.

A silicate layer was developed as follows. The arrays were placed in an ultrasonic cleaner filled with deionized water to remove any glass particles. The arrays were then degreased ultrasonically in acetone for 5 min, then soaked in 0.1 M HCl for 2 to 5 min depending on the inner diameter of the tubes. For example, the 1.33 mm and 0.91 mm I.D. tubes were treated for 2 min while the 2.0 mm I.D. were for 5 min. According to Blodgett, the HCl acid leaches out the surface layer of PbO leaving behind the SiO_2 film. We have not attempted to measure the thickness of the SiO_2 film but have noticed after the hydrogen treatment the various radiant colors from the glass under a fluorescent light. Without the SiO_2 layer, the various colors are not present.

The arrays were again cleaned ultrasonically in deionized water, blown dry with Ar and baked in air at 140°C for eight hours. The final ultrasonic cleaning was essential to remove loose glass particles that might adhere to the walls and clog the tubes during the drying process.

Hydrogen Reduction

The reduction was performed in a vacuum-tight stainless steel cylinder 40 cm long capable of handling up to fifteen 10 cm by 10 cm by 2 cm thick arrays spaced 1 cm apart. A schematic

diagram of the entire layout is shown in Figure 3. The gases were preheated inside tubes coiled around the cylinder while thermocouples monitored the temperatures at various locations. A desiccator and particle filter were attached to the gas line to assure the dryness and purity of the H_2 gas used in the reduction process. Figure 4 shows the chamber in the furnace.

To ensure uniform surface reduction of the tubes, we mounted the arrays such that the gas flow was along the axis of the tubes. The arrays were flushed with helium at a rate of about 4 ml per sec while the temperature was raised at a rate of 100°C per hr. Generally the temperatures of the chamber and the furnace were allowed to reach equilibrium overnight before passing H_2 into the chamber.

The conductive layer (about 100 nm thick) was developed according to a similar H_2 reduction recipe by Blodgett. Hydrogen at 350°C was admitted at 15 ml per sec for a period between six to eight hours. Then H_2 was replaced with He while the oven was allowed to cool to room temperature. Figure 5 shows the cylinder opened after an H_2 run. Figure 6 shows two converters, one prior to treatment (the left one) and one after treatment. For electrical contact a thin conductive epoxy layer was applied on the ends as described earlier. Resistances of the 0.91 mm I.D. arrays are shown in Table 4.

Our continuing efforts to develop efficient gamma-ray PbO glass tubing converters have led us towards the use of small inner diameter and thin wall tubes. The smallness of the tubes has then led us to an investigation of the improvement of uniform conductivity on glass surfaces. We investigated the problem by adjusting the H_2 treating recipe for different conditions. It was determined that our previous treating process[2] could be optimized to give maximum conductive uniformity within the tubes. We found that by lowering the H_2 reducing temperature from 400° to 350°C, the resistance of a 5 cm by 5 cm by 2 cm thick (0.91 mm I.D., 0.096 mm wall) converter was actually reduced from 270 MegΩ to 300 KΩ ! The applied voltage gradient measured with microprobes within the tubes was extremely constant. We also found that the period needed to H_2 fire the glass to achieve good surface conductivity could be reduced from eight to six hours since the measured resistances

were the same for either treatment. Qualitatively, for 80% PbO glass our results agree with Blodgetts's 60% PbO glass, i.e., the H_2 reduction temperature lies within a range of about 100°C where conductivity can be maximized. For 80% PbO glass this temperature range is between 275° to 375°C as compared to 380° to 480°C for 60% PbO glass.

Conclusion

Table 5 shows in chronological order the various tubings from which we have made glass arrays. In the achievement of high efficient gamma detection arrays the trend is in the direction of smaller and thinner-wall tubings. The measured gamma detection efficiencies of the corresponding arrays are listed in column 6 for gamma ray energies of 511 keV. Details on how the efficiencies were measured are given elsewhere.[2] For example, we began the development with 2 mm I.D., 0.43 mm wall thick tubes which gave a gamma detection efficiency of 3.5%. We have reached an efficiency of 10.6% for 0.91 mm I.D., 0.096 mm wall tubes. Currently, we are investigating even smaller ones that have 0.5 mm I.D., 0.01 mm wall thickness. Our goal is to obtain efficiencies close to the total gamma absorption of the glass. The total absorption for 511 keV gammas by a 2 cm thick, 0.91 mm I.D. converter is about 30%.

Acknowledgments

We would like to thank various staff members of LBL who assisted us, especially Dr. R. Kopa and Mr. P. Miller who designed our hydrogen treatment container and system. It is also a great pleasure to thank Dr. M. Tripp of SKIA Corporation and Mr. P.P. Lin of Litton Industries who made some conductive converters which provided us with the impetus to continue this project.

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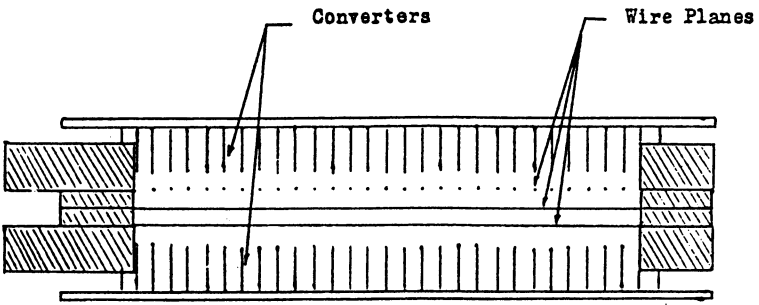

Figure 1. Cross-sectional view of converters coupled to a conventional three-wire plane multiwire proportional chamber—the entire assembly is placed in a gas-filled medium, such as 30% CH_4, 70% Ar.

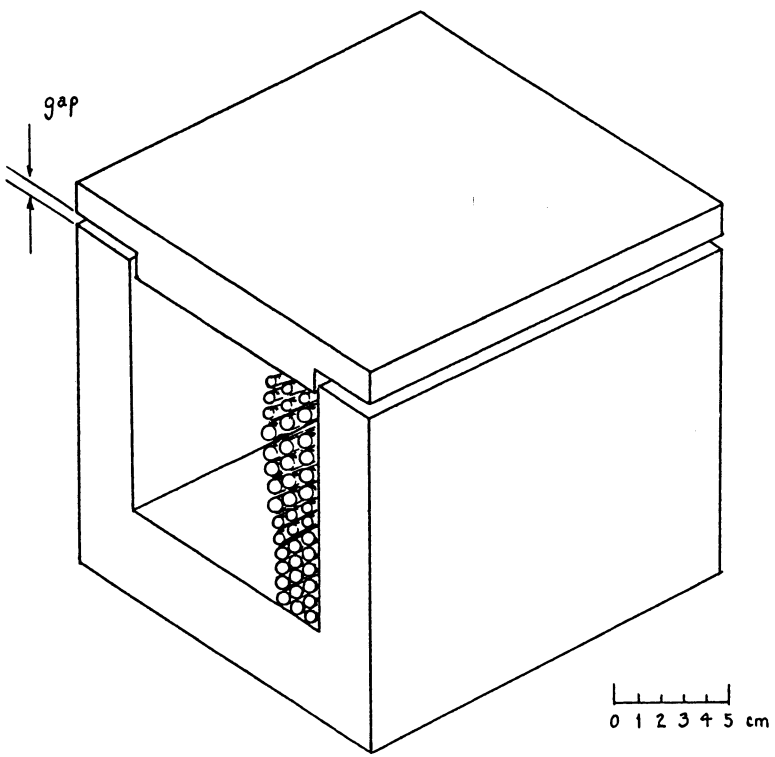

Figure 2. Graphite mold in which glass tubings are stacked for fusing into a boule

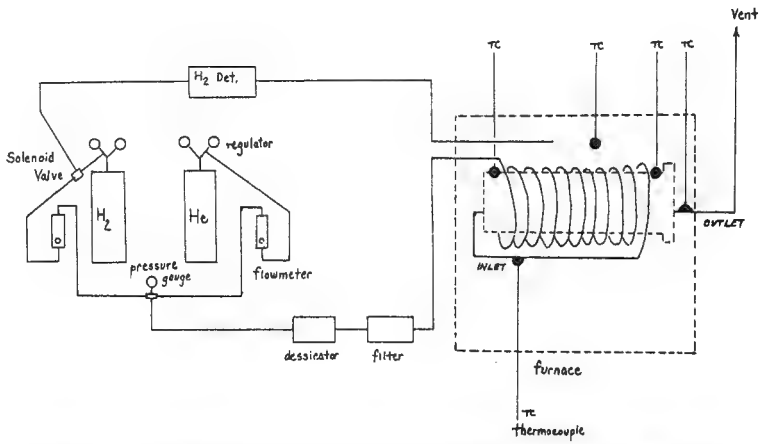

Figure 3. Schematic diagram of the hydrogen treatment system

Figure 4. Hydrogen treatment chamber in furnace—with inlet gas tubings wrapped around chamber to preheat gases—thermocouples are mounted at various locations on the system.

Figure 5. Cylinder is opened after a hydrogen run. Glass array 2 cm thick is still mounted with stainless steel brackets. A collimated baffle was used to concentrate the H_2 flow across a 5 by 5 cm area.

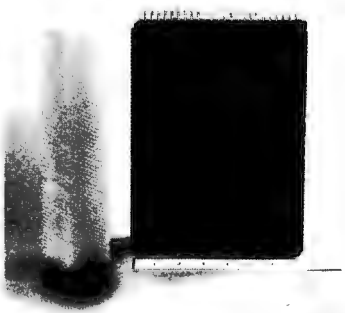

Figure 6. Lead glass arrays (1.33 mm inner diameter, 0.129 mm wall thickness, 2 cm thick—the left one has not been H_2 reduced.

Table 1. Composition of lead glasses used by Garner Glass Co. for tube drawing

Type of glass	PbO percentage (%)	Density (g per cm^3)	Composition of glass
Corning 8161	51	4.02	2% RbO, 2% BaO, 5% K_2O, 40% SiO_2, 51% PbO
Kimble EG-16	59	4.30	less than 0.1% MgO, CaO, Na_2O, 0.35% Al_2O_3, 6% K_2O, 35% SiO_2, 59% PbO
Nuclear Pacific Hi-D	79	6.2	small percentage of As, approximately 1% Al_2O_3, 19% SiO_2, 79% PbO

Table 2. Dimensions of tubes drawn by Garner Glass Co.

Type of glass	PbO percentage (%)	Inner diameter (mm)	Outer diameter (mm)	Wall thickness (mm)
Kimble EG-16	59	2.00	2.86	0.43
"	"	1.20	1.70	0.25
Corning 8161	51	1.00	1.40	0.20
Nuclear Pacific Hi-D	79	1.33	1.59	0.129
"	"	0.91	1.10	0.096

Table 3. Stannous chloride treatments were performed with hexagonal arrays (59% PbO by weight) of 6 cm sides. The arrays were immersed for 15 min in various concentration of $SnCl_2$ solutions and were baked 410°C for 15 min. A temperature rise of 50°C per 15 min was used.

Array Dimensions		Converter	Concentration of $SnCl_2 \cdot 2H_2O$ per	Array
Inner Diameter (mm)	Outer Diameter (mm)	Thickness (cm)	1000 ml H_2O (grams)	resistance (Ω)
2.0	2.9	1.5	10	3.9Meg, 23Meg
2.0	2.9	0.5	10	430K, 230K,45K
1.2	1.7	1.5	20	2K
1.2	1.7	1.5	5	10K
1.2	1.7	1.5	3	∞

Table 4. Resistances of lead glass arrays from H_2 reduction at 350°C for 6 to 8 hr – all arrays are 5 by 5 cm^2 thickness as listed.

	Tube Dimensions		Array	Array
PbO percentage (%)	Inner Diameter (mm)	Outer Diameter (mm)	thickness (cm)	resistance (Ω)
79	0.91	1.10	1.0	200K
,,	,,	,,	2.0	300K
,,	,,	,,	4.0	700K

Table 5. Gamma detection efficiencies from various PbO glass converters – details on how the efficiencies were measured are described in Reference 2.

PbO percentage (%)	Tube Dimensions: Inner Diameter (mm)	Tube Dimensions: Wall thickness (mm)	Converter thickness (cm)	Approximate Number of tubes per cm^2	Gamma detection efficiency (%)
59	2.00	0.43	1.5	14	3.48
"	1.20	0.25	1.5	42	4.76
51	1.00	0.20	1.5	67	6.00
79	1.33	0.129	2.0	50	8.90
"	0.91	0.96	2.0	100	10.6

DRILLING HOLES IN GLASS

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Introduction

Glass, because of its brittle and abrasive nature, poses serious machining problems, especially when drilling straight, clean holes through glass components. Chipping, cracking, drill burn-out and excessive wear will occur, unless great care is taken.

A steel or carbide twist drill that may be used routinely on steel, aluminum or wood will not work on glass! The ideal tool is without question the diamond-edged drill which actually abrades away the glass particles instead of drilling the glass itself.

There are two basic drill types: solid and core. It is universally accepted that diamond core drills are superior in performance to solid drills because of the importance of through coolant in drilling. With a core 0.031 inch (0.75 mm) or smaller, however, it is impossible to force sufficient coolant through the tiny orifice. Hence, most units under 0.031 inch are of the solid type.

A diamond drill is essentially a metal shank to which diamonds have been affixed in one of three ways: impregnation, plating or electro-deposit.

1. The impregnated (or sintered) diamond drill. The diamond section consists of a prescribed quantity of natural or synthetic diamond, sieved to the desired size, mixed with a holding matrix and bonded to the steel shank. The depth of diamond is usually 1/8, 1/4 or 3/8 inch.

2. The plated diamond drill. A single layer of diamond particles, either natural or synthetic, is plated with nickel or another material to the end of a metal shank. The diamonds are more

exposed than those in impregnated drills and, consequently, cut faster. Once the layer of diamond wears off, the drill must be discarded.

It should be remembered when using plated drills, that only the top, or crown, of the drill does the actual cutting. Although diamonds may be visible on the side of the drill when the crown area is worn clear of diamond, the drill will no longer cut. Some machine operators look at the drill and, seeing diamonds on the side, attempt to continue drilling and are usually puzzled when the drill fails to penetrate the glass.

3. The electro-deposited drill. Here, the diamond section is built up in layers on a ceramic core by means of an electrolytic process. The core is then discarded, leaving a hollow drill with all the advantages of a sintered drill plus, because of its thinner wall, an increase in the penetration rate of the drill.

The Diamond

In a paper presented some years ago we stated that synthetic diamonds were slowly taking the place of natural diamonds in drill applications. Today, virtually all drills are made with synthetic diamonds. There are special types that have been engineered for this purpose.

For plated drills many manufacturers use GE Synthetic Diamonds of the MBG (T) type in 120/140 grit or De Beers ECD type synthetics. For impregnated type drills De Beers SDA or GE MBS are used.

Combinations of some or of all of the above types are not uncommon and are often used to produce drills for special materials.

We believe that, beyond better availability and lower cost, the main reason for the switch to synthetic diamonds is uniformity in grit, shape, and size. This is important not only in obtaining close tolerances in size, but also in avoiding the problem of the "High Diamond," a problem we will cover later. It is possible also to produce a fairly thin wall drill with synthetics, an important factor when high penetration rates are required.

Diamond Grit

Diamond grit is available in sizes ranging from a coarse 60/80 to a fine 270/320 mesh. Choice of mesh is largely determined by the size of the drill and the material being drilled. It is highly important that the correct grit size be used. A coarse 60 mesh diamond drill will not give the same performance as a 150 mesh unit. Generally speaking, coarser grit sizes are used for faster stock removal (i.e., higher productivity) without much concern for surface finish.

Diamond Concentration

Although the term "diamond concentration" may be familiar to very user of diamonds, its meaning and significance are not widely understood. Concentration refers to the quantity of diamond in the drill. Consider two drills with 120 mesh-size diamond grit. One has a 100 concentration, the other a 50. The 100-concentration drill contains twice as many diamond particles as the 50 unit. It is understandably expensive. High concentration does not, however, automatically lead to better performance. Correct concentration depends upon the material being drilled, feed rates, and other variables.

Drilling Equipment

When the goal is to drill chip-free holes consistently, every component in the drilling process is important. The basic equipment required is a sturdy, vibration-free and well-made drill press, capable of developing the proper speeds and, at the same time, allowing sensitive feeds. The drill press must be mounted on a firm base and its spindle must be periodically checked for runout. The workpiece should be securely held and positioned. Moreover, the spindle bearings must be in good condition and the feed sensitive enough for the operator to "feel" the cut.

Not too long ago there were only a few machines capable of quality drilling small holes (under 1/8 inch or 3.18 mm). Then we developed our SL-1 drill press which is capable of drilling

holes down to 0.031 inch with coolant moving through the drill center.

This machine was manually controlled, but then we developed the Lunzer SL-1A, an automatic drill press which, following setup, drills a hole at the touch of a button and then automatically retracts at the completion of the operation.

In the past we had always recommended drilling by the "pecking" method, where the operator constantly lowers and raises the drill to ensure a steady supply of coolant at the cutting point. Now, with the SL-1A automatic drill press, we can drill straight through the workpiece. The operation is faster and the drill lasts longer. This only applies to core drills, because it is still necessary to use the pecking method with solid drills.

Ultrasonic Drilling

When small, deep holes are required, say, in the range of 1 mm to 2.6 mm diameter and as deep as 24 inches, we recommend an ultrasonic rotary drill of the type manufactured by Branson Sonic Power Co. This machine is also very useful in trepanning or grinding.

Pecking Method

Many operators favor a pecking action when drilling holes in glass. There are pros and cons to this method. When using a *solid* diamond drill, it is essential that the workpiece be submerged in coolant or that a good supply of coolant can at least be directed at the drill head. The up-and-down motion of the drill, in and out of the hole, helps to pump out the abraded waste material from the hole. This is highly desirable. Moreover, since the drill is flooded by coolant, it remains relatively cool and, hence, lasts longer. The pecking method does, of course, slow down productivity.

When using a conventional core drill, constant pressure of the quill along with the correct speed of rotation; produces favorable results. In the past we had also recommended a pecking action for core drilling, but we have learned that when good coolant pressure (60/80 psi) is maintained through the

center of the drill, the drill will remain cool, debris will be forced out of the hole and core hang-up will be minimized. One of the main reasons for not using pecking method with core drills is that the pecking action itself often breaks the core from the workpiece. This can lead to clogged or broken drills, or to damaged workpieces.

Core Hang-Up

This is an important subject. Core hang-up generally takes place just at the point of break-through. As the core breaks away from the workpiece, especially when the workpiece is not properly supported, there is usually a rim of unground or unabraded glass at the very bottom of the core. Being unground, it is wider than the core drill's I.D. This unground rim causes the core to stay firmly within the drill.

By waxing down the workpiece with a minimum of wax adhesive and by properly supporting the workpiece under the drilling point, it is possible to drill completely through the workpiece, have the core remain in position and also obtain clean holes.

Remember, good coolant pressure, a suitable coolant type, and a minimum of pressure at break-through will help to prevent core hang-up problems. Whenever possible, a dial gauge should be used so that the operator knows exactly the point of break-through, and so can reduce the downward pressure of the quill and ease the drill through its last few thousandths of an inch of travel.

Finally, a "disintegrating" core drill with the drill's I.D. offset to one side (of the drill) also helps to prevent core hang-up. With this type of design, the actual core material is smaller than the drill's internal diameter, and can easily be washed away by coolant pressure.

Be sure to tell your supplier what you will be drilling. He can then deliver the correct diamond size and matrix. Also ask for the drill to be made to your largest tolerance – i.e., if ordering, say, a 1 inch drill with a given tolerance of ± 0.005 inch, request that the drill be made 1 inch + 0.005 inch and -0.000 inch. This will give you longer drill life.

Coolant

Coolant is as important as the drill bit itself. We do not recommend drilling dry with diamonds!

Coolant serves two purposes: first to keep drill and glass cool; and second, to flush away abraded glass particles that could otherwise impede the operation of the drill. With the use of an additive, coolant flow is increased, thus aiding in carrying away the abraded material.

The ideal procedure is to supply coolant under controlled pressure through the center of the drill. Next best is to drill with the workpiece submerged in coolant. With solid drills, a copious flow of pressurized coolant to the outside is good. The practice of "squirting" coolant onto the workpiece and drill point is utterly useless.

In our experience oil-based coolants keep the drills cleaner than water. We use and recommend Vantrol, made by Van Straaten Chemical Co. But there are also several other good products, such as Crystal Cut #322 marketed by Hangsterfer Corp. of New Jersey.

The smaller the drill, the greater the coolant pressure should be. Water direct from the mains gives 40/60 psi, which is not adequate for small hole drilling, such as 1 mm. It is also difficult to introduce additives when taking water from a tap, so, when possible, a pressure tank should be used to enable additives to be introduced and to control the coolant pressure flow.

As a rule of thumb, we recommend a pressure of 80 psi for a 1 mm core drill. For a 1 inch drill, 20-30 psi is adequate.

Drill Speeds

Experience has taught us through the years that there is no set speed for diamond drilling. Drill speed depends on the glass, the operator, the pressure and other variables. Today, we recommend a variable speed control for the drill press. The operator will soon determine the best speed for the specific job he is doing.

Chipping and Break-Out

Care, experience an good equipment are required to minimize chipping.

The most successful method of avoiding chipping is to flip-drill. This however, is not always feasible. It also presents additional problems, such as realignment of the workpiece. In some instances, drilling with an undersized drill followed by reaming with a reamer of finer grit size not only enables you to hold tight tolerances but also removes any small amount of chipping that might occur at break-through.

The application of facing plates will also reduce chipping. But this application is costly an is not always practical.

Dual size drills have also been successful. Here, the drill portion of the head is below required finish size, while the section immediately behind or above the head tapers out to the correct finish size. This results in a hole reamed to finish size while at the same time removing any chipping. With this type of drill a "catchment" is required beneath the workpiece to trap and return the coolant to the outside of the drill during the reaming action. If a catchment is not provided, the coolant will escape and the reamer section of the drill will run dry with disastrous results to both workpiece and drill.

Holding Hole Size Tolerances

Most manufacturers produce drills to a plus or minus 0.002 inch tolerance. Tighter tolerances can be obtained on request. It is nonetheless often found that while the drill measures "on size," the resultant holes are over size. This is brought about either by bad drill alignment, machine run-out or a badly ground drill.

First, check the machine spindle head to be sure there is no run-out. Check that the drive belt is not too tight or too slack since either condition affects spindle motion. Also be certain there is no foreign matter on the drill seat, to cause misalignment of the drill.

Check the drill for head concentricity. If the head is not absolutely concentric with the tail or mounting portion of the drill, it will produce oversized holes. The fact that the head size "mikes" correctly means little.

A single high diamond will also produce over-size holes, but may not be detected when the drill is "miked." So it is prudent to check the drill head with a loupe. If a high diamond is found, switch on the drill press and very lightly touch the drill head with a fine file, to chip off the high diamond without damage to the drill surface.

You have, no doubt, often heard it said, "Just drill into a scrap piece of glass and your drill will align itself." This is correct to some extent. The drill appears to be running true. But to be certain, place a dial indicator against the head of the drill and rotate the spindle by hand to be sure there is no run-out. Be sure the dial indicator is fitted with a nylon-protected finger.

Finally, if you must drill straight through with no backing, drill carefully! Watch your dial gauge, and when you come near the end of the hole, ease up the pressure and increase your drilling speed.

There is no substitute for experience. An operator's first holes can be ragged and drill life short. But as he or she proceeds and gains an understanding of speeds and pressure, the quality of the holes improve and drill life is lengthened.

THE MEASUREMENT OF STRESS IN GLASS

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Method and Equipment

When glass is strained it becomes doubly refracting and behaves as a uniaxial crystal. The birefringence is due to (a) the change in the spacing between atoms which causes negative birefringence and (b) the distortion of the outermost electrons which produce positive birefringence.[1] It has been shown that the birefringence is directly proportional to the intensity of the stress. Thus, stresses in glass can be determined photoelastically, i.e., by measuring the birefringence.

The photoelastic technique measures the difference between the two principal stresses that are normal to the light beam. The third principal stress, which is parallel to the light beam, has no influence on the measurements. Hence, stress calculations using photoelasticity require some prior knowledge concerning the stress state.

The photoelastic technique commonly used to measure stresses in glass is the Senarmont method.[2-4] The general optical system used in the Senarmont method is shown in Figure 1. The sequence of optical equipment is as follows: light source, green filter, polarizer, sample, Senarmont compensator and analyzer. The green filter, usually having a maximum transmission of 546 nm, is used to make the light source more monochromatic. The analyzer is rotatable and it is also useful to have a rotatable polarizer. The polarizer and analyzer are initially crossed, and the slow vibration direction of the Senarmont compensator is oriented in the same direction as either that of the polarizer or that of the analyzer. For example, in

Figure 1 the vibration directions of the polarizer and analyzer are at 135° and 45°, respectively, and the slow vibration direction of the Senarmont compensator is at 135°. The sample is oriented so that the two principal stresses that are of interest are at 45° to the polarizer and analyzer. For example, in Figure 1 they are parallel to the X and Y axes, i.e., in the 0° and 90° positions. The optical retardation δ is then measured at various points of interest in the sample by rotating the analyzer.

The difference between the two principal stresses, $\sigma_1-\sigma_2$, is related to the sample length t traversed by the light beam, the stress optical coefficient K and δ by:

$$\sigma_1-\sigma_2 = \frac{\delta}{tK} \tag{1}$$

The sign of the quantity $\sigma_1-\sigma_2$ can be determined by removing the green filter and inserting a first-order red retardation plate in place of the Senarmont compensator, which is removed. The retardation plate is oriented with the slow vibration direction in either the 0° or the 90° position. Color patterns will then appear which are associated with the sign and the magnitude of the retardation. Usually for a given optical setting, a glass sample having a known negative or positive $\sigma_1-\sigma_2$, e.g., tempered glass, is initially used to quickly check the relationship between the color patterns and the stress sign.

The orientation of the two principal stresses at the points of interest can be determined by crossing the polarizer and analyzer and removing the compensators and green filter.[5] Dark extinctions will then occur in sample regions where (A) the two principal stresses are parallel to the analyzer and the polarizer resulting in an extinction defined as an isoclinic, and (B) $\sigma_1-\sigma_2$, resulting in an extinction defined as an isotropic point. The isotropic points are independent of the position of the crossed polarizer and analyzer with respect to the sample, while the isoclinics are dependent on the position of the crossed polarizer and analyzer. Hence, sample rotation can be used to differentiate isoclinics from isotropic points since during sample rotation the old isoclinics will disappear (new isoclinics may form in other regions) while the isotropic points will remain.

Measurement of Tempering Stresses

Glass is generally tempered by heating the sample of a suitable temperature and subsequently cooling the sample by forcing air over the surfaces. The surfaces then cool faster than the interior, generally resulting in biaxial compression in regions close to the surface and biaxial tension in the central regions.

The photograph in Figure 2 shows the cross section of a piece of tempered glass. The three principal stresses are σ_1, σ_2 and σ_3. Regions 1A and 1B are in biaxial compression with $\sigma_1=\sigma_2<0$ and $\sigma_3=0$. Region 2 is in biaxial tension with $\sigma_1=\sigma_2>0$ and $\sigma_3=0$. The sample is oriented so that σ_2 is normal to the plane of this paper and therefore parallel to the light beam. In this orientation the quantity $\sigma_1-\sigma_3=\sigma_1$ can be measured photoelastically.

The results of photoelastic stress measurements with the sample in Figure 2, using the Senarmont method, are plotted in Figure 3. The curve in Figure 3 is typical for tempered glass. The maximum compressive stress, 11,000-11,600 psi, occurs at the two outer surfaces, while the maximum tensile stress, 5,100 psi, occurs at the central region of the sample. The two black lines in Figure 2 that separate region 2 from regions 1A and 1B are neutral axes where $\sigma_1=\sigma_2=\sigma_3=0$.

Measurement of Cord Stresses

Cord is a microscopic planar or linear glass defect which has a different composition and therefore a different thermal expansion and contraction than the matrix glass. This results in internal stresses that form during cooling from the fabrication temperature.

Figure 4 shows an example of a corded glass structure in one region of a glass sample. In this sample the cord was found to be approximately planar with a variable thickness and intercord spacing. In the region shown in Figure 4 the cord thickness and intercord spacing are on the order of 2 μm and 20 μm, respectively.

At a sufficient distance from the surface, in this case on the order of the intercord spacing, the cord and matrix are biaxially stressed with $\sigma_1=\sigma_2$ and $\sigma_3=0$. σ_1 and σ_2 are parallel to the plane of the cord and σ_3 is normal to the plane of the cord.

The stress distribution in the cord and matrix is assumed to be that given in Figure 5. Regions in which a considerable amount of diffusion has occurred between the cord and the matrix will have a smoother stress profile. Nevertheless, Figure 5 is sufficient for the purposes of this discussion. In the cord,

$$\sigma_{1,c} = \sigma_{2,c} = \sigma_c = -\frac{E\Delta\alpha\Delta Tb}{(1-\nu)(a+b)} \tag{2}$$

while in the matrix,

$$\sigma_{1,m} = \sigma_{2,m} = \sigma_m = \frac{E\Delta\alpha\Delta Ta}{(1-\nu)(a+b)} \tag{3}$$

where a is the cord thickness ($\sim 2\ \mu$m), b the intercord spacing ($\sim 20\ \mu$m), E the elastic modulus ($\sim 10^7$ psi), α the thermal expansion difference between the cord and matrix, T the difference between room temperature and the glass set-point temperature and ν Poisson's ratio (~ 0.22).

The stresses in the cord were measured with samples that had been sectioned, ground and polished to ~ 0.010 inch in thickness such that the plane of the cord was normal to the plane of the sample, as shown in Figure 4. In this orientation during the Senarmont photoelastic stress measurements the light beam was parallel to σ_2, and the quantity $\sigma_1-\sigma_3=\sigma_1$ was measured.

The stress measurement results indicated that the cord was in biaxial compression; thus the cord has a smaller thermal expansion than the matrix. The most highly stressed cord was in a state of stress less than 1500 psi, and most of the cords were in a state of stress less than 300 psi. The matrix stress was usually too small to be measured, but would be opposite in sign (tensile) and approximately one-tenth that of the adjacent compression cord stress.

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4. H. Rawson, *J. Soc. Glass Tech.* 42, 119 T-124 T (1958).
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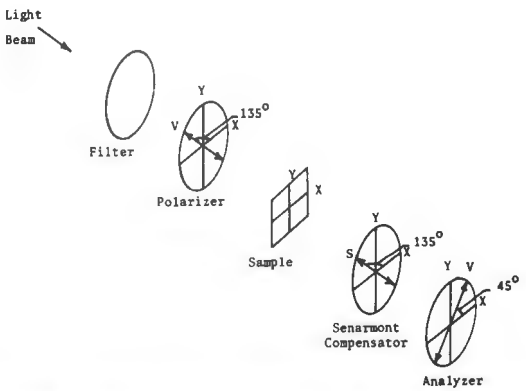

Figure 1. General optical system used in the Senarmont method for measuring stresses in glass. V = vibration direction, S = slow vibration direction.

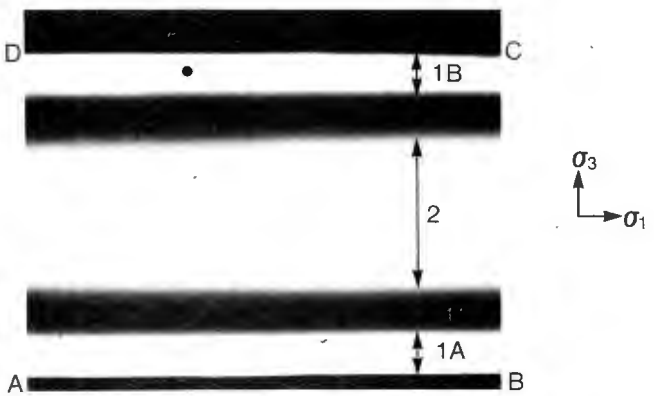

Figure 2. Photograph showing the cross section of a tempered glass sample. The glass was tempered by forcing air over surfaces AB and CD which are normal to the plane of this paper. The three principal stresses are σ_1, σ_2 and σ_3. σ_2 is normal to the plane of this paper. Regions 1A and 1B are in biaxial compression with $\sigma_1 = \sigma_2 < 0$ and $\sigma_3 = 0$. Region 2 is in biaxial tension with $\sigma_1 = \sigma_2 > 0$ and $\sigma_3 = 0$. Photographed with plane polarized light and crossed polars at 16X.

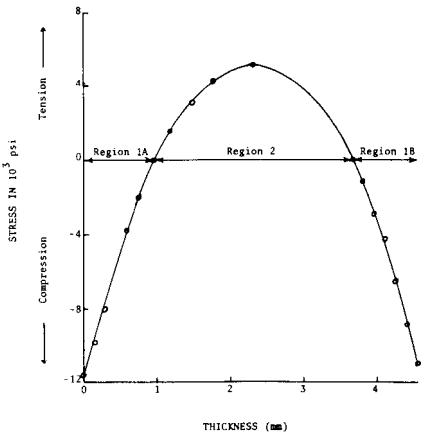

Figure 3. Results of stress measurements of σ_1 from surface AB to surface DC of sample shown in Figure 2.

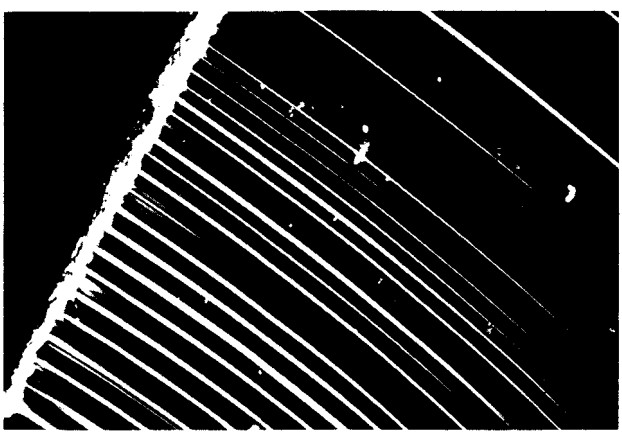

Figure 4. Photomicrograph showing the presence of cord (bright striations) in a glass sample. σ_1 is parallel to the bright striations, σ_3 is normal to the bright striations, and σ_2 is normal to the plane of this paper. Photograhed with plane polarized light and crossed polars at 200X.

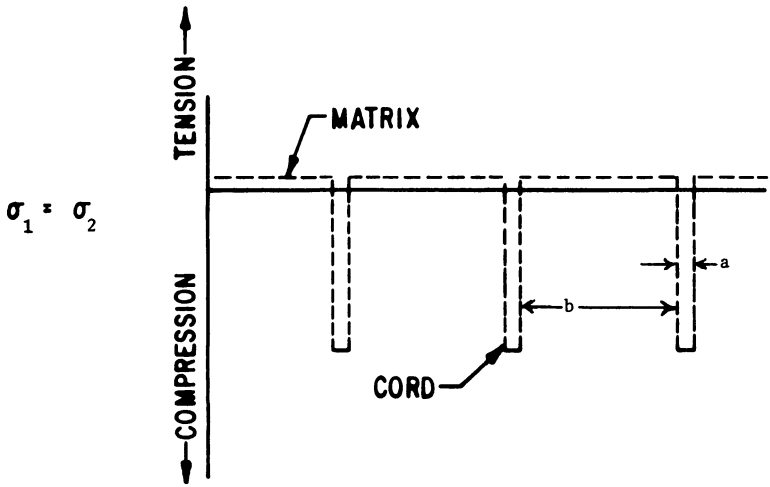

Figure 5. The approximate qualitative distribution of stresses σ_1 and σ_2 in the cord and in the matrix.

AUTOMATIC QUARTZ SEALING CIRCULAR BURNER ASSEMBLY

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This paper is the result of our recent need for a burner that would seal a domed quartz plug inside an ampoule. While under vacuum, the burner had to be automatic and made a leak tight seal.

It had to be able to seal both 64 mm and 74 mm ampoules. The seal had to be made in a static position within five inches from an "O" ring seal.

The current method for making the seal was with a hand torch (Figure 1). This method was time-consuming and required a steady hand and much patience. The very hot concentrated flame caused some sublimation which was undesirable in a clean area.

Our objective was to have a burner with automatic pre-set gas flows and programmed cycles so when the operator pressed the buttons, the burner would function by itself.

Most of our experience in designing and building burners in the past had been for lead glass. Using the same basic approach, we experimented with our first quartz burner.

The first change was to go from natural gas to hydrogen for our fuel gas.

We had previously developed a circular burner that would seal in a static position by angling the jets to produce a swirl.

Our first prototype burner had jets around the entire circle. When we tried our first seal in a glass lathe in a static position with a fore pump to supply vacuum, an interesting thing happened. When the quartz heated under vacuum, it began to gather making further sealing impossible.

We solved this by removing the jets on one side and making the seal in two cycles. By this method, we are able to accomplish a seal. However, it presented a centering problem.

The first seal would crowd the ampoule against the opposite wall making the second seal more difficult because the plug was in contact with the wall.

The problem of the off center seal was that the seal would not be even and sometimes leak on the side that was crowded against the wall.

If there was a gross difference between plug and ampoule size, a suck-in would occur on the opposite side.

To solve this, we removed jets from each side of the burner (Figure 2). This allowed us to seal the top and bottom first and then the sides. At this time, we found that accurate centering of the burner was very important.

By having the burner exactly centered on the side and slightly closer to the bottom, we could overcome gravity and slightly raise the plug on the first cycle.

From these experiments, our proposed specifications were:

1. Mechanism would mount on a pump station and be out of the way in retracted position.
2. Ring burners will open vertically and clear 74 mm ampoule by 1/2-inch while advancing and retracting.
3. Burners will be interchangeable for 64 mm and 74 mm seals.
4. Positioning adjustments one-inch vertically and one-inch horizontally (front to rear).
5. Sealing position adjustment five-inches horizontally (left to right).

A problem that sometimes occurs with internal mixing automatic burner is backfire on shutoff. The solution to this is to delay the oxygen to shut off after the hydrogen.

When the first automatic burner (Figure 3) was installed, we had to establish times and flow setting that would make a good seal but not be too hot and cause a suck-in.

The burner functioned well mechanically, however, it soon became evident that a good centering system of burner to glass was necessary. At first we tried measuring. But this was difficult and required a steady eye.

The glass to burner alignment was not predictable. It was dependent upon the end cut of the ampoule as to how it would heat in the compression fitting when vacuum was drawn.

Our final solution was an iris that would attach to the outside of the burner ring and close on the ampoule (Figure 4). To center, we would bring the burner forward with the fires off, attach the centering iris to the burner and make adjustments.

We found that even though the seal was made in a narrow area with a sharp flame 0.031" jets had no sublimation occurred on the ampoule (Figure 5). This could be due to the fact that as soon as the outside wall was heated under vacuum, it moved away from the flame and was not overheated.

The most satisfactory seals (Figure 6) were ones that slightly deformed the inner plug evenly around its circumference.

How much fire? When we proposed using the 0.031" jets in a single row, some expressed doubts that the burner would be hot enough to make a vacuum tight seal. We felt confident that it would work provided we could have enough flow.

On our first burner, we used a 90 CFH oxy and a 50 CFH hydrogen flow meter (Figure 7). With this combination, good seals were made, however, the oxy flow meter was nearly full on and tended to bounce. This was corrected by changing to a 120 CFH meter.

The old hand seals had much deformation and were oversealed to avoid any change of leak. With our automatic seal, we wanted the capability of oversealing so that we could control the seal by reducing time and heat.

The existing spec on the ampoule only put a tolerance on the end that fit the compression seal. In order to meet this, the manufacturer had paddled down the ends on some of the ampoules.

At this point of seal, however, there was a gross difference in size. These needed exact centering to avoid a suck-in (Figure 8). From this experience, we established a rule of thumb. If the distance between the plug and ampoule was twice that of the ampoule wall, it was undesirable to make in production.

The repeatability of the process was also dependent on the consistency of wall thickness. On ampoules and plugs on some of our early tests, we used a very hot fire for 30 seconds on the first cycle and 20 seconds on the second cycle. (Less time is required on second cycle due to pre-heat of the first.)

This worked fine until we came upon a thinner ampoule and a resulting suck-in through both walls. This was something we had not seen before. The off-center seals had sucked but only through the outer wall forming a blister which popped in between.

When this happened, we knew we had enough overseal capability and could back off on heat.

We chose to reduce heat and increase time. This accomplished a good seal with some inner distortion but not the hot spots caused by overheating.

The hot spots always occurred when the burners overlap from the first cycle. To further reduce this, the end jets were shortened by 0.100".

These suck-ins were dubbed "hernia's" for which we were always on the lookout. They could be stopped before bursting if you were quick enough on the kill switch.

Due to urgency of project, all debugging was done in production area. This limited us on the number of practice seals we could make.

Before turning over to production, we did three things:

1. Changed the spec on the quartz tubing to a tighter tolerance.
2. Provided iris centering.
3. Cut down on fire and sealed for a longer time.

These measures solved the suck-in problem and we have had a history of good seals.

April 29, 1974

Figure 1. Side view of hand sealed capsule

Figure 2. Prototype burner

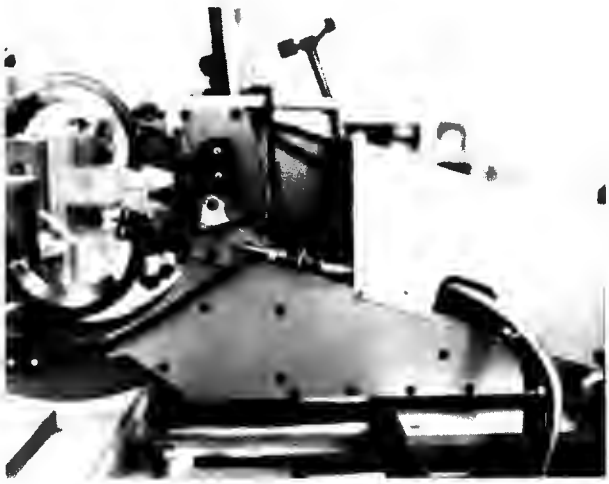

Figure 3. Automatic burner

Figure 4. Close up of centering iris in position

Figure 5. Side view of automatic sealed capsule

Figure 6. Side view of good seal

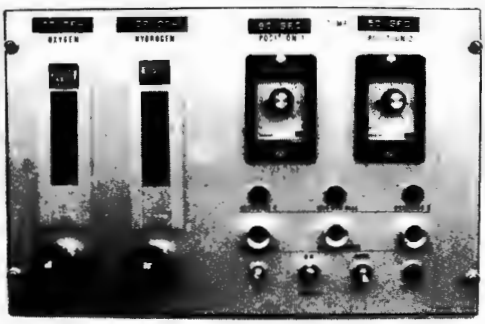

Figure 7. Control panel

Figure 8. End shot of suck-in

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