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ON THE  
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# MANUFACTURING METHODS AND VARIETIES OF FUSED SILICA

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This paper draws heavily on previous publications and various other inputs from Dr. G. Hetherington, Mr. P. Browell, Mr. John Winterburn, and others associated with TSL Thermal Syndicate, England.

It is a brief overview of past and present manufacturing methods utilizing different raw materials and producing a variety of end products having somewhat different characteristics.

All of the finished material to be discussed is properly called "Fused Silica", despite the fact that traditionally the transparent varieties have been called "Fused Quartz", and the translucent and opaque varieties have been called "Fused Silica". The earlier nomenclature arose from the fact that the transparent varieties were generally produced by fusing small pieces of clear quartz crystal while the translucent varieties were produced by fusing high purity silica sand; and because of the nature of the crystals and the ease with which interstitial gases could be removed from either of these materials, the end product was either transparent or translucent. In any case, the product is Fused Silica and should be called, "Transparent Fused Silica", "Translucent Fused Silica", "Transparent Synthetic Fused Silica", etc.

You may have noted that we have already mentioned three starting materials including, by inference, chemical starting materials from which the synthetic varieties are made.

The manufacturing processes can be based on flame fusion or electrical fusion. Fusion can also be done in a "plasma" flame. Fusion can take place at atmospheric pressure, under vacuum, or in the presence of specific gases. Therefore, depending on the method of fusion, the raw material, and other conditions, several types of fused silica may be produced.

These are: transparent, translucent, transparent synthetic, water free synthetic, sand surface opaque, etc.

Opaque or translucent fused silica is produced by fusing high purity "quartz" sand. There are deposits of sands of sufficient purity for fusing in several places in the U.S., but the principal source is the Ottawa Beds in central Illinois. Sand from these deposits, contains 99.8% + SiO<sub>2</sub> after washing.

Our Company uses a process developed in the early 1900's by Dr. F. Bottomley wherein a graphite resistor is surrounded by high purity silica sand. Heat from the resistor fuses the sand outward and a hollow boule is formed. Excess sand in the furnace body acts as a thermal insulator. When a sufficient weight is formed, the boule is removed from the furnace and is blown into metal or graphite molds. The surface of the boule material which was next to the hot electrode is highly glazed. The exterior surface is rough and generally sandy because of unfused or partly fused sand which clings to the glassy part. Because the fusion takes place at atmospheric pressure and because of interstitial gases in the sand, the glassy portions contain myriad fine bubbles.

The combination of bubbles and sand surface gives the finished product a milky, opaque appearance; and so items made by this method are usually called, "Opaque Sand Surface Fused Silica". This method is used to produce tubing and pipes as well as trays, tanks, crucibles, and retorts.

While in the plastic state, which incidentally lasts about a minute after the boule is removed from the furnace, the silica can be rolled into plate by means of a rolling mill. Rolled plate of this kind has a sand surface, but this can be ground off if necessary.

Opaque fused silica articles can also be made by centrifugal molding processes to produce tubes or closed end containers. Either resistance heaters or electric arcs are used for fusion.

Relatively large articles can be produced by the centrifugal process; for example, bell jars, 1 meter diameter x 2 1/2 meters long and with wall thicknesses on the order of 1/2 to 3/4".

Tubing can be produced by several processes. The simplest one uses a heat pot, a die, and a mandrel.

Crystal or sand is fed in at the top and tubing is withdrawn from the bottom as material is fused in the pot. This process is generally known as the "Hanlein" process after its inventor, and is commonly used to produce low quality tubing generally containing surface striae, internal air lines, and bubbles. Transparent and

translucent tubing can be produced by this process, or modifications of it, depending on the starting material.

A common method of producing better quality transparent tubing is to prepare a high quality cylindrical billet by a centrifugal molding process or by fusing in a vacuum chamber or both. This billet is then redrawn by suitable equipment to the desired tubing size. Some manufacturing processes redraw the billet in a horizontal lathe; others use vertical redraw machines.

Naturally occurring raw material for transparent fused silica can come from several sources. Some years ago the principal source was in Brazil, but other sources have been developed in Madagascar, Angola, United States, and Canada.

Quartz crystals can be very small or very large.

In the less-developed countries, mining, cleaning, and grading is done by hand.

Quartz crystal used for fusing has relatively good purity. The principal impurity is alumina. Impurity shown is in parts per million.

An alternate source for starting material for transparent fused silica is byproduct crystal from feldspar mines in North Carolina.

Synthetic fused silica can be produced by burning silicon tetrachloride in an oxy-hydrogen flame.

The vapor phase silicon dioxide produced in the burning process deposits on the hot end of the boule which is simultaneously rotated and withdrawn in order to produce a rod shaped ingot of transparent synthetic fused silica. Other processes based on this principal produce discs or pancake shapes rather than rods.

Recent developments in the fiber optics industry have produced fused silica from soot. The soot is the result of the conversion of silicon tetrachloride or other silicon based chemicals. After the formation on the inside of a tube or on the end of a rod, the soot is sintered and then fused to a solid. In this process, minute quantities of dopants can be added to alter optical properties.

The materials produced by the various fusion methods could be classified into 4 types. That is: electrically fused quartz crystal will produce infrared grades. Flame fused crystal will produce ordinary transparent grades; vapor phase hydrolysis of silicon tetrachloride will produce "wet" synthetic; oxidation of silicon tetrachloride using a plasma flame will produce a "dry" synthetic.

These comments apply to transparent materials. The opaque materials are usually fused electrically, but except for the infrared transmitting properties required in tubing for electric radiant heaters, the transmission properties are generally not of interest.

Starting materials which are fused in an oxy-hydrogen flame have high water content picked up from the combustion products; but on the other hand have slightly lower impurity contents than during the flame fusion process. Because of the lower impurity content, the transmission in the ultra violet is generally better than in electrically fused varieties. On the other hand, the high water content blocks infrared transmission, principally at 2.7 microns. Conversely, electrically fused materials will have good transmission in the infrared, but generally not quite as good transmission in the UV.

Synthetic materials produced by burning silicon tetrachloride in an oxygen-hydrogen flame resulting in silicon dioxide, hydrochloric acid, and water have a high water content; and therefore, high absorption in the infrared. The water content in "wet" synthetic material may be as much as 0.1 weight percent. An exception to this is the synthetic material produced in any oxygen plasma. This type of material has exceptionally good transmission at both ends of the spectrum because the starting material is generally very pure silicon tetrachloride, and the water content present in ordinary synthetic fused silica is replaced by chlorine.

Other optical properties change with method of manufacture. The electrically fused material will generally show high granularity; the flame fused material will show some granularity, but will be an improvement over electrically fused. The synthetic materials will show practically no granularity and are excellent materials for high quality optical applications.

One of the effects of varying water contents resulting from different manufacturing processing is to alter the viscosities and, therefore, the softening points, annealing points and strain points of the materials. Of course, in addition to water content, other impurity content also has the same effect.

The annealing point for electrically fused transparent silica is about 110° C higher than for high water content synthetic fused silica. The strain point is about 120° C higher.

A typical annealing cycle for transparent fused silica raises the

temperature to 1050° C over about 4 1/2 hours, holds at that temperature for about 1/2 hour and then gradually cools to room temperature.

Fused silica tends to devitrify at elevated temperatures, and this normally begins at about 1000° C. It can be accelerated by surface impurities, for instance; finger prints or by internal impurities, for instance; pieces of furnace refractory, etc.

The effects of the devitrification are not noticeable at elevated temperatures.

A series of slides is presented to show the progress of devitrification as a sample of tubing cools, after being heated for 24 hours at 1450° C. At 1000° C none of the effects of devitrification are visible. The sample of tubing is placed on a cold surface, and as the tubing is placed on a cold surface, and as the tubing cools, the effects of devitrification spread.

If the fused silica is maintained above about 300°C, the effects of devitrification are not noticeable. It is only in cooling through 275°C that the crystalline structure changes and devitrification becomes evident.

As is mentioned earlier, transmission properties of fused silica can be altered by adding impurities; for instance, addition of small amounts of impurities can block ultra violet so that, for instance, UV lamps can be produced which do not generate ozone.

This has been a review of some of the methods of manufacture of various types of fused silica and a brief look at the effect of those methods on the physical properties of the end products. For most of us, producing fused silica is a difficult process requiring ultra pure raw material, high energy input and considerable care in manufacture.

For certain people from more advanced cultures, it's easy!



## FLASH LAMPS FOR THE DESTRUCTIVE MODE

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In the past 30 years a substantial body of information has been accumulated on non-explosive reactions in shock fronts. This information suggests that the effect of a shock wave in producing a chemical reaction goes beyond the simple effects of pressure and temperature. There may be direct interaction between the shock front and the individual molecules which influence their chemical states.

Using optical spectroscopy as a diagnostic tool, the Shock Dynamics Group at Washington State University is conducting experiments to identify spectroscopic features which are sensitive to compression and search for evidence of the dependence of these features on the rate of compression and for evidence of catastrophic shock fronts.

Figure 1 depicts the 14-meter gas gun used to produce the shock waves in the sample. The sample is contained in a sapphire cell mounted in a fixture at the end of the barrel in the muzzle room. Measurements of spectra involve a sequence of events initiated by the impact of the projectile face with the target face. The required light source is a Xenon flash lamp, especially designed to provide light to the sample at impact. Light is transmitted from the sample to a grating spectrograph and the resulting spectrum is recorded using either a rotating mirror streak camera or an electronic image convertor camera. The gas gun is capable of firing the projectile at speeds in excess of 3,000 ft/sec, about the speed of a high powered bullet.

Figure 2 shows the flash lamp which is the heart of these experiments. The flash lamp presents some complicated and unique design problems. A few of the requirements are:

1. A bright, broad band light source with substantial output in the ultraviolet.

2. A lamp geometry which will accommodate some rotation of the impactor and still be in view and face through the cell.
3. Lamp must stand at least one test flash of 50 Joules before it is flashed to destruction at 200 Joules.
4. Exposed electrodes must be protected from the partial vacuum of the target chamber to prevent discharge outside of the tube envelope.
5. Flash lamps must be inexpensive since they are destroyed in each experiment by projectile impact.

Figure 3 shows a sketch of the overall projectile and target assembly. The flash lamp is glued into the target holder with its curve portion exposed and elevated so that the side looking mirror on the projectile will direct the light of the flash through the cell just at impact.

The envelope is constructed from fused quartz and consists of a 3 mm by 5 mm curved portion sealed to 12 mm diameter tubes which contain the electrodes. The larger diameter (12 mm) tubing which holds the electrode assembly helps prevent tube failure during the test flash and provides a well for the sealing material for the tube and high voltage electrode. The electrodes are made from 1/8-inch thoriated tungsten. These are cut from heli-arc electrodes, a commonly available item.

All the tubing for the flash lamps are cut to eight-inch lengths and washed with a 10% solution of hydrofluoric acid for about 30 seconds. The curved portion of the lamp (Fig. 4) is made using a carbon-forming gauge to achieve the exact radius and also location of the bend to seal the larger tube.

The actual construction of the flash lamp presented some interesting problems because of the geometry and tight tolerances. The large tube is made by heating and tapering to one side more than the other. The ends are ground down to match the size of the smaller tubes. The outside diameter of the small tube is in the same plane as one side of the large tube. After all parts are cut to size, they are dipped in hydrofluoric acid again to clean the ground surfaces.

Figure 5 shows the sealing fixture for sealing the parts together. This fixture was made to hold flash lamp pieces while making



seals. One side is sealed by heating both surfaces and sealing together leaving the final working out of seal until after the other side is sealed. Seal other side by using quartz rod to fill in gap. Work out both seals using standard quartz working techniques.

Because this lamp is test-fired at the maximum design energy of 50 Joules, all seals and surrounding areas must be free of bubbles visible to the naked eye.

All bloom must be removed.

The electrodes are pressed into brass plugs which center the electrode and act as a dam for the sealing material. "Torr seal" has been found to be the best sealant. One brass plug has a filling tube added.

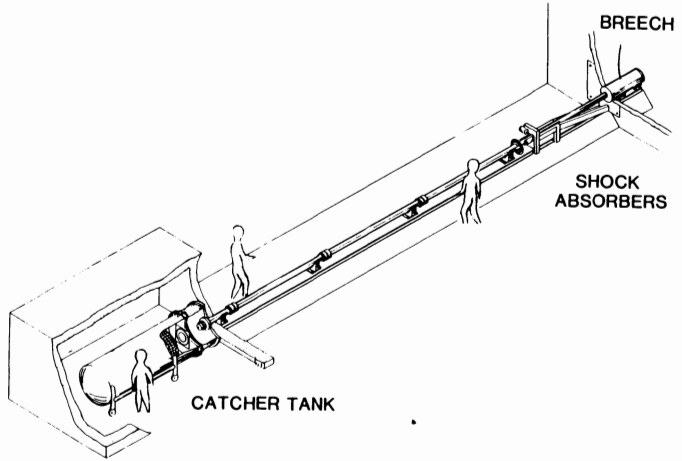
The assembled flash lamp is rough pumped to about one micron of vacuum for two days and then back-filled with Xenon to between 450 and 700 Torr. Many of the lamps back-filled to 700 Torr fail during the test firing.

Quartz envelopes are supplied at about \$15.00 and about an hour of technician's time is spent in assembling the tube. This has provided an inexpensive and reliable flash lamp for spectroscopy in shocked materials.

One early design required using 1/8 thoriated to quartz seals. This process was not successful. Shock Dynamics insisted that we use thoriated tungsten which is another reason we went to copper cup and Torr seal.

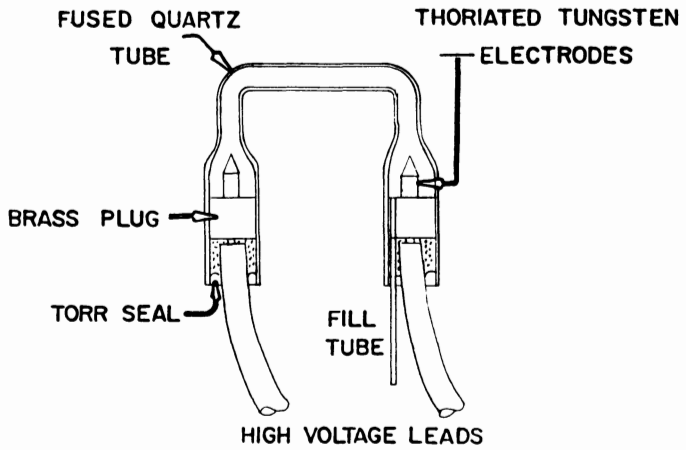
Several different styles of flash lamps were tried with varying degrees of success.

Acknowledgement is made to Jack Snowden of Technical Services, Division of Sciences, Washington State University, for the graphics.

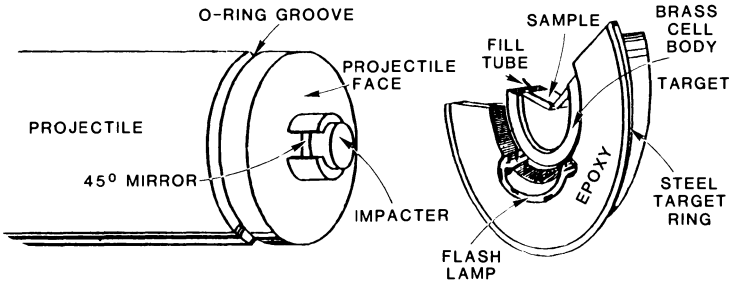


**Figure 1**

**CURVED UV FLASH LAMP**

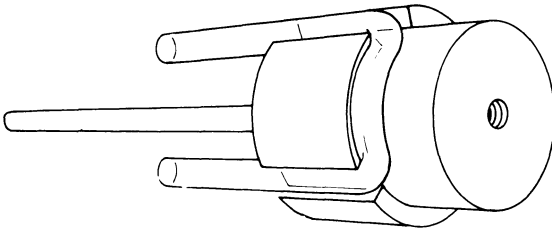


**Figure 2**



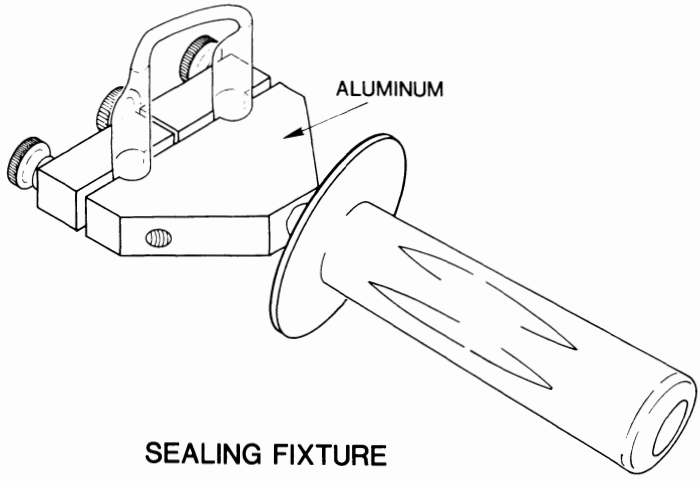
### PROJECTILE AND TARGET

**Figure 3**



### FORMING GAUGE

**Figure 4**



**Figure 5**

# HELIUM-NEON LASERS; THEIR PHYSICS AND CONSTRUCTION

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## INTRODUCTION

The following report is intended to illuminate the engineering and construction of helium-neon (He-Ne or HeNe) lasers. Whereas laser plasma tube construction is of the realm of the glassblower, an understanding of principle, design and terminology may be appreciated and useful. As reliable production of uniform and flawless lasers is rather capital intensive and can ultimately involve automated glass-working, this paper may be of only casual interest.

### Part One

#### HELIUM—NEON LASER PHYSICS

An intense and unique emission of light is exhibited by a helium-neon *lasing medium*\* when placed in a geometrically proper *optical lasing cavity*, at a prescribed pressure, and when *excited* by some optimum electrical discharge. Although the outer shell electrons of both helium (He) and neon (Ne) are *pumped* (shifted to outer, higher energy orbitals around the nucleus) by direct collisions with the free electrons of the discharge, the *laser action* occurs as a result of electrons in the outer (*excited*) metastable levels of Ne atoms falling to lower energy orbitals, the energy difference being emitted as photons. The He is added to the lasing medium to assist pumping by the resonant transfer of its energy to the Ne. This resonant transfer of energy is indeed the principal pumping mechanism for the Ne. There are three distinct transitions and thus three distinct wavelengths of photons emitted by Ne; they are  $3.39\mu\text{ m}$  (infrared),  $1.15\mu\text{ m}$  (infrared), and  $0.633\mu\text{ m}$  (red).

When the population of excited atoms exceeds the population of unexcited atoms a *population inversion* exists, and upon reaching some critical ratio the medium will be capable of lasing. As the decay time for

\*Key words which are a part of the huge redundant jargon of laser science will be italicized when first used in the text.

excited Ne is much shorter than for excited He, the Ne upon reaching its ground state is quickly pumped again by the excited He. By pumping, the population inversion is sustained and *continuous wave (cw) emission* results.

An excited Ne atom is stimulated by an appropriate photon to emit a photon exactly resonant with the stimulating photon (this is *first order coherence*). In the process, the Ne atom's deexcited electron falls to a lower energy level. Both the stimulating and the stimulated photons leave the Ne atom in step with each other and in the same direction (this is *second order, or spatial coherence*). This is *stimulated emission*. Placing this lasing medium in an optical cavity (between highly reflective multi-layer dielectric — *quarter wave stack* — mirrors) allows the generation and amplification of an oscillating cascade of emissions.

The degree to which stimulated emission rather than absorption (*radiation trapping*) occurs is indicated by the *gain coefficient*. Laser gain will vary inversely with *plasma cavity* diameter as long as the pressure-to-diameter ratio is kept constant. Laser gain will also increase in direct proportion to pressure. A HeNe laser oscillating at  $\lambda = 0.633 \mu\text{m}$  has about a two percent gain per pass.

For any laser a power supply must be tailored, as there is an optimum current density. Too high a current density can excite Ne atoms to higher, nonlasing orbitals, or can deexcite metastable electrons by a superelastic collision process whereby the energy is transferred to electrons in the discharge. At some higher current density the glow discharge in the medium will arc due to the ionization of the lasing medium.

Energy loss also occurs by the lasing medium's colliding with the cavity walls. This is taken advantage of in bringing the Ne atoms which have just emitted photons fully to their ground state where they can once again resonate with excited He. Bringing such atoms to their ground state is inversely proportional to plasma cavity diameter and occurs at a rate dependent upon diffusion. As some atoms are stripped of their electrons, these ions can recombine with electrons only at the wall where the discharge density is zero.

Since the optical cavity is many times longer than the wavelength of the emission, any of many possible standing waves may establish themselves in the cavity, depending upon the cavity diameter and separation and curvature of the cavity mirrors. These standing waves

result in the structure of the beam produced to be described as of a particular *mode*. Of note, the *Gaussian* ( $TEM_{00}$ ) mode results in a beam producing a patternless spot. Other modes are seen to be patterns made by discreet beamlets.

It is by varying cavity geometry (principally bore diameter) that mode is controlled. Since spontaneous emissions may occur in any direction, an optical resonating cavity is established with mirrors. All oscillations other than those parallel to the resonator axis will dissipate into the walls. Although cavity diameter may range up to a few centimeters, most commercial lasers have diameters of one to six millimeters for reasons of mode control.

Maintenance of mirror alignment for *closed cavity* lasers (those with mirrors sealed to the plasma tube) is achieved by affixing (typically one of) the mirrors to a *tilter mechanism*. The higher gain plane parallel resonator, and concentric resonator mirror alignments wherein mirror radius equals one half of the resonator length are very sensitive. Less sensitive arrangements are typically used with spherical mirrors of radii up to ten times cavity length, or a flat and spherical mirror of long radius. These latter examples (termed confocal and hemiconfocal respectively) make efficient use of *cavity cross section*.

A window inclined at Brewster's angle (a *Brewster's window*) will transmit without reflective loss, any light which is polarized in the axis of the laser and normal to the angle of the window. Brewster's angle is dependent upon the refractive index of the window material. As reflective losses increase in proportion to deviation from planes normal to the window angle, the preponderance of the emission is polarized by the window. This is *linear polarization*, and can be achieved with a closed cavity laser by incorporating a Brewster's window just inside of an end mirror. Photons reflected back into the medium can be trapped by Ne. Upon stimulation by the oscillating beam, the Ne thus pumped emits the photon thereby amplifying the linearly polarized output.

Lasers in general may exhibit the phenomena of *spatial* and *temporal coherence*. To the extent that different parts of the beam are in phase with or correlate to one another, the output exhibits spatial coherence. To the extent that diverging wavelengths emanating from a laser illuminated pinhole are in phase with one another, the output exhibits *temporal coherence*. Both spatial and temporal coherence can be

demonstrated interferometrically, and are characteristics of a given output to degree and are not absolute, binary things. Temporal coherence is proportional to monochromaticity and thus inversely proportional to the oscillation bandwidth (which for HeNe lasers may range up to about 500 Hz).

The *directionality* or non-divergence of a laser output is again a matter of degree, and inversely proportional to the beam diameter.

## Part Two

### HELIUM-NEON LASER CONSTRUCTION

HeNe lasers are either of *open* or *closed* (optical) *cavity* design. An open cavity laser requires external mirror(s) (*output coupler(s)*) to close the optical cavity. Such a laser tube would terminate with Brewster's window(s). The mirrors of a closed cavity laser are sealed directly to the tube.

*Soft-sealed* lasers have optics and/or other components attached either with an adhesive or by an arrangement of clamps and gaskets. The adhesive of choice would be an epoxy formulated for high vacuum use. As the laser tube may become quite hot in use, it is important to also consider this in seal design. By selecting an epoxy with a pot life on the order of thirty to sixty minutes, the extraction of a considerable amount of volatiles can be accomplished by degassing the epoxy in a vacuum chamber with a small mechanical pump. 'Filling' the epoxy with up to 50% of an inert substance such as powdered aluminum, will also help avoid contamination from and through the adhesive. Numerous substances, among them water, can diffuse through epoxy.

*Hard-sealed* lasers are assembled without the incorporation of adhesive nor elastomer seals. This is accomplished by the use of glass to metal and solder glass seals. Atmospheric gasses can diffuse along a heavily oxidized interface of a glass to metal seal, thus Kovar seals should be light gray and tungsten seals should be tan. Spotty or black-spotted seals should be rejected. Electronic grade tungsten (as opposed to welding grade 'stingers') should be used for electrical feed-thrus, as lesser grades may have microscopic striata through which gasses may pass.

Solder glass seals are used to hard-seal the optics into their receptacles. Careful matching of the thermal coefficients of expansion of the



optic, the solder glass and the substrate material will result in a strong hermetic seal without imparting birefringence to the optic as a result of compressional or tensional strain. The solder glass may be applied either as a paste, or preferably as a pre-formed ring which is more simply inserted into place with the optic. As the various solder glasses have prescribed heating schedules, a microprocessor controlled programmable oven is the means of choice to effect the seal.

The laser plasma tube should be constructed of precision bore capillary. For lasers of certain designs it is convenient to use all 7052-type glass to avoid graded seals. To facilitate construction, tungsten or stainless steel mandrels can be centerless ground and then coated with Aquadag and used to maintain the trueness of the bore during sealing operations.

The discharge to the cathode should be diffused by the incorporation of a piece of glass tubing to act as a shield. Without such a shield the discharge may focus upon some one or more regions of the cathode causing pitting and hot spots on the cathode and consequential sputtering of cathode material into the bore of the plasma cavity and onto the end windows or mirrors of the tube.

Proper cleaning of all metal parts in the laser is critical, as impurities are incorporated into all machined surfaces. The most serious contaminant is cutting oil which becomes encapsulated into microscopic tears in the machined surface which are formed and then pinched shut by successive passes of the metal working tool. The cleaning process employed must remove the worked surface in such a manner as to avoid further penetrating contamination. Electropolishing can be very effective. Chemical etching is another, more typical means. As an example, aluminum may be prepared as follows:

- 1) Degrease (preferably vapor degrease) parts with a solvent
- 2) Brush (preferably ultrasonically clean) parts with hot soapy water
- 3) Rinse parts with hot water
- 4) Etch parts in a potassium hydroxide solution (two hours in a one molar solution at room temperature will remove approximately 0.002 in. from all surfaces of most types of aluminum)
- 5) Rinse with cold water

- 6) Neutralize in a 10% nitric acid solution for up to ten minutes
- 7) Rinse with cold water
- 8) Rinse extensively with distilled water
- 9) Rinse with methanol to displace water
- 10) Bake off adsorbed water at up to 300° F overnight (preferably in a vacuum oven)
- 11) Store in a clean vacuum container prior to assembly

This procedure will produce a uniform matte finish. Being thus roughened, the aluminum's surface area is greatly increased, resulting in better cathode performance. Seamless tubing should be utilized, as welded tubing will etch unevenly and can still be contaminated with welding flux.

Virtual leaks may be inadvertently incorporated into a laser by impurities not being completely removed from components, from air and oils incorporated into the metal surfaces, diffusion of volatiles from and through elastomers and adhesives, volatiles adsorbed onto components during storage, fluxes, corrosion, and fragments from equipment used to clean the components (as paper, cotton and bristles). Such contaminants can result in a film forming on the intracavity surfaces of the optics. Debris in the plasma cavity will disperse considerable light, diminishing gain, as may readily be apparent. These problems may be avoided only by careful materials preparation, storage, assembly, and filling in a scrupulously clean environment. Such environments are provided only by cleanrooms, clean boxes, or workstations with HEPA filtered laminar air flow. In all cases non-particle shedding laboratory garments (including for beards and hair) and non-powdered disposable gloves or finger cots must be used. Cosmetics should not be worn. Critically cleaned components should be baked at up to 300° F overnight to drive off adsorbed moisture, and then stored in clean vacuum chambers until ready for assembly.

Consideration must be given to preventing particle liberation in laser assembly, shipping and use. Ground glass surfaces which may contact other surfaces should be fire polished.

Once assembled and affixed to the filling manifold, the laser tube is evacuated to a hard vacuum (about  $10^{-6}$  torr). This evacuation can be accelerated by warming the tube with a heat gun which drives off residual adsorbed gasses. The tube is then checked for leaks with a tesla coil, or better, a helium leak detector. Once evacuated, a few torr of

research grade oxygen is admitted to the tube and an alternating electrical discharge is applied to arc-clean the plasma cavity and simultaneously lightly oxidize the cathode. After several cycles of this arc-cleaning, the tube is once again evacuated to a hard vacuum and then filled with the lasing medium. For  $\lambda = 0.633 \mu\text{m}$  (red) emission the optimum  $^3\text{He}$  to  $^{20}\text{Ne}$  ratio is about 5 to 1. Optimal total gas pressure can range from  $4/d$  torr to  $5/d$  where  $d$  is the plasma cavity diameter in millimeters.  $\text{TEM}_{00}$  output predominates from a  $1.50 (\pm 0.15)$  mm diameter plasma cavity with hemiconfocal optics. Note that the  $^3\text{He}$  isotope is used as it is more efficient at resonantly pumping the naturally occurring  $^{20}\text{Ne}$  isotope. Suppliers offering laser quality gasses (in excess of 99.99% pure) typically also offer premeasured HeNe laser gas mixtures.

It should be mentioned that ultimately the laser is only as good as the filling station used to charge it. It must be scrupulously clean and may incorporate an activated charcoal filter chamber and/or cryogenic traps just ahead of the pumps.

Once filled and prior to 'tipping off', the laser should be connected to a suitable power supply and tested for proper function. If lasing does not occur, the filling operation should be repeated. Testing should be repeated after tipping-off to verify that a leak has not been made in the tipping-off operation. Should such a leak have been formed, the tube can be opened by carefully scoring with a diamond pencil around the entire circumference of the filling tip and cracking the tip off by 'hot sticking' the scratch (applying the hot, softened end of a piece of cane to the scratch). Water should not be applied to the scratch, as it will be sucked into the tube when the tip cracks off. A fresh filling tube is quickly fused to the laser tube using a microtorch and hydrogen and oxygen fuel. As the combustion by-product of this fuel mixture is water, it is advisable to bring the tube back down to vacuum as soon as possible, to avoid allowing the water to disperse into the tube.

After tipping-off the laser from the filling station, the getter is fired. Getters are of two general types; the dispersive barium getter and the nondispersive zirconium getter. Having scrupulously cleaned the laser, it makes good sense to use the nondispersive type getter which incidentally is much better at sorbing the sorts of contaminants likely to be found in especially soft-sealed lasers. If provision is made for electrical

feed-thrus, a resistance heater activated getter may be used, otherwise the getter is fired by radio frequency induction heating. To increase the shelf life of the laser it may be advisable to *soft-fire* (partially activate) the getter after construction and *hard-fire* the getter immediately prior to shipping or use. Detailed instructions for this may be obtained from the getter manufacturer. As with all components, getters should be stored in clean vacuum containers prior to assembly of the laser tube. If the getter is to be fired by induction heating, the laser tube and RF antenna must be constructed in such a manner that other metallic components of the laser are not excessively heated. This is especially important with soft-sealed components, as the adhesive can be destroyed by excessive heating.

Once assembled and the getter is fired, the laser should be operated continuously until the output stabilizes. This is called '*aging-up*', for the output increases with time. This may take from eight to forty-eight hours. After having aged-up, the laser should reach peak output within fifteen minutes of starting-up cold.

Lasers of some designs can be easily opened-up and refurbished when necessary, but other lasers cannot be repaired effectively. They can be disassembled and components can be salvaged, but unless they can be reassembled after critical re-cleaning, and with a fresh getter, the lasing medium and optics will inevitably become contaminated, and the laser will eventually fail again.

Corporations employing technicians and utilizing automated machinery, RF and oven heating, are today producing HeNe lasers in high volume and at low cost. There is at most the flame-working of one or two components which again is accomplished by a technician. Automation has thus excluded the glassblower and his or her talents from all but the initial design stage of this latest generation of HeNe lasers. Indeed, the advances made in solid-state 'laser-on-a-chip' technology may make HeNe lasers obsolete in the very near future.

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## ULTRASONIC MACHINING (Impact Grinding)\*

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### ABSTRACT

This presentation will describe ultrasonic machining (impact grinding) of hard brittle materials in general and its use in optics at LLNL.

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Ultrasonic machining or impact grinding is the use of ultrasonically induced vibrations delivered to a designed tool combined with an abrasive slurry, to produce accurate cavities of regular and odd shapes in hard brittle materials such as: fused quartz, glass, crystal, ceramic, carbides and various metals. Ultrasonic machining is a non-thermal, non-chemical, non-electrical process, and creates no change in the metallurgical, chemical or physical properties of the substrate.

The ultrasonic vibrations are produced by coupling an electronic generator, a magnetostrictive package, a transmitting cone and a tool holder-tool combination (figure 1). The 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 silver soldered to the transmitting cone and the two together are referred to as the transducer. The transducer converts the high frequency power into mechanical energy, oscillating linearly .001", 20,000 times per second. A tool is silver soldered to a tool holder and this combination is connected to the transducer by means of a 1/2 - 28 stud. The tool holder-tool combination receives the mechanical energy from the transducer. The tools are normally made of 304 stainless steel, because of its machinability, wear rate and soldering qualities. For singular operations and ease of fabrication, brass can be used.

The length of the stroke at the tool face is determined by the type of tool holder selected. There are two types: the non-amplifier and the amplifier. The non-amplifier tool holder is merely an extension of the transmitting cone, varying in diameter from 1 to 3 inches and producing the same length of stroke as the transmitting cone (Figure 2a). The non-amplifier tool holder is used with grinding compounds with a particle size of less than .001" such as #600 boron carbide. This combination has a slow cutting rate but produces a finish of 15 micro-inch or less and can hold tolerances of .0005" or better. The amplifier tool holder ranges from 1/4" to 3-1/2" in diameter and uses three basic shapes: the tapered cone which is the most widely used, the double cylinder and the chisel. This tool holder expands and contracts within its own length, thus amplifying the stroke to a maximum of .0025" (figure 2b). The amplifier tool holder is used with grinding compounds having a particle size no greater than .0025" such as #240 boron carbide. This combination has a fast cutting rate and produces a 25 micro-inch finish. Sometimes it is necessary to use both operations described to obtain the required size and finish. This method of machining a cavity is limited in size and shape only by the size of tool holder and the ability to produce the desired tool shape.

The most important item of the ultrasonic machining process is the abrasive. The widely used abrasives are: boron carbide, silicon carbide and aluminum oxide. We have selected boron carbide for our operations because of its ability to cut the harder materials without excessive break down of the particles. Under normal usage, boron carbide will last about 200 hours in comparison to 60 hours for silicon carbide. The abrasive is suspended in water at approximately 50% by volume and is fed to the work piece at a rate of 7 to 7-1/2 gallons per minute. This high volume of slurry acts as a coolant for the tool face and the work piece as well as maintaining a fresh supply of abrasive to the cutting area and removing the cut particles. The fast motion of the tool face drives the abrasive into the work piece creating the desired cavity. Since the abrasive is the cutting surface of the tool, it determines the overall size of the cavity in relation to the tool design, the finish, and the cutting rate. It has been found that a vacuum assist system will increase the efficiency especially when machining blind

or small holes. This is accomplished by pulling a vacuum through the center of the tool holder-tool combination.

Some of the applications of ultrasonic machining are: slots, precision holes, small holes, blind holes, odd shaped holes and multiple hole and pin patterns. An example of these operations follows: starting with a polished piece of fused quartz, .060" thick with parallel faces, two slots .118" wide, 3-1/4" long and .032" apart to a depth of .030" and joined at one end were machined to within a tolerance of .0005". The requirements were that the slots be parallel to the surfaces and have sharp square corners at the bottom. A very important factor was that these parts be easily reproducible because there would be a need for a number of them. This was accomplished by making a stainless steel tool to the necessary shape and ultrasonically machining the part in one operation (figure 3).

Precision holes and small holes are the most called for in the field of optics fabrication. As an example of precision holes: fused quartz discs 3" in diameter and polished on one surface to a 20th wave flatness are used by the Laser Group in large quantities (figure 4). The requirements are that precision holes, varying in size from .022" to .170" in diameter be machined through the disc at a 45° angle, with a clearance hole .375" in diameter on the back side. There must be no chipping on the front surface where the hole comes through or any distortion of the optical flatness. As is evident from figure 5, ultrasonic machining does not have any effect on the optical surface. This two stage hole is done in a single operation. The machining of an elliptical hole through a like substrate at a 45° angle was also done by ultrasonics, since there was no other way. The actual machining time of these parts is from 3 to 5 minutes. The smaller the hole the longer the time.

The producing of holes from .010" to .200" by means of ultrasonics has become routine. These may be through holes or blind holes and can be easily accomplished with this method. Blind holes with any desired shape at the bottom are readily reproducible. Since the tools to make these holes are simple to fabricate as compared to obtaining diamond tools, the time and expense saved is appreciable. Time study charts and graphs are available to compare ultrasonic machining with conventional methods and in most cases it is found that ultrasonic machining is

much faster, especially for small holes, blind holes and naturally for those of odd shapes.

The simultaneous machining of multiple holes in a precise pattern is one of the unique capabilities of ultrasonic machining (figure 6).

The light absorption sample in figure 7 is a lamination of two different glasses and could not have been made in any other way. A brass tool was made by drilling the required number of holes to leave 50 % of the surface of the sample in tact and penetrating the bottom layer to a depth of .010", to a total machining depth of .050." Since it was necessary to use fine grinding compound this operation takes about 6 minutes of machining time. This method was also used to produce precision pins in great quantities in a single operation. The diffusion cell in figure 8 is a good example of the use of ultrasonics in the field of optics. Over 40 % of the mass of this part was removed without making any changes in the optical surfaces of the original precision block.

For some other examples of the use of ultrasonic machining see figures 9 and 10.

Ultrasonic machining is a safe operation. One potential hazard to the operator is excessive physical contact with the tool holder-tool combination and the slurry when the machine is in operation, this could cause serious tissue damage. However, there is no reason for an operator to be in contact with either while the machine is running. Secondly high pitched noise can cause discomfort if the machine is not properly tuned.

Finally, sometimes there is no other way. Ultrasonic machining can be used for the routine machining of holes and producing discs in various sizes with great accuracy, good finish and high production rate. The real value of ultrasonic machining is doing the jobs that cannot be done any other way, namely, the machining of small holes, blind holes, coring, multiple hole and pin patterns or unique shapes in non-conductive materials with high precision and a rapid cutting rate.

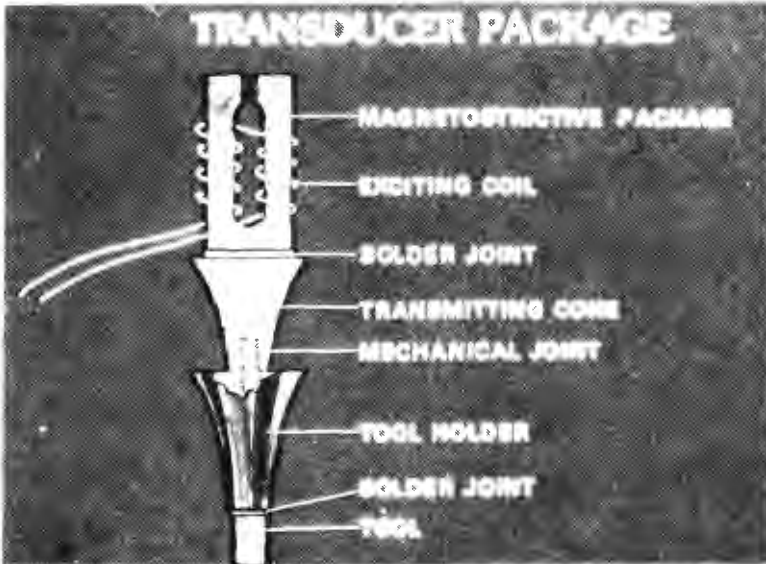
#### **ACKNOWLEDGEMENTS**

My thanks to Steve Brown of Bullen Ultrasonics, Inc. for supplying some of the substrates depicted in this report.



**DISCLAIMER**

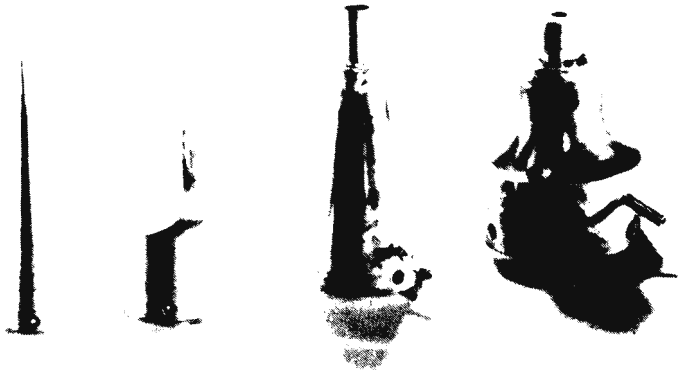
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**Figure 1. Transducer Package**



**Figure 2a. Non-Amplifier**



**Figure 2b. Amplifier**

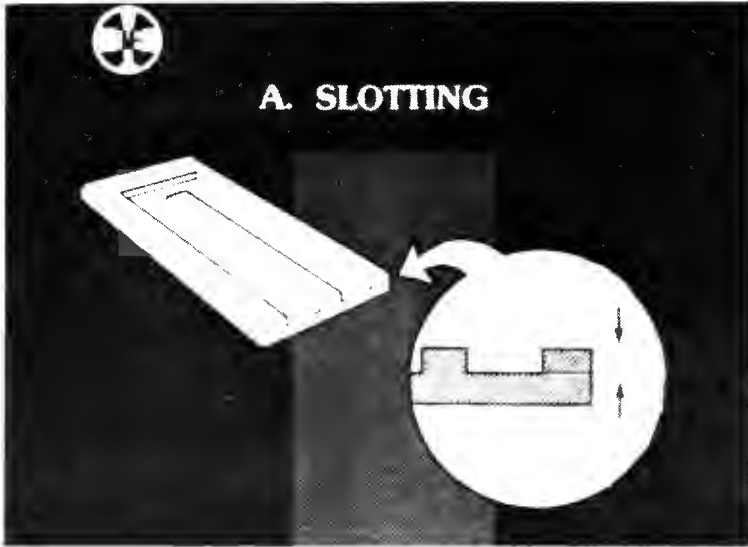


Figure 3. Expansion Cell

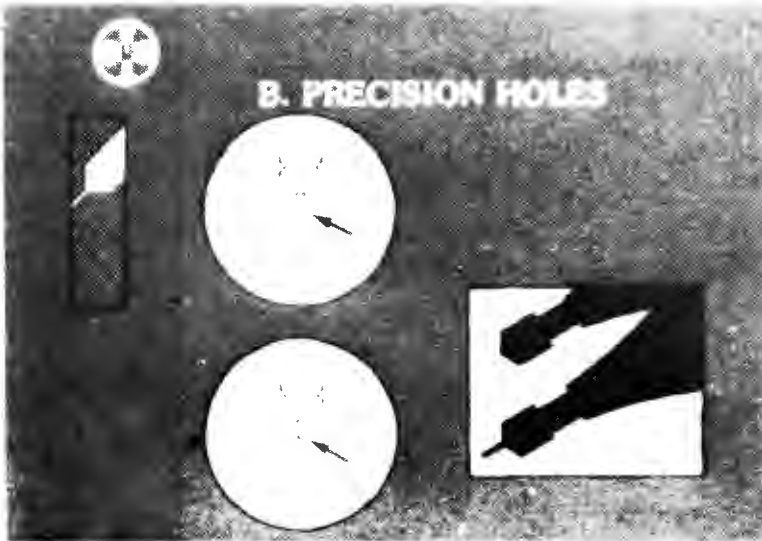
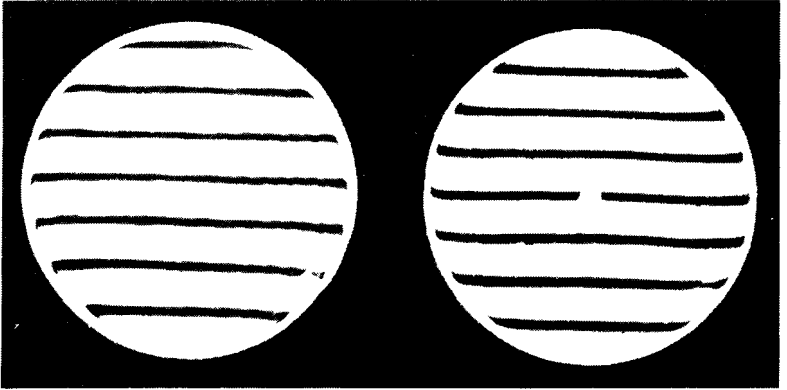


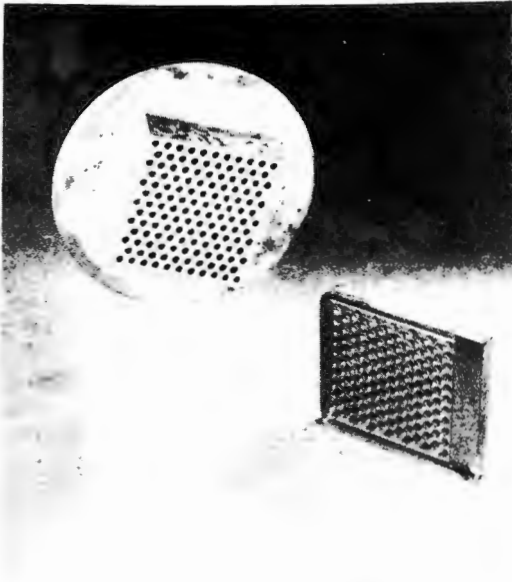
Figure 4. Scraper Mirror



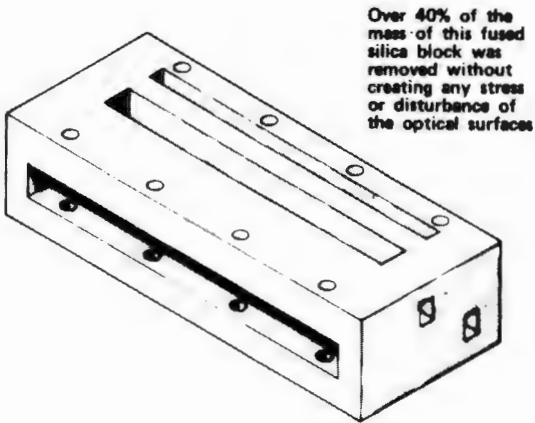
**Figure 5. Interferometer Comparison**



**Figure 6. Multiple Holes**



**Figure 7. Multiple Pins**



Over 40% of the mass of this fused silica block was removed without creating any stress or disturbance of the optical surfaces

**Figure 8. Diffusion Cell**

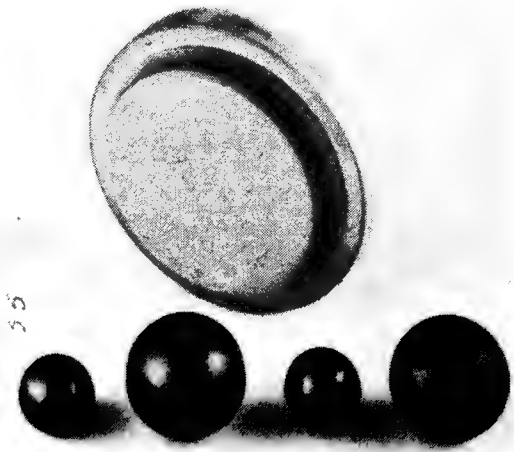


Figure 9.

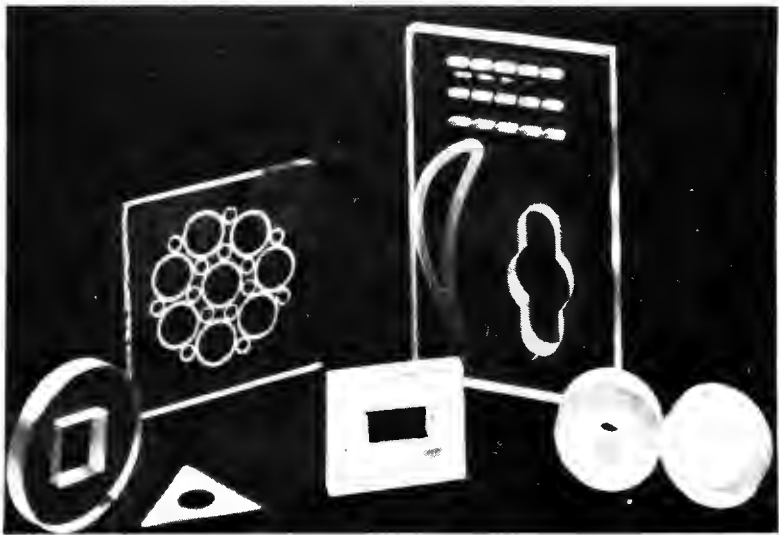


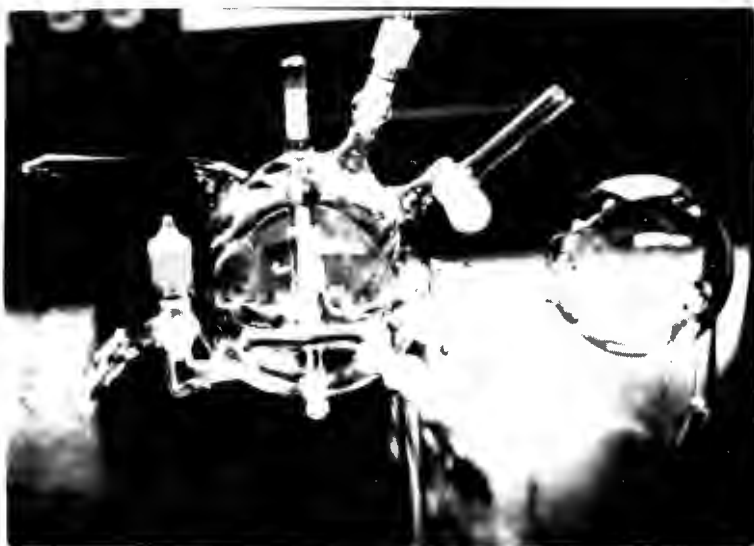
Figure 10.

## CONSTRUCTION OF A LOW FRICTION GAS MIXING BULB

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When I was first asked to redesign a gas mixing bulb that would have very little friction or no glass turning on glass, I did what I hope most of us do, and that is go to the cumulative index and see if someone had built one before. As luck would have it I found something of an almost appropriate design but it still had glass to glass contact. So I set out to improve the design and that is what this paper is about. The desired piece was to be 1 liter with vanes inside that could be rotated from an external source, in this case a magnetic stirrer. I felt that the best surface for these to rotate on would be Teflon®. The first slide (Fig. 1) shows the end result. Now I will back up and explain how I got them inside. One primary concern was to make sure that the finished flask could be oven annealed. The first step was to build the inner part with the vains keeping everything as light as possible. The 4-vains were cut from flat glass. In some cases I have used pieces cut from tubing that I flattened myself. These pieces are then attached to the 8 mm rod of the central shaft (Fig. 2). Then the rod is attached to the tube that is to contain the 3" stirring bar. Care must be taken to make sure that this is in the exact center of the bar, if not it will either not rotate or cause an undue amount of vibration. Now the bottom rod is sealed to the tube. The stir bar is then sealed to the tube, finished to the desired length and oven annealed. While this was taking place, I had the machine shop make my two teflon bearing pieces, so that they would fit loosely over the 8 mm rod and also fit rather loosely into a 13mm tube. At this point a 13 mm tube was sealed onto each end of a 1 liter flask (Fig. 3). The flask was then cracked in half using a diamond pencil and heat shock (Figs. 4 and 5). We are now ready to assemble the whole thing. I usually leave the flask mounted in the lathe after cracking off and then use some corrugated paper at each end to hold the inner piece. The flask is then sealed back together, and any additional stopcocks or arms are sealed on taking care that at

least one of them is 13 mm. After the flask has cooled the corrugated paper is removed from the bottom and the inner part is moved to the opposite extreme. The bottom 13 mm tube is now closed off and the whole flask is placed in the oven for annealing. The Teflon® pieces were inserted by using a piece of wire and a rod in the inverted position, as shown in Figures 6 and 7. The finished flask (Figure 1) is then ready to be installed in the vacuum line.



**Figure 1**





**Figure 2**



**Figure 3**



**Figure 4**



**Figure 5**



**Figure 6**



**Figure 7**



# FABRICATION TECHNIQUES AND PROPERTIES OF OPTICAL FIBERS

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## INTRODUCTION

Optical fibers are thin strands of glass which can transmit light over long distances. They have resulted in many improvements in communication for applications ranging from data transmission and instrumentation to telecommunications. The advantages offered by optical fibers over conventional metal cables include faster data transmission rate, smaller lighter cables, elimination of “noisy” electrical interference and greater security in information exchange. Basic properties of optical fibers and methods of manufacture will be described including the major contributions of Hewlett Packard to the present technology.

## FIBER PROPERTIES

Glass optical fibers are pulled or “drawn” from a glass rod called a preform. They are coated with several protective coatings and then cabled in a manner similar to wire. A finished cable consists of a doped silica core and pure silica cladding, a soft first coating, a harder inner jacket, fibrous strength members and an outer jacket. (Fig 1) Important properties of optical fibers which will be described include core size, numerical aperture, attenuation, bandwidth and strength.

Core size describes the physical cross section of the light transmitting part of the fiber. The core is silica, modified by doping with other elements to achieve a higher refractive index than the surrounding cladding. (Fig 2) This change in refractive index causes light to be totally internally reflected and transmitted along the fiber. The size of the core directly affects how much of the light source can be collected by the fiber. Larger cores will collect and transmit more light.

Numerical aperture is the sine of the angle of incident light which can be collected and transmitted by the fiber. Higher

numerical apertures are obtained by doping to a higher refractive index and enable more light to be transmitted. (Fig. 3)

Attenuation is a measure of the light lost during transmission through the fiber. This loss is caused by scattering from defects such as bubbles or inhomogeneities in the glass and by absorption due to impurities in the glass. Eliminating impurities are a major concern in optimizing attenuation of fibers because metallic and hydroxyl impurities (which form from water vapor) cause high attenuation at levels less than a few parts per million. (Fig. 4)

Bandwidth is a measure of how fast information can be transmitted along a fiber. This speed is limited by the spreading or dispersion of a light pulse as it propagates along the fiber and is controlled by the doping profile. A parabolic profile enables transmission of pulses without spreading which allows faster pulses to be sent without interference. (Fig. 5)

Another important fiber property is strength, which must be sufficient to allow tensile stress and bending during installation and use. This property will be discussed in more detail later.

Two major distinct types of optical fiber are used for data communication and telecommunication. (Fig. 6) Data communication systems need to be relatively inexpensive and therefore use light emitting diodes instead of lasers. They require larger cores and higher numerical apertures to collect enough light. However, data transmission is generally slower than telecommunications, allowing use of lower bandwidth fiber and due to shorter transmission distances, attenuation may be higher. In addition, data link fibers must be stronger because of rougher treatment and harsher environments encountered in applications such as manufacturing.

Telecommunication fibers can have smaller cores and lower numerical apertures because high power lasers are used as light sources. However, they must have low loss for long length transmission and high bandwidth for fast rates of information transfer. Strength and fatigue resistance are not as critical because once cables are installed in ducts, they are usually under much lower stresses than datalink fiber and will not degrade due to stress corrosion.

## PERFORM FABRICATION

There are several methods of manufacturing the preform rod which are similar for both data and telecommunication fiber. The Outside Vapor Phase Deposition process developed by Corning consists of the deposition of soot on the outside of a mandrel followed by sintering into a solid glass rod. Another method called Vapor Axial Deposition has been developed by the Japanese which also deposits soot on the outside but in a vertical configuration rather than horizontal. The method used at Hewlett Packard was developed by Bell Laboratories and is an inside soot deposition process called Modified Chemical Vapor Deposition.

In the MCVD deposition process, glass particles are formed on the inside of a quartz tube by reacting gaseous chlorides with oxygen. (Fig. 2) These gases are obtained by "bubbling" oxygen through a liquid such as silicon tetrachloride which evaporates and is "carried" by the oxygen to the tube. The refractive index dopants such as germanium and phosphorus are introduced into the tube in the same way. The gases and liquids must be free of metallic impurities and water which will absorb light in the fiber. To maintain this purity, the gas delivery system must be stainless steel, quartz or teflon and completely sealed from the air. The tube must be high quality fused silica and is purchased from Heraeus Amersil. It must have the best dimensional control possible and be free of bubbles and inclusions to produce a fiber with good dimensional control and low attenuation. Starting tubes are about 25 mm OD and have 1-3 mm thick walls.

A standard glass lathe and oxy-hydrogen torches are used to fabricate the preforms. The reaction gases are introduced into the tube through a rotary seal and are exhausted at the other end through a larger diameter section which is open to the atmosphere. The torches are used to heat and react the chloride gases with oxygen, forming oxide soot particles and chlorine gas which is exhausted as the waste product. The particles deposit in a thin layer (25  $\mu\text{m}$ ) on the inside of the tube on the downstream side of the torch. The torch moves along the tube at 0.2 cm/sec as the tube rotates at 30 RPM and fuses the layer of soot into a glassy layer. Reaction temperatures are between 900°C and 1300°C and fusion occurs at about 1700°C.

After reaching the end of the tube, the torch returns quickly to the start and repeats the deposition "pass" to form another layer which is repeated many times to build up the desired thickness. The amount of dopant gas can be changed for each pass, changing the composition of the glass particles formed. A refractive index profile is formed across a cross section of the layered deposit by this technique. Control of this profile shape and the deposit thickness is important for uniformity in the finished fiber, therefore the entire operation is fully automated. A computer controls the fire carriage movement and the reactant gas flows. Control of the temperature is also very important for uniform chemical reaction and deposition. It is controlled by measuring the tube temperature with an optical pyrometer which is in a feedback loop with the computer to control the hydrogen and oxygen flows to the torch. The diameter of the tube is controlled by measurement with a TV camera and feedback to an internal pressure system in the tube.

After the desired thickness of deposit is attained, the reactants are stopped and the torch temperature is increased to cause the tube to collapse into a solid rod. This is done gradually over several passes and is the most difficult part of the preform fabrication process because the tube must be collapsed into a solid rod without deformation or entrapment of gas bubbles. The doped deposited layers are always a softer glass than the substrate tube and have a greater tendency to deform. This tendency will increase with increasing dopant levels and will change with the type of dopant. For instance, aluminum oxide is softer than germanium oxide and only a few percent phosphorus or boron will lower the softening point of the glass several hundred degrees.

After final collapse into a rod which is one meter long and 1.0 to 2.0 cm in diameter, the rod is separated at the exhaust end of the tube and removed to be pulled into fiber. Careful handling of the preform is necessary to achieve high strength fiber because most fiber breakage is caused by particles on the surface of the glass. Therefore, preform fabrication is often done in a clean air flow and preforms are stored and transported in clean boxes.



## **FIBER PULLING**

Preforms are “pulled” into optical fiber on a fiber drawing tower which can be 10 to 50 feet high. (Fig. 7) The preform rod is mounted near the top in a collet and heated to above the softening point (1900°C) by a zirconia induction furnace. The draw is started by dropping through a small piece of the preform which is pulled down and guided to the takeup reels. After the draw is started, the pulling is done by a rotating capstan and the perform is drawn into 5-20 km of fiber continuously at the rate of 1-2 m/sec. The physical and optical properties of the preform do not change during drawing. Therefore the properties of the fiber are controlled by the perform, except for the outside diameter which is reduced by about 100 times as the preform is pulled from 10-20 mm diameter to 100-200 um diameter fiber.

Fiber must be protected after drawing to maintain high strength, therefore a soft silicone coating is applied as the fiber is pulled before any handling of the fiber. High quality fiber is drawn in a clean air environment to prevent particle contamination and the fiber and coating must also be dimensionally uniform. A computer controls the preform feeding and capstan speed to control fiber diameter. The furnace temperature and coating diameter are also controlled in the fully automated process. Fiber is wound on reels, and application of outer jackets and testing are performed in other areas.

## **MECHANICAL PROPERTIES**

After drawing, the properties of the fiber are fully established and the next step is optical and mechanical testing. Optical properties have already been described. Mechanical properties, however, are equally as important. Silica glass is intrinsically very strong, exhibiting strengths of over one million pounds per square inch. However, the surface is easily scratched or flawed by small particles which greatly reduce the strength. For instance, a particle of only 1 um size reduces the strength to less than 100,000 psi. Particles can originate in the preform, the drawing furnace or the ambient air around the preform and drawing tower. Upper limits of strength are tested destructively in short lengths on an Instron tensile tester, and this “fast fracture” strength is usually characterized by a distribution called a Weibull plot. (Fig 8)

Strength can also be tested on a “prooftester” by stressing the fiber to less than the expected average breaking strength. This test is performed continuously and destroys only that part of the fiber which is very weak. Therefore, all fiber which survives this test will be guaranteed or “proven” to have at least the test strength when stressed later in use. All Hewlett Packard fiber is prooftested to insure its durability for datalink applications. High strength has been a major emphasis of HP’s research efforts to provide fiber which can be used in environments where high stresses are expected. This has resulted in the achievement of long lengths at higher strengths than are typical of commercially available fibers. For instance, a typical run of four preforms yielded more than 40% of the fiber in average one km lengths or greater when prooftested at 300,000 psi. This is strong enough fiber for most applications and compares very favorably to typical prooftest levels for standard telecommunication fiber of 50,000 psi.

In addition to achieving and testing fiber for initial strength, it is also important to maintain high strength in the fiber over the time period of its use. This can be difficult due to moisture in the atmosphere which attacks glass when under stress and causes flaws to grow and the fiber to break. This is particularly detrimental for datalink fiber which is often stressed in use and can be installed in hot, humid environments which accelerate this attack, called static fatigue or stress corrosion. Telecommunication fiber is not as susceptible to static fatigue because the environments are not as harsh and the fiber is not normally under stress once it has been installed.

Standard fiber under high stress can degrade in strength to one half the original value in a year or two in normal environments and much faster in extreme environments. Hewlett Packard has developed coatings which are applied on the silica surface of the fiber during drawing to seal the moisture away from the glass, preventing stress corrosion and the resulting strength degradation. At normal temperatures (22°C) and humidities (50%), coated fiber is stronger than uncoated fiber after about one month under stress. (Fig 9) The lifetime improvement for fiber in harsh environments is even greater which can be seen by tests in 98°C water where uncoated fiber fails in one day, while coated fiber lasts many months. (Fig 10) To show an extreme example of

how this coating protects the fiber, tests were carried out in concentrated HF acid. An uncoated fiber breaks in less than two minutes, while coated fibers have survived for more than 12 months.

Coated fibers also have another strong advantage over conventional uncoated fibers, which is their resistance to diffusion of hydrogen into the fiber. Hydrogen causes absorption and therefore increased attenuation. Hydrogen can be generated in a chemical reaction in moist environments between some of the metals in protective cable structures. This has been shown to increase attenuation beyond usable limits in many installed cables. The attenuation of uncoated fibers in hydrogen at room temperature doubles in less than one week at the operating wavelength, while the coated fiber shows no increase for many months. (Fig 11)

Hydrogen is also found in environments containing decayed organic materials such as swamplands and oil and gas wells. The resistance to hydrogen degradation in combination with the high strength and resistance to static fatigue has made HP coated fiber ideal for use in oil and gas exploration. High strength fiber is needed in this application because 10km fiber cables are lowered into deep wells where the cable requires 200,000 psi strength to support its own weight. Static fatigue resistance is needed because the environment can be an aqueous liquid at temperatures up to 200°C which causes breakage very rapidly at these high stresses.

## **ALTERNATIVE DOPANTS**

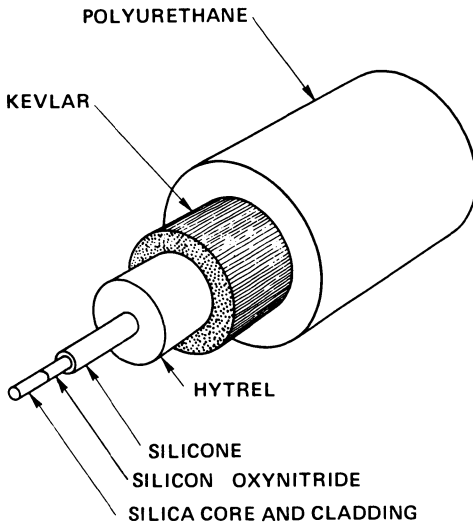
Another area where research at Hewlett Packard has concentrated is alternative dopants to reduce the cost of the preform materials. Germanium is the most common dopant in silica optical fibers. This is an expensive and rare natural resource which contributes 80% of the preform materials cost while germanium oxide makes up an average of only 6% of the preform. This is partly due to the inefficient chemical reaction which converts germanium chloride to germanium oxide and results in waste of the germanium. This problem can be overcome if a dopant is used which has a more efficient chemical reaction.

Aluminum is a dopant which reacts very efficiently and is also a lower cost material. In addition, aluminum reaction chemistry

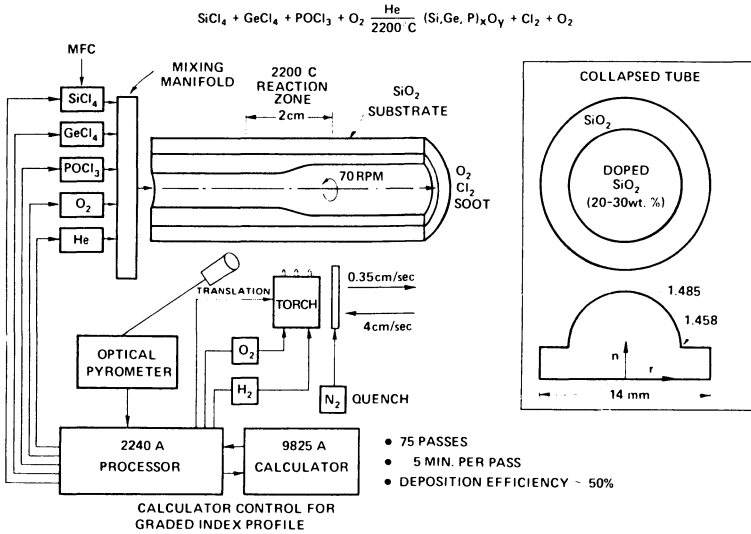
enables more uniform doping profiles to be achieved in the fiber which improves optical properties. HP has been able to dope fibers with enough aluminum to eliminate germanium as a dopant. (Fig 12) There are some disadvantages to aluminum doping which causes the glass to be soft and therefore more difficult to collapse uniformly. There is also a tendency for the glass to crystallize at high doping levels which scatters light and increases fiber attenuation. The addition of phosphorus suppresses the crystallization and has enabled the achievement of reasonably low losses at fairly high numerical apertures. (Fig 13)

Higher numerical apertures can also be obtained by depositing a doped "cladding" before the core is deposited, which is doped to decrease the refractive index. This results in a larger refractive index difference between the core and cladding, thereby increasing the light collecting ability of the fiber. The addition of boron and fluorine have been shown to "depress" the cladding refractive index and reduce costs when compared to fibers doped only with germanium. Combining the aluminum doped core and a fluorine doped cladding results in a high numerical aperture datalink fiber at about one fourth the materials cost of a germanium doped fiber. (Fig 14)

In summary, datacommunication and telecommunication fibers are fabricated using similar techniques. However, the requirements of the different information systems necessitate the use of fibers with very different properties. In particular, reliability in harsh environments and efficient refractive index dopants are needed for datalink applications and technical advances in these areas have provided cost effective solutions to these problems.

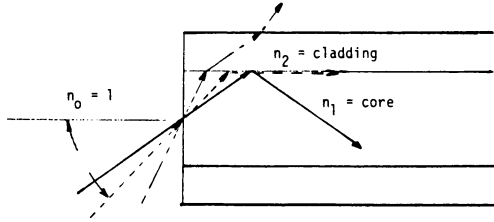


**Figure 1 Cabled Optical Fiber**



**Figure 2 MCVD Preform Fabrication**

NUMERICAL APERTURE



$$N.A. = \sin \theta_c = \sqrt{n_1^2 - n_2^2}$$

N.A.	$\theta_c$
0.1	5.7°
0.2	11.5°
0.3	17.5°

Figure 3

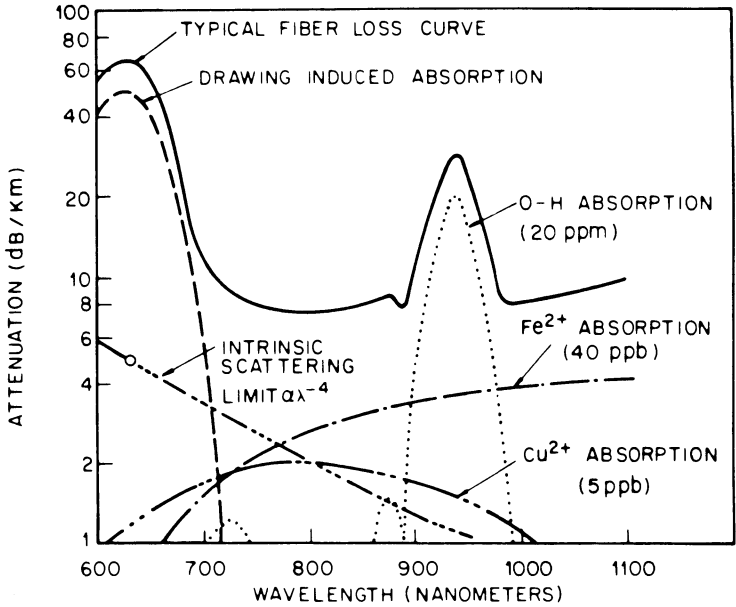


Figure 4 Sources of Fiber Attenuation

# REFRACTIVE INDEX PROFILES

$$n(r) = n(0) \left[ 1 - 2\Delta \left( \frac{r}{a} \right)^a \right]^{1/2}$$

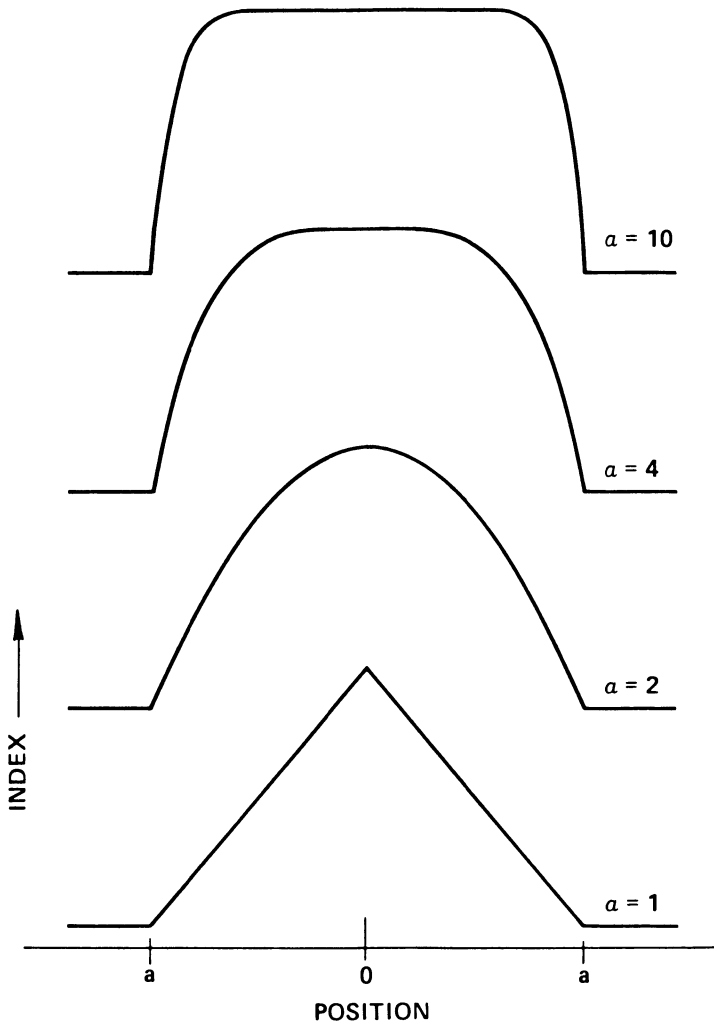


Figure 5a

## PULSE DISPERSION

P. D. is proportional to refractive index profile

$$n^2(r) = n^2(o) [1 - 2\Delta (\frac{r}{a})^\alpha] \quad f_{3dB} \approx 0.374/2 \sigma$$

α	P.D. (ns/km)	Bandwidth (MHz-km)
2 (parabolic)	< 0.3	> 3000
10	~ 20	50
∞ (step)	~ 50	20

**Figure 5b**

### ***DATA COMMUNICATION***

**LIGHT EMITTING DIODE**

**LARGE CORE**  
90-100 μm

**HIGH NUMERICAL APERTURE**  
0.30

**LOWER BANDWIDTH**  
200-500 MHz-km

**HIGHER ATTENUATION**  
1-5 dB/km

**SHORTER LENGTHS**  
.1-1.0 km

**HIGH STRENGTH**  
200,000 psi

### ***TELECOMMUNICATION***

**LASER**

**SMALL CORE**  
10-50 μm

**LOW NUMERICAL APERTURE**  
0.10-0.20

**HIGH BANDWIDTH**  
1.0-2.0 GHz-km

**LOW ATTENUATION**  
0.3-1.0 dB/km

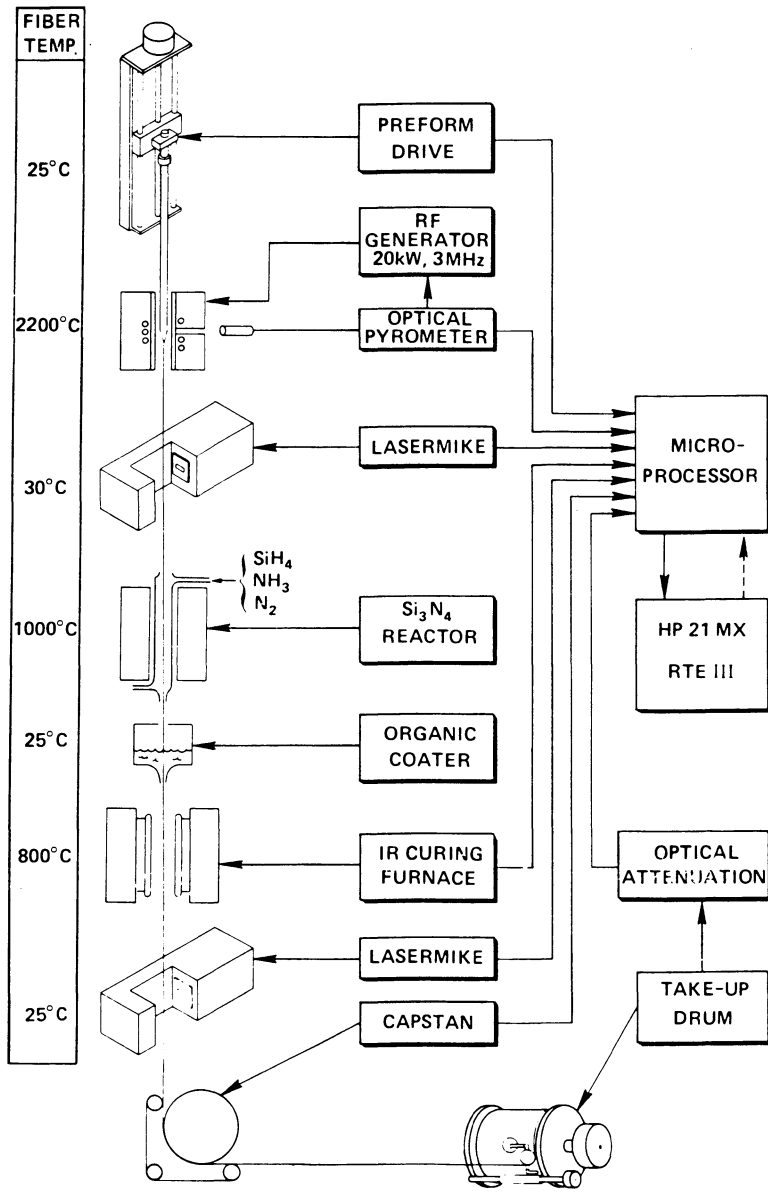
**LONG LENGTHS**  
10-20 km

**LOWER STRENGTH**  
100,000 psi

*C. Scott  
HPL/MRL*

**Figure 6**





**Figure 7 Fiber Drawing Tower**

# WEIBULL PLOT

## SILICON OXYNITRIDE COATED FIBER

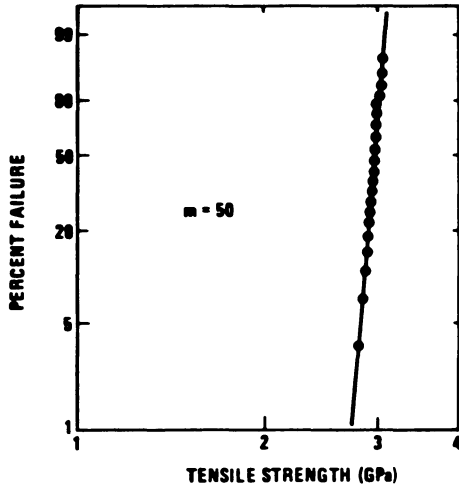


Figure 8

### STATIC FATIGUE AT 22°C, 50% R.H.

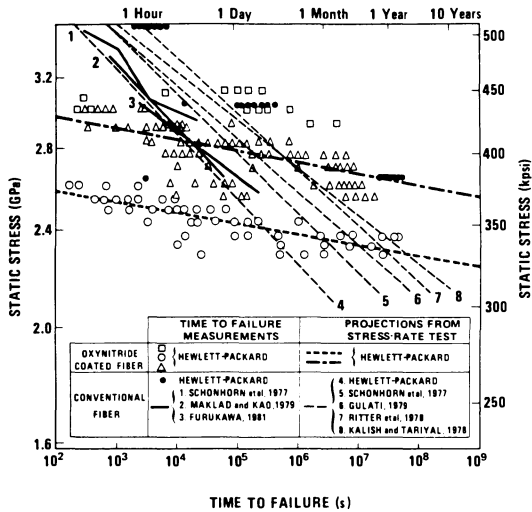
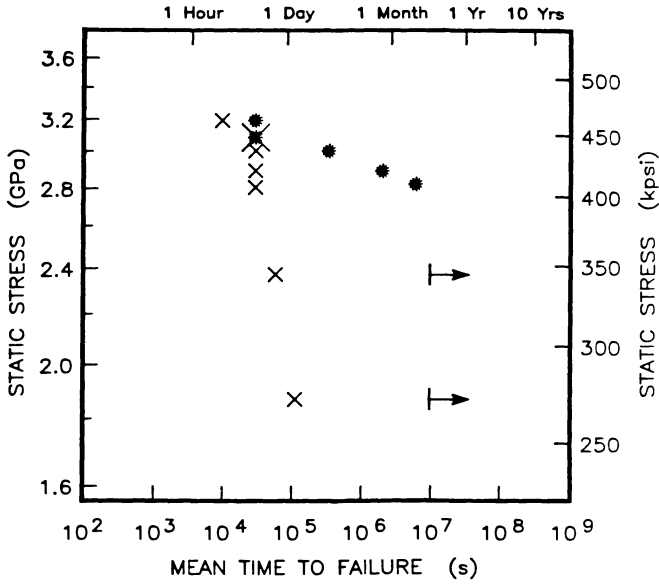
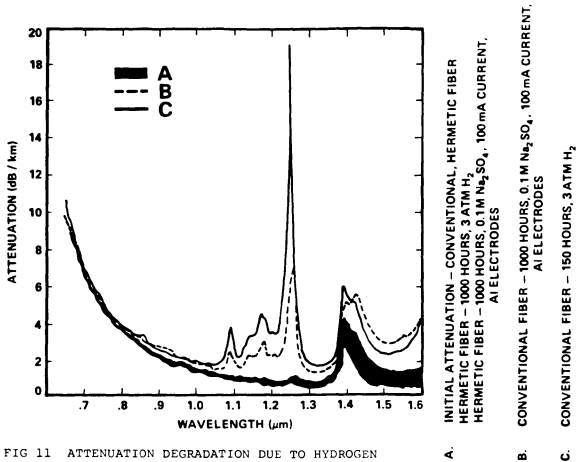


Figure 9



**Figure 10** STATIC FATIGUE LIFETIME IN 98 DEG C WATER. ARROWS INDICATE TEST DURATION OF HP HERMETIC FIBERS STILL UNDER TEST.  
 ● HERMETIC FIBER  
 X CONVENTIONAL FIBER



**FIG 11** ATTENUATION DEGRADATION DUE TO HYDROGEN  
 A. INITIAL ATTENUATION - CONVENTIONAL, HERMETIC FIBER  
 B. HERMETIC FIBER - 1000 HOURS, 3 ATM H<sub>2</sub>  
 C. HERMETIC FIBER - 1000 HOURS, 0.1M Na<sub>2</sub>SO<sub>4</sub>, 100 mA CURRENT, Al ELECTRODES  
 A. CONVENTIONAL FIBER - 1000 HOURS, 0.1M Na<sub>2</sub>SO<sub>4</sub>, 100 mA CURRENT, Al ELECTRODES  
 B. CONVENTIONAL FIBER - 150 HOURS, 3 ATM H<sub>2</sub>  
 C. CONVENTIONAL FIBER - 150 HOURS, 0.1M Na<sub>2</sub>SO<sub>4</sub>, 100 mA CURRENT, Al ELECTRODES

**Figure 11** Attenuation Degradation Due to Hydrogen

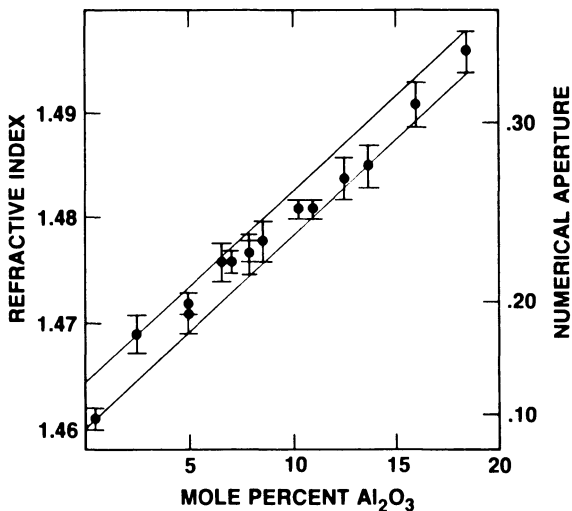


Figure 12 REFRACTIVE INDEX VS. COMPOSITION FOR  $\text{Al}_2\text{O}_3$ ,  $\text{P}_2\text{O}_5$  DOPED  $\text{SiO}_2$  FIBER ( $\text{P}_2\text{O}_5 = .5\text{-}6\%$ )

### ATTENUATION OF $\text{Al}_2\text{O}_3$ , $\text{P}_2\text{O}_5$ DOPED FIBERS

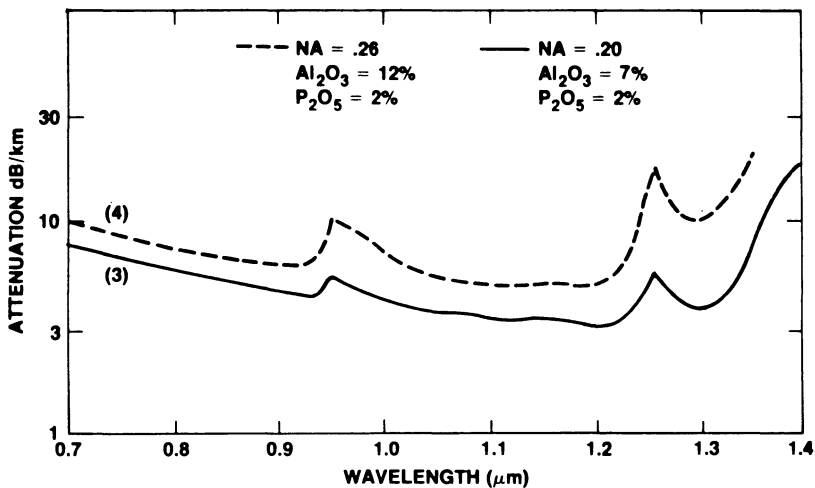


Figure 13

# LOW COST HIGH PERFORMANCE OPTICAL FIBER

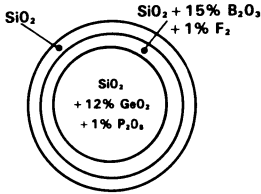
**CONVENTIONAL DATA LINK FIBER**



**MATERIALS COST:** 9.2c/meter  
**FABRICATION RATE:** 550meters/hour  
**INDEX PROFILE:**



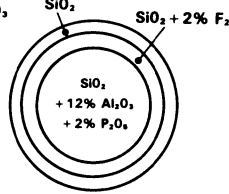
**BORON/FLUORINE CLAD HPL FIBER**



**5.0c/meter**  
**550meters/hour**



**ALUMINUM CORE HPL FIBER**



**2.5c/meter**  
**550meters/hour**



HPL/MRL  
 C. Scott

**Figure 14**



# A MULTIPURPOSE COIL WINDING MACHINE

Gary S. Coyne

California State University, Los Angeles California

## INTRODUCTION

In movies that have a laboratory scene you will probably see a coil; in the ASGS symbol you will see a coil; and, if a table were covered with scientific glass apparatus, the piece that would attract the eye first would be the piece with the coil.

There is something about coils that is instantly compelling and attractive, but the job of making coils can be frustrating or tortuous.

The procedure of winding coils, like any task, requires skill and practice. Whether the glassblower rarely or frequently makes coils, the coil-winding process poses particular challenges if the coil must meet specific length or space requirements.

This paper discusses the mechanics of coil winding, and details the development of a coil-winding machine that has total variability, reliability, and accuracy.

## THE MECHANICS OF COIL WINDING

First let me speak on the mechanics of coil winding.

When a tube is bent, the inside diameter is following the radius of the bend, as we can see in figure I. The outside diameter, however, is stretched to allow for the greater distance that the same unit length of tube must now follow. In (a) you see a series of lines that are parallel. In (b) these lines now are the radii of a semi-circle. The distance between the lines on the outside is greater than that on the inside. In (c) you can see this has resulted in a thinning of the glass.

At a certain diameter of the glass tube and/or the radius of the bend, the outside glass will not stretch; rather, it will pull toward the inside diameter and, in cross-section, will have a "D" shape as seen in Figure II(c). This may also be due to improper or insufficient heating. Blowing into the tube may be able to correct this.

For many years I wound coils in the following manner: (Figure III) I had a mandrel of carbon or metal that was attached to a wooden handle. One end of the tube about to be wound was inserted into a hole at the end of the mandrel. The mandrel was placed on rollers at the edge of a table and was at an angle to the floor such that, as the mandrel was rotated, the coil formed down the mandrel until one ran out of glass or mandrel.

It was important to provide even heating and as long as one didn't alter the angle of the mandrel, the distance between coils was fairly well maintained. Trial and error, and experience, would guide the angle of the mandrel to the floor and this governed the number of coils per unit length.

If a constant number of coils per unit length were required, and one needed many of the same type of coil, one could always get a pre-grooved mandrel (FIG IV).

### **THE NEED FOR INVENTION**

The above process had worked well for me for many years. However, one day I was asked to make a coil that required eight feet of tubing and had to fit into an area some four inches wide.

The latter requirement was something I could deal with, but eight feet of tubing was a length that couldn't be easily wound over the edge of a normal table; the end of the tube would drag on the ground, distorting the coil as it formed.

I knew it was possible to wind a length of tubing, stop, join another length of tubing, and continue; but I had never been successful at stopping and starting a coil smoothly. I didn't want to deliver an item that would look as if it had been made by one of my beginning students; I wanted an alternative. Since I was not aware of any device to help me, I would have to invent what I needed.

### **THE PROCESS OF INVENTION**

Hanging an eight-foot length of tube would have required drilling a hole in the floor or setting up a tall ladder. Neither of these ideas seemed to have much merit. It was obvious that I would have to feed the glass horizontally.

My early tries were very educational, but not successful. If the



mandrel were perpendicular to the rollers (FIG V), it would turn fine; but while holding the mandrel in one hand and the glass in another, I could neither move the flame nor move the coiling tube down the mandrel. On the other hand, if the mandrel were at any angle to the rollers (FIG VI), the mandrel moved in or out during rotation, like a bolt in a nut.

My solution became apparent; if I couldn't get the coil to form down the mandrel, I would have the mandrel move up the forming coil.

In my former method, because the glass moved down the mandrel, I had to chase it with the torch. But if the mandrel moved, then I could fix the flame, set the rollers at a pre-set angle, the glass could be fed to the mandrel horizontally, and all I would have to do is turn the mandrel.

To try this, I set up two rollers that were parallel to each other and laid across them a shaft that held the mandrel (FIG VII). The glass tube was supported perpendicular to the mandrel. I then lit the fires and rotated the mandrel. It didn't look very good, but I could see the idea was right.

After a few more frustrating attempts and experiments, I discovered that the glass needed to be fed to the rotating mandrel at approximately one-half the angle of the rollers. But I had other problems: the rollers didn't want to stay still, and unless the two rollers were absolutely parallel, the dissimilar pitch resulted in an inconsistent mandrel movement, and this prevented consistent coils.

The final machine is shown in Fig VIII. There is only one set of rollers, solving the problem of parallelism. The other supports are steel-ball casters that are free-moving in all directions.

A protractor is drawn below the rollers (Figure IX). At any given angle, the mandrel will travel a given and reliable distance. This allows me to pre-calculate how many turns I may need in a given distance, set it, and get it.

The mandrel shaft is heavy to insure stability (FIG X). If using a large mandrel, a counter-balance is sometimes required to prevent the shaft from tipping (Fig XI).

Depending on the orientation of the rollers to the mandrel shaft, one can obtain either clockwise or counter clockwise coils.

By empirical experimentation I developed a graph that shows

the distance traveled and the angle of the rollers required for that distance. As Figure XII shows, to get a distance of 20 mm from the center of one coil loop to the center of the next coil loop, I could set the rollers at 25° and I would get it.

Here you see, in Figure XIII, five coils made of the same glass size, all on the same mandrel. But the first coil is at 15°, the second at 25°, the third at 35°, the fourth at 45°, and the fifth at 55°.

Incidentally, it is easy to take a mandrel that has pre-cut grooves, measure the distance from groove to groove, set the roller angle accordingly, and not lose the use of that mandrel.

Figures XIV and XV show the full set up in use. Here you see the coil winding machine, ribbon burner, and glass support. The glass support is used to hold the glass horizontally and thereby eliminated length as a restriction in coil winding.

## CONCLUSION

As you can tell, I went to a great deal of trouble to make a machine that could do a lot more than make the one coil for which it was developed. Right from the beginning, I saw a need to have greater control in making coils than the happenchance pattern I had been using. I wanted variability, reliability, and accuracy. Although still requiring skill and some practice, this machine has shown that it can do all I expected it to do.

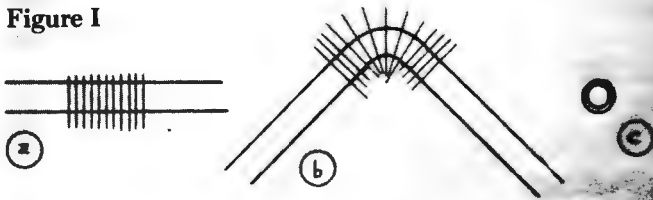
There are very few glassblowers who don't have their own particular techniques for one job or another. It is this allowance of variation and individuality that can be so rewarding to glassblowers. Because of the many new and different things we are asked to create, and because we so often strive to make things better and/or easier, we often call upon ourselves to invent.

I'm sure that the percentage of inventors in this room is only surpassed at an inventors' symposium.

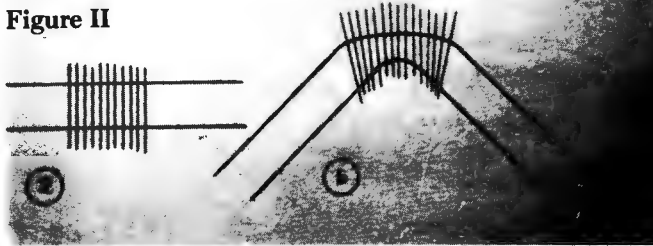
We have seen how some inventions like the glass lathe, which was initially developed to aid the inexperienced glassblower, have grown into tools that few glass shops are without. Necessity is the mother of invention, but the invention can create new capabilities. The lathe was invented to simplify some benchwork, but now there are tasks done on lathes that cannot be done at the bench.

I do not, by any means, wish to imply that I rank my coil-winding machine on the same level as a glass lathe. Rather, I wish to put it, and every invention, into a proper perspective. For me, my coil-winder is an aid; because of its variability it is also a tool.

**Figure I**



**Figure II**



**Figure III**

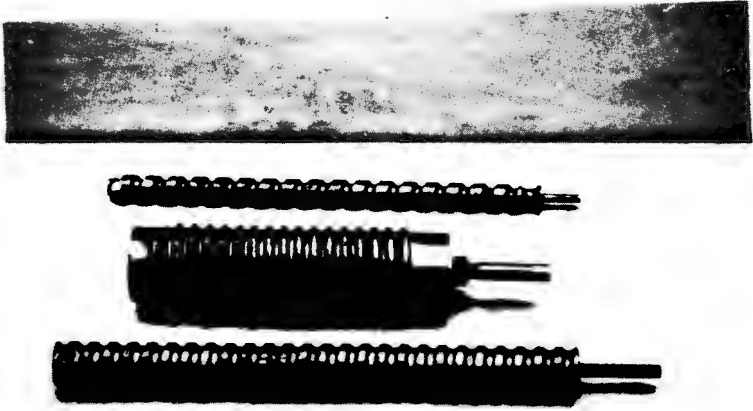


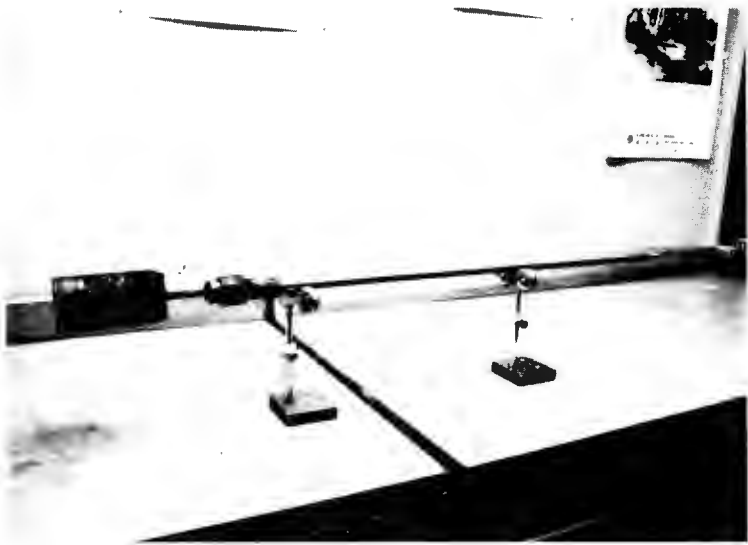
Figure IV



Figure V



**Figure VI**



**Figure VII**



Figure VIII

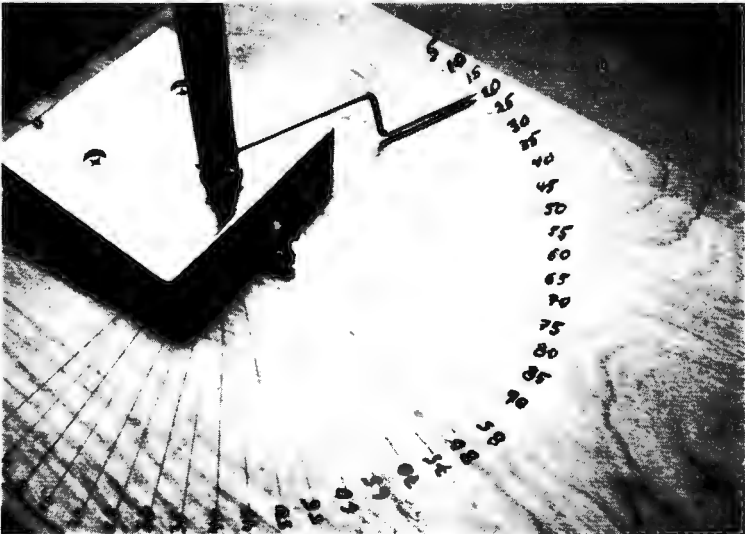


Figure IX

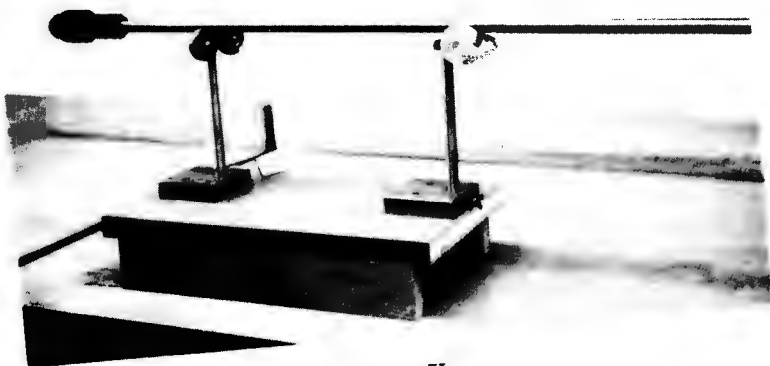


Figure X



Figure XI



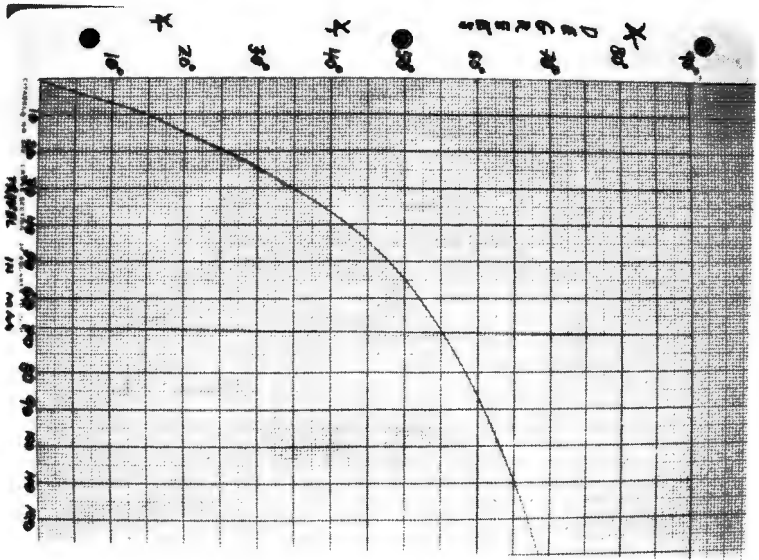


Figure XII

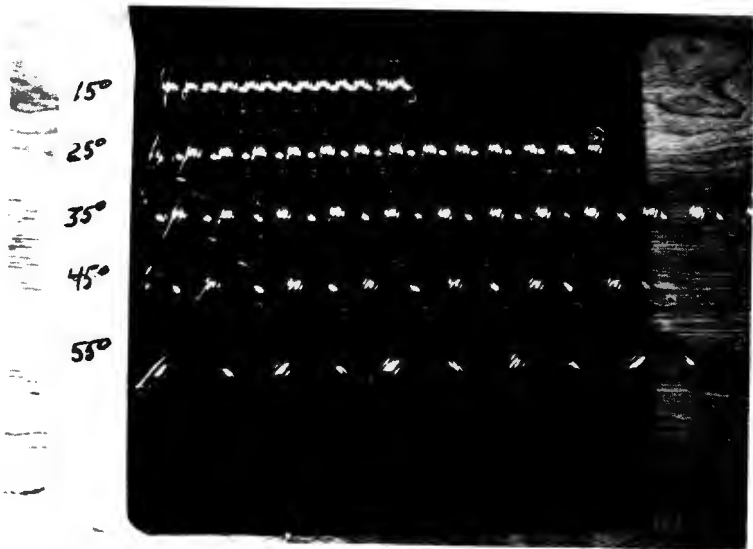


Figure XIII



**Figure XIV**



**Figure XV**

## THE EVOLUTION OF A GLASS, P.T.F.E. SCREW-THREAD VACUUM VALVE AT WAIT, WESTERN AUSTRALIA

Glen Whiting, W.A.I.T.

Perth, Western Australia, is around 12,000 miles from everywhere and not an easy place in which to obtain out of the ordinary or non-standard type laboratory glassware. So when confronted with the prospect of having to build an all glass vacuum system that would be contamination free for carbon 14 dating within the School of Chemistry at WAIT (Slide 1), it was decided that we should try to produce our own P.T.F.E. glass vacuum valves, of which we would require a large number (Slide 2).

Having no idea at all of how the commercial valves were manufactured, I had to start from scratch and develop my own line of thought on the matter.

For ease of production of the P.T.F.E. component, I decided to try an internal glass thread, my first idea being to have our engineering department make up a stainless steel former to the dimension of the P.T.F.E. plug, and to try with the aid of the glass lathe to tool a glass blank onto it.

Sad to say, this technique failed! or, at least, I could not make it work. Many failures later I decided to try a new tack.

Recalling a method of vacuum forming, precision bore glass tube for syringe barrels that I had used some years ago, I had our engineer make yet another metal mandrel from silver steel, this time with an axial hole through its length (Slide 3), I had this mandrel polished to as near a perfect finish as our technician could achieve with the tools at his disposal. Next, I shaped up on the glass lathe some close fitting glass envelopes, or parrisons, in heavy walled borosilicate tube (Slide 4).

The pre-heated mandrel was then placed inside the glass pre-form and this was then plugged onto a vacuum swivel in the glass lathe. A support rod already in the tail stock was fused to the test tube end of the pre-shape. With the mandrel and glass set to rotate in the lathe, a soft flame was applied to heat the mandrel and dry moisture from the envelope. At this point the vacuum was

applied to the envelope and the flame used to soften the glass just above the thread at the top end of the mandrel.

The flame was moved along the mandrel and glass blank to keep place with the collapse of the glass under reduced pressure from the vac-pump. On reaching the end of the mandrel, the whole unit was given a final warm over to partly anneal and then allowed to cool under vacuo.

The next step should have been to screw out the mandrel from the perfectly shaped glass envelope, but this was not to be the case. At this point I found that my vac-pump was faulty, that the mandrel had oxidized, and that the glass had stuck fast (another failure).

Back to the mechanical workshop for yet another steel mandrel! At this point a friend from the glass shop at Murdoch University, Mr Denis Clair, suggested that he had seen an aqua-dag coating used on metal tools while attending a Society Symposium at San Jose, California. So the new mandrel I coated with Aqua-Dag and polished this new tool to as smooth a finish as possible. This time I achieved a result.

After removal of the waste glass from the top end, above the thread, by diamond saw, and a tubular stem joined axially at the other (Slide 5), a side tube was fitted to the barrel via a suck, seal and pull method. This gave a good shape to the inside of the barrel, with no lumps or bumps to touch or mark the P.T.F.E. plug.

Our P.T.F.E. plug was produced by our workshop engineer, Mr. Fred Murphy, from high density P.T.F.E. Rod stock, fitted with three viton O rings, one as a seat seal and two to seal against the atmosphere. The knobs or handles on the P.T.F.E. plugs were made from aluminium radio knobs.

I should point out perhaps that the steel mandrel was made with a slight, but uniform, taper from end to end to allow for ease of removal from the glass shape.

## CONCLUSION

While the Aqua-Dag coating on the mandrel tends to give a grainy appearance to the inner surface of the glass barrel, the valves I have produced here are holding better than  $10^{-5}$  vacuum.

They are easily reproduced and in a place where anything in the glass line, apart from flasks and beakers, has to be ordered in from overseas, we can produce valves at will.

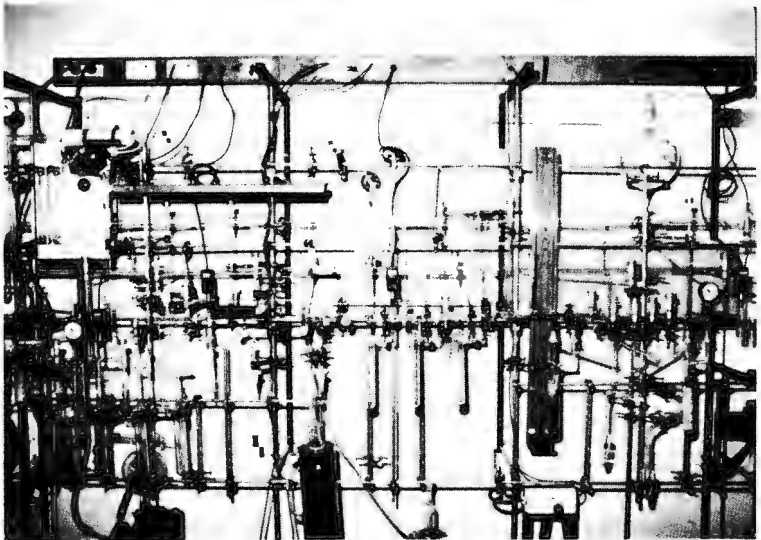
We have also adapted the same technique to produce vacuum couplings to allow easy attachment and removal of traps and reaction vessels to the same carbon 14 system (Slide 6).

### **An Apology**

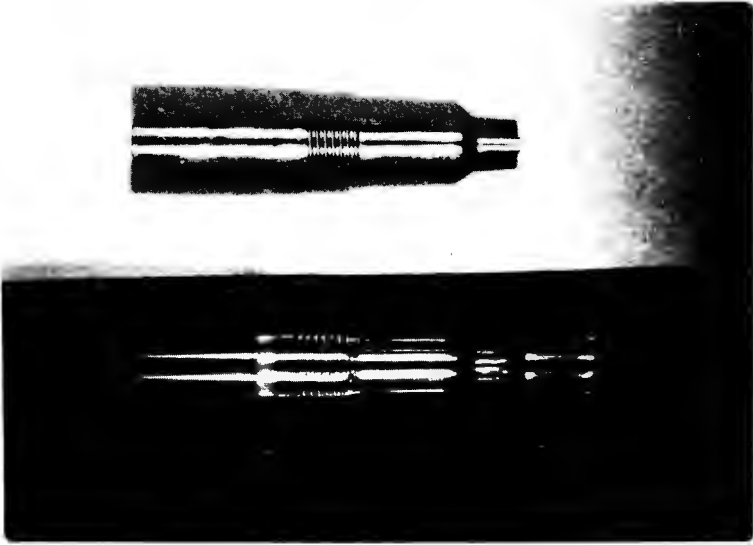
Ladies and Gentleman, I regret my inability to attend this the 29th Symposium of the Society, but distance, workload and cost are a problem. I hope you are having a successful symposium and that this short paper will prove of interest and benefit to some of you.



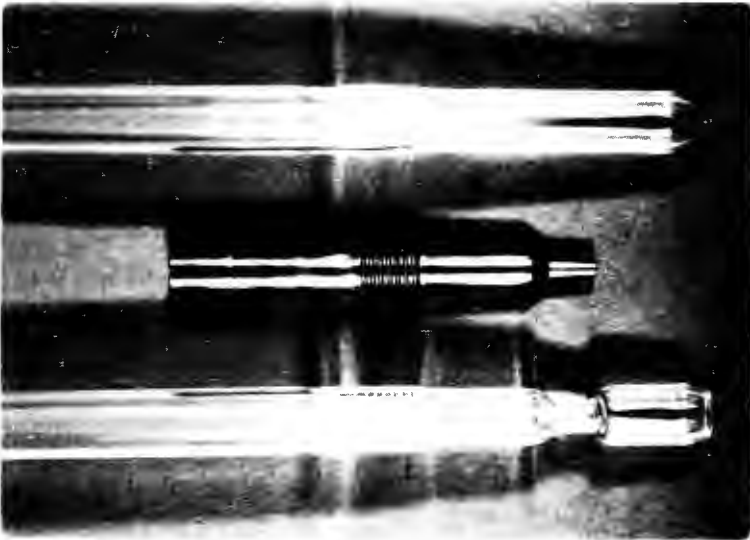
Slide 1



Slide 2



**Slide 3**



**Slide 4**



Slide 5



Slide 6



## A UNIQUE APPLICATION OF GLASS MOLDS

Donald W. Gagnon  
Kimble Division of Owen-Illinois

The main portion of the presentation was a seven (7) minute film on the C.A.P. process. The film was produced by Universal Cyclops and shown in Chicago in conjunction with the 1983 I.R. 100 award.

A summary of the film and/or process follows:

A new consolidation process that uses glass containers instead of metal for post-atomization storage and billet preforming of powder metal will yield a high-quality product for less cost. The new method is called CAP (consolidation by atmospheric pressure).

The consolidation process is made cleaner by using glass. Simplicity is the main advantage over the HIP (hot isostatic pressing) method.

Both methods of powder manufacturing begin with vacuum induction melting to prepare the metal for atomization. The liquid metal is poured into a refractory pot on top of the atomization chamber and then is passed through a ceramic nozzle where it is atomized by a vortex of argon gas, forming a very fine homogenous powder. Rapid solidification techniques can be applied to cool the powder at rates approaching 100,000C per sec. The solidified metal powder then is collected in a water-cooled bed at the bottom of the chamber and screened to the size specified by the customer. This step removes oversized and non-atomized particles.

In the HIP process, the screened powder is placed in metal containers, which are welded closed and placed in an isostatic press where an external gas pressure of 15000 psi is applied and the temperature raised to over 1,090C. Once the process is complete, the formed billets must have the containers machined or chemically milled away.

The CAP method begins by chemically treating the powder to clean and promote diffusion bonding of the particles. After chemical activation, the powder is placed in glass instead of metal containers as used in the HIP process. Once filled, all gas is

evacuated from the glass bottles, which are sealed by annealing the filler neck. The filled bottles then are placed in refractory crucibles, surrounded by sand and heater to over 1,090C in a conventional furnace for consolidation.

Heat and atmospheric pressure on the vacuum bottles causes the powder to solidify. As cooling takes place, the bottles shatter into small pieces leaving the billet intact. After being removed from the crucible, the billet is ready for hot working or sale as billet stock. Sand remaining in the crucible is strained to remove the glass particles and used again.

Cyclops officials said metal HIP containers are "more expensive and not as reliable" as the glass bottles. Since the metal molds require welding, weld slags can contaminate the metal powder. The bottles eliminate contaminants and lack any surface joints.

As with other consolidation methods, all steps in the CAP process can be done in an air-free environment. The powder can be manufactured without ever seeing outside air.

Since most of the powder produced from this process is gas turbine related, the quality is very important. All of the areas are very clean. Most engine manufacturers require metal powders fabricated in air-free environments.

Billets and basic preforms account for most of the work being done by the CAP process.

In the future, the glass bottles will be made into more complicated and near-net shapes to minimize the amount of wasted material. Owens-Illinois, which did the development work along with Cyclop, is working on new molds and designs, and company officials believe a strong market will develop for near-net shape bottles.

## MEMBERS IN ATTENDANCE

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