Proceedings

THE TWENTIETH SYMPOSIUM

ON THE

ART OF GLASSBLOWING

1975

THE

AMERICAN SCIENTIFIC GLASSBLOWERS SOCIETY

Proceedings

THE TWENTIETH SYMPOSIUM
ON THE
ART OF GLASSBLOWING

Sponsored by

THE AMERICAN SCIENTIFIC
GLASSBLOWERS SOCIETY

MARRIOTT MOTOR HOTEL
PHILADELPHIA, PENNA.

JUNE 15 - 19, 1975

CONTENTS

OFFICERS AND DIRECTORS
OF THE
AMERICAN SCIENTIFIC GLASSBLOWERS SOCIETY
1974 - 1975

OFFICERS

President—THEODORE W. BOLAN
Philips Laboratories, Briarcliff Manor, New York

Past President—BILLIE E. PAHL
The Dow Chemical Company, Midland, Michigan

President-Elect—EARL R. NAGLE
State University of New York at Albany, New York

Secretary—OWEN J. KINGSBURY, JR.
East Carolina University, Greenville, North Carolina

Treasurer—JOSEPH W. BAUM
Sterling-Winthrop Research Institute, Rensselaer, New York

SECTIONAL DIRECTORS

RUSSELL STANLEY
JOHN A. GLOVER
ARTHUR DOLENGA
J. H. OLD
JOSEPH J. FOX
ROBERT B. TOBIN
FERN STODDARD
WILLIAM STEIN
ALFRED H. WALROD
WILLIAM A. WILT
MIHALY L. KOROSI
VINCENT C. DEMARIA
ROBERT G. CAMPBELL
CZESLAW DEMINET
ELMER ANDERSON

PAST PRESIDENTS

J. ALLEN ALEXANDER
President Emeritus

KARL H. WALTHER
ARTHUR DOLENGA
ALFRED H. WALROD
*JONATHAN W. SECKMAN
RICHARD W. POOLE
WILLIAM E. BARR
CHARLES J. CASSIDY
WILLIAM A. GILHOOLEY
M. HOWE SMITH
BILLIE E. PAHL

*Deceased

EXECUTIVE SECRETARY
WILLIAM A. GILHOOLEY

OFFICE MANAGER
DOLORES I. SITES

The American Scientific Glassblowers Society
309 Georgetown Avenue
Wilmington, DE 19809
Phone 302 798-4498

SYMPOSIUM COMMITTEE

Chairman—HELMUT E. DRECHSEL

Technical Papers—THEODORE W. BOLAN

Exhibits—ANDREW J. WARGO

Workshops—ROBERT L. MOLE
NEIL J. BETCHNER

Audio Visual—CHARLES A. BENNETT

Banquet—RUSSELL STANLEY

SEALING QUARTZ AND BOROSILICATE GLASS PARTS BY THE USE OF SPECIAL GLASS COATINGS

MRS. KITTY ETTRE
Vitta Corporation
Wilton, Connecticut 06897

ABSTRACT

This paper discussed the problems associated with sealing quartz and borosilicate parts. Typical applications were demonstrated and the relationship between the thickness and composition of the glass sealing layer were described for special cases.

CERTIFICATION . . . YES OR NO

JOSEPH W. BAUM
Sterling-Winthrop Research Institute
Rensselaer, NY 12144

Since our meeting of 1974, certification of professional and technical groups has gained impetus in this country. Some members of our society have expressed the desire for a certification program. Some general arguments in favor of certification are:

It establishes minimum standards of competence for those who have special fields of employment.

It tends to improve professional competence in a given field.

Certified people gain a form of peer recognition.

It can improve the financial status of certified people.

It provides an important credential and this draws favorable attention when applying for a job or a promotion.

Certification is national in scope and does not limit mobility between the states.

If periodic re-certification is required, certified people are encouraged to stay up to date in their field through continuing education.

Let us look at the various terms used by states and professional groups:

Licensing is administrated by the states, i.e., doctors.

Registration is also in the domain of the governmental agency, i.e., registered nurses.

Accreditation is not used or applied to individuals. Colleges, etc., are usually accredited for a program of study.

Certification is a means of providing recognition and assurance that an individual has the right blend of basic and specialized knowledge and experience to function effectively in a given area of specialization. It is always a voluntary program.

A program was started in the sixtys, through the seminars at Alfred University, to start a certification for glassblowers. Due to the youth of our society and other factors, we were not ready to establish this program. With an additional seasoning of ten years, let us look at a tentative voluntary program. This proposal is not a complete edition but offers the start of a program.

A certification board under the Education committee could be appointed. They would consist of our past presidents, members of the Board and honorary members from industry and technical schools. Exams would be written and be available at our annual meeting. Members not attending the annual meeting would take the exam in their immediate area.

Time and place of exams would be in FUSION. A demonstration of ability would be possible at the annual meeting or at a "friendly" lab or college.

This proposal would be expanded under the Certifying Board. A grandfather clause for a master glassblowers certificate, may be established for one year to include those members with years of experience and top references. It would effect those with a minimum of 12 years working experience. They would be certified without an exam or demonstration if their application meets with the Board's approval. After one year, an exam and demonstration would be necessary. For a regular certificate, a level of four years experience and two years education at a technical school or college would be necessary.

Certification should be a recurring necessity to make sure our members keep up with our changing technology. It might be necessary every three years. It would be a two part program. The applicant could take an exam or through a continuing education program gain credits for certification. Credits would be given on a point basis and each member must notify the certifying board of his activity. Credits would be given for the following:

1. For related courses taken at a technical school or college.
2. For attendance at seminars, conferences, workshops and symposiums.
3. For papers given at the above meetings.
4. For approved adult education courses.
5. For certain employer sponsored courses and study courses.

The Society would endeavor to set up or develop seminars, such as those held at Alfred University, and other programs to aid members to gain these credits.

The two years of higher education requirement for those members living in New York State can be partially gained through the Empire State College. This college awards college credits towards a two year Associate of Applied Sciences degree. This is based upon past higher education and learning experience in your chosen field of work. Those interested should contact the nearest office of the college. Other states may have similar programs in affect.

Fees for the exams would be fixed to make them self supporting. Fees for certification might be $30 initially. For re-certification — $20.

GLASSY MATERIALS FROM PLUMBITE TREATED COTTON

BY

TRUMAN L. WARD AND RUTH R. BENERITO

PRESENTED BY

AUSTIN C. F. MASON

Southern Regional Research Center[1]

New Orleans, Louisiana 70179

INTRODUCTION

This report concerns glassy materials produced by heating sodium plumbite treated cotton fabric while the fabric was in contact with a source of silicon. Analyses indicate that the new glassy products contain considerable amounts of carbon. According to our available literature, carbon has been used only in trace amounts as a coloring agent or to produce foamed glass. In neither instance was the carbon necessary to the formation of the glass.

It would sound more scientific to say that the glassy materials were expected. Actually, they were the result of double serendipity.

The first bit of serendipity involved treatment of the cotton fabric which was subsequently used in making the new glassy materials. That treatment consisted of soaking the fabric in an aqueous solution of sodium plumbite, washing out the excess plumbite solution and drying the fabric. The lead added was readily varied from a few percent, which was expected, to an unexpectedly high 40% by varying the time the fabric was in the plumbite solution. No other metal and no other salt of lead gave such high add-on to cotton.

The second bit of serendipity involved production of glassy materials from the treated fabric. While attempting to ash the fabric in a porcelain crucible in a muffle furnace, hopefully for analytical purposes, we found that it changed to a glassy material. A temperature of 600°C. for 1 or more hours while the fabric was in contact with the smooth side of a material having a high silicon content, such as glass or porcelain, was sufficient to produce the new glassy material. The fabric was placed in contact with the silicon material prior to commencement of heating and heating was carried out in a muffle furnace which supplied even heat and somewhat limited oxygen. When either the silicon source material or the oven was heated to reaction temperature prior to introduction of the treated cotton, or when the oven door was kept open, the fabric burned and produced metallic lead rather than the desired glass.

EXPERIMENTAL

Formation of the glass seemed to involve several recognizable steps. The plumbite treated cotton first turned black indicating a skeleton containing carbon. Further heating above about 400°C. turned the skeleton

[1]One of the facilities of the Southern Region, Agricultural Research Service, U. S. Department of Agriculture.

reddish yellow and then greenish yellow. Continued heating produced the glass.

The yellow skeleton shown had little strength although scanning electron microscope pictures of Fig. 1 revealed at low magnification a structure similar to that of the starting fabric. The nature of the weave is clearly visible in A and the light allowed through can be seen as a pattern of light spots in the back lit picture C. Higher magnification showed as in B and D the open, lacy look of the fibers in the skeleton. The fibers appear to be composed of crystals joined together. Usually in the heating process this fabric skeleton acted as if it were attracted toward the glass on which it rested. This caused it to bend around and conform to the shape of the holder. Further elevation of temperature caused this skeleton to change to a glass which bore the fabric weave pattern.

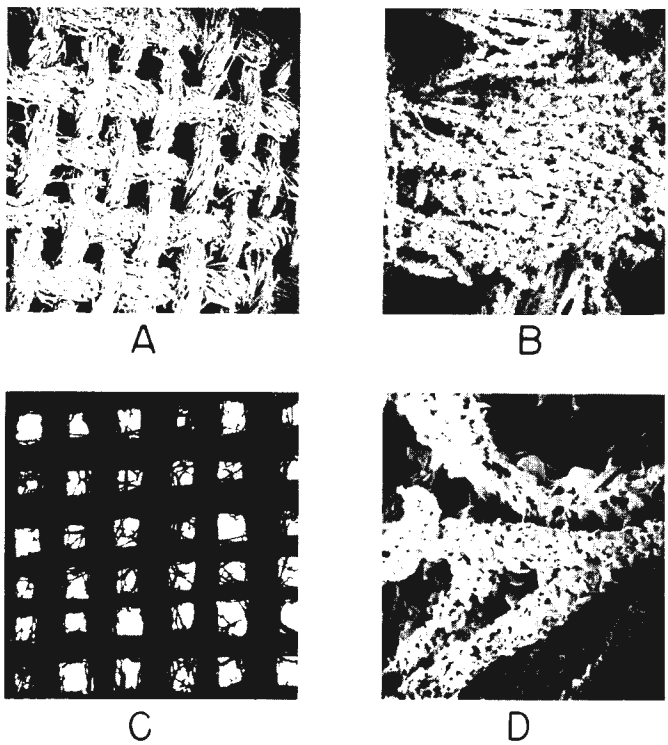


Figure 1

Cotton fabric with lead contents ranging from 4 to 40% were tested and all formed glassy materials. In Fig. 2 the glass shown as A was made using cotton printcloth containing 4% lead on a Pyrex dish. B had 8%

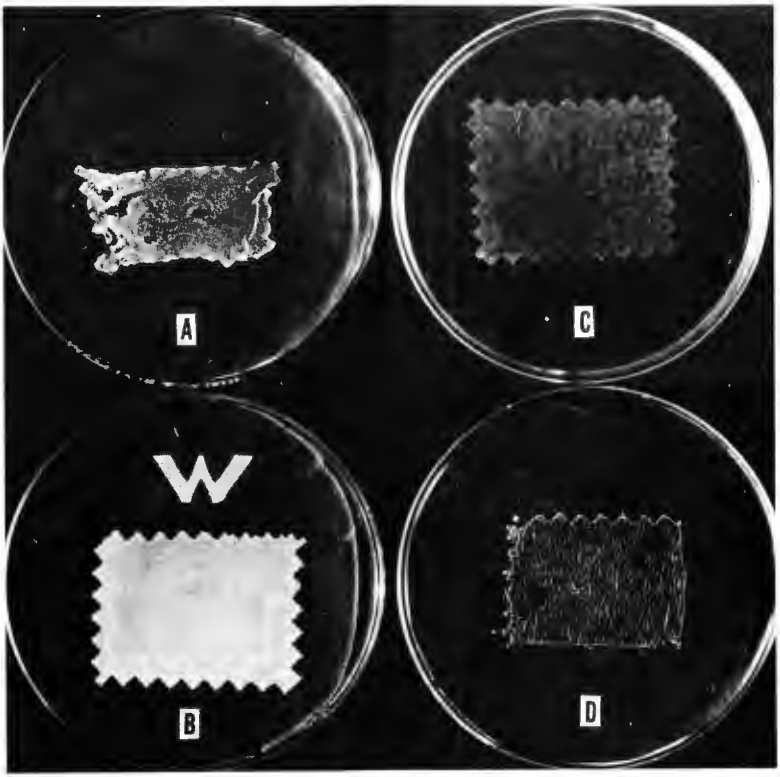


Figure 2

lead, C had 15%, and D had 30%. The glassy materials resisted concentrated HCL, HNO_3, H_2SO_4, aqua regia, and 50% NaOH for 48 hours with no apparent damage. They also did not appear to be harmed by repeated heating to 500°C.

When the lead content of the plumbite treated cotton was of the order of 20% or higher as with D, the glassy material separated in flakes from the silicon glass on which it was made. Careful examination of the flakes revealed the fabric weave pattern was still present in the new material. The flakes were hard enough to scratch soft glass and stainless steel. They did not melt in a bunsen flame at temperatures approaching 1500°C.

Cotton fabric with lead contents in the range of 4 to 15% formed a translucent glass on the surface of a Pyrex brand beaker, test tube, porcelain crucible, or other glass as shown in samples labelled A, B, or C. The translucent glass adhered firmly and had both the shape and fiber pattern of the cotton fabric from which it was made. This is best seen in B which had 8% lead. It could be written on with an ordinary lead pencil and the writing erased with the pencil eraser.

Fig. 3 shows a beaker and a test tube with several labels made by holding the leaded fabric in place against the vertical surface by using aluminum foil while heating. The percentage of lead in the starting fabric is written in pencil on the labels.

Figure 3

A closer look at these glasses is provided in Fig. 4 which shows microscopic views of the glasses front lit (top row) and back lit (bottom row). A and D were made from 8% leaded cotton, B and E from 15% and C and F from 30%. The back lit pictures clearly show the openings of the weave pattern as lined up light spots. The front lit pictures of the top row show the surface character which appears rough and fibrous when the lead content is low and explains why these samples can be written on. The 15%, or middle sample surface is at the margin of lead level for write-on labels. The higher percentage of lead as with the sample on the right gives a product that flakes off although it does still retain the weave pattern. The sample with the least lead is the most opaque and it seems almost as if you can distinguish carbon particles based on the amount of lead present, i.e., the left hand sample contains the least lead and is the most opaque with the most carbon.

All high silicate glasses served well as sources of silicon for the new glassy materials. In Fig. 5, the top row was made using cotton with 30% lead. The lower row had 15% lead. Soft glass (A & B) distorted some from the heat and produced a less transparent product than did Pyrex® (C & D), porcelain (E & F), Vycor® (G & H) or similar high temperature glasses.

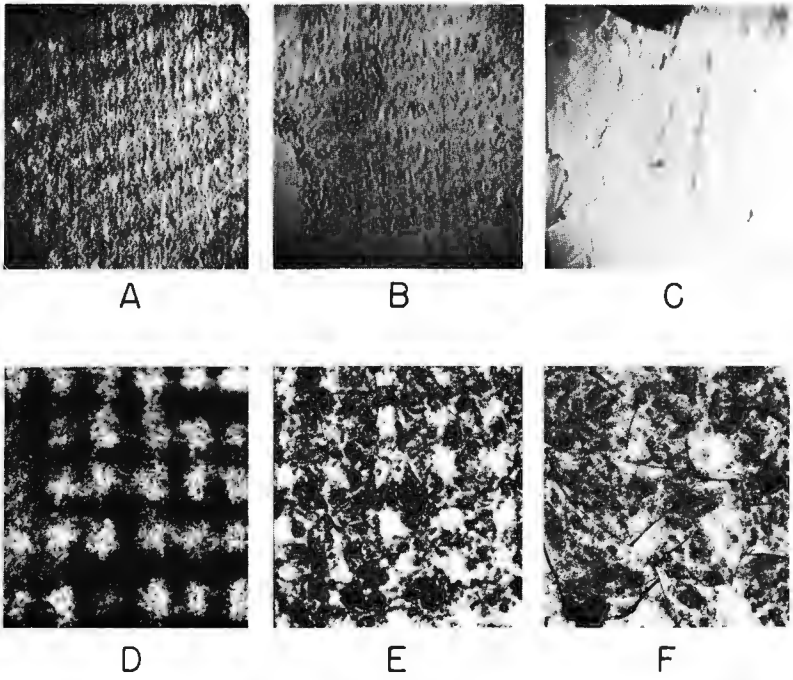


Figure 4

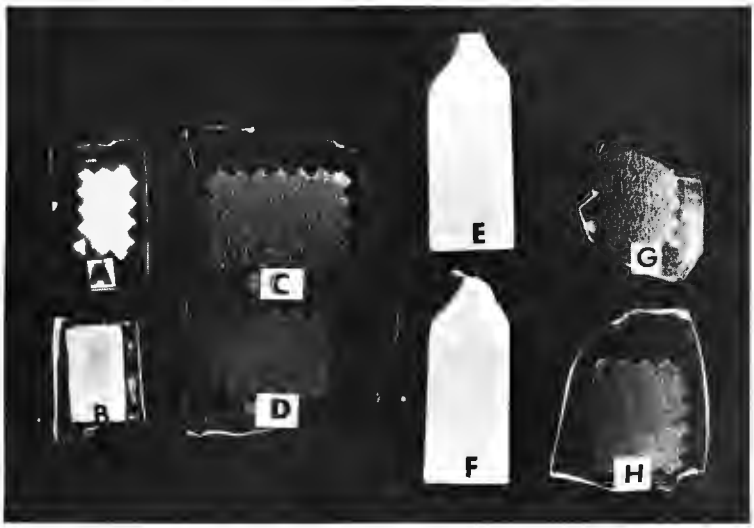


Figure 5

Sand, silicon dioxide, silicic acid, water glass, calcium silicate, and sintered silicon were all tried as sources of silicon. None of these substances in combination with the plumbite treated cotton fabric produced a glass. The thickness of the final product did not seem to be related to the fabric thickness.

Since new glassy materials made from low lead content cotton adhered firmly to glasses of different composition and properties, the fabric might be expected to form a glasslike link between two otherwise difficult to join glasses. Glasses such as Pyrex, Vycor, porcelain, and soft glasses were joined together as illustrated in Fig. 6.

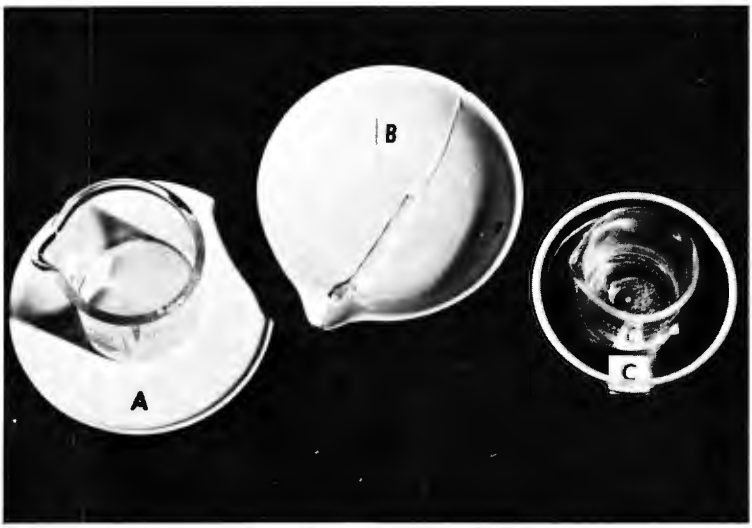


Figure 6

A piece of the plumbite treated cotton fabric was placed between the pieces to be joined and they were heated to a temperature of 600°C. A shows Pyrex joined to porcelain, B is soft glass joined to porcelain, D is Pyrex joined to pyrex. The glasses joined could be either alike or different. The time required at the highest temperature varied somewhat with the rate of heating and the size of the objects to be bonded, but was usually of the order of one hour. Pyrex could be joined to quartz by this procedure although the dimensions of contact between the pieces were more limited due to the large difference in coefficient of expansion.

After cooling, the bond was so strong that under applied force, the joined glasses ruptured more readily than did the bond itself. This can be seen in B where the porcelain was removed as the glass rod was broken away.

The glassy products could be caused to change color as shown in Fig. 7. The original clear color could be changed to gold (A), deep purple (B), silver or grey (not shown) by varying the length of time that the glass was exposed to the direct flame of a bunsen burner. When the new glass was adhered to the mother silicon glass, as in the low lead labels, the color could be removed by heating that side of the glass opposite from the film. This effect is evident in the center of sample B. Reheating any of the colored glasses in the muffle furnace to 600°C. caused them to return to the original colorless state. This is shown as the two pieces of B which have the arrows on them. This color reversal process is repeatable. The colored glassy film is not a good electrical conductor as an elemental lead coating would be.

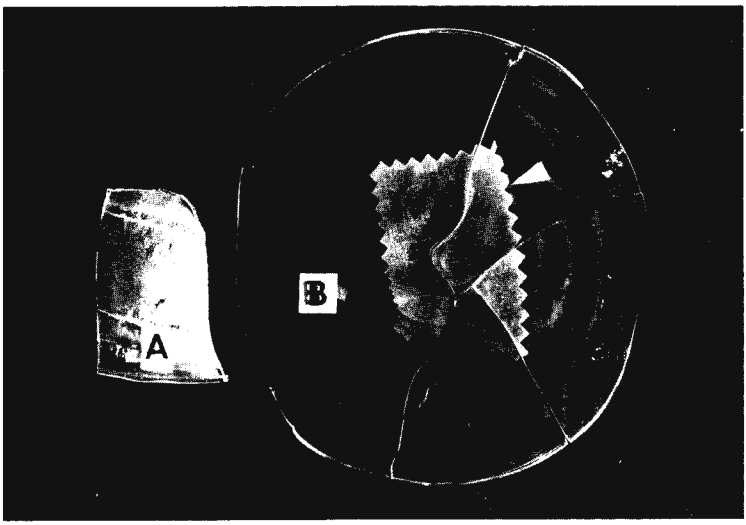


Figure 7

A conclusion that can reasonably be drawn is that only sufficient silicon atoms migrated from the glass holder to the leaded cotton skeleton to form the new glass. The glass holder was left roughened by this procedure as shown in Fig. 8. When the new glassy material was removed, as it was when the lead content was high, the glass holder was etched with the fabric weave pattern as shown here. Another conclusion that can reasonably be drawn at this point is that carbon in the skeleton of the plumbite treated cotton fabric participated in the formation of the new glass.

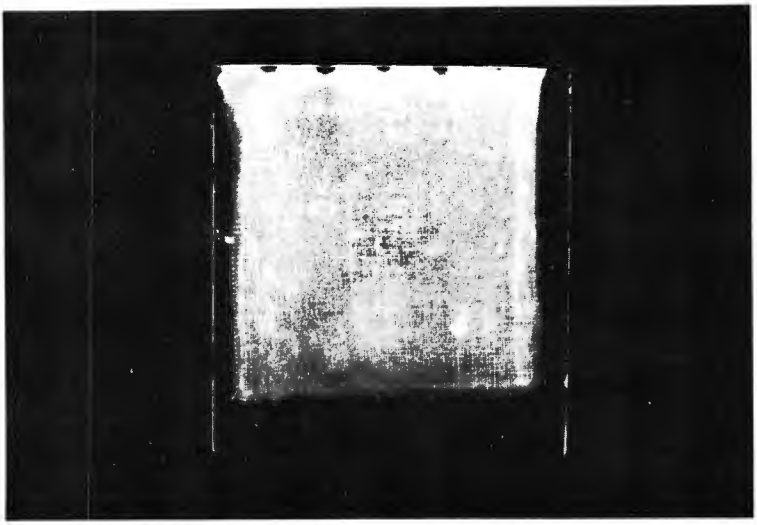


Figure 8

In a usual glass surface, each silicon atom is bonded in a network with oxygen atoms. In the case of these new glassy materials, it appears that the lead oxide from the cotton fabric with the carbon in it helps break the silicon to oxygen bonds allowing new bonds to be formed outside the surface of the old glass and in the carbon skeleton of the new glassy material. As heating continues, some carbon may be expelled as CO_2. The resultant new glassy material then usually contains carbon and shows the grid pattern of the fabric from which it originated.

A glass made from cotton fabric containing 15% lead and that same glass heated with a bunsen flame to turn it silver in color were analyzed by electron emission spectroscopy. The amount of lead and carbon relative to silicon is higher for the new glass than for Pyrex glass. After turning silver, the amount of siilcon and oxygen compared to lead and carbon increased, which seems to indicate that the color is not due to elemental lead. The lead is in the same oxidation state as before it turned silver in color.

X-ray fluoresence also indicated the presence of substantial carbon although in this procedure the silicon and lead are measured directly and the carbon calculated. A calculated ratio of atoms shows 30 oxygens to 10 carbons, 3 silicons, and 1 lead. This rate indicates that at least some of the carbon must be serving only as a filler or backbone.

A third and final conclusion is that since higher lead content results in a glass that releases more readily from the surface of the old glass, the lead is important in the formation of new stable bonds, and probably results in a greater difference in coefficient of expansion between the new product and the silicon material on which it was made, thereby facilitating release between the two.

SUMMARY

These new glassy products and their mode of preparation are not usual for glasses. It does seem that, at least initially, the new materials have skeletons of carbon that may not be chemically bound to either silicon or lead. Some of these materials are clear and some are translucent. The materials are formed by extracting enough silicon from the surface of the mother silicate glass to create new bonds. Under intense heat in a reducing atmosphere a shiny thin layer forms which appears to be metallic but which has low electrical conductivity and which analytical procedures indicate is not pure lead. This color may be removed by heating again in the muffle furnace. The new materials are acid and alkali resistant. They exhibit possible uses as labels, adhesives, mild radiation shields, and decorations.

ACKNOWLEDGEMENT

The authors wish to thank W. Goynes for Electron Microscopy; Biagio Piccolo for X-ray fluoresence; Donald Soignet for ESCA; and Austin Mason for consultation and help throughout this investigation.

Trade names are given as exact experimental conditions and not as endorsement over other similar products.

SOME PROPERTIES OF GLASS BALLOONS

DAVID ROGERS CROSBY
100 Chestnut Street
Haddonfield, New Jersey 08033

Tethered glass balloon with average diameter of 100 millimeters and buoyancy of 54 milligrams. Calculated buoyant life is 36 years. Average thickness of glass envelope is 5.4 microns.

INTRODUCTION

In recent months, a technique has been developed for constructing glass balloons having a diameter of about 100 millimeters and a buoyancy of about 54 milligrams. Because a glass envelope is much tighter to helium than the usual plastic envelope, new uses for small balloons may now arise. This paper describes the new balloons and considers possible applications for them.

The envelope of a balloon must be light-weight and preferably gas tight. Steel and aluminum are excellent gas barriers[1][2] and may eventually be used in small balloon-like cells as part of air-buoyant foam. Glass and metals share the disadvantage that their envelopes must be permanently inflated at the factory, as simple films of glass or metal cannot be repeatedly folded and compactly stored without damage to the film. In contrast to this, is the excellent folding capability of plastic film.

Compared to glass, metals have the disadvantage of a narrower working range of temperature, so their hot-forming is relatively difficult. However metals have attractive cold-working characteristics and superior resistance to thermal and mechanical shock.

The first passenger balloon (1783) had a paper-lined linen envelope, but within a few days a rubber-coated silk envelope was also used.[3] In the 1940's, plastic envelopes of nylon, polyester, neoprene and polyethelene came into use. In the 1950's, composite metal-plastic film such as aluminized polyester were developed for balloon envelopes.

Helium (isolated as an element in 1895) was first used in balloons in 1921.[4] The light-weight inert helium molecules are much more difficult to contain within a thin envelope than the slower partner-seeking molecules of air or methane. All envelopes of fabric, rubber or plastic perform poorly as containers of helium, compared to glass envelopes. The greater hardness, greater density and higher softening temperature of glass compared to the plastics are all indicators of the greater tightness of glass.

For example, a 100 mm (3.94 inch) diameter helium-filled rubber balloon has a theoretical buoyant life of three or four hours, compared to many years for a glass balloon of the same diameter. The buoyant life of a balloon increases rapidly with size. A helium-filled passenger balloon of 12 meters diameter using a plastic envelope, could have a buoyant life of a year. In brief, the chief limitation of passenger balloons of all sorts is their poor maneuverability in adverse wind, the chief limitation of conventional small balloons is the shortness of their buoyant life, and the chief limitation of glass balloons is their fragility.

BALLOON GASES

The interior of a balloon is filled with a gas lighter than the ambient air. Under conditions of constant temperature, the six lightest gasses are: hydrogen, helium, methane, ammonia, neon and acetylene. Also heated gases are used in balloons. Hot-air passenger ballooning is now a thriving sport in several countries. Although in principle, most any gas can be heated to be lighter than air, the convenience of air results in its widespread use in hot-gas balloons.

A balloon can be made with a vacuum in the interior, and a US Patent[5] issued in 1966 describes such rigid ceramic balloons. Few if any of this design appear to have been constructed. Two of its advantages are: stress in wall is independent of temperature, and the stress in wall decreases with increased altitude. The nominal operating stress in the wall

of a rigid vacuum balloon would be twenty or more times that of a pressure-inflated fixed-volume balloon. A slight flexibility in the rigid material of a vacuum-filled envelope could generate dangerous tensile stress.

Balloons using a light gas now mostly employ helium or hydrogen, as all other gases have a relatively low lifting ability. As hydrogen is dangerously combustible, helium has been used in the glass balloons constructed on this project.

FIXED VOLUME VERSUS VARIABLE VOLUME BALLOONS

The glass balloons constructed on this project are here treated as being of fixed volume. As the differential pressure across the envelope changes in normal operation, there is a change in the balloon volume of perhaps one part in 1000 due to the elasticity of the glass. This variation we disregard.

A fixed-volume balloon has a predictable elevation, knowing the barometric pressure and air temperature profile and with negligible vertical air currents. At this stable elevation, the air density and balloon density are equal. It is a pretty sight to see a suitably-ballasted glass balloon glide into its stable elevation within a room and hold this elevation within a few centimeters. Without ballast, the stable elevation of one of these glass balloons is calculated at 734 meters above sea-level.

A fixed volume balloon may burst from excessive differential pressure before it reaches its stable elevation. In the era of the dirigibles (1900-1937) safety valves were sometimes installed on their gas bags to forestall such mishaps. Balloons employed up to 1000 meters in elevation need have their volume increase by only about 12% to avoid excessive pressure. Balloons used in scientific research may experience a volume increase by a factor of 1000 or more over the ground-level volume. Such balloons may have elaborate arrangements to facilitate this volume increase.

The buoyancy of a fixed volume balloon is proportional to air density, which permits the balloon to be used to demonstrate several scientific principles, and to indicate changes in air density and changes in altitude, so that it supplements the barometer and becomes an attractive teaching device. However fixed volume balloons may burst because of high interior pressure due to high ambient temperature. Also glass balloons may collapse and fracture due to low interior pressure caused by low ambient temperature.

A glass balloon may be made variable volume by building in a bellows-like section. In the 1920's, a bellows-like mid-section was sometimes incorporated in passenger balloons that were otherwise spherical in shape.[6] A small amount of variable volume could be obtained by constructing a glass balloon in the shape of an oblate spheroid.

DESIGN CONSIDERATIONS FOR GLASS BALLOONS

Table 1 has been computed for glass balloons of 100 mm diameter, as well as for 10 mm and 1 mm diameter.

TABLE 1

PARAMETERS FOR GLASS BALLOONS

Diameter mm	Mass of Air mg	Wall Thickness microns	Wall Stress atm	Buoyant Life years
100	631	5.4	184	36
10	0.613	0.54	184	0.36
1	0.000631	0.054	184	0.0036

The second column indicates that the mass of air (at 20°C) displaced by the balloon varies as the cube of the diameter. This follows from the geometrical formula for the volume of a sphere.

The third column concerns the wall thickness. Increasing the diameter by a factor of 10 increases the allowable glass weight by a factor of 1000, but since the wall area increases by a factor of 100, the wall thickness can only increase by a factor of 10. For all three diameters listed, the weight of glass in the envelope was taken as 68% of the weight of the displaced air. The balance is made up of 14% helium, 9% for glass in the seal and 9% for buoyant force.

The fourth column shows that the stress of 184 atmospheres (2700 pounds per square inch) is independent of diameter. This follows since the cross-section of the wall resisting the internal pressure, and the total internal force producing the stress both vary as the square of the diameter. A fixed internal pressure of 1.04 atmospheres is assumed.

The last column, based on permeation rates of helium thru glass measured by V. O. Altemose, shows the theoretical buoyant life to improve as the square of the diameter.[7][8] The end-of-life point was taken when the internal pressure drops to 1.00 atmospheres. The buoyant life is for the common soda-lime glass (Corning 0080). A buoyant life shorter by a factor of from 100 to 1000 would be expected from a typical borosilicate glass. A buoyant life longer by a factor of about 100 would be expected from an aluminosilicate glass (as used in radio tubes).

The balloon of 1 mm diameter has a theoretical buoyant life of only 1.3 days. When the diameter of the balloon increases by a factor of 10, the volume of the balloon and the volume of allowable lost helium each increase by a factor of 1000. The increased wall thickness reduces the rate of helium loss by a factor of 10. The product of these two factors, 10,000, is divided by the factor of 100 for the increased permeation area, resulting in a net increase in the buoyant life by a factor of 100.

The theoretical buoyant life can be improved by a factor of 2 or 3 by increasing the original load of helium so the internal pressure is 1.08 or 1.12 atmospheres, but at the cost of a more probable failure of the glass in tension. A 10 mm diameter balloon with 0.54 micron wall, has a wall thickness equal to about 2000 diameters of an oxygen molecule.

BOUNDARIES FOR BALLOON OPERATION

The calculated boundaries for the operation of a typical fixed-volume glass balloon of 100 mm diameter are shown in Fig. 1 plotted against temperature and elevation. The zero-stress boundary indicates the conditions for the same pressure on each side of the envelope. The balloon would withstand compressive stress if its shape were a true sphere with uniform wall, but as a practical matter the envelope is not self-supporting and any compressive stress is likely to produce a dimple in the envelope that develops into a fracture. This zero-stress line assumes the internal pressure is 1.04 atmospheres at 20°C and sea-level. The line indicates that at sea-level the lowest operating temperature is 9°C, and that at 20°C the lowest elevation is 340 meters below sea-level.

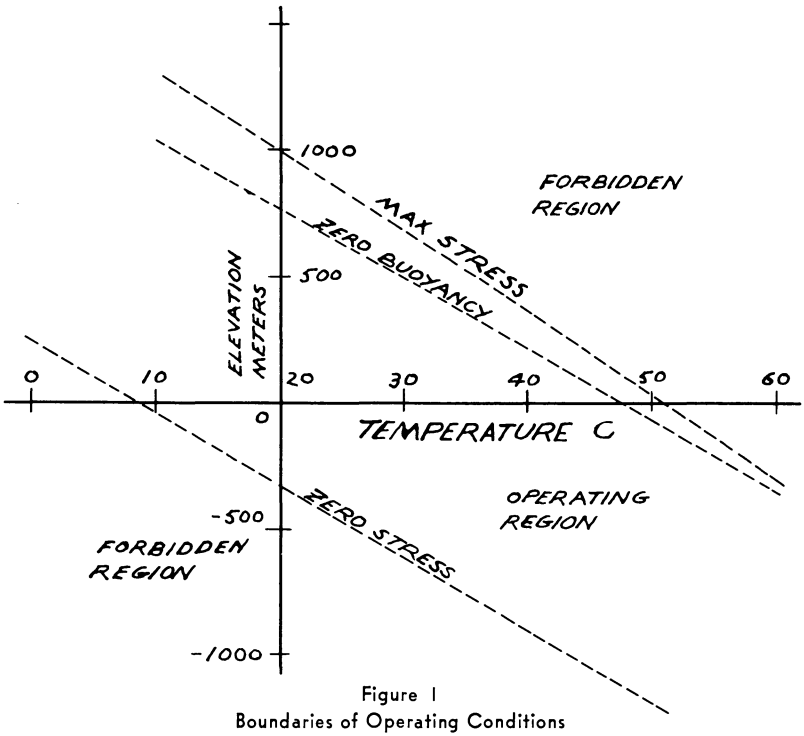


Figure 1
Boundaries of Operating Conditions

The maximum-stress line indicates the conditions for the internal pressure to be greater than the external pressure by 0.15 atmospheres. Based on a small amount of experimental data, this is the condition at which the average envelope fails in tensile stress. It corresponds to 690 atmospheres (10,100 pounds per square inch) stress in the wall. As many balloons have weak areas, the maximum-stress line as shown is often optimistic. The plot indicates that at sea-level the highest operating temperature is 51°C and that at 20°C the highest operating elevation is 1000 meters above sea-level.

The zero-buoyancy line indicates the condition for the average density of the balloon to equal the density of the ambient air. For conservative operation, stay well away from the two pressure dependent lines, but this zero-buoyancy line may be visited.

The above buoyancy calculations assume dry air. Adding water-vapor to dry air produces a mixture with a density less than the dry air, as the molecular weight of water-vapor is only 18 compared to 29 for dry air. A 100 mm diameter balloon with a 54 mg buoyancy in dry air at 20°C, will have a buoyancy of only 50 mg in saturated air. For further precision in determining the operating boundaries, a correction can be made for the deviation of the barometric pressure from normal.

EXPERIMENTAL BUOYANT LIFE OF GLASS BALLOONS

The several dozen glass balloons constructed to date have mostly been broken by careless treatment or by intended rough treatment such as exposure to cold air. Some failed after a few hours or a few days, due to leaky seals. A few have failed suddenly while carefully stored in covered glass jars, expressing the well known tendency for glass in tension to fail unpredictably. There is no clear evidence that any of the balloons have lost helium by permeation thru the glass wall. One envelope has retained its buoyancy for six months.

The typical balloon can be held in the hand and tested by being gently squeezed. If it feels too soft, it may have a leaky seal, or be properly sealed but with less than normal charge of helium. Occasionally a balloon will escape just after the sealing operation and rise to bump the laboratory ceiling. Such fugitives are captured with the aid of a step-ladder.

When a balloon bursts due to excess interior pressure, the fragments fall in a circle not much larger than the balloon diameter, as the movement thru air of a small or thin particle encounters a large air-resistance per unit of particle mass.

GLASS BALLOONS AS AIR FLOW INDICATORS

A glass balloon is an excellent device for studying air circulation in a room. By trimming its ballast, the glass balloon can be made to stay at constant altitude in still air. Such free-floating balloons will search the room for channels of air flow. At a cool or warm wall, or at an operating floor-lamp, the balloon may find a circulation loop, make several round-trips in that circulation, and then move on. At a cold wall, there is a cascade of cool air falling off the wall, but this air returns in a broad upward-flowing stream a meter or so from the wall. A free-floating balloon may find in a room unexpected air exits or inlets.

A tethered glass balloon is useful to indicate both direction and strength of air flow at a fixed location in a room, the longer the tether the more sensitive the indication. Outdoor air velocities, even on a still day, may be so great as to put excess stress on a tethered glass balloon.

GLASS BALLOONS AS INDICATORS OF AIR DENSITY

The ratios below are for a balloon with trailing tether weighing 83 mg per meter. A trailing tether permits the balloon to rise until a length of tether is supported equal to the net buoyancy of the balloon, as the tether is not fastened at its base. The ratios are calculated for experiments made at elevations near sea-level. These ratios assume negligible variation in forces due to vertical air currents, and negligible variation in forces due to any electrostatic charge on the balloon.

(1) A one percent increase in the air density produces a tether lengthening of 76.3 mm. Here no assumption is made of fixed pressure, temperature or elevation. All may change. This relation directly reveals changes in air density with time or with base elevation, which is inherently impossible to do by observing a conventional barometer.

(2) For a rise in base altitude, the tether shortens by a factor of 1,120. This relation is useful in getting the height of multi-story buildings, when the temperature is substantially constant.

(3) For a rise in the mercury barometer reading, the tether lengthens 10.0 times that distance. This relation is useful in getting changes in the barometric pressure over a period of time, at a fixed location and constant temperature.

(4) For each centigrade rise above the original temperature, the tether shortens 26.0 mm. This relation is useful in getting small temperature changes, as within a room, at constant pressure and elevation.

The ratios given in (1), (3), and (4) above when multiplied by 1120 give the elevation sensitivity for a free-floating balloon that is adjusted by ballast to be in equilibrium at the start of the test.

GLASS FOAM BUOYANT IN AIR

A lighter-than-air foam can be constructed using glass balloons such as have been built on this project. Such a foam has two phases: the glass balloon phase to impart buoyancy, and a second bonding phase to structurally unite the balloons and give rigidity to the entire slab. The bonding phase can be of plastic foam or of glass foam. A commercial lighter-than-air glass foam based on a buoyant cell of 30 mm diameter might have a density as low as 90% of that of air. Using closely packed buoyant cells of two different diameters, even lower densities would be expected. This two-phase foam has the advantage in fabrication that the helium is near room temperature at the time the buoyant cell is sealed.

Although the fixed-volume glass balloon is now available for air-buoyant foam, metal cells and cells of composite construction might be employed. A bellows action allowing a volume change of 20% would enable a buoyant cell to operate safely over the temperature range —10°C to 50°C.

Slabs of buoyant foam might be used as controllable clouds to raise the temperature at the earth's surface (such as in an orchard) during

cool nights. A disc-shaped cloud of 100 meters diameter and 5 meters thickness, tethered at an elevation of 20 meters, could be hauled down and stored behind a wind-breaker during the day and on windy nights.

CREDITS

This on-going project, now underway for about five years, has greatly benefited from numerous comments of friends, and from experienced workers in the glass industry. Prominent among those who, without a professional background in glass technology, made stimulating observations are: Isaac Abeyta, John Liggett, Aristid Grosse, Alexander Rehe, Howard Branson, and Mildred Glenn.

Most of my exposure to glass working techniques came from my association with John Giacometti who did most of the early laboratory work, as well as important parts of the later laboratory work. Some other professionals in the glass industry who also made valuable contributions are Wayne Wynn, Jasper Freeman and John Rossi. The project was facilitated by glass preparation done by Wilmad Glass Co. and by Precision Electronic Glass Co. Important references to the literature of glass technology were supplied by the Corning Glass Works.

REFERENCES

1. R. M. Barrer, "Diffusion In and Through Solids", University Press, Cambridge, 1941
2. W. Jost, "Diffusion in Solids, Liquids, Gases", Academic Press, 1960
3. "History Of Flight", Amer. Heritage Pub. Co., 1962
4. C. W. Seibel, "Helium, Child Of The Sun", Univ. Press of Kansas, 1968
5. A. Axelson, "Evacuated Hollow Spheres", US Patent 3288398, Nov 1966
6. R. H. Upson and C. F. Chandler, "Free and Captive Balloons", Ronald Press, NYC, 1926
7. V. O. Altemose, J. Appl. Phys., Vol. 32, No. 7, July 1961
8. V. O. Altemose, Proc. Symposium Amer. Sc. Glassblowers Soc., 1962

ABOUT THE AUTHOR

D. R. Crosby was born in 1911 at Lockport, New York. For 36 years he was employed as an electronics engineer, specializing in circuit theory, applied mathematics, and applications of computers to engineering problems. Since 1971 he has been self employed. He is a licensed Professional Engineer and a member of the American Scientific Glassblowers Society.

LOW TEMPERATURE (I - R) OPERATED BOROSILICA LIGHT INTEGRATING SPHERE

EDWIN E. ECKBERG
ECKLUX R & D Vacuum Laboratory
P. O. Box No. 5102
Boise, Idaho 83705

INTRODUCTION

Light integrating spheres have been used extensively. The ULBRICHT sphere[1] is mentioned as one of the early forms designed and developed several decades ago. Prior to that, when most light sources were relatively small, their radiation was measured with a Bunsen or the improved Count Rumford type of photometer.[2] These measurements were quite accurate, however, since the source lamp was considered as being a "point" source. Hence, the inverse square law was applicable in such cases. But with the advent of larger and extended light sources such as powerful incandescent beacons and tubular lamps, such as the fluorescent and/or the sodium vapor lamp, the spot source definition no longer held. It was for this reason that the design and use of the light integrating sphere was worked on and progress was made. In the early 1960s the A V C O Research Laboratories devised an early form of the so-called high gain light integrating sphere. The surface of their model was rendered lenticular with the forming of small dents. The interior of the sphere was made highly specular with a thin film of aluminum. It was useful and offered some gain. Shortly thereafter I was contacted to work on the development and improvement of this type of integrating sphere; but larger sizes were wanted, and higher gain factors. Also, it was advised that the improved integrating sphere, when completed, would be used in the infrared. At our Sixteenth Annual Symposium, in Milwaukee, Wisconsin ('71) the result of these efforts was covered, up to that time.[3] But, on the other hand, it was during this development period that strong and valid indications justified the decision that the high gain light integrating sphere (later) would be modified to allow a low temperature environment. Liquid nitrogen for one example. At that time, indeed, the writer submitted a design. It was quite similar to the one we will discuss in the present case. FIG. 1. The sketch illustrated one method that might accomplish this low temperature problem. But the design was allowed to remain dormant for an extended period of time. Then, about a year and a half ago, it was given serious study and was accepted for development.

GENERAL — SPECIFICS — AND, DETAILS

Figure No. 1 illustrates the scheme of the low temperature operated light integrating sphere. The innermost of the three glass spherical flasks is the gold thin filmed integrating sphere itself—which is rendered lenticular. The middle glass flask is used to form the boundary of the coolant compartment which surrounds the integrating sphere. The third and outermost glass flask is to provide the boundary for the insulating compartment—a dewar type of arrangement. To maximize the insulating charac-

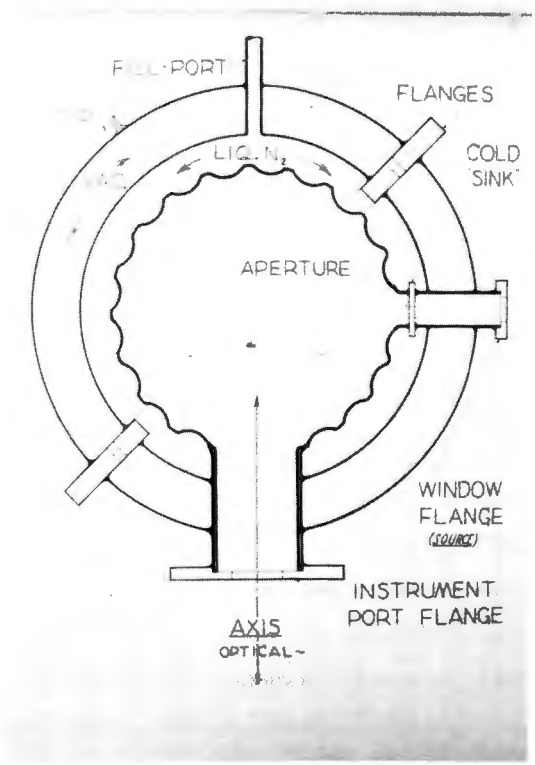


Figure 1

The Low Temperature (I-R) Operated Borosilica Light Integrating Sphere. Schematic one-line sketch showing a vertical sectional view of the L'TIRIS (an acronym for "Low Temperature Operated Integrated Sphere.")

ter of this dewar jacket the outer surface of the middle flask, and the inner surface of the outermost flask are thin-filmed aluminum. An opaque film is used and allowed to age several days. As far as the point at which it is best to stop glasswork and the assembly of same to do the filming, well . . . I can only say that the gold film for both the integrating sphere and the inner walls of the instrument port tube is best done after all subassembly of glassblowing components. But, the inner surface of the outermost sphere can not await complete and final assembly, the outer surface of the middle flask must be done at a point in the procedure that permits an access to its outer surface. I'll refer to this a bit later. The two large ring-type and mated flanges, obviously, are required in order to assemble this unique form of device. The main port tube with its attached instrument port tube flange are attached to the bottom of the integrating sphere, centrally positioned. The latter flange mentioned, or the instrument port

Figure 2
Round Bottom Spherical Borosilica Flasks.
(As used on the construction of the L'TIRIS.)

tube flange, is attached to whatever instrument is to be used for detection and measurement of the radiation being investigated. As stated previously, this radiation can be of low energy — extremely low.

A source port tube having a window flange leads from the integrating sphere completely through to the outside of the entire assembly. This source port tube, therefore, requires two ring seals, both extended. Suitable window material (not shown) is positioned on the window flange. One example of such material is calcium fluoride. Another is magnesium fluoride. A cold "sink" plug or flange is an integral part of the source port tube sub-assembly. The sink is to be located as that region that will be cooled by the liquid coolant. The sink plug (or flange) is provided with a coarse orifice—say, about one-quarter of an inch diameter. But, surrounding this hole are eight equally spaced tapped holes (2-56 TAP). These tapped holes will be used to attach various sized "aperture" discs (not shown). Each of the discs, of varied aperture diameter, may be considered as quite analogous to the "f/stop" adjustment on ordinary cameras.

At the very top of the unit is a fill tube which leads down into the liquid nitrogen compartment. An air bleeder tube (not shown) is to be used during any filling cycle to permit the entrapped atmospheric gases to escape from the compartment as it is being filled.

Any meagre mention of the theoretical which I will present is to appear near the end of this paper. At the moment, however, we must remark that the "critical angle" will determine, largely, the final effectiveness of the assembly's performance when completed and used — all other

factors involved being considered as being of optimum value or character. This critical angle is maximized by the simple means of using a wider or rather as large a diameter, as feasible, for the heavy wall instrument port tube. Too, the length of the instrument port tube should be kept to a minimum. It can be mentioned that a computer study which was made prior to construction gave promise that an angle of twenty degrees for the critical angle would be successful. The unit illustrated has an angle in excess of 35°.

GLASSBLOWING AND OTHER MATTERS

As we give first glance at this assembly we can honestly admit that it presents problems in glassblowing, machining, and mechanics/physics that, virtually, border on the impossible. However, as the old saying goes, . . . "the impossible takes a little time." And I like to think that that is what research and development is all about. At least it is true that the challenge is there for one to accept. Candidly, about nine near-complete trial efforts were carried forward before the tenth unit proved a success. The actual procedure which was followed is now outlined: Round bottom long vial neck borosilicate flasks were used for all three spheres. The innermost was first worked in a horizontal lathe in order to attach the proper sized instrument port tube. Since this tube will bear the entire assembly weight it is of extra heavy wall thickness. The beginning of the source port tube is next sealed on at the side, or ninety degrees from the instrument port tube's axis. This axis is also the optical axis. Finally, on careful removal from the glassblowers lathe, this is given a full anneal cycle. In my case, the vertical glassblowers lathe, or machine which is available has the electric annealing furnace enclosing the entire workpiece space. FIG. 3. Diameters in excess of twenty inches can be handled.

Figure 3
Vertical Glass Lathe With Electric Anneal Furnace.

Figure 4
Electric Annealing Furnace.

Therefore, following the anneal cycle, the vertical rotation and vertical positioning of the sphere proved a convenient one for the forming of the lenticles over the entire surface of the flask. For a safe method of forming these lenticles please see Reference 4. I have found that the use of a polariscope is of real help. At any rate, the spherical zone being worked must be kept well up near the softening point of the glass. The workpiece, of course, is rotated (by machine), during heat up periods — and stopped just long enough to form a few lenticles. The more difficult areas to be lenticulated are those which surround any and all of the tubulations. It is my personal practice to do these first, then the remaining area seems relatively simple. While forming the lenticles, the entire sphere must be held above 500°C. And, the temperature at the spherical "zone" actually being worked, or lenticulated, is best raised to the higher temperatures which nears the softening point of the glass. If this latter temperature be as high as 650° to 700°C., then it will take far less time to raise the temperature to its softening point and form a lenticle. I have made as many as five or six lenticles during one stoppage, and the overall temperature of the entire workpiece has not fallen below 550° or 525°C. But, again, these temperatures should be monitored by thermocouple-junction method if possible. This removes the guess work.

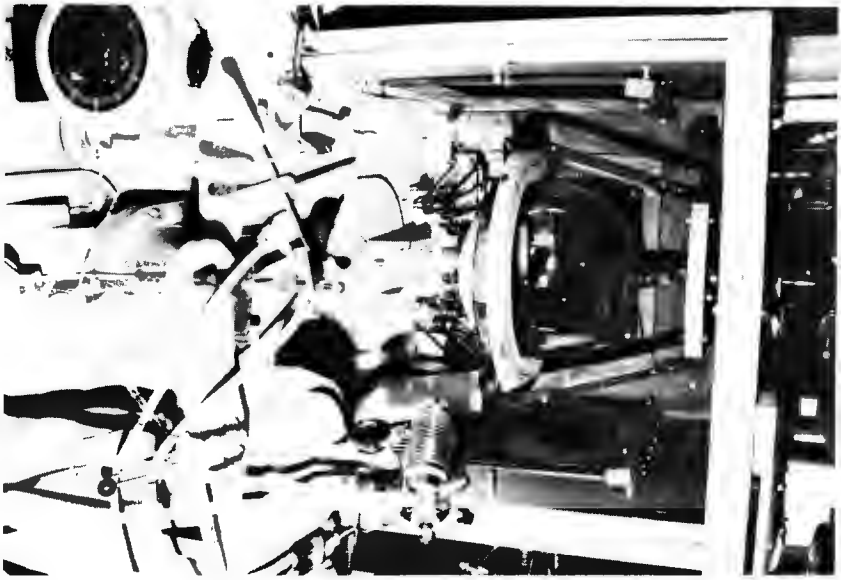


Figure 5
Vertical Glass Lathe in use.

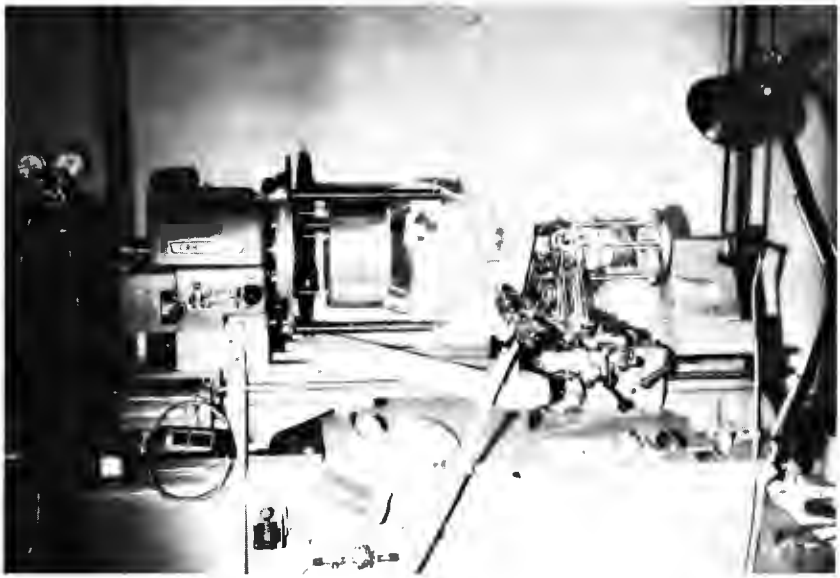


Figure 6
Horizontal Glass Lathe (HOMEMADE) in use.
(Note: 10 jet LITTON fires — adjustable.)

The next set of operations involves both of the outer spheres. Each flask, in turn, must be cut exactly in half at the indicated 45° angle with respect to the main axis. And, since we are not sure that the cut edges will result in a perfectly circular one — until after they are made — we must do the cutting prior to the making of the two ring-type metal flanges which are to abutt the halves together in final assembly. The cut can be non-circular after these cuts are made. Not much, of course, but enough to control (rather affect) the dimensions for the grooves in the two ring-type flanges that are to receive the cut edges and be sealed. (Not shown in sketch). There are two such grooves in each ring-type flange face which will receive the cut edges of the two divided flasks, and space enough in each groove for their proper sealing. But, before the cutting, it is necessary to cut off the necks and tool the bottom halves of each of the two, to fit the outside dimension of the instrument port tube on the integrating sphere. (NOTE: These tooled holes are to form the outer member of the two main ring seals — later.) Also, on the top of the middle flask a heavy wall tubulation is sealed on, this being the start of the "fill" port tube. This project necessitated the making of an improvised wet wheel glass cutting machine.[5] When making the cuts (45°) it was observed that fluid lubricant/coolant must be applied to the interior of the large sizes, as well as the commonly-known outside jet spray, during all cutting operations. The spherically shaped flasks are best worked, or cut, by seating them into a semi-rigid bowl. I found that a pet-food tray (plastic) or bowl, was excellent. The cut line can be set forth using masking tape and a water proof ink lining. Then, as a maximum cut is completed. (controlled/limited to the length of the pendulum type of swing support) the wheel can be returned to its starting position, the workpiece rotated to uncut portions and another cut may be made. With a 14 inch wheel, it took me about four passes to cut a 22 liter flask in half. I experienced no losses on this cutting arrangement.

At this stage the components that are at hand permits some subassemblies to be made — more glassblowing. This involves five ring seals, with some of them requiring an extension (same size of tube) by the butt seal method along with the ringseal blowout preceeding the three-piece united ring seal itself. Thorough annealing cycles follow each and all completed seals. Proper heatup time is, of course, necessary prior to making each of the successive seals.

This paper was to have included coverage of the very important glass stress strain consideration. But, in deference to another Paper Presentation at this A.S.G.Society's 1975 Symposium, this is presently deleted. Just a few remarks: The initial and the several failures in the development of this low temperature glass integrating sphere were not all the result of an annealing problem. Nor, indeed, were they the result of any normal stress. It can now be recalled that many mechanical problems presented themselves. Space forbids mentioning all of them. But the chucking arrangements, the thermocouple(s) and their respective insertion into the workpiece with the necessary swivel provision being made, and, the ring-type flange-to-flange plus glass-to metal-flanges, with the fastener pressures required for both vacuum and fluid tight sealing . . .

well, some failure was to be expected. But, as far as the glass is concerned a full consideration must be given to the mass and the size of the glass, (in view of its geometry), the heatup time factor, the temperature determination, and the proper or sufficient amount of heat for the mass of glass being heated. Then, a maintenance, or the holding of that temperature condition within the safe limits of the stated temperature range during any and all times that fusion or actual glassblowing is to be performed. It is true that we should possess the ability to "get the feel of any glassblowing problem." At least with time and experience. So it proved out in this particular case. But to return and complete the glassblowing on this device. We have the choice of three methods to effect most glassblowing operations: The off-hand method; the vertical glassblowing lathe or machine; and, the well-known horizontal glassblowing lathe. Generally, since no machine can equal the "feel" of the human hand, I have usually preferred the off-hand. But, because of the size and the weight the machines must be used. The vertical type is excellent in view of production and economy of floor space. However, for the individual project, and the problem that gravity can impose, it is best, in most cases, to use the horizontal glassblowers lathe.

First the source port tube of the innermost lenticular sphere is cut at a length that allows the lenticular sphere to be fitted down into the lower half of the middle half flask. The source port tube can then be ring sealed to this middle half flask, and the tube extended by butt sealing. The latter extension is, of course, the same size as the original source port tube. Then the first of the two large main ring seals onto the heavy wall instrument port tube can be made. Similarly, the lower half of the outermost half flask is port tube ring sealed, and extended. Same size tube abutting ring seal. To complete the assembly of the entire lower half of the whole unit (glass-wise), the second large main ring seal is made. All of this takes a lot of preliminary chucking and fixture modification to enable or insure a safe and true rotation of the work pieces; along with provisions for positive means of blowing into any or all of the three compartments. This blowing is very important. It effects strong seals that would otherwise strain and crack, irrespective of any amount of annealing. It is quite natural to overlook the amount of time that these preliminary matters consume.

Now that the lower half of the glass work is assembled (AND ANNEALED) it may be used to facilitate the concluding glassblowing operations — those involved in the assembly of the upper half of the entire assembly. But, in order to use the lower assembly, the instrument port tube flange must be affixed; and, the ring-type flange with the grooves must be sealed to the two half flasks of the lower glass assembly. A special low temperature putty type of epoxy[7] is used for these, and for all subsequent seals — excepting the two flat mated faces of the two ring-type flanges themselves. This sealing compound is excellent, and is conveniently mixed and applied. The upper fill tube of the upper half of the middle flask is cut to a suitable length to allow the proper and symetrical fitting of the two upper half flasks. The ring-type flange for these two half flasks can now be attached and sealed, and the two completed half assemblies, both the lower and the upper halves, can be abutted and the

fill tube ring seal with its extension tube can be made. Of course our assembly is only a temporary one, for after the fill tube ring seal is made, the upper half (glass only) of the assembly — with the flange removed, is given a full anneal cycle. Following this annealing, all seals can be made using the low temperature epoxy compound. The cut edges of the half flasks (four in number) are optionally covered with a narrow stripping of paper. (e.g. masking tape is suitable for this). The paper is best applied as a bit wider that the actual wall thickness of the cut edges. This will allow one to turn the edges of the glass cuts, onto both inner and outer spherical surfaces of each, respectively. The turndown, or turnup, can be approximately one-sixteenth of an inch. This paper tape will function both as a mechanical and a thermal buffer. This about completes the assembly. The fasteners, or the screws used to adjoin the two ring-type large flanges together are spaced at intervals not in excess of 18 degrees. This would total twenty. Too, when engaging these screws they are cautiously tightened by hand only. We are near its conclusion.

The vacuum compartment when the final assembly is made can be evacuated. Thus, the two ring-type large flanges are brought together with great force. At this time, the screws may be tightened. Only a few inch-ounces of torque for each is required. Even hand tightening is sufficient.

Before concluding, a few words on the experimental and the theoretical may be of interest. The entire system of instrumentation involved in the use of this low temperature light integrating sphere includes: The optical instrument being used for the identification and the measurement of the unknown (and known) wave length; a source of radiant energy, both known or/and unknown; a set of aperture discs; liquid fluid refrigerant such as liquid nitrogen; and, an ultra-high and ultra-dry vacuum system[9] with added monitor and metering paraphernalia.

Figure 7
Completed L'TIRIS

The early and fine work of Planck, his Law and his formula for radiation will form the basis for most of the evaluations which will result from the use of this low temperature integrating sphere. The lowering of the temperature during the use of this device could very well result in several confirmations of present-day theories. These results, and the progress that can follow may appear seemingly slow. However, with today's sophisticated computor facilities, the progress can appear in our lifetime.

This paper is concluded with a simple remark: Glass is glass — and, yet though we make it with certain skills, another may break it but, we are quite ready to remake it . . . and better, too!

REFERENCES

(1) Encyclopaedia-Brittanica (1965) ULBRICHT SPHERE. Vol 17, page 844-d.

(2) Count von Rumford. See SIR Benjamin Thompson, 1753-1814.

(3) A.S.G.S. Proceedings, 1971, Milwaukee, Wisc. "Glass Integrating Sphere."

(4) Same as Ref. 3.

(5) Pendulum, or Swing-type Wet Glass Cutting Machine.

(6) General Electric's GLYPTAL. Red; No. 8001. Clear; No. 1202.

(7) The Biggs Company, El Segundo, California. A & B Putty Epoxy.

(8) A.S.G.S. Proceedings, 1973. Denver, Colo. "Thin Film Depositioning."

(9) Unified System Vacuum Pump. =(SIEGBAHN — ECKBERG MOLECULAR PUMP.)
See; Molecular Vacuum Pump, British Patent No. 332,879.
Professor Manne Seigbahn. (Nobel Prize Recipient.)
and; Molecular Vacuum Pump, U.S. PAT. No. 2, 954,157.
E. E. Eckberg. U.S.V. Pump, U.S. PAT. No .3,104,802.

TRAINING AND EXAMINATIONS IN GLASSBLOWING — B.S.S.G.

R. A. L. MASON, *Hon. Secretary, B.S.S.G.*
Royal Holloway College, Egham
Surrey, England

When the British Society of Scientific Glassblowers was founded, it was the wish and the intention of the founder members to establish a qualification process for glassblowers.

After the initial formation of the British Society of Scientific Glassblowers, in the early 1960's, it was decided to form a sub-committee of the main Council to look into and formulate an examination and training process for glassblowers. This sub-committee was entitled the Board of Examiners. At this time there was considered to be a real need for a specific training programme, followed by a formal examination, that could be used by both industrial and educational establishments as a standard —in much the same way as our G.C.E. exams are the accepted standards for academic training. There was already in existence a glass course included in the City and Guilds Technicians Course, and it was the intention of the Board of Examiners that any courses devised by them would form an extension of this basic course. This would cover the needs for those technicians who wished to become more specialised in glass than other aspects covered in the technicians courses. At this time generally, there was a growing cry for certification in all subjects, and the British Society of Scientific Glassblowers felt that they, via their Board of Examiners, could answer this cry.

Members were elected to the Board of Examiners from each of the regional sections of the British Society of Scientific Glassblowers. Whilst no hard and fast requirements were laid down, members of the Society treated the matter with seriousness, and the members elected were all glassblowers of long standing and high repute. Thus the Board of Examiners was born. In the early days of the Board of Examiners, much time was spent in meeting the technical colleges, training boards, industry etc. to find the true requirements for a glassblowing course. The Elementary Course was thus formulated, and a syllabus and training scheme was published. The standard achieved would be on the level of constructing a condenser, although other pieces are obviously included too. The training to the elementary level takes approximately 8 weeks for a full time student. Initially, it was Isleworth Polytechnic, London, that adopted the training schedule and syllabus of the B.S.S.G., but there are now other colleges too that use the scheme.

Following on from the Elementary Course, are Stages I and II respectively. Stage I includes practical work on construction of a filter pump, soxhlet extractor and McLeod gauge. Again, the course for this is approximately 8 weeks, following on from the Elementary Course. Stage II, practically, deals with stop cocks, double bulb splashead etc. Approximately 8 to 10 weeks would be allowed for this Stage.

At all levels to this point, (i.e. Elementary, Stages I and II) the associated theory is taught. The examinations are of a practical type, and the theory is examined by means of an oral examination. No written exams are held at these levels. The examinations are held at intervals during the year, to coincide with courses being run. For the Elementary Examination, one Examiner is present, the Examiner being a member of the Board of Examiners.

For Stages I and II, the Board requires that two examiners must be present at the exam. Certificates of the B.S.S.G. are awarded to the successful candidates.

Following the success of the courses and syllabuses, the Board of Examiners continued in their work of constantly up-dating, revising and extending the courses, and the Lathe I course and syllabus was drawn up. The practical work consists of centrifuging straight joins up to 75mm, parting by melting and thermal shock, forming bulbs up to 50mm diameter, forming re-entrant and conical bulbs and multiple bulb fractionating column, flanges up to 150mm, and setting up of the lathes. This is again usually an 8 to 10 week course.

It is true to say that at all levels, revision of previous work is included and any up-dating processes are carried out.

Following these levels, was the Examination, Certificate of Competence. This was, until very recently, the highest level of examination of the B.S.S.G. This examination course has a much wider range of theory included, and on the practical side, glass to metal seals, making of sinters, glass bellows lathe work and situ work, are all included. At the conclusion of the course (approximately 10 weeks again) practical, written and oral exams are conducted by two examiners.

No student may progress from one level to the next until he has passed the examination at the previous level, unless exempted by the Board of Examiners.

The B.S.S.G. courses and syllabuses are now followed by 4 major colleges around the country, operating full time courses. The course tutors are not employed by the B.S.S.G. but by the college itself. The examinations are carried out at the college where training has been received. Each student pays an examination fee to the B.S.S.G. The times for exams vary from 1 hour for Elementary to 1 day for the Certificate of Competence. The courses, over the past few years have become well attended and the levels of success have been promising.

The marking of examination pieces takes place to a format laid down by the Board, in order to achieve uniformity of marking between all examiners. More recently the B.S.S.G. training schemes and syllabuses have been adopted by the Government Training Centres. These centres are set up by the government for the retraining of older men and women into trades of varying types. Often where people are made redundant, or where firms move from one area of the country to another, leaving many without jobs, the government sends these people to the training centres where they are taught an entirely new trade. One such course is in glassblowing, and the B.S.S.G. have been called upon for help and guidance.

Another new venture for the B.S.S.G. was undertaken in 1971, when the B.S.S.G. was approached for help in the setting up of basic glassblowing training in Malawi. The approach came from the Inter-University Council for Higher Education Overseas, who wanted to run a short course of 8 weeks duration to train selected technicians in elementary glassblowing. Whilst the B.S.S.G. were prepared to assist, they could not undertake to send someone out to Malawi without careful consideration of courses etc. and final details were arranged with I.U.C. to meet the requirements. I was fortunate in going as course tutor and examiner, sponsored by I.U.C. for the 8 week period. As a result of this course, the most promising students were recommended to come to the U.K. to undertake full time courses leading to the higher levels of examination.

The success of the course in Malawi has resulted in the B.S.S.G. in conjunction with I.U.C., operating courses in Nigeria in 1974 (to Elementary level) with another course planned for July/August of this year. This year another Elementary Course will be run, together with the higher levels of examination for those students who passed the elementary course in 1974. The second examiner will be a member of Nigerian origin, who has been trained in this country and who has satisfied the Board of Examiners of his competence to act as an examiner. The demand for courses of this type from other overseas countries, on the B.S.S.G. has increased tremendously over the last year, which alone speaks for their success. The qualifications of the B.S.S.G. are held in very high regard by these overseas countries.

The B.S.S.G. have also assisted in the training of foreign students in the medium of placing them with a member of the B.S.S.G. for a specified period, where they are able to work with the member and so learn the skills of glassblowing. Examinations at the various levels are also arranged for students who have been trained in this way.

So far, mention has only been made of the success of the training programmes and examinations of the B.S.S.G. Like all other societies, members have differing opinions. Many of the older members regard glassblowing as an art—not something which ought to be examined and taught at college—but something that one learns over a period of years, by apprenticeship. Many of these men are very talented glassblowers, superb at their trade, and wish for no more. There are then, on the other side, the younger members, who being brought up in a modern society have seen the need for certification. They, perhaps are more aware of the changes coming about by technological developments, they can see the growing need for a glassblower needing more knowledge than simply glassblowing—the need to be a glass technologist or a glass engineer—this is what industry requires, so this is what they seek. So, the B.S.S.G. could be 'split' into these two definite catagories. This is why, when in 1972, the B.S.S.G. wanted to formulate the Institute of Glass Engineers, members were very divided in their views. Some saw it as the finishing of the B.S.S.G. and some saw it as perhaps a new era for the Society. The main aims of the proposed Institute were really to provide a means whereby glassblowers could, if they wished obtain formal qualifications of a level equivalent to a British degree. After very much discussion, ground-work,

meetings etc. the members were finally invited to vote on whether they wanted to form a further body (in addition to the B.S.S.G.) called the Institute of Glass Engineers, or whether they wished to further the B.S.S.G. itself, by asking the Board of Examiners to draw up very much higher levels of examination.

On voting, members called for the latter of these two suggestions, and the Board of Examiners took on the very onerous task of drawing up such a syllabus.

They realised the very serious nature of the work, and resolved to call on outside experts, if needed, at any stage.

The new level of examination, or grading, is called Master Glassblower. The nature of the examination is theoretical in addition to practical—the main emphasis being placed on the theory. Guide lines have been taken from the courses operated by the new Open Universities—where course units are studied, in various subjects, and a good level of competence has to be achieved in each of the Course Units. Considerable work has been done on preparing the syllabus content for the exams, and in drawing up regulations etc. It has been agreed that the level is to be high, if the qualification is to have the desired meaning, and so members must have a minimum of 12 years experience in glassblowing, before they may apply.

There are 21 course aspects of glassblowing and associated topics, covered in the syllabus, and all material for these courses and draft information guide lines are at present being drawn up. These will, in due course, be published in the B.S.S.G. Journal, and be available in book form for members. It is the intention that the examinations covering these various course units will be held once or twice a year, but for a one year period (from March 1975 to March 1976) present full members of the B.S.S.G. who satisfy the regulations (i.e. 12 years experience) may apply to the Board of Examiners for Master status. The application must be endorsed by two members, and by the member's employer etc. The applicant must give evidence to the Board that he has knowledge of the topics which would normally be examined. The reason for this 12 month period, was to allow some of the older members of the Society who are of the highest repute to achieve what was obviously a deserved title without the formality of taking the courses and the examinations. There is still a lot of work to be done—suitable courses have to be set at various colleges around the country—specialists will have to be enlisted to give certain lectures etc. There are problems—the Board of Examiners however feel that these are not unsurmountable—and they are keen to achieve for members this much higher level of qualification that is so often called for.

As yet, there has been little backing from outside bodies. None from our government—glassblowers are a minority group, and it is hard to get any sort of backing for small groups such as this. Colleges are not terribly quick to adopt new courses unless they know there is a definite need for them.

We still have some members who think there is no need for this higher level—but they still can have their membership of the B.S.S.G.

as it is now—they don't have to apply for Master grade, or to take any exams. The new, younger members however, have everything to gain—the future is theirs. They will be in the unique position of having a profession which calls for a high level of manipulative skill, but for which there will, at last, be an academic qualification which carries degree status. Whether we like it or not, this is a changing world—the emphasis is becoming more and more on paper qualifications, proving, by certification that you are of a set standard. The B.S.S.G. via their Board of Examiners has over the years tried to achieve for its members the status that the glassblower so rightly deserves—and with the formation of the Master qualification—will open many avenues for the glassblowers of the future.

NEW MATERIALS FOR RELIABLE INSTRUMENTATION

Dr. Josef Francel
Chief, Product Development
Owens-Illinois, Inc.
Toledo, Ohio 43666

ABSTRACT

Reliable instrumentation is the basic building block for any effective process control and efficient automated production. New materials technology contributed a great deal to improve the reliability of instrumentation by miniaturizing electronic components. Microelectronics have also reduced weight, cost and power consumption.

This presentation will cover the latest advances in upgrading:

1) Component bonding
2) Interconnections
3) Electrical characteristics
4) Chemical characteristics
5) Thermal characteristics
6) Strength and performance

Particular emphasis will be placed on new advances in Solid Planar Dopant technology.

INTRODUCTION

Reliability of instruments is a measure of one's confidence that the instrument will perform as intended for specified extended period of time. Quantitatively this confidence level is usually established by statistical analyses. These analyses require extensive numbers of samples be tested for long durations of time. The number of samples surviving the specific conditions of the test is related to the total samples in determining the probability of achieving 90%-99% confidence levels of reliability. The failure modes of components not surviving the test are analyzed and then better components, better designs, and improved connections are developed. In the past, a search for new materials and a new system of materials were required to achieve higher reliability of devices. Accordingly, a close working relationship had to be established between material supplier and user early in the system concept phase and had to continue through its life cycle. As a consequence in the field of instrumentation, a new breed of technical teams was organized. These teams include the system designer, the reliability engineer and the materials and process engineer from the staff of suppliers. The key link and the essential interface between good, reliable instruments and the materials technologists is the most essential member of this team — the reliability engineer. He must monitor the activity of evaluation and failure mode analyses, and he must effectively transfer this knowledge to the supplier who in turn is responsible for development of more reliable systems of materials. The most reliable instruments were built in this way.

In my talk today, I intend to illustrate by specific examples how these new teams work and what dramatic improvements in reliability were achieved in this country.

1. THE NICHROME RESISTORS IN SEALED IC DEVICE

The nichrome resistors are nickel chrome alloys similar to heating elements which in this example were deposited on silicon metal with thermally grown silicon dioxide (silica glass). The resistor is protected (passivated) from the attacks of surrounding vapors by a layer of silicon monoxide. The resistors are very thin (100-200Å), 0.5 mil wide and have values of 2 to 6000 ohms. The resistor and integrated circuit (IC) are sealed by glass in an alumina standard package (DIP — Dual In Line Package). This package is used for very high reliability critical equipment. It is also known under the term HERMETIC, high reliability with standard designation HiRel.

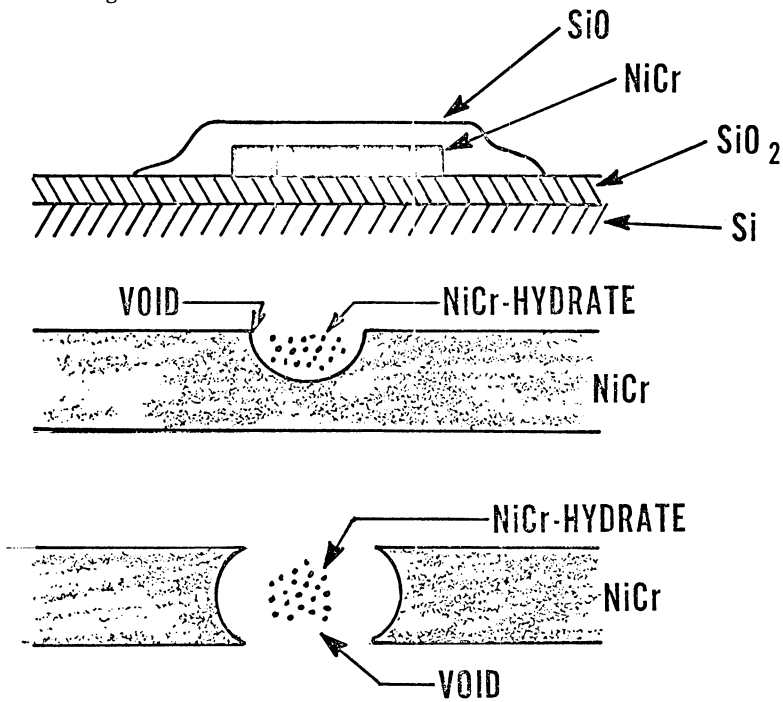


Figure 1
Nichrome Resistor

In testing many devices, an unexpected statistically significant number of failures occurred despite the application of the best protective technique — the best package and the best protective coating. The reliability engineer requested screening of the devices by high-temperature treatment under load (current applied). This procedure in the past used to

eliminate the "bad" devices. However, in this case another unexpected significant failure occurred even after "burn in." Then the reliability engineer examined very closely the source and the location of failure origin. He found small voids in the thin film of nichrome resistor. These voids were arc of circles with the middle portion of some new material. The reliability engineer requested identification of this new strange phase — by all modern techniques (Scanning electron microscope, electron microprobe, ion microprobe, electron diffraction, gas chromatograph). The new phase was nickel chrome hydrate. How can any hydrate (bound water) be formed in an isolated hermetic package? This was the most important question. The mechanism (method) of hydrate formation had to be understood before any corrective action could be initiated. Many experiments were carried out by device and reliability engineers, and finally the answer was pinpointed. The hydration was due to condensation of small amounts (picograms) of atmospheric moisture sealed into the package during final closing step of the base and lid. Based on these findings several obvious solutions were naturally considered: sealing in dry atmosphere, purging furnaces by super-dry air. These procedures were rejected because they could not guarantee elimination of moisture from the protective coatings. At this stage the material engineer entered the problem solving team and developed a super desiccant and also creatively defined the process of application of this new material.

At first the basic solution of obtaining a desiccating (drying) action inside a small cavity appears to be almost trivial. However, when all of the constraints of package size and long-term reliability (zero defects) were considered it was found to be a challenging problem for a creative material problem solver. For example, the mechanical testing alone called for gas exposure, ultrasonic vibration, exposure to the environment of a flash X-Ray machine, and scribing by a sapphire needle point. The material which passed literally hundreds of tests was PolyAl™ — trademark for a material developed by Owens-Illinois. This material can be described as a colloidal suspension of extremely fine alumina particles. The material when properly applied to a flat alumina lid of an IC package, gives (yields) a deposit of porous aluminum oxide that is both very tightly bonded (chemically and mechanically) and a very active desiccant. Furthermore, PolyAl™ properly applied does not show signs of cracking and delaminating. The alumina lid has to be broken first (strength test) before the desiccant film would show any signs of delamination. In addition, thermal shock from 65-175°C had no effect on any of the samples. This material will adsorb moisture quickly — in a few minutes. In hundreds of thousands of hours of testing many components performed in different locations, no nichrome failures have been reported in a hermetic package containing properly activated PolyAl™ desiccant. This type of reliability was achieved through coordinated efforts of a team of designer, reliability engineer and material supplier engineer.

2. REACTIVELY BONDED METALLIZATION

Reliable components of instruments use the most reliable conductors. Presently the noble metal base conductors have a high level of confidence with extended periods under continuous use, and under adverse environ-

mental conditions. As in the previous case (No. 1) and also in this second case the substantial advances were accomplished by a team designer, reliability engineer and materials supplier scientist.

Adhesion of a gold conductor to the substrate was achieved in the past by selective additions of special glasses. The vitreous part of the conductor system penetrated the alumina surfaces — forming relatively good interfaces. The problem was centered more on the opposite side of the gold conductor region, which is used for wire, beam lead and solder bonding. Too much glass caused localized bonding interrupted by islands of no bonds. The processing of gold conductors had to be controlled to a degree which in manufacturing can be achieved only with difficulty and excessive appropriations. Supplier scientists analyzed this conductor bonding problem and developed a new generation of conductors. These conductors are known in the electronic industry under several designations:

Au99+™
Pt99+™
Fritless Gold
Reactively Bonded Metallizations

These terms explicitly indicate the main attributes of these materials. The bonding to substrates is based on the reaction of additives with the alumina to form a copper aluminate spinel. The elimination of frit from these conductors offers the following significant advantages:

1. Denser films
2. Improved bonding
3. Improved soldering
4. Improved wire and beam lead bonding
5. High thermal dissipation
6. Sharp line boundaries

Although all of these contribute to the higher reliability, the improved wire bonding can be selected to illustrate the impact of new materials on reliable instrumentation.

The printed conductors are connected to the active devices by wires. The strength of this wire bond is obviously essential and without it the

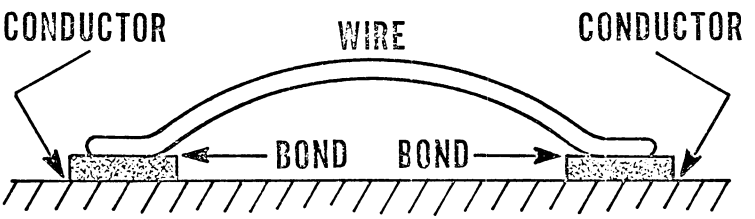


Figure 2
Gold Conductor with Wire Bond

electronic device will have many inactive components. Typical values of average bond-strength for old metallization usually do not exceed ten pounds. The bond strength of Au99+ is increased by a factor of 2 to 5 over similar gold with frit. The thermocompression bonding (heat and pressure) is also significantly improved with high reproducibility and with less sensitivity to thickness and firing temperature variations.

The present new electronic technology could not achieve the high levels of reliability of devices without the new conductor materials from Electro Oxide Corporation and Owens-Illinois. This development of improved material is a continuous activity from which all users will benefit.

3. ADVANCES IN SOLID PLANAR DOPANT TECHNOLOGY

The third and last case of our discussion will describe innovations related directly to the basic building block of the solid state technology. Silicon with p- or n-type regions are manufactured by diffusion of boron or phosphorus. The boron diffused into silicon produces p-type semiconductor and phosphorus in silicon gives n-type semiconductor. The silicon thin plate, or wafer, rests on a quartz boat which is sealed into a tube. The boron sources can be gases, liquids and solids. The boron diffusion is carried out at temperatures from 900-1200°C.

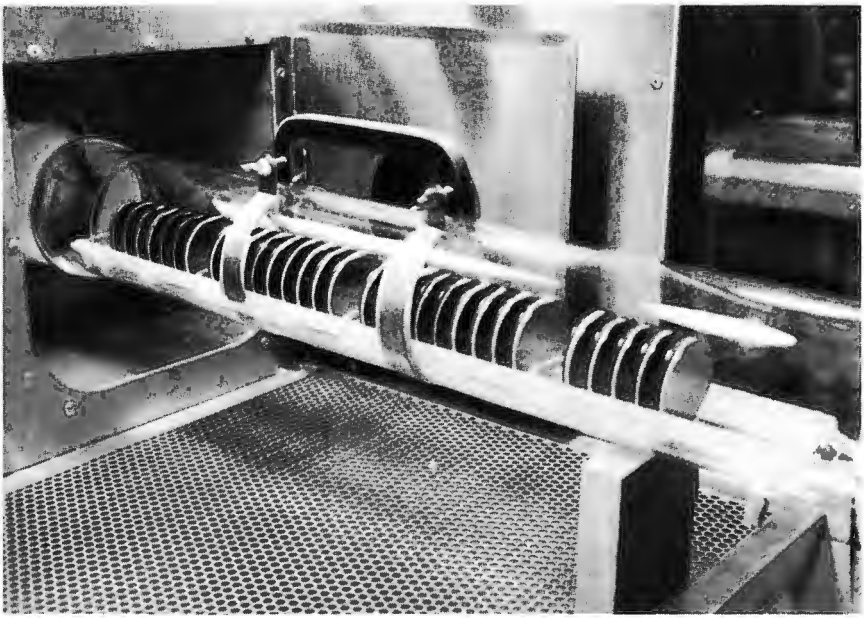


Figure 3
Tube with Silicon and Dopant

Solid planar diffusion sources have been used in the semiconductor industry since 1970. The main reason for solid dopants is the never-

ending increase of the diameters of silicon wafers. The most important contribution of solid dopants is their ability to produce a uniform concentration of boron across the 2-inch to 3½-inch diameters used today and 4-inch to 6-inch diameters required for solar cells of tomorrow. The uniform concentration in turn is closely related to the reliability of the final active device which is the basic block of the modern instrumentation. The solid planar dopants of yesterday required a stabilization cycle to minimize the adverse effects of the high initial volatilization rate. Furthermore another cycle was frequently needed to assure reliability of these sources. This cycle is known as the reactivation cycle — which is, as the name suggests, a step to activate the dopant to be effective by etching away the depleted layers of boron. The theoretical lift of many hundreds of hours is reduced by a significant factor because of the activation, stabilization and etching cycles. Furthermore, these cycles are also expensive because production time is lost. The circuit designer and reliability engineer here again challenged the material scientist to develop a new material free of the limitations of the solid dopant.

A new high-purity planar dopant source has been developed by Owens-Illinois and tested by ITT. It is marketed by Electro Oxide Corporation, Palm Beach Gardens, Florida, under the trademark BORON+™, and it is presently being evaluated by all the leading semiconductor producers in the U. S. and abroad.

BORON+™ is a glass-ceramic [with boron present in its oxidized state (B_2O_3)] made of high-purity raw materials. BORON+™ needs no periodic reactivation and virtually does not contain any alkalies and heavy metals. The active life is now approaching thousands of hours. The variations of deposition of boron are less than 2.5%. The uniformity of boron diffusion across the surface is excellent. This high uniformity is a manifestation of the excellent distribution of boron in the BORON+™ solid dopant.

Both the circuit designer and the reliability engineer require high control in manufacturing sophisticated IC components. BORON+™ developed by glass materials engineers provides the proper source to make devices more exactly alike and whose characteristics can be predicted more accurately. Once we can make reliable active devices, the path to the reliable instrumentation is clear of many obstacles.

CONCLUSIONS

These three contributions of a team (designer, reliability engineer, material scientist) are illustrations of how high reliability devices were made and how a high level of confidence in expected long-term function of the systems is being obtained for the users sitting in this audience.

REFERENCES

1. Scott A. Bonis, *Drying Up A Semiconductor Package,* Electronic Packaging and Production, Vol. 14, No. 2, Pgs. 46-56, 1974.
2. B. R. Smith and R. L. Dietz, *An Innovation in Gold Paste,* Proceedings 1972, International Microelectronic Symposium, 2A-5-1, 1972.
3. J. J. Steslow, J. E. Rapp and P. L. White, *Advances in Solid Planar Dopant Sources for Silicon,* Solid StateTechnology, Pgs. 31-34, January 1975.

FABRICATION OF SINGLE-FILAMENT, TUNGSTEN-HALOGEN BULBS

ARTHUR DOLENGA AND JOHN C. HILL
Research Laboratories
General Motors Corporation
Warren, Michigan 48090

ABSTRACT

Tungsten-halogen bulbs are currently used in projection, studio, and floodlighting applications. Their greater light output makes them attractive for other applications such as automotive lighting.

We have made tungsten-halogen bulbs from quartz and other materials for which the type, geometry, preparation, cutting, and cleaning techniques are important. Molybdenum-foil current leads are sealed into the quartz envelope on a vertical machine using nitrogen or forming gas (85% nitrogen and 15% hydrogen) as cover gases.

The bulbs require special processing prior to filling with a halogen compound and either argon or krypton. The final room temperature pressure of about six atmospheres requires a calibrated volume and liquid nitrogen immersion. Sealing off must be done with proper safety precautions.

Testing yields performance figures in terms of life and interesting failure modes which have been observed due to water cycling, metallic impurities, and insufficient or excessive halogen.

A short movie will be used to illustrate the fabrication, processing, and testing sequences described in the paper.

INTRODUCTION

The first patent information describing tungsten-halogen bulbs was published about 16 years ago.[1] Since that time many advances have been made in this technological area. Bulb efficiency, life, and reliability have been greatly improved. In fact, double-filament tungsten-halogen bulbs which were previously thought to be impracticable have been on the market for several years.

A typical incandescent bulb has a large glass surface area and a gas fill at about atmospheric pressure or slightly below. Tungsten losses occur through filament evaporation. The tungsten atoms move to other adjacent parts of the filament or they collide with gas molecules and then either return to the filament or move away from it and deposit on the bulb wall. This wall deposit of tungsten absorbs light, and hence limits the minimum size of a normal incandescent bulb. The gradual loss of tungsten from the filament also causes thinning of the filament, and failure of these thinned segments normally is the ultimate cause of bulb failure.

If a halogen is added to the gas fill and if certain bulb wall temperature conditions are met (450 to 900°C),[2] a regenerative cycle is estab-

lished which prevents tungsten deposition on the bulb wall. An illustration of such a regenerative cycle is shown in Fig. 1. As tungsten evaporates from the filament it reacts in the vapor phase with the halogen to form volatile tungsten halides.[3,4] In the very high temperature regions near the filament the tungsten halides decompose, and the tungsten returns to the filament and releases halogen for further regenerative reactions. The returning tungsten deposits randomly onto the filament. With diffusion and redistribution of tungsten occurring as in a typical incandescent bulb,[5] there is no increase in life which can be attributed to the regenerative cycle; its prime purpose is to prevent bulb blackening.

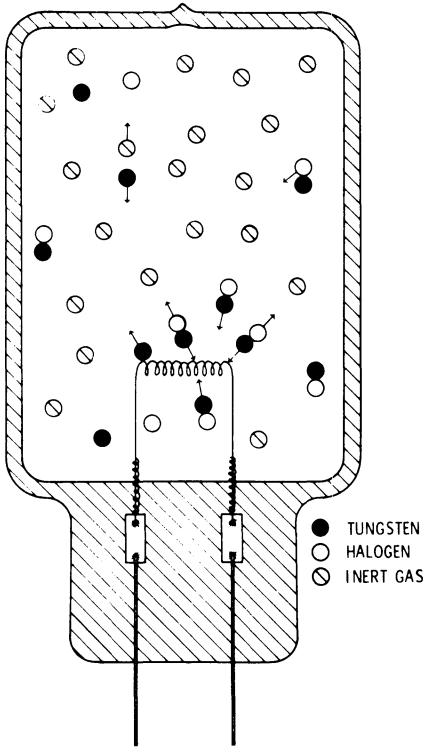


Figure 1
Simplified representation of tungsten-halogen regenerative cycle

Inasmuch as there is no blackening, this regenerative cycle permits the use of smaller bulbs which are able to withstand higher pressures. The room temperature fill pressure can be increased to at least six atmospheres. We estimate that there is at least a further, three-fold increase in pressure at normal bulb operating temperatures. This increased pressure (primarily of inert gas) retards the rate at which tungsten evaporates from the filament (Fig. 1). This in turn retards the rate of thinning and

therefore lengthens life. It has been found that bulb life is proportional to operating pressure,[6] at a given filament operating temperature. This potential for increased life is a major advantage of tungsten-halogen bulbs.

These bulbs were investigated at our laboratory using equipment and techniques which would be similar to commercial production practice. A description of the resulting quartz bulbs with a tungsten-halogen regenerative cycle is presented.

ENVELOPE FABRICATION

I. *Glass*

Since the bulb wall temperature must be fairly high for the regenerative cycle to function, the glass must be a high-softening-point material such as fused silica, fused quartz, or Vycor. Low hydroxyl, alkali, and metallic impurity levels are also required to avoid disruption of the halogen cycle. Metallic impurities are especially deleterious because they can combine with the halogen to form stable halides which deposit on the bulb wall. This phenomenon can remove enough halogen from the system to disrupt the regenerative cycle. Surface deposits can also promote devitrification, which can diffuse the light from the filament and lead to envelope fracture. For the best combination of properties, we chose General Electric Type 204 clear fused quartz tubing.

The bulb envelope was 10 mm O.D. with a 1 mm wall thickness, and the tubulation was 4 mm O.D. with a 0.5 mm wall thickness. The tubulations were hand cut to 145 cm using a glass scoring knife, while the envelope cylinders were cut to size (27 mm) on a Dyna-Cut glass slicing machine using a Wale (S-120-SR) rubber bonded silicon carbide cut-off wheel. The sealing of the tubulation to the envelope was done as a lathe operation or by using either of two custom built tungsten-halogen bulb making machines. Both of these machines were made to our specifications. The original was made by Kahle (Fig. 2); the other was made by Yorktown [7] (Fig. 3).

Once the tubulation seals were made, the envelopes were rinsed in an Alconox solution followed by two rinses in distilled water. All three rinses were in an ultrasonic tank at a temperature of about 55°C. The envelopes were dried in an oven at 200°C and packed in dust-free boxes to minimize contamination before further processing.

II. *Electrical Components*

A tungsten filament, tungsten slip-coils, molybdenum current leads, and molybdenum foils for connecting the filament and current leads form the electrical components as shown in Fig. 4a. The filament design depends on intended bulb usage and will not be discussed here. The hairpin shape of the molybdenum current leads helps to stabilize them in the weld-pocket during the press-seal operation described below. The shape of the molybdenum foil is the most interesting feature for the glassblower. Its thin, lenticular geometry allows formation of a vacuum-tight seal with quartz glass even though molybdenum and quartz have a coefficient of expansion difference of 43 x 10^{-7} cm/cm°C. The resulting seal is similar

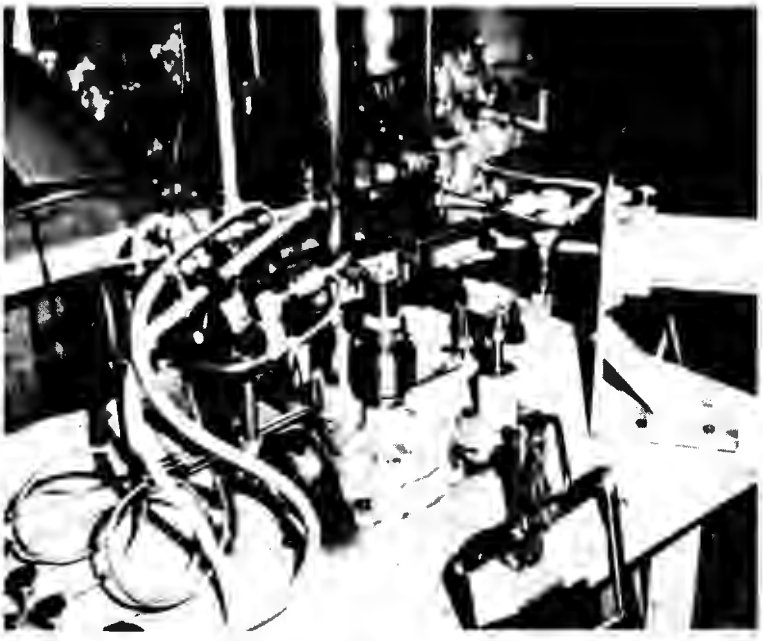


Figure 2
Photograph of Kahle machine

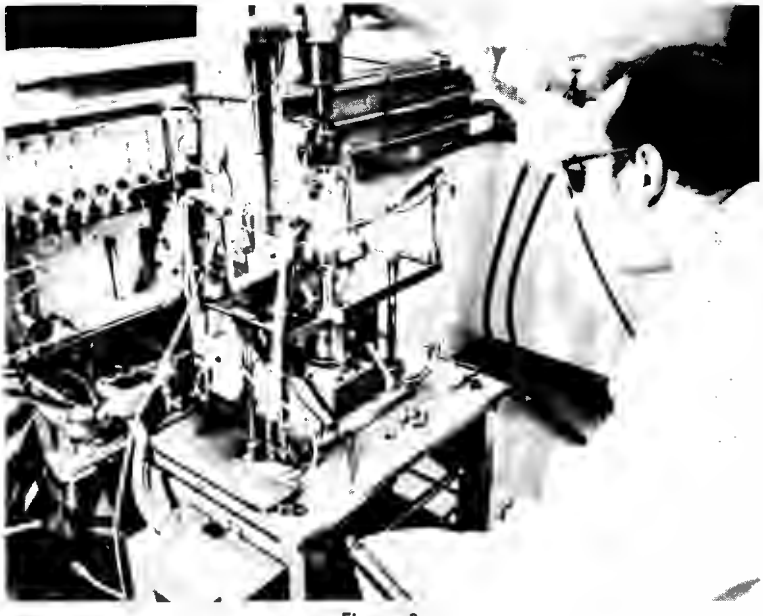


Figure 3
Photograph of Yorktown machine

to a feathered edge Housekeeper seal between various glasses and copper or stainless steel. The filament (with the slip-coils on its legs) and current leads are welded to the molybdenum foils using platinum-plated tabs as fluxing agents (Fig. 4b).

The completed lead-sets are then cleaned by boiling gently for 2 to 3 min. in a KOH solution (200 g/litre) followed by several water rinses to clean the tungsten. Soaking for 2 to 3 min. in a HCl solution (1 + 1), likewise followed by several water rinses, cleans the molybdenum foils. After a rinse in methanol the lead-sets are allowed to dry. As an alternative to this liquid chemical cleaning the lead-sets can be fired in a dry hydrogen atmosphere at 1100°C for about 20 min. A high flow rate of hydrogen (at least 1 m^3/hr) is recommended.

III. *Press-Seals*

Inasmuch as the halogen cycle can be severely disrupted by impurities, the formation of a vacuum-tight press-seal between the quartz envelope and lead-sets is critical. Both the Kahle and Yorktown machines were used to make satisfactory press-seals. Both machines are vertical lathe units with chucks driven by a spline bar. The quartz envelope is clamped in the upper chuck and the lead-set is clamped in the lower chuck. On the Kahle machine, the chucks stopped before the press-seal was made by hydraulically-driven dies. On the Yorktown machine, the dies rotated with the chucks so that pressing could be initiated without the time lag associated with stopping. It is important that pressing starts while the quartz is sufficiently hot and soft enough to flow completely around the molybdenum foil. At the same time excessive heating can cause oxidation of the foil and result in a leak between the foil and the quartz. Therefore the Yorktown machine usually produced a higher yield of satisfactory press-seals. In both cases the die closure was activated when the quartz was at the proper temperature.

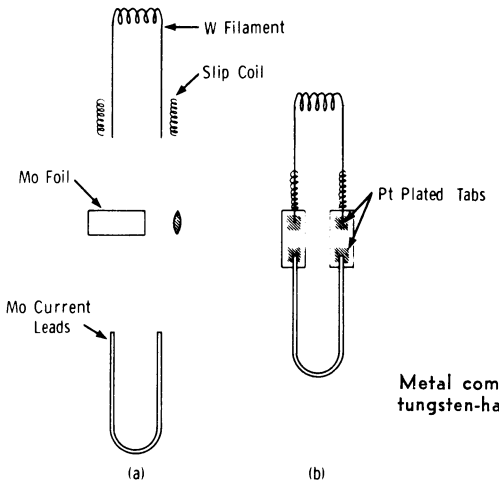


Figure 4
Metal components (a) and assembly (b) of a tungsten-halogen bulb

In order to minimize oxidation, a protective flow of cover gas was introduced from above through the envelope tubulation (Fig. 5). Both nitrogen and forming gas (85% nitrogen and 15% hydrogen) prevented any observable oxidation provided there was no turbulence or any other disruption of the flow around the lead-set. A tapered weld-pocket (Fig. 5) helped to maintain a smooth gas flow and prevent mixing of air with the cover gas.

The design of the dies which make the press-seal is critical. The need for complete flow of quartz around the molybdenum foils has been mentioned above. This can be accomplished if there is some extrusion of the soft quartz beyond the ends of the dies; however, too much extrusion causes torn foils. We have found that the cross-sectional area of the opening between the closed dies should be approximately 90% of the cross-sectional area of the quartz tubing. Another hazard is leaving an inner "V" at the top of the press-seal area (Fig. 6a). Such a V results in catastrophic failure (by explosion) of bulbs pressurized to six atmospheres. Rupture is believed to initiate at the V. This can be prevented by having sufficient upward extrusion to eliminate the V (Fig. 6b). The cross section of a die design which provides the proper amount of extrusion is shown in Fig. 7.

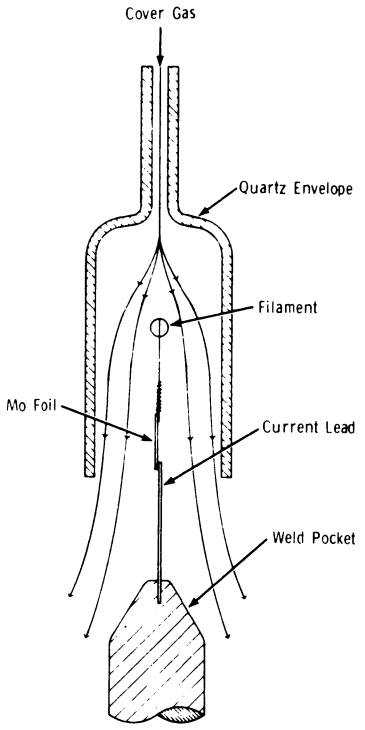


Figure 5
Cover gas flow around metal components

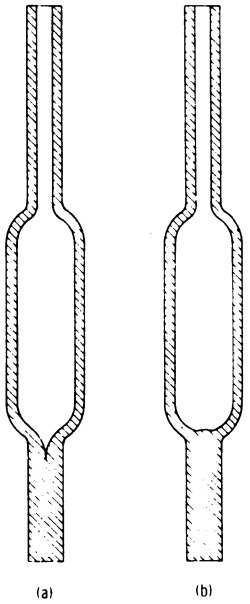


Figure 6
Improper (a) and proper (b) cross-sections of press-seal area

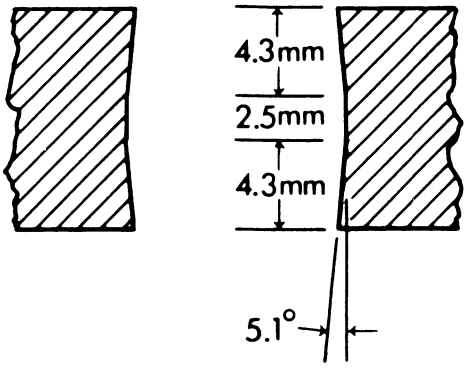


Figure 7
Cross section of dies for the press-seal

FINAL PROCESSING

The standard manufacturing procedures for incandescent light bulbs are not adequate for tungsten-halogen bulbs. The amounts of impurities left in standard bulbs interfere with the halogen cycle. Oxygen is particularly bad since it combines with hydrogen liberated from halogenated hydrocarbons (typically used to supply the halogen) to form water, which drastically shortens bulb life via the well known water-cycle. Therefore, we modified the standard procedure as described below.

It is common to heat the filament to incandescence (this is called flashing) in the presence of forming gas to reduce the oxides normally on the surface of the tungsten. This forms water in the vapor phase which is subsequently removed by evacuation. This type of processing is not completely satisfactory inasmuch as some of the oxides and other contaminants are redeposited on the cooler bulb wall rather than being removed by the evacuation. If a vacuum outgassing is used to remove envelope contaminants after flashing, this will still allow some redeposition of contaminants on the filament. We found that separate cleaning of the filament and the bulb is insufficient to completely remove all of the various contaminants. Therefore, we developed an improved procedure,[8] whereby flashing and outgassing are performed simultaneously. The processing manifold on which the bulbs are mounted (Fig. 8) is filled with forming gas; the valve shown in Fig. 9 is closed to isolate each bulb. The remainder of the manifold is then evacuated. The wall of the first bulb is heated by torch to just below its softening point. Then the filament is flashed at 6V AC in a cycle of 3 sec. on, 3 sec. off, and 3 sec. on, while maintaining the heating by torch. The valve to the bulb is opened while the wall is kept hot by the torch for another few seconds until evacuation is complete. The process is repeated for the other bulbs on the manifold. The entire procedure is then repeated at 9, 12, and 14 V to remove contaminants sequentially.

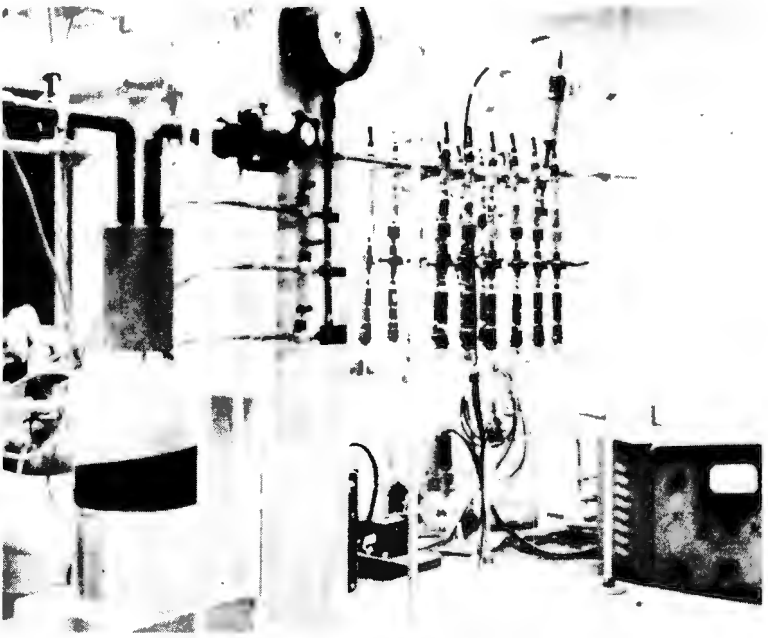


Figure 8
Photograph of processing manifold

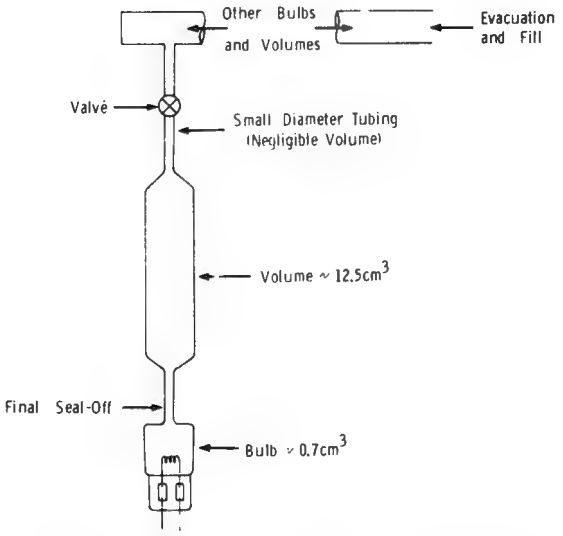


Figure 9
Metered volume system for controlling final fill pressure

The method described in the previous paragraph has several advantages. The oxides and other contaminants normally removed from the filament during flashing are not allowed to migrate to the bulb wall since it is sufficiently hot to prevent adsorption or absorption. The contaminants are immediately removed by evacuation leaving both the filament and interior bulb surfaces clean. We found that by using these techniques a considerable increase in bulb life was achieved over commercial halogen bulbs. At this stage the bulbs are ready for final filling with a mixture of halogenated hydrocarbon and inert gas. We found that the best results (longest life) were obtained with krypton containing 0.05% by volume methylene bromide (CH_2Br_2). The final fill pressure of six atmospheres was achieved in two steps. One atmosphere of the fill gas was admitted to each bulb and its attached metered volume (Fig. 9), then a valve was closed to isolate each bulb and its volume from the processing manifold. The metered volume was carefully determined so that the amount of fill gas in it and the attached bulb would create a pressure of six atmospheres if contained in the bulb alone. Each bulb was immersed in liquid nitrogen for 45 sec. to condense all of the fill gas into the bulb. The bulb was then sealed off from its tubulation with a Tescom Little-Torch with a number 5 cross-fire tip. A delicate torch adjustment was required to permit the tubulation seal-off to be made close to the boiling liquid nitrogen. After warming to room temperature the final pressure was consistently 6 ± 0.5 atm. The bulbs were then complete and ready for life testing.

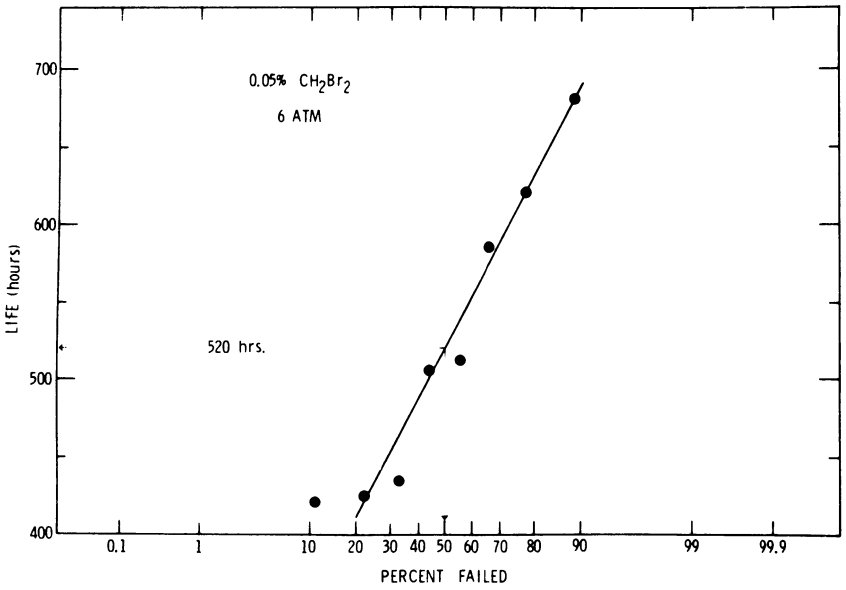


Figure 10
Weibull plot of life vs. percent failed

LIFE TEST RESULTS

The bulbs were mounted on a test stand and operated at 14.00 ± 0.05 V DC, which produced a filament temperature of 3250°K. A timer in a series circuit with each bulb automatically recorded the number of hours to failure. Although average life is the generally accepted criterion of bulb quality, significant information on failure modes and remedies was obtained during the development phase of this work. This information is summarized in Table 1. Only those bulbs which remained clear with essentially unreduced light output to end-of-life were considered acceptable.

For a given batch of bulbs, life is conveniently determined by plotting time-to-failure on a Weibull plot as shown in Fig. 10. A straight line is then drawn through the data points as shown. The intersection of the 50% failure point with this line is then the rated life. In the example shown the life was 520 h.

This life figure is the best we achieved. To put it in perspective, the life of commercial bulbs tested the same way by us averaged about 250 h. The processing described above thus resulted in an approximate doubling of tungsten-halogen bulb life.

TABLE 1

FAILURE DATA FOR TUNGSTEN-HALOGEN LIGHT BULBS

Failure Mode	Causes	Corrections
Filament burnout	H_2O cycle	Reduce hydroxyl content of quartz
(early, in coil region)		Reduce oxidation of metal parts
		Check purity of fill gases
Filament burnout (early, on leg)	Excessive halogen	Reduce halogen content
Blackening of envelope	Insufficient halogen	Increase halogen content
	Halogen depletion by impurities	Check purity of all components
	H_2O cycle	See above
Cloudy envelope	Air leak	Improve press seal
		Check Mo foil temperature
	Devitrification	Clean bulb exterior before operating
		Check for improper handling

CONCLUSIONS

It was possible to double the life of tungsten-halogen light bulbs by giving careful attention to processing. In addition to choosing components with minimal inherent impurities, extensive cleaning procedures were used to maintain cleanliness throughout fabrication. Protection of metallic parts by cover gases during the press-seal operation is mandatory. Simultaneous flashing of filaments and outgassing of envelopes further reduces impurity levels. With the proper fill gas and operating pressure, the life of tungsten-halogen bulbs can exceed 500 h.

ACKNOWLEDGMENTS

The authors would like to thank Joseph Palazzolo, George Rice, and John Price for their technical support and Richard Dusman for the die design. We would also like to thank Tom Persing and Gale Craig for their numerous consultations.

REFERENCES

1. E. G. Fridrich and E. H. Wiley, "Electric Incandescent Lamp", U. S. Patent 2,883,571 (1959).
2. J. R. Coaton, *Proc. Inst. Elec. Engrs.* (London) *117,* 1953 (1970).
3. L. N. Yannopoulos and A. Pebler, *J. Appl. Phys. 42,* 858 (1971).
4. L. N. Yannopoulos and A. Pebler, *J. Appl. Phys. 43,* 2435 (1972).
5. H. Horster *et al, Phil. Tech. Rev. 32,* 155 (1971).
6. B. A. Moys, *Trans. Illum. Eng. Soc.* (London) *29,* 13 (1964).
7. Yorktown Tool and Die Co., Yorktown, Indiana 47396.
8. A. Dolenga and J. C. Hill, "Improved Processing of Tungsten-Halogen Light Bulbs", U. S. Patent Application S.N. 391,883.

SEALING SAPPHIRE TO GLASS

M. Howe Smith
Technicon Instruments Corporation
511 Benedict Avenue
Tarrytown, New York 10591

In this paper I will attempt to bring to light various methods of sealing sapphire to glass and some of the various glasses we have been successful in using.

Sapphire is a material that is used in many ways. With the invention of micro miniature parts, it serves well as substrates, tubular hose connection, infrared windows, etc. Sapphire is now being made in various sizes and precision shapes. A new process developed by Tyco's Saphikon Division of Tyko Laboratories, Inc., Waltham, Massachusetts, has made it possible to obtain sapphire tubes, rods, and plates grown to very close tolerances.

Sapphire is unlike glass in that it is a single crystal material having a melting point of 2053°C (3727°F). The expansion varies according to axis crystal is grown or cut. Without going into a long discussion or explanation of expansion at various temperature ranges, we will take a nominal of 56 x 10^{-7} to 65 x 10^{-7}. We find that parallel to C axis we have largest expansion, 60° to C axis next and perpendicular to C axis lowest expansion.

We had a problem, which most would not have when sealing glass to sapphire. Our problem was to find a glass that was resistant to chemical attack. In our systems we run anything from Sodium Hydrx. to Nitric Acid, and many reagents. We were not looking for a vacuum-tight seal, but one that was tight to leakage of fluids.

We checked the list of best known glasses from Corning and Kimble, where expansions were closest to that of sapphire. Corning's we used 7740 as a master against:

	7800
	7280
	7052
	7520
Kimble	1N-3
	N-51
	EN-1
	KG-33 as master

Tests were made by weight loss over a certain time lapse, using various reagents and acids at elevated temperatures to speed results. We finally decided on Kimble 1N-3 as having the best chemical resistance with an expansion in the range we were looking for of 56 x 10^{-7}. The one problem encountered with 1N-3 was that it is only in melt once every year or two depending on orders.

As background to some of our work, we have made seals of Corning's 7800-707 to Pyrex, and 7280 Boron free glass. Kimble's glasses of N-51 and 1N-3 were used both of which are used in our small tube sapphire seals.

In the making of seals to small sapphire tubes, sizes .060 O.D. x .040 I.D. and smaller, we found sapphires had to be clean. Trichroethylene was used as a degreaser and methanol as a final rinse, and air dry. We also have used Freon as a final cleaning agent. To accomplish our cleaning, we put parts in beaker with cleaning fluid and float in our small ultrasonic cleaning unit.

Sapphires tubes must not have any sharp edges. Therefore, edges must be mechanically radius or flame polished. Care must be used in flame polishing sapphire, as sapphire melts and does not soften as glass. Too much heat will cause white crystals to appear, which would be a cause for a cracked finished seal.

In sealing sapphire tubing to glass tubing, Figure 1, straight in end seal will be the hardest to perfect. Sapphire tubing should be closed off on end, so one might blow into glass tubing, working glass in well around sapphire using a good amount of heat. We used a Tescom torch with hydrogen and oxygen, although gas oxygen could be used with a larger tip size. The side seal should be worked in well also, although it is not as critical as an end seal. Most of our holes are drilled into glass tubing as our operation requires a sharp transition from sapphire to glass with bore remaining straight.

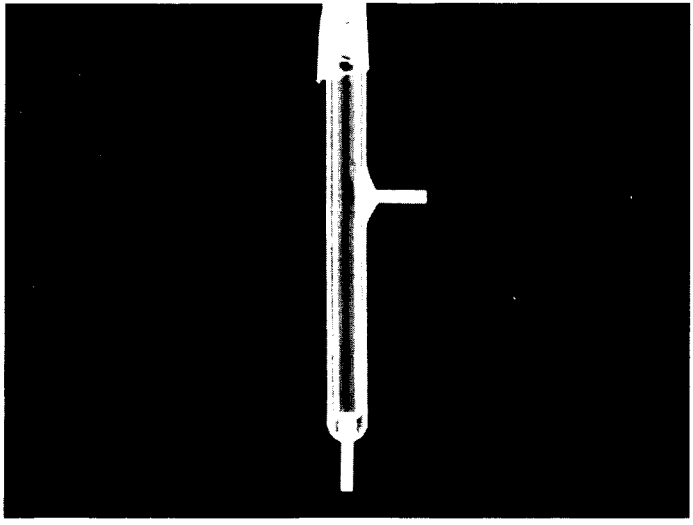


Figure 1
Sapphire Tube Seal

Another method we have found that makes a very good seal and stands up to our acids and reagents is a structural adhesive put out by the 3M Company. Two types we have tested, and are now in use are: #2186, which cures well by heating at 360°F for ½ hour, color white; and #2214, which cures at 250°F for 40 minutes, color metallic gray. Both are very fine adhesives. We have never been able to break a bond after it has been cured, but have broken the glass. In using the adhesive, one has to be careful not to use too much, as too much build-up causes high stresses in the glass on each side of seal, and could cause breakage. Parts have been tested under vacuum and have been leak-free to 10^{-11}. This testing was done by Ted Bolan of Philip Laboratories.

Another process of sealing sapphire tubes in small parts is by what we call our monolithic process. This we have developed and is patented by Technicon. This is a process whereby we mold all parts into a glass block or tube, using 1N3 glass, after which all core material is leached out by acid leaving perfect configuration of channels and openings wanted.

Figures 2 and 3 are some examples of our monolithic process, bore size of which are .5mm to 1 mm in diameter. We also work with sizes down to .25mm, Figure 4.

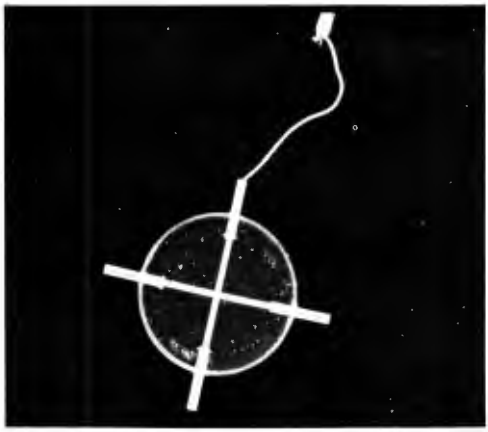


Figure 2
Sapphire Tube Seal
Monolithic

Figure 3 — Sapphire Tube Seal — Monolithic

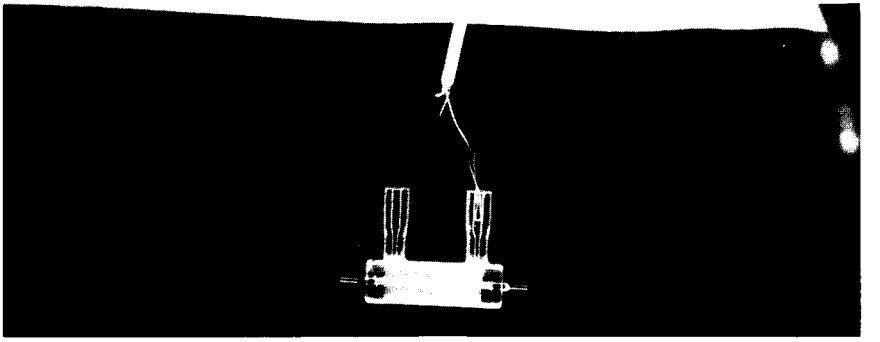


Figure 4 — Sapphire Rod Seals

Methods of sealing windows are different than what I have just discussed. Windows of sapphire are usually sealed by the induction method. That is, transferring of heat from a metal or carbon body into the sapphire and then to the glass that is to be sealed to the sapphire. Glasses used are 7520 and 7052 Corning.

Tube to be sealed to window is ground square on one end and polished so surface is flat with good sharp edges. The better the surface finish, the better the seal will wet with minimum amount of heat applied. If no polishing equipment is available a good fine ground finish will work. End should be cleaned by a slight etch in dilute H.F., rinsed in distilled water, and a final rinse in Methanol.

Glass tube to be sealed to sapphire should be at least .020" smaller than O.D. of sapphire window, so that during sealing operation glass will not flow over edge of window. If the seal flows over edge of sapphire window it could cause part to crack.

One method that can be used with a glass lathe is to make a vacuum holder per sketch (Figure 5). This should be made from stainless steel or nichrome. A ring burner is used behind flange to heat holder, while holding sapphire window in place by a vacuum being drawn thru tube. Chuck tube to be sealed to sapphire window in tail stock of lathe.

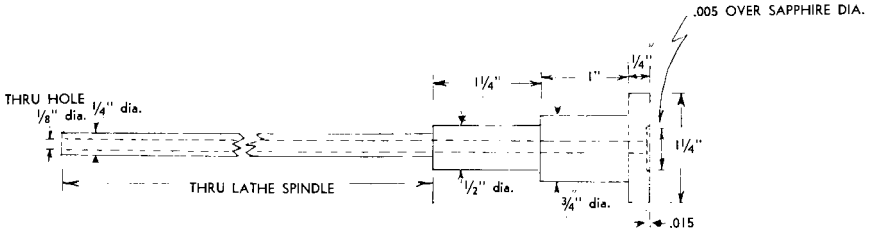


Figure 5 — Sapphire Window — Lathe Fixture — Nichrome or Stainless Steel

Bring tube to be sealed in close to sapphire, heat holder to a light red, bring in glass until it touches sapphire, applying light pressure until you see a wetting of glass and sapphire all the way around the tube. Allow to soak with heat on without further pressure until a seal without a re-entrant is cbtained, bring temperature down slowly. When color has gone from holder, take piece from holder and put into pre-heated furnace. If many parts are to be done, hold furnace just above strain point of the glass. When parts are completed, run furnace to annealing temperature allowing to soak for a suitable time according to thickness of material used. Windows to ½" in diameter may be sealed by this method. Another method of sealing is by R.F. using a nichrome or carbon holder, with slight recess for sapphire to fit into. Set holder on a non-conductive block such as transite. Assembly should then be set on a lab jack or another means of adjusting vertical height of sapphire window and glass tube in R.F. coil. Coil diameter and number of turns needed to do the job should be determined by the size of generator being used. Sealing is accomplished in same method as previously reviewed, except use a small weight on top of glass tube to give downward pressure until seal is accomplished. Take weight off and soak part as in lathe seal.

For the most part, time and temperature is a matter of the individual R.F. unit one is using. I have made sapphire window seals to 3" diameter using this method.

Sapphire, as a good source of U.V. transmission for windows and with the development of sapphire tubing a very strong material, resistant to abrasion and acid attack. I am sure with the invention of other glasses that are compatible with sapphire, we will see more uses for sapphire and glass combinations in our daily work.

Therefore, we will be challenged to find other methods of sealing it into glasses. I hope that this paper will have stimulated some new ideas that might help you solve some of the problems you might encounter in the use of sapphire and glass.

DATING THE TORCH WITH RESPECT TO THE EARLIEST SIGNIFICANT PIECE

RAYMOND J. KELLER

Glassland
Whitehouse, New Jersey

The title of the paper is "DATING THE TORCH WITH RESPECT TO THE EARLIEST SIGNIFICANT PIECE". The purpose is to establish the history of the lamp. There is much evidence to support the idea of early lampworking.

In this context, lampworking implies the use of a torch, and involves the use of glass rods and tubes. Lampworking is not presently defined in dictionaries, and its history is either vague or for the most part nonexistant.

If we consider how far back torches, lamps, or flames with a degree of regulation may go, we can start with the Egyptians and early metalworkers of the prechristian era, when fine filigree in Gold, Silver, and other metals was accomplished. The Egyptians worked in both glass and metal, and the humble beginning of a torch or lampworking may have even preceded them.

To better understand history in general, and the cultures which produced early work, lampworking should be defined, and its use established wherever and whenever it existed. Our study has centered on the goblet because it exhibits the greatest degree of lampworking and therefore may be the most significant of any early work. Our reproduction was made entirely on the lamp, and there is every indication that the original was made in the same fashion some 400 years ago.

Furnaces and pot melts for glass dates back to antiquity. So do rods, threads and tubes, the materials for lampworking, which accompanied most glass making furnaces.

The Egyptians used threads for surface decorations on their vessels. The Romans likewise used rods for latticino and millifiore, a technique developed 2000 years ago.

For a proper evaluation of the goblet, the fundamental differences between blowing glass on a lamp and blowing glass from a furnace should be understood.

The lampworker uses preformed glass rods and tubes of various lengths and diameters and works directly in the open flame from a torch. The torch, or lamp, which produces the flame permits the use of a fuel and a supplementary gas, such as air, and offers a degree of regulation to provide localized and intermittent heating. Lampworking in glass is unique and distinctive for these reasons. Blowing glass in a furnace operation requires the use of a long, hollow, iron blowpipe, on which glass is gathered or wound, and drawn from a pot or tank of molten glass which is contained within a furnace. The gather, once drawn, is rotated and blown to a desired size and shape. (FIG. 1)

Offhand glassblowing from a furnace.

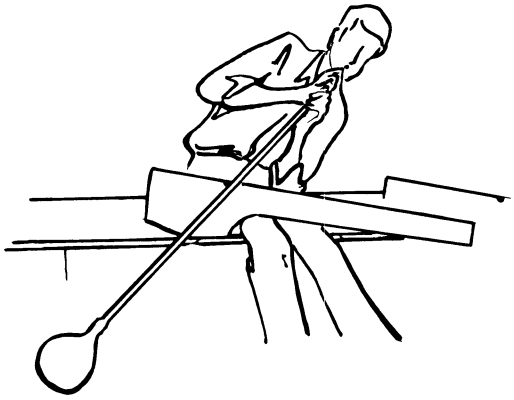
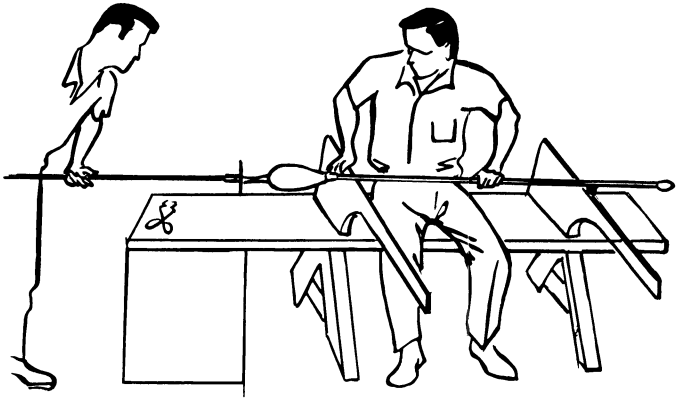


Figure 1

After blowing, the piece is transferred to a long iron rod known as a pontil rod for finishing. The pontil rod traditionally is attached to the bottom of the piece, and the work released from the blowpipe by scoring it, thereby setting up a stress which causes separation from the pipe by a gentle tap. (FIG. 2)



Attaching a pontil rod to a base.

Figure 2

Furnace operations often include the use of a glory hole, which is a separate heating chamber containing no glass melts but operates at a higher temperature than the melting furnace and facilitates finishing or reheating.

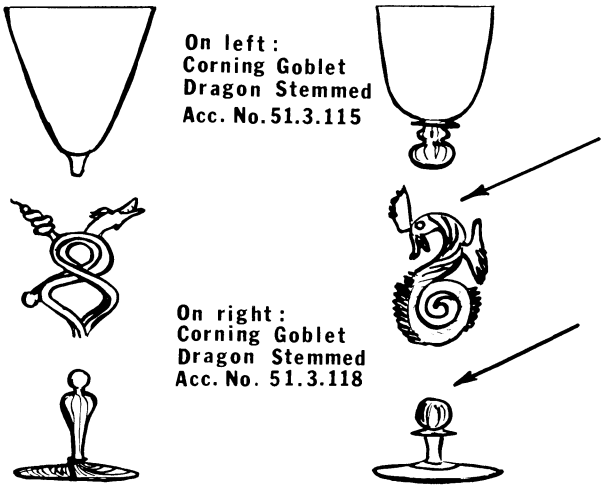
The finished piece is finally separated from the pontil rod itself by another gentle tap and the results from the attachment leaves a very distinctive mark. These pontil marks are so indicative of hand crafting that many glass enthusiasts seek them out.

It should be stated that a great deal of expertise can be exercized with a blowpipe and a gather of glass, as well as with a torch and the use of rods and tubes, and in some cases it may be difficult to distinguish the difference, however we are concerned only with those pieces that clearly indicate lampworking, such as the dragon goblet.

Had the goblet been made from a furnace in the conventional manner, as explained, it should have a pontil mark, which is conspicuously absent in this case and becomes an important point.

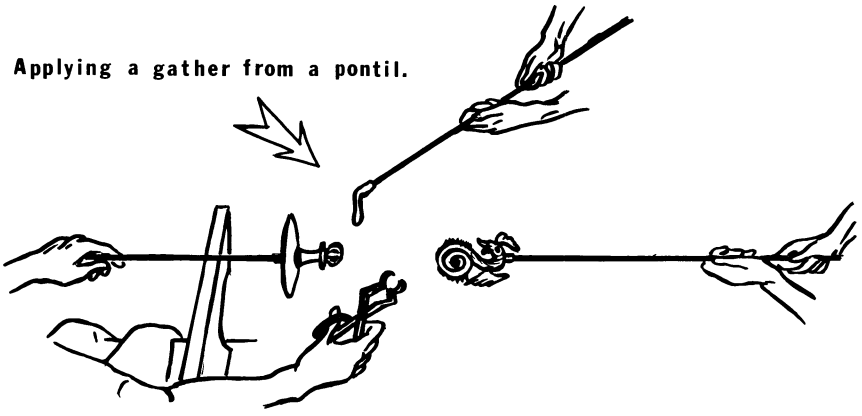
Next the detail in the dragon head contains white eyes with a tiny black dot in each, 2 separate rows of fine white teeth, layers of rods to form tassels on the top and gills on the bottom, and all clearly indicate the use of a lamp.

Another comparison can be made between furnace and lampworking techniques where the receptacle, the body, and the base are joined. (FIG. 3) The joining of separate components using furnace techniques requires two men working in concert, each supporting his section on the end of a long pontil rod or blowpipe. (FIG. 4)



Arrows indicate where more glass was added before joining.

Figure 3



Joining 2 pieces of a Dragon Stemmed Goblet using a furnace technique.

Figure 4

In lampworking these seals can be made by one man, supporting short pieces in each hand, consequently there are marked differences (FIG. 5). Goblets with the same drag-motif, from the same time period, bearing pontil marks and attributed to furnaces display an entirely different look, where joined. In the figure 8 of the body itself there are two layers of rods with convoluted lines of color. The convolutions or spirals were probably made by enclosing threads of color around a central clear rod and encasing in a tube and in turn, sealed on a lamp, and twisted into a spiral. What does seem apparent in the body itself, is that one layer was first shaped and the second layer added later by following the

Sealing components of a dragon stemmed goblet on a lamp.

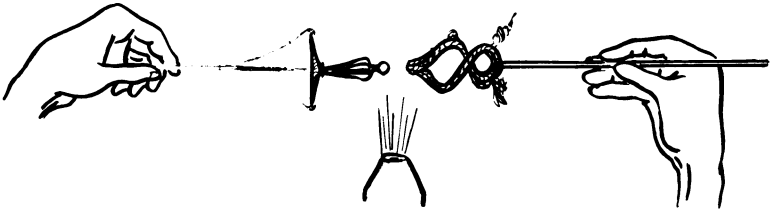
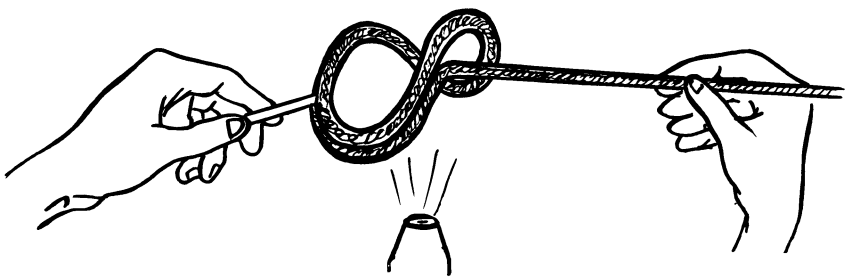


Figure 5

first configuration with a threaded second similar rod and applied by torch. (FIG. 6) It is apparent because the line movement between the layers are not the same, and had they been formed at the same time they should correspond and be identical.

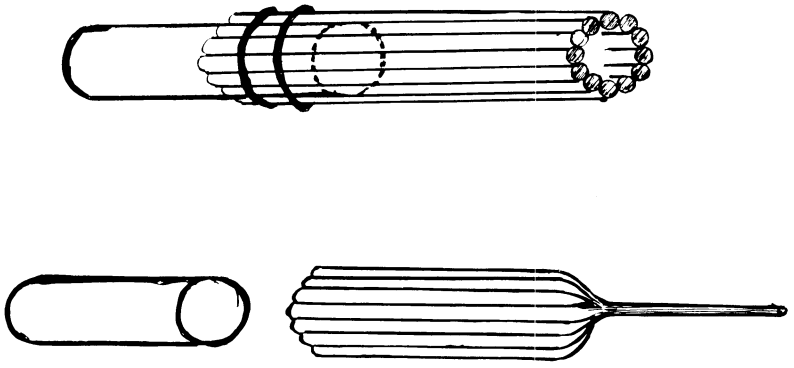
There are line interruptions in the spirals where the head and tail join the body. This indicates localized heating. Probably the biggest and best distinction can be made between lamp and furnace techniques by the lamp offering the advantage of being able to apply a localized, concentrated, flame when desired, in order to heat a particular spot or area. The departure here is very sharp from the furnace where the entire piece, or certainly the biggest part of it, must be subjected to a broad or general heating when exposed to the furnace or glory hole. There is more evidence of localized heating where the body sits atop a ball of crystal glass on the stem, the ball contains lines which trail off. The lines appear to be from the body itself, and again, it retains the character of lampworking because of localized heating. The next area of study is the lined stem and base. These lines may be achieved in a furnace operation through the use of a rib mold into which a gather is blown, the mold itself being lined. There are some existing furnace pieces which contain lined stems, however the stems are heavier and there is not the same line defination, nor do these lines extend into the bases themselves as in the dragon goblet.

In a lampworking operation these lines may be accomplished by starting with a lined tube. The tube is made by lining rods around a cylinder or core of an appropriate size and banding together. (FIG. 7) The end is then sealed and drawn down to a smaller diameter, the cylinder is removed, another similar end pulled down at an appropriate length, thereby forming the lampworkers familiar point, but one which contains lines. The stem and base can be made by applying the conventional



Applying a threaded rod to a figure 8 by lampworking.

Figure 6



Forming lined tubing from rods. Millicane method

Figure 7

lampworking techniques of heating, pulled down for the stem, sealing off, blowing the bulb, and flaring for the base. (FIG. 8 & 9) The spirals in the base are made by twisting while the bulb is being blown.

There are other more subtle, but equally compelling signs indicating the piece was lampworked, but the aforementioned points would seem enough to justify serious consideration as to the actual origin or nature of its method of manufacture. The evidence appears overwhelmingly in favor of lampworking.

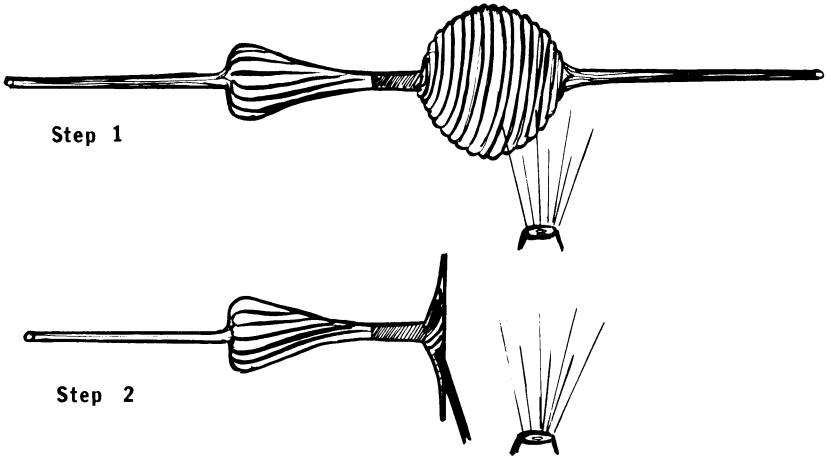


Figure 8

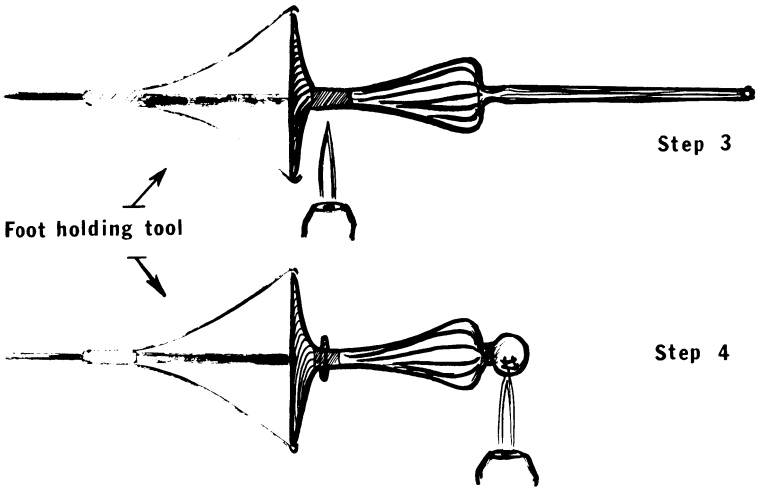


Figure 9

Lamps did exist, and were used during the 16th century for bead making and enameling by the Venetians. The goblet is a clue to how sophisticated they were because of the scope of regulation required to complete the piece, from a fine, sharp needle flame for the collar separating the base and stem, to the configuration of the 8, or blowing the receptacle, which requires a broader adjustment.

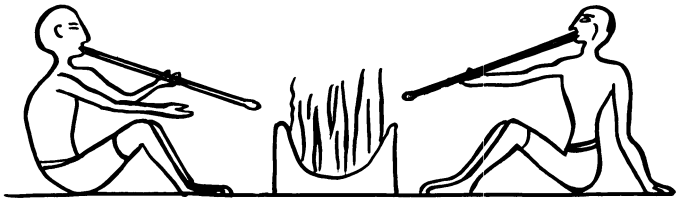
Another study which involves early work and the use of the lamp for a reproduction was made, and a paper entitled "SNAKE THREAD GLASS" was published in 1965 by Weidmann describing his use of lamps for reproductions from the Roman Period. He mentions the use of a bunsen type burner to achieve a chain link effect which was applied to the surface of his particular reproduction. The chain link effect was made by drawing a thread or fine rod, and when cooled, and able to be supported with the hands at either end, it was exposed to a flame in the critical areas, in a line, and bent in succession, so as to periodically and methodically reverse its direction in order to acquire the look of a chain link assembly. Once fashioned these pieces of thread or rods were picked up by a gather from the furnace and appear as surface decorations on the finished product.

These things are of little consequence however until we find the earliest significant piece and firmly establish both the lamp and its contributions.

As far as the earliest lamp and what it may have looked like, consider the simplicity of the device itself. A stream of air, mouth blown, and directed through a tube into a flame as small as that of a candle can reach temperatures well in excess of 2000 degrees F., high enough to melt certain minerals or small amounts of glass. There are pictures on the

walls of some excavations of early Egyptian tombs depicting just such an activity, where men appear to be blowing at, through, or into a fire. (FIG. 10 & 11) The first blow tube was probably a reed. Other drawings also show the use of bellows by the Egyptians for metalworking. Soldering is still being done by blowpipes today where air is supplied by mouth, in more primitive areas, by jewelry craftsmen, and the blowpipe is standard for identifying rocks and minerals, even in advanced societies, using the same system, because of ease, convenience and simplicity.

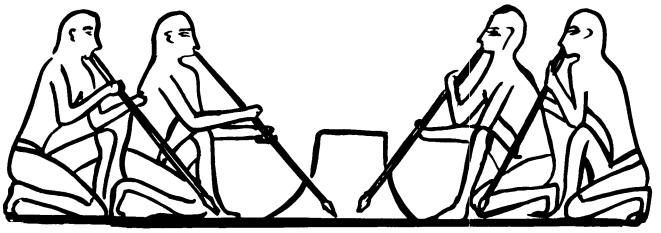
Painting at Beni Hassan, as given by Wilkinson. From Antique "Drinking Glasses" by A. Hartshorne.



Early use of the blowtube and fire by the Egyptians.

Figure 10

Illustration from M. Garnier's book, taken from Cailliaud. From "Antique Drinking Glasses", by Albert Hartshorne.



Ancient Artisans engaged in blowing operation.

Figure 11

The first fuels were probably made from animal fat, or tallow and contained in reservoirs carved out of rock.

The Romans extracted oils from plants for cosmetics, and some oils may have served as fuels. They also developed foot pedals, which they used, and through a pulley arrangement achieved shaft rotation for early grinding operations. They may have applied it to the production of a blower.

The invention of electric motors and compressors had an influence on torches and their development, as did later technologies, which brought other liquified and compressed gases as fuels. Today's sophisticated torches may accommodate as many as 2, 3, or more gases simultaneously, however, we are not so far removed from the very earliest in principle.

The final consideration as to what the contribution of the lamp may have been deserves a look at the proliferation of beads which has come out of all ages, and appear as the very earliest of any work from the past. Inconsequential as they may seem, they belong in the realm of the torch, and are suggestive of the amount of activity carried on in that area.

It may well be that the use of the blowpipe, and a study of minerals and rocks, led to the understanding of the chemistry of glass in the very beginning.

SOLAR ENERGY

FRANCIS P. FEHLNER
Research and Development Laboratory
Corning Glass Works
Corning, New York 14830

ABSTRACT

The application of solar energy to solving the present "energy crisis" requires the solution of a basic economic problem. The present cost of solar collectors is too high for solar energy to compete with fossil fuels.

A solution to this problem is being sought by designing collectors which combine higher efficiency with lower cost materials. An example of such a collector is the evacuated tubular collector of Corning Glass Works.

The Corning collector consists of a flat plate absorber, which is coated with a selective, optically absorbing film, mounted in a 4″ diameter glass tube. The assembly is evacuated to less than 10^{-4} Torr pressure and sealed off. Heat collected by the absorber plate is transferred to a heat-transfer fluid which flows in metal tubes attached to the absorber plate. The tubes pass through the vacuum jacket via glass-to-metal seals.

The evacuated tubular collector offers up to 2½ times the performance of a standard flat-plate collector in solar climate control applications which include the production of hot water, space heating, and space cooling. In this way, the operational cost of solar energy is being reduced.

A MODIFIED JET SEPARATOR FOR A GAS CHROMATOGRAPH — MASS SPECTROMETER INTERFACE

DR. MARSHALL GORDON
Department of Chemistry
Murray State University
Murray, Kentucky
AND
WILBUR C. MATEYKA
Mateyka Scientific Glassblowing
Lexington, Kentucky

In the study of environmental problems the chemist requires the analysis of such diverse substances as air and water pollutants, pesticides, smokes and fuels, all of which require the separation and identification of components of extremely complex mixtures. The Gas Chromatograph has the ability to separate complex mixtures. The Mass Spectrometer has the sensitivity and specificity to provide identification and quantitative determination of these components. Therefore, the coupling of the two methods has provided the chemist with one of his most powerful analytical tools. The operational parameters of both Gas Chromatographs and Mass Spectrometers are similar in regard to sample size and temperature requirements, and both separation and analysis occur in the vapor state. However, one incompatibility exists between the two methods: namely, the relatively large volumes of carrier gas used in Gas Chromatography must be accommodated to the requirement for reduced pressure in the Mass Spectrometer. Several interfacing techniques have been developed which minimize this problem.[1]

One such technique, the use of a jet separator, has been available for several years. Figure 1-A is a schematic of one type of jet separator currently in use at Murray State University. Basically, it consists of two fine capillary jets sealed in line with each other in a chamber that has a vacuum outlet. Figure 1-B is an enlargement of the junction of the jets showing the operation of the separator. As shown in the diagram, the gas stream from the chromatograph, consisting of sample and carrier gas, enters the evacuated chamber through the Gas Chromatograph jet. The lighter helium atoms tend to diffuse outward into the area of reduced pressure more rapidly than the heavier sample molecules and are, therefore, pumped away, leaving the sample molecules to travel straight on and enter the Mass Spectrometer jet which is connected to the Mass Spectrometer ion source. The efficiency of the separator depends on the flow-rate of the carrier gas, the precise alignment of the jets, the relative size and separation of the jets, and the pumping speed of the auxillary vacuum system. Data concerning these parameters are currently being calculated, but are not ready for publication.

Difficulties were encountered in using some of the commercial jet separators available. First, the bores of the jets are extremely small with

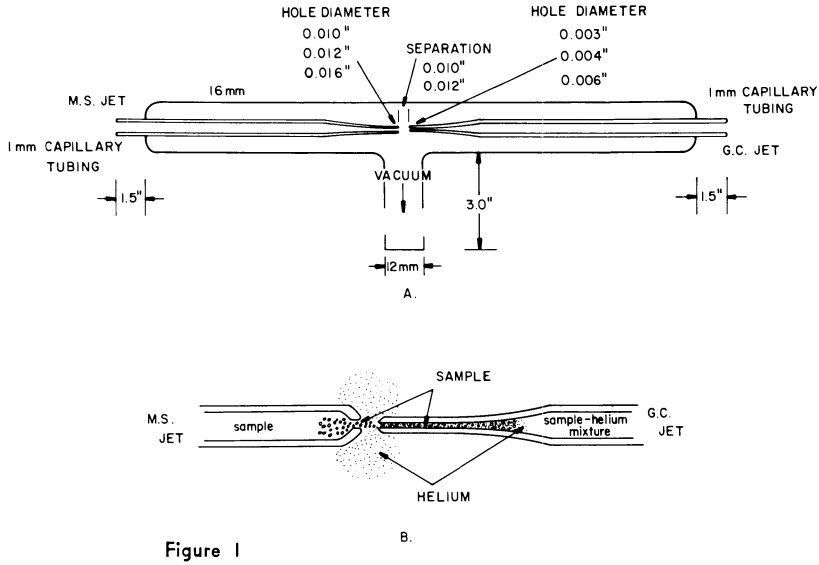


Figure 1

thin walls, making them difficult to clean and easily broken upon being subjected to minor mechanical shock. Secondly, the volume of the line connecting the separator to the Mass Spectrometer often allows the separated components to become mixed together before they reach the point of ionization.

The first problem was solved by using the separator shown in Figure 1-A. By using jets of larger bore with heavier wall thickness, cleaning and breakage problems were minimized while sacrificing only a slight loss of sensitivity in the system. The second problem still remained. To eliminate both problems, a second separator was designed which could be mounted through a CAJON ULTRA-TORR[2] fitting, directly into the ionization chamber of an Hitachi RMU-7 Mass Spectrometer as shown in Figure 2. A schematic of the separator is shown in Figure 3. This arrangement positions the end of the separator inside a Quartz funnel which leads to within about 1 centimeter of the filament, thereby eliminating any mixing of sample components before they are ionized.

In the operation of conventional separators such as that shown in Figure 1, the separator is inclosed in an oven and the line from the Chromatograph and that to the Mass Spectrometer are wrapped with heating tape to prevent the sample from condensing. The nature of the connection used in the modification prevented the use of an oven. Thus, it was necessary that the heating element and thermocouples be built into the separator as shown.

The preparation of the jets is shown in Figure 4. The Gas Chromatograph jet, Figure 4-A, is 1mm capillary tubing drawn out and cut at the proper size. The size is determined by gauging the bore with music wire of the proper diameter. The end is ground square and beveled with 400

Figure 2

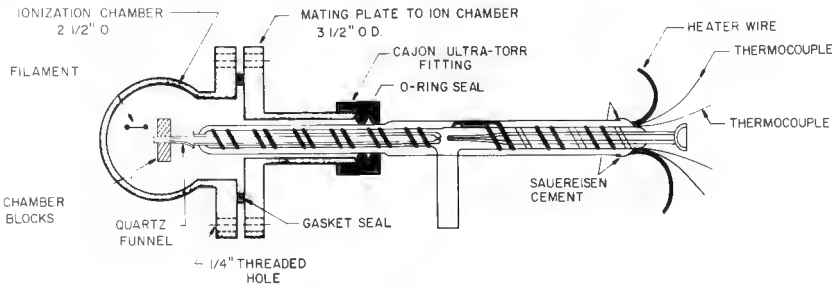


Figure 3

mesh carborundum paper, it is then polished to a high gloss with 600 mesh carborundum paper. The Mass Spectrometer jet is made from 5mm tubing which was heated and thickened to a sharp constriction as shown in Figure 4-B. It was gauged, ground and polished in the same manner as the Gas Chromatograph jet. The smaller outside diameter of the Mass Spectrometer jet allows room for sealing, as the vacuum chamber is only 6mm I.D. at the Mass Spectrometer end. It was also hoped that the larger I.D. would permit more efficient pump-out of the jet. Beveling the ends of the jets permits the use of heavier wall thickness, for strength, while still permitting adequate clearance for efficient pump-out of the carrier gas.

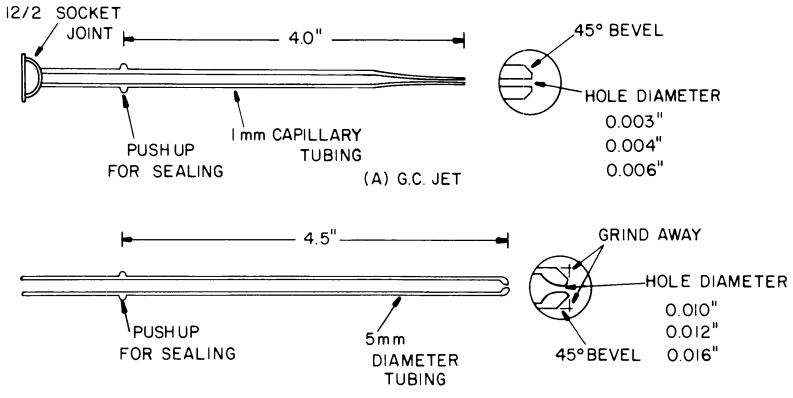


Figure 4

Figure 5 shows the construction of the Mass Spectrometer end and the attachment of pump-out sidearm of the separator. The vacuum chamber is cut to length and the sidearm is trimmed so that the chamber centers inside the outer jacket. Because of the sidearm, the heater wire and the first thermocouple must be wound around the vacuum chamber before it is sealed into the outer jacket. The insulation on the thermocouple and the binder in the asbestos on the heater wire will burn, causing problems during the sealing operations; therefore, it is best to run

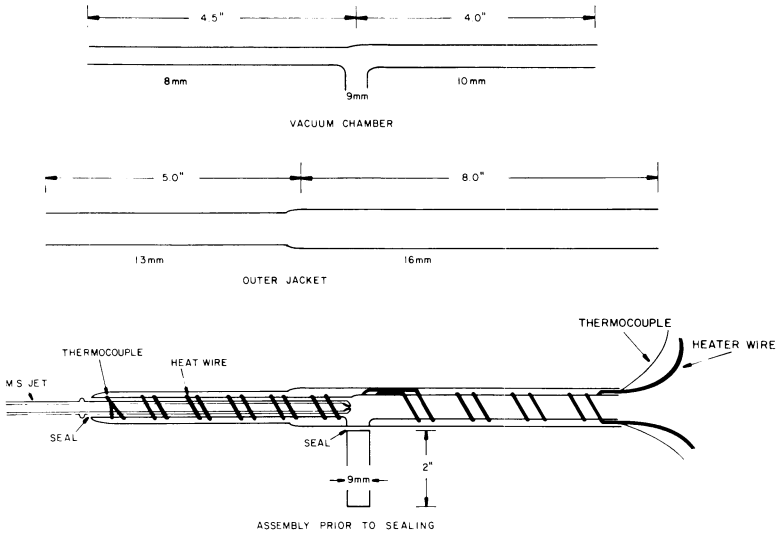


Figure 5

the assembly through the annealing oven before attempting to seal in the jet and the sidearm. The only available thermocouple wire was insulated with cotton sleeving. To insure against the possibility of shorting out after the insulation is burned away, the thermocouple was wound around the heater wire as shown in Figure 6. This technique induces an A.C. voltage (about 0.5 millivolt) into the thermocouple when the heater is operating, but it creates no problem when using a regular potentiometer. If a solid state digital readout potentiometer is used, it might be best to filter out the A.C. voltage. After the jet is sealed in and the sidearm is attached, the assembly can be oven annealed. (This is the last operation which can be annealed in the oven.)

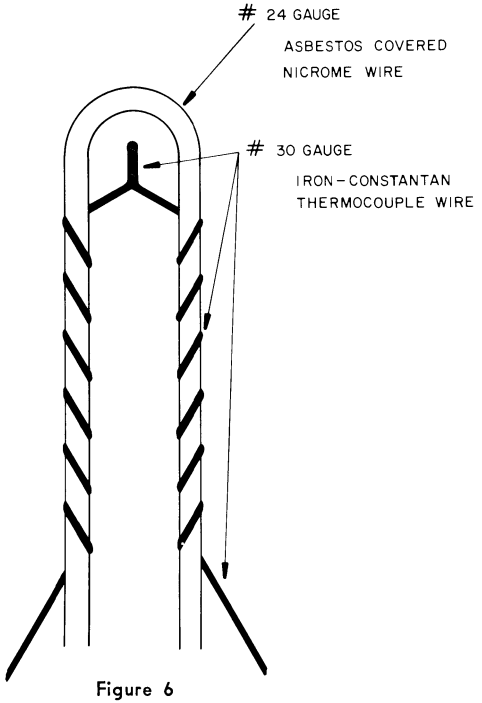


Figure 6

Next, the outer jacket is cut to length, the heater wire and thermocouple are folded back out of the way, and the Gas Chromatograph jet is sealed in as shown in Figure 7. Because of the critical nature of the alignment and spacing of the jets, a jig was used to hold the jets in position during the final sealing operation. Figure 8-A is a schematic of the jig. It consists of a piece of 3/32″ aluminum rod, a 2″ length of stainless steel hypodermic tubing[3] the size of the Mass Spectrometer jet, and a length of music wire the size of the Gas Chromatograph jet. The music wire is inserted through the hypodermic tubing and folded back on one end. This end is inserted into a small hole in the end of the aluminum

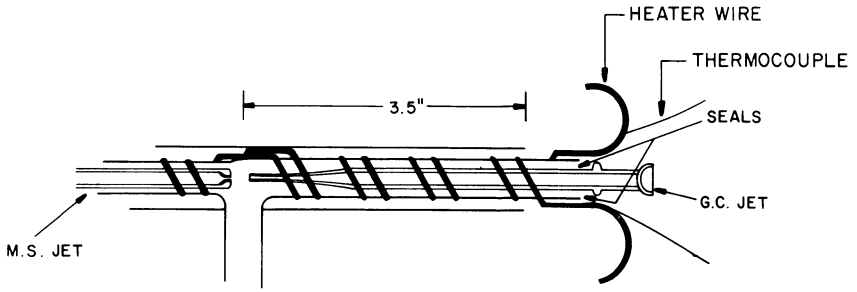


Figure 7

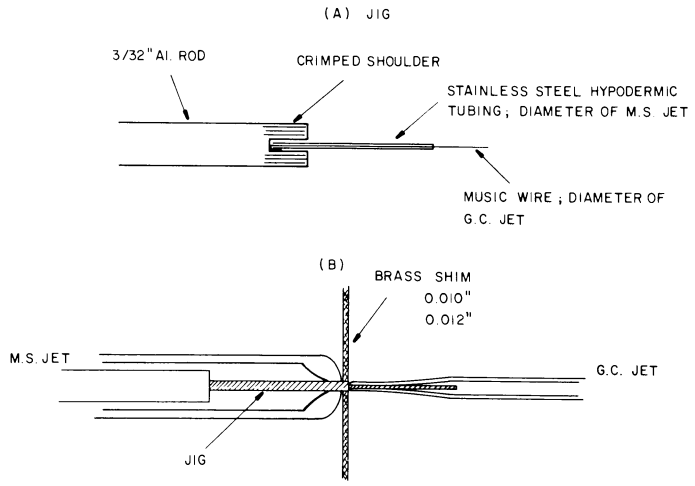


Figure 8

rod. The assembly is then crimped with a crimping tool to hold it all in place. The jig is then positioned in the Mass Spectrometer jet, and a brass spacer is inserted through the sidearm and placed over the jig as shown in Figure 8-B. The Gas Chromatograph jet is inserted into the vacuum chamber, carefully threaded onto the jig, and the final seal is made. The Gas Chromatograph jet must be pressed firmly against the spacer shim while the sealing and annealing operations are performed to insure proper spacing. Care should be taken to localize the heat in the sealing area and away from the jig. Because of the close fit, any expansion of the jig will crack the jets. After the seal has cooled, remove the jig. If the shim does not fall out on its own, do not attempt to force it out, as this can chip the faces of the jets; simply dissolve it away with dilute nitric acid. Do not attempt to oven anneal the assembly as any sagging of the jets will cause misalignment and ruin the efficiency of the separator.

The last operation consists of inserting the second thermocouple and cementing all wires in place using Saureisen #8 Ceramic Cement[4] as shown in Figure 9.

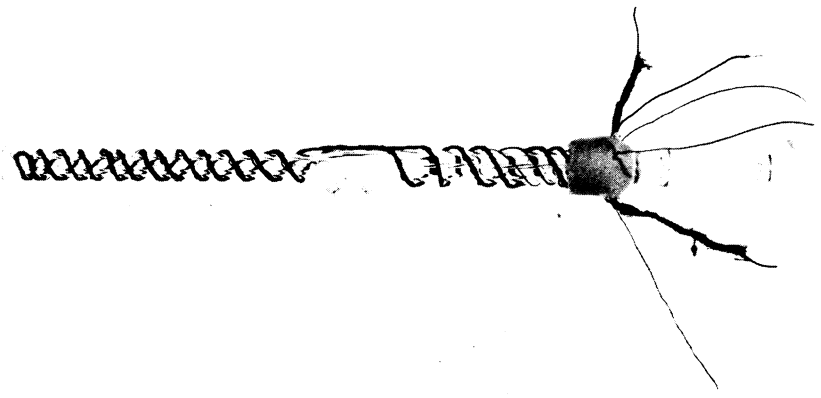


Figure 9

CONCLUSION

Although the use of this device is limited to a specialized scientific instrument, we believe that some of the construction techniques will prove useful in other glassblowing operations. It is also hoped that the above discussion will provide useful insights into the design and construction of jet separators which can fulfill the specialized needs of scientists in your organization.

ACKNOWLEDGEMENT

The authors would like to express their appreciation to the Environmental Protection Agency, which supported this work through Grant #R-802964-01.

REFERENCES

1. Merritt C. Jr.—Applied Spectroscopy Reviews 3(2), pp. 263-326, 1970; and references therein.
2. Registered Trademark, Cajon Company, Solon, Ohio.
3. Available from Tubesales, Forest Park, Georgia.
4. Available from Saureisen Cements Company, Pittsburg, Pennsylvania.

A SIMPLE GLASS TEFLON CO-AXIAL VALVE

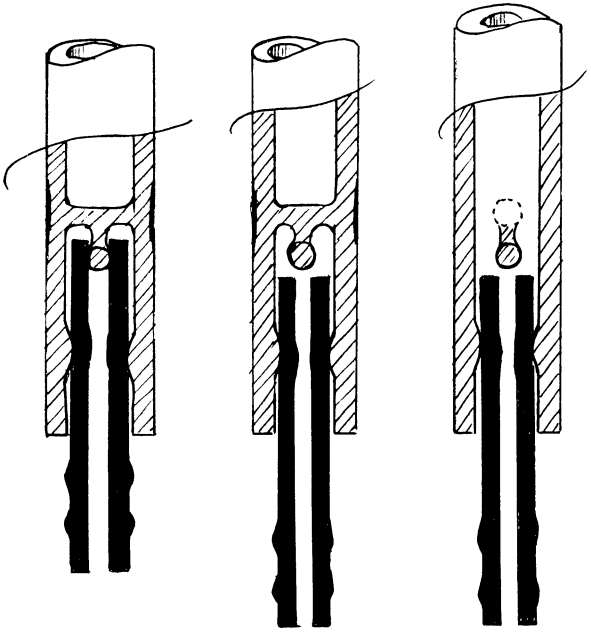
ALF LUNDBERG AND BÖRJE WICKBERG

Glass Workshop and Department of Organic Chemistry 2
Chemical Center, University of Lund, P.O.B. 740
S-220 07 Lund 7, Sweden

A glass-teflon connection, which is described separately, can be adapted to serve as a simple, efficient and non-sticking valve, which is very convenient for many purposes, for instance for chromatographic columns where small dimensions and low dead volume are desired.

The valve (Fig. 1) is made from a piece of glass tube and a teflon tube. The glass tube has a ring-shaped constriction, through which the teflon tube can be pushed by applying firm pressure (after the first pass it will slide more readily). A glass rod with a slightly smaller diameter than the i.d. of the teflon tube is melted in one end to form a ball with a slightly larger diameter than the i.d. of the teflon tube. The rod is then formed to a T-shape (Fig. 2) by working the softened glass with a pair of tweezers. The T should fit rather snugly into the glass tube and is po-

Figure 1



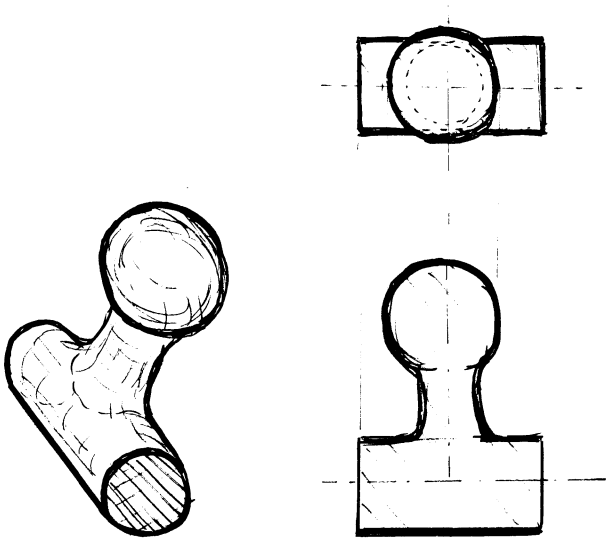


Figure 2

sitioned there as indicated in Fig. 3. By heating a small area of the glass tube, one end of the cross-bar is made to stick slightly to the wall of the tube. The tube is then turned around and allowed to cool somewhat so that the ball and rod part will come into correct position (Fig. 4). The second end of the cross-bar is then carefully fused to the glass wall (Fig. 5). When the glass has solidified the first end of the cross-bar is per-

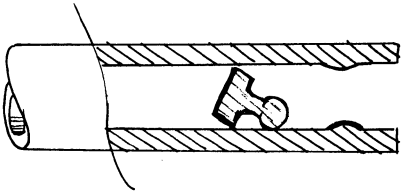


Figure 3

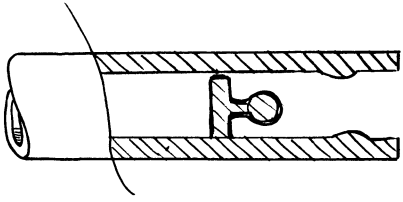


Figure 4

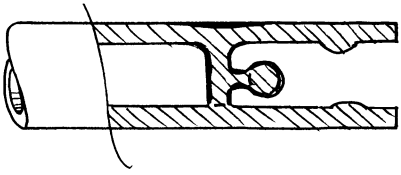


Figure 5

manently fused to the tube wall (Fig. 6). Introduction of the teflon tube for the first time is made easier by using a little silicone grease and warming the glass in a hot water bath. The projecting end of the teflon tube can be provided with a grip as indicated in Fig. 1 by softening it in a small flame (Hood!) and pushing it together at two points separated by a few mm. The dimensions indicated in the figure are arbitrary; we have also made micro valves with a 1.2 mm o.d. teflon tube.

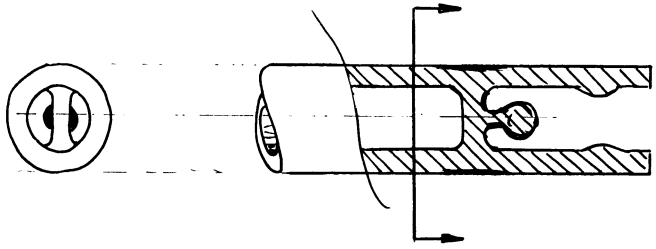


Figure 6

A FLEXIBLE GLASS TEFLON TUBE CONNECTION

ALF LUNDBERG AND BÖRJE WICKBERG

Glass Workshop and Department of Organic Chemistry 2
Chemical Center, University of Lund, P.O.B. 740
S-220 07 Lund 7, Sweden

Many laboratory operations require a flexible yet chemically resistant connection between different vessels. Teflon tubes are often ideally suited for the purpose provided they can be attached reliably to glass. A very simple but efficient connection can be made by pushing the teflon tube through a slightly narrower constriction in a glass tube (Fig. 1). If the constriction is made properly, the connection will be gas-tight, non-slipping and will still allow adjustment of the teflon tube in an axial direction. Minor leaks, for most purposes harmless, may appear if the temperature is allowed to decrease more than 50°. The teflon connection can be used for venting the head space or for siphoning the contents of a flask. In the latter case the tube is simply pushed below the surface of the liquid. Connections of this type have been used for instance for attaching a glass pre-column to a gas chromatograph (1), for transferring solutions of diborane and organo-metal compounds, for feeding eluants to low-pressure chromatographic columns and for passing nitrogen into low-volume photochemical reactors. Connections of the dimensions shown in Fig. 1 have also been used under suitable circumstances at pressures above 10 atm.

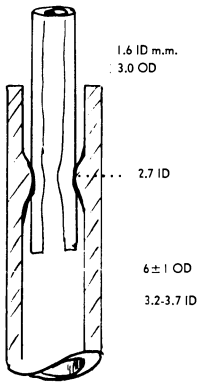


Figure 1
A Flexible Glass Teflon Connection
"Wickbergare"

NOTE

(1) Gunnar Bergstrom, Chemica Scripta *4* (1973) 135

THE LASER AND YOU

GEORGE BATLEY

Lab-Crest Scientific Division
Fischer & Porter Company
Warminster, Pennsylvania 18974

In greetings to one another again at this 20th Annual Symposium, let us welcome a new friend, the Laser. After many years of a mysterious incubation period, the laser has finally emerged: it is here. It will soon become a part of our every-day life so we might as well get used to it, familiarize ourselves with it, and treat it as a "friend", for it will be, and a very good one, too.

As we look over the past half-century with all its advances in technology probably one of the greatest single boons to the glassblower trade has been advent and developments of the electric sign industry. We have thousands of shops today in each and every part of the country, lighting up our cities, sometimes like a giant bill-board but in most cases, with a spectacular and colorful display of the sign-benders art and science. Many glassblowers actually get their start from training as a sign-bender and there are very few that aren't completely acquainted with the bends and splices, the tubulation and tip-off, the sealing-on of electrodes, and other basics of this art. Most of you are probably also familiar with the evacuating, bombarding, fill and aging procedures required to complete the unit as a workable sign-lite.

One of the more lightly-taken acronyms for the laser (no pun intended) refers to it as "just a glorified sign-lite tube". This certainly can't be applied to the rod family, or the solid-state or crystal members, but for the gas lasers, today the biggest and fastest growing branch of this new field, the term does have some surprising elements of truth. Several of these units are merely sophisticated sign-lite tubes. There is one whale of a difference, though, and it is where the glorification or sophistication inference enters the picture: the laser in most fundamental concept requires that light generated in the tube be "coherent" in nature.

Coherent Light. Just what do we mean when we speak of light as being coherent? Well, we mean that those countless billions of protons created as light in the sign-lite tube by an electrical discharge through a rarefied gas must be generated in such a well-disciplined order so as to be in-phase with one another and to possess the wave-pattern characteristics of a radio signal, or other electromagnetic oscillation we are more familiar with.

We can sit in a movie theater without realizing we are in absolute darkness for half of a two-hour program. The light that activates the screen is actually "pumped" at us through synchronization of a shutter and frame-feed mechanism in such a way that we are unaware of anything other than continuous light, and to the human eye, a perfectly natural set of conditions.

We can watch our television sets in similar fashion without an awareness that the light produced by the action of an electron beam on various phosphor materials is actually "pumped" at us by a vertical and horizontal "scanning" process in a line or dot pattern. Although each line or dot is activated only a few times a second with almost instantaneous "decay" of the light in between scans, the human eye sees the result as a continuous and complete picture, perfectly "normal" in every detail.

To produce "coherent" light in a gas-laser tube one of the first requirements is to incorporate some means of "pumping" the impulses too, to obtain the phase properties desired. In a mixture of gases such as helium-neon, the most widely used combination today, there is an automatic inherent built-in pumping mechanism; it involves the transfer of an electrical charge from one gas to another, and, because of the electrical polarity, of a highly directional nature and in a continuous-wave pattern. If a portion of this output is confined to a small, straight-line, "lasing cavity" of precise dimensions, and if mirrors are also incorporated in precise dimensions at the ends of the cavity to reflect the beam back and forth, the resulting effect is production of an intense, polarized, monochromatic beam of light possessing the wavelength and frequency properties required for modulation. The beam of light escaping through a small aperture in the center of one of the mirrors has some amazing properties, and is our "laser beam".

The "sophistication" of the laser with respect to a sign-lite tube involves mostly:

1. The use of a mixture of gases rather than a single one, in proper proportion and at a specified fill-pressure.
2. The use of "off-set" electrodes to permit the incorporation of the mirrors. The cathode in this case is usually designed to act as a sheath for the tube and doubles as a reflector to help confine the maximum amount of light produced.
3. The incorporation of the mirrors, and in some cases other optical devices.
4. The precise geometry required for the lasing cavity, in most cases today ranging from one to one-and-a half millimeter in diameter and from five to thirty inches in length.
5. The glass compositions. Although there is still considerable activity in the lead-glass types employed in the sign-lite industry, the trend today is toward borosilicate glasses, or the more common electrical glasses, and even to quartz.

This oversimplified description of a helium-neon gas laser tube as introduction heads up to a question all of you must have had at one time or another, "How does all this affect me"? It is the intent of this paper to take a look at this question in an attempt to provide some answers, especially in three areas of activity.

First, your work. Some of you may be already fabricating some of the envelopes or components for either the laser tubes or detector instruments used in a laser system. And, whereas the helium-neon plasma tube is an important segment of the industry, it is by no means the only one.

There are today several different laser systems even in the gas-laser field, but most of them involve pretty much the same basic construction features. Whether you are, or are not directly involved in such work, the laser is going to have an effect on your future activity, probably as a tool of the trade. Pulsed lasers are already being used in the garment industry to cut cloth to pattern, and in the semi-conductor industry to cut thin wafers of quartz to size and configuration: it is only a question of time before the science catches up to intermediate materials such as glass. These same lasers are also being used to drill holes in material such as diamond, or metals, or ceramics: it will progress to glass. And, the same equipment can be used on an assembly line for welding and other heat-treating work, in the case of electron tubes actually right through the wall of the envelope glass. Somewhere between the temperatures required to vaporize quartz during cutting and those used for welding metals on a production line, it is not inconceivable to think of a laser "torch" to replace conventional fires or electrical and RF energized equipments used in most glass-working operations today. It should be noted in passing that these above-mentioned uses of the laser have come about in most cases because of remarkable economies produced and other tangible or intangible improvements rather than just the use of something new. There will be other uses of the laser on our production floors, mostly for alignment of work and equipment and for the automatic inspection of product in three-dimensional perspective: but, even more so, for applications still undreamt of at this writing.

In the second area, your every-day living. You will probably note the laser's impact on our lives more quickly in this direction than in any other. If you have had occasion to watch construction work at close hand, you may have already observed a thin beam of red light being used to replace the more conventional transit and surveyor's chain and rod or the mason's and carpenter's plumb line, or chalk line, or level. The laser is completely revamping construction practice. In the super-market you may have already observed a special pattern on several national brand-name packages, the universal product-code marking already being applied in readiness for the combined automatic cashier and inventory control system that will be a laser read-out and which is about two years away from wide-spread usage. Before our twenty-first annual symposium, you may be the owner of a home recorder and play-back system that will attach to your television set and permit the recording and playback of any program you desire in both audio and video, or play programs purchased like records and referred to as "video" discs. It is already in the mass-production planning stages. You may already be reading a daily newspaper or a magazine for which the printing plates were "cut" by a laser, or for which transoceanic photographs were transmitted by a laser system. If you should have occasion to need certain types of surgical or dental treatment, you may come face to face with some of the amazing techniques the medical and dental profession are employing with lasers, but even if not, there is an excellent chance that some of your pre-surgical or post-surgical tests were analyzed in the laboratory using instruments with laser components. Tomorrow's new products, such as the

video-phone, will unquestionably operate on electro-optical principles, lasers.

The introduction of the laser opened the door to science and industry for use of wavelengths four orders of magnitude shorter than any other electromagnetic energy available up to that time. This is a tremendous advancement, almost unbelievable: a ratio of 10,000 to 1. For several years after its introduction the laser was laughingly referred to in some quarters as a "solution looking for a problem". There is no such inference today except from sheer stupidity: the "solution" has discovered an untold number of "problems". Lasers have produced temperatures several thousand degrees hotter than the sun and material densities a thousand times greater than any natural condition on earth, the key to tomorrow's cheap and plentiful "energy" through nuclear fusion processes already achieved in the laboratory on a limited scale. Our military is employing lasers for surveillance, tracking, weaponry and communications purposes, and there will be others. It has been said that there is almost nothing performed today with other electromagnetic frequencies that can't be performed better and cheaper with lasers. The laser has been called "the most versatile tool in the tool-box of tomorrow's engineer".

And this is where our third and most important area of discussion is concerned. The laser is here, but now that we have it, what are we going to do with it? The "solution" has already found countless "problems" but it has been estimated that there are still several thousand yet undiscovered and limited only by the imagination. You may not become involved in actual manufacture of these devices, or their components, or associated products, but if you have an imagination you could come up with some new use for them.

One of the more important advancements in the fiber-optic field has been discovery of a thermal crack-off method to produce optimum end-finish on these materials, used in "piping" light over vast distances: this crack-off is an old stock-in-trade technique of the glassblower. As one of the classical poets once observed: "The strongest rays of the hottest noon-day sun is completely obscured by one small, insignificant cloud". The laser has its "cloud" too: the most powerful rays of a potential "death-ray" machine can be harmlessly deflected off-target by such an insignificant device as the pocket mirror. Laser light is highly directional: during landing maneuvers on the moon our astronauts determined that total divergence of a laser beam from earth to moon amounted to about eighteen inches. And yet the light is extremely intense: another team of astronauts could clearly observe a small green light signalling at them as they passed in orbit thirty miles over-head. It would seem possible, for instance, to combine several tiny mirrors with the directional and intensity properties of a laser beam and bend the beam into a sign-lite pattern. One of you might make the first laser sign-lite, or an equivalent display: operating costs would certainly be minimal. It isn't too far-fetched to think of a possible laser waveguide tube, with a laser beam replacing the electron beam and maybe even a fiber-optic helix replacing the conventional filament. The first such helix would have to be fashioned by a glassblower. Possibilities are unlimited and in any conceivable direction,

and in these days of skyrocketing inflation it is comforting to know that there is still one product line still showing a reverse trend. Lasers are getting cheaper, as demand grows, as newer and better means of manufacture are developed, and as new and better methods are discovered for lasing a beam of light. Laser application is already extending well beyond the visible range into both the infrared and ultraviolet spectra and some chemical lasers are said to have a laser output that actually exceeds the electrical input required to achieve it. It is hard to speak of lasers without using the superlatives at one time limited to theater marquees or billings.

We in the Lab-Crest Scientific Division of the Fischer & Porter Family are quite excited about lasers: for supplemental business in our precision-glass specialty lines, for new and better tools for our manufacturing facilities, and for applications in new generations of the instruments that are our life-blood. We would like to pass some of our enthusiasm along to you, and in closing we'd like to leave you with this message: THINK LASER.

CAN RESIDUAL STRESS LIMITS DEFINE THE ANNEALED STATE FOR GLASS?

H. E. HAGY
Corning Glass Works
Research and Development Laboratory
Corning, New York 14830

ABSTRACT

Annealing of glass has two primary objectives: (a) reduction of residual stress and, (b) modification of structure. Most annealing is concerned with stress reduction, but exact definition through stress limits has been avoided by the industry.

A new ASTM Task Group has been formed under Sub-committee E41.01 which will attempt to define annealing on a quantitative stress basis for laboratory glassware. The need for a sound definition is developed through a discussion of glass strength and fracture probability. A federal specification is cited as a possible precedent for establishing a residual stress limit. Advanced stress measurement procedures must be described, preferably in ASTM documents, to support this program.

Although this assignment will be difficult, the new ASTM group is committed to preparing a useful document for the protection of both consumer and producer.

INTRODUCTION

Annealing of glass can be defined as a thermal process with two primary objectives: (a) reduction of thermal residual stress and/or (b) modification of structure. This discussion is concerned with stress reduction which is by far the more prominent objective in glass manufacturing. Certain applications for glass call for accurately defined physical properties, such as refractive index and density, which can be trimmed to exact value with the proper heat treatment. It is this kind of consideration which underlies the structure modification objective.

Once stress reduction is stated as a purpose, the question naturally arises, "What is the maximum permissible stress?". This is a difficult question to answer. The problem is that the strength of glass is such a variable property, greatly dependent on the severity of surface damage. Furthermore, the working stresses for most glass articles are difficult to define. These circumstances lead to conservatism. One cannot find in the printed word an exact, universal stress limit to define the annealed state for glass. Personal experience has indicated that, in general, annealed glass stresses are kept below 1000 psi.

We have entered into an era of "consumerism", having the objective of safer products. This is as it should be, provided that product specifications are guided by reason rather than emotions. Good product specifications are in the best interest of both consumer and producer. But consider injury or damage caused by broken glass that results in litigation. Fre-

quently the annealing of the glass is questioned. How can this question be resolved without firm guide lines?

This problem recently received formal recognition from ASTM. A task group on Thermal Residual Stress in Glass has been formed under the jurisdiction of Subcommittee E41.01 on Glass Apparatus within the newly formed ASTM Committee E41 on Laboratory Apparatus. As chairman of this task group, it is a pleasure to present some background on the problem and discuss the work ahead, as we see it. Our goal is to produce a document representing an ASTM specification for maximum permissible thermal residual stress in glass apparatus. We have received support from the American Scientific Glassblowers Society in that several members have been seated on the task group.

QUANTITATIVE STRESS LIMITS — A NEED

During use, an article of glass will be subjected to all kinds of thermal and mechanical stresses. In addition, the glass surfaces will undoubtedly be scratched and bruised, thus lowering strength. This surface damage is the most significant factor in determining the strength of the glass, and to be practical, it must be assumed that the worst will happen.

Consider the tensile strengths reported by Orr[1] for several specific abrasions as tabulated in Table 1. Strengths are tabulated for two different failure times to show the effect of static fatigue. This phenomenon of lowered strength with time is explained by the fact that cracks can propagate in glass with lower velocity at lower stress. The point made by these data is that the strength of glass can be reduced to below 1000 psi with severe surface damage. Even modest abrasions restrict the strength to a few thousands of pounds per square inch.

TABLE 1

EFFECT OF SPECIFIC SURFACE ABRASIONS AND TIME ON GLASS STRENGTH

Abrasion Description	Approximate Strength, psi	
	1 Min Failure	1 Day Failure
Air Blast 70 Grit Silicon Carbide	5800	4100
NO 180 Silicon Carbide Cloth, Hand Scratched	3800	2300
NO 80 Silicon Carbide Cloth, Hand Scratched	3400	2100
NO 60 Silicon Carbide Cloth, Hand Scratched	2700	1700
Steel Wheel Cuts — Dry	2000	1200
Steel Wheel Cuts — Wet	1500	900

It is logical that thermal residual tensile stresses correlate with field breakage somewhat like the curve in Figure 1 describes. Failure incidence increases as does the stress. But where do we draw the line to limit breakage? Deciding this is much like defining a speed limit on our highways. It is arbitrary, but at the same time it should be a satisfactory compromise between a reasonable flow of traffic, on one hand, and risk of accidents, on the other. The same can be said of prescribing an annealing stress limit. It must be a compromise between what a producer can reasonably and economically accomplish and the minimum risk to the consumer.

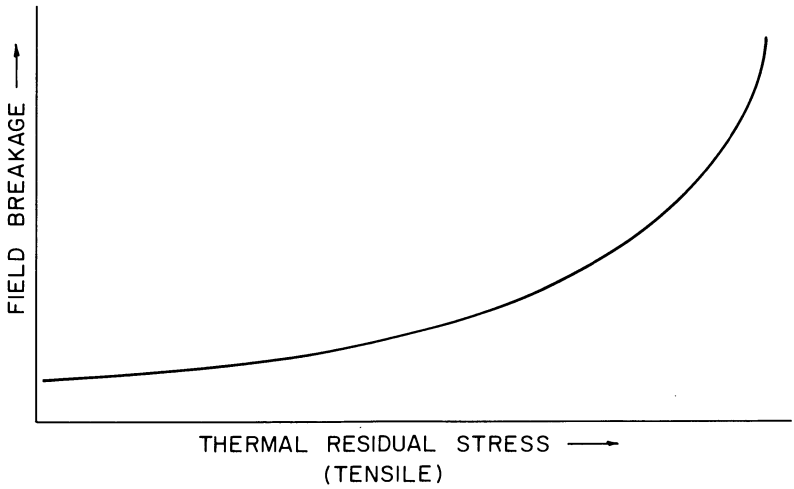


Figure 1
Probable relationship between field failures and thermal residual stress.

Emphasis is being placed on tensile stress in this discussion. Since glass is very strong in compression, higher levels might be considered as permissible in the ASTM document. This is a decision the task group must make.

MEASUREMENTS — POLARIMETRY

Certainly a document which specifies a stress limit must give some information on stress measurement. Although other stress-measuring techniques for glass shall be considered, it is clear to the task group that the stress-optical effect must be a primary tool. It has the advantages of being suitably sensitive and nondestructive.

The basics of the stress-optical effect must be remembered however. When polarized light, with its axis of polarization at 45° to the principle stresses, passes through stressed glass, optimum conditions for the stress-optical effect are produced. When this condition is met, two light rays, parallel to the principle stresses and hence at a right angle to each other

are produced. The two rays travel at different velocities through the glass and on emerging from the glass, one ray will be retarded relative to the other. This optical retardation can be expressed as follows:

$$\Delta\lambda = K\ (\sigma_1 - \sigma_2)\ p, \qquad (1)$$

where

$\Delta\lambda$ = optical retardation, nm,
K = stress-optical constant, nm/cm/psi,
σ_1 and σ_2 = principle stresses, psi, and
p = path length (glass thickness), cm.

This formula simply states that the optical retardation measured in a polarimeter is proportional to the algebraic difference between the principle stresses and the path length (thickness) of the glass. The proportionality constant, K, is the stress-optical constant. This is a function of the chemical composition of the glass. Hence, for accurate stress determinations, the correct stress-optical constant must be used.

One must never forget that a polarimeter measures a retardation that relates only to the principle stress difference. For example, if multiaxial (drumhead) stress exists, no retardation results. In this situation it would be a gross error to deduce that the stress is zero. In many cases polarimetric observations are made at the edge of a glass article, in which case the stress normal to the surface must be zero, and the stress calculation is on sounder gorunds. However the stress may vary along the light path chosen, in which case the retardation observed is related to the algebraic average stress. Polarimetry has significant limitations!

Two ASTM documents now exist for reference in polarimetry. Designation C148 instructs how to measure optical retardation with glass strain discs in a polariscope and also with a rotational analyzer polarimeter, known as Senarmont or Friedel. Designation F218 gives more detailed information on rotational analyzer polarimeters. But neither of these documents describe how to convert retardation to stress. These procedures will certainly be helpful to the task group efforts, but it appears that a great deal of amplification and further detail will be necessary for determining stress in the various glass apparatus geometries that must be considered.

A PRECEDENT

In deciding on a thermal residual stress limit, the task group will be on firmer, more comfortable ground if a precedent of significance is used as a guide. Perhaps Federal Specification NNN-C-940 for glass graduated cylinders is a precedent that could serve well in this regard. It requires the following for annealing:

> "4.2 *ANNEALING.* Ring strain shall be no greater than Temper NO. 1 when tested as specified in 4.4.1 (ASTM C148). Any longitudinal strain, if present, shall be faint and highly diffused."

Here the word strain refers to optical retardation, not the usual correct physical definition relating to elastic deformation.

Since a very common way to evaluate optical retardation is with strain discs, as described in ASTM Designation C148, the ASTM definition of strain discs was consulted. ASTM Designation C162-71, "Standard Definitions of Terms Relating to Glass and Glass Products" states the following:

> "*STRAIN DISC*—a disc of glass having a calibrated amount of birefringence at a specified location, and used as a comparative measure of the degree of stress."

This is confusing and contradictory. First, ASTM Designation C148 never mentions the word stress in relation with the strain discs. Secondly, to derive stress, as in Equation (1), we must know the path length, stress-optical constant, and the optical retardation of which the strain discs give us a comparative measure.

It is a fair assumption that the federal specification really intends to limit the stress, since breakage is probably the worry behind the strain limit, and stress is what breaks glass. If this is the case, we can go a few steps further to determine the stress level implied. Temper NO. 1 is defined as an optical retardation of 22.8 nm. Knowing the stress-optical constant and thickness of the glass strain disc, the stress can be calculated.

A nondestructive method for determining the stress-optical constant of the strain disc glass was devised. A strain disc, originally supplied by the Glass Container Association of America, was subjected to diametral compression as shown in Figure 2. The tensile stress, σ_1, and the compressive stress, σ_2, at the center of the disc combine additively to produce the resulting optical retardation as given below.

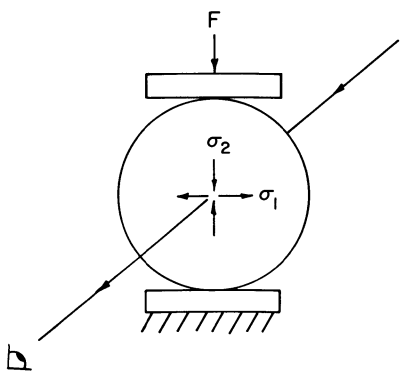


Figure 2
Diametral compression loading scheme for strain disc, indicating principle stresses.

$$\sigma_T = \sigma_1 + \sigma_2$$

$$\sigma_T = \frac{2F}{\pi Dt} + \frac{6F}{\pi Dt} = \frac{8F}{\pi Dt}, \qquad (2)$$

where

F = applied force, lb.,
D = disc diameter, 3.483 in.,
t = disc thickness, 0.091 in. (0.232 cm), and
σ_T = total effective stress (principle stress difference) psi.

The optical retardation at the center of the disc was measured as a function of applied load. Experimental data shown in Figure 3, when

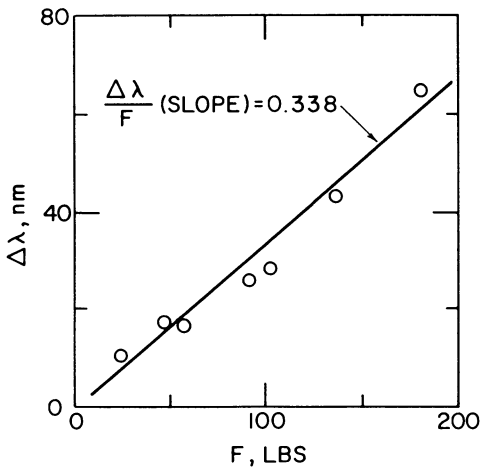


Figure 3
Optical retardation—load curve experimentally derived for strain disc.

fitted to a straight line by regression analysis, yielded a retardation — load slope, $(\frac{\Delta\lambda}{F})$, of 0.338 nm/lb.

Combining Equations 1 and 2, the stress-optical coefficient is given by the following expression:

$$K = 0.155\ D\ (\frac{\Delta\lambda}{F}). \qquad (3)$$

Substituting the measured values for the diameter and slope, the stress-optical constant for this strain disc is found to be 0.182 nm/cm/psi, a very reasonable value for a soda-lime glass, the most likely composition.

The stress level in the strain disc may now be calculated using a single stress version of Equation (1):

$$\sigma = \frac{\Delta\lambda}{pK} = \frac{22.8}{0.232 \times 0.182} = 540 \text{ psi}$$

One can say that 540 psi is a reasonable value in light of the defect-strength data in Table 1.

THE TASK AHEAD

The Task Group has many aspects to consider in drafting a specification. For stress limits,

(a) Should there be one unqualified limit?

(b) Should a higher compressive than tensile stress limit be invoked?

(c) Should stress limits be a function of the type of ware and how it is used?

(d) Should the size of the ware have a bearing on permissible residual stresses?

In addition, the experimental techniques that are recommended for measuring the stresses are very important to the final document. Direct and comparative polarimetry will be considered and, most likely, used. Polarimetry under oil immersion is messy but may be necessary in certain situations. It may be that abrasion-fracture criteria can be employed. That is, if stress measurements are impossible or inconclusive, ware acceptance could be granted on the basis of surviving a specified abrasion and water immersion. New techniques are welcome and will be given serious and thorough consideration.

REFERENCE

1. Leighton Orr, "Practical Analysis of Fractures in Glass Windows", Materials Research and Standards, MTRSA, Vol. 12, NO. 1, p. 21.

IN ATTENDANCE

The following are on record as having attended the Twentieth Symposium on the Art of Glassblowing held at the Marriott Motor Hotel, Philadelphia, Pennsylvania, June 15-19, 1975. As fully paid registered participants, these persons are entitled to a copy of the Proceedings.

REGISTRATION LIST — MEMBERS

Adams, Lemond ..... 901 W. Main, Urbana, IL 61801
Agam, Benjamin ..... Dept. of Chemistry, Tel-Aviv University, Ramat-Aviv, Israel
Alexander, J. Allen ..... 20 Merwood Dr., Upper Darby, PA 19082
Alexander, Lawrence J..... 208 Brown Ave., Iselin, NJ 08830
Angel, Alfons A. ..... Oak Ridge Rd., Bethel, CT 06801
Anderson, Robert H. ..... Old Turnpike Rd., Fitzwilliam, NH 03447
Arias, Arthur ..... 7920 West Park, Houston, TX 77036
Armstrong, Gilbert ..... 5N432 Neva Terr., Itasca, IL 60143
Baral, Melvin ..... Box 72, Ferry Rd., Bridgeport, NJ 08014
Bardo, Charles S. ..... 339 Lisbon Ave., Buffalo, NY 14215
Baum, Joseph W. ..... 200 Highland Ave., Rensselaer, NY 12144
Bayne, Eugene H. ..... 10284 W. Winston #1, Baton Rouge, LA 70809
Becke, Helmut ..... 6 Banner Rd., Ottawa, Ont., Can. K2H 5T2
Bennett, Charles A. ..... Frankford Arsenal, Pitman-Dunn Res. & Dev. Lab., Philadelphia, PA 19137
Biermann, Hugo C. ..... 1903 Cherry Place, Edgewood, MD 21040
Blessing, S. David ..... 17305 Wilshire, South Bend, IN 46635
Boger, Walter C. ..... Rt. 7, Box 239, Chapel Hill, NC 27514
Bolan, Theodore W. ..... Scarborough Lane Apt. 3C, Wappingers Falls, NY 12590
Boussert, Christian ..... 42 Gnarled Hollow Rd., E. Setauket, NY 11733
Brandler, Frank ..... 2 Fountain St., Clifton, NJ 07011
Brereton, Walter ..... University of New Brunswick, Physics Dept., Fredericton, NB, Canada
Brewin, Thomas A. ..... 22 Naples Rd., Hamilton, MA 01982
Brewin, Thomas A., Jr. ..... 7560 Cathedral Oaks, Goleta, CA 93017
Brosious, Edward C. ..... 18 Turnor Ave., Hamden, CT 06517
Brown, Allan B. ..... 353 Warrenville Rd., Manfield Ctr., CT 06250
Brunfeldt, Bob ..... 1324 Greystone, Bartlesville, OK 74003
Burcar, Thomas H. ..... 792 Kenray Ct., Cincinnati, OH 45215
Campbell, Clair ..... 1429 Zettler Rd., Columbus, OH 43227
Campbell, David R. ..... 4816 Wilhar Rd., Powaukee, WI 53072

Campbell, Robert G. .....Univ. of Windsor, c/o W. R. Filbin & Co., 2436 Bagley Ave., Detroit, MI 48016
Carter, Frank B. .....373 S. Main St., Andover, MA 01810
Chaconas, Peter G. .....11607 Hitching Post Lane, Rockville, MD 20852
Christie, Henry L. .....308 Almonte St. (Box 419), Almonte, Ont., Can. K0A 1A0
Cicero, Philip .....308 West Empire Ave., Benton Harbor, MI 49022
Cleckley, Derald .....2104 N. Fisher Ct., Pasadena, TX 77502
Cloninger, Jerry A. .....2816 Arden Way, Smyrna, GA 30080
Costea, Dumitru .....48-06B-46th St., Woodside, NY 11377
Cristaudo, Lorenzo .....RR4, Carbondale, IL 62901
Danko, Robert .....118 5th St., NE, Barberton, OH 44203
Davis, David A. .....631 Sequoia Dr., Webster, NY 14580
DeAngelis, William M. .....59 Whipple Rd., Wilton, CT 06897
DeCesare, James V. .....Shell Development Co., 3737 Bellaire Blvd., Houston, TX 77025
DeFlorio, William .....117 Robbins Dr., Carlisle, MA 01741
Dehn, William R. .....15931 Batson Rd., Spencerville, MD 20868
DeMaria, Vincent C. .....48 Union Tpke, Wharton, NJ 07885
Deminet, C. .....26037 Marine View Dr., Kent, WN 98031
Denton, Tom .....909 Forreston Dr., Richardson, TX 75080
DeWolff, W. N. .....The UPJOHN Co., Unit 7266-25-1, Kalamazoo, MI 49001
Dicu, Mihal .....48-06-46th St., Woodside, NY 11377
DiGiacomo, Robert .....198 Westford, Chelmsford, MA 01924
Dolenga, Arthur .....44045 Donley, Sterling Heights, MI 48078
Dollé, André .....261 Fern Dr., Elk Grove Village, IL 60007
Domizio, Arthur .....44 Nichols Ave., Yonkers, NY 10701
Doody, Thomas J. .....7930 Woodglen Ln., Downers Grove, IL 60515
Doring, Horst .....7D Radsvagen, Huddinge, Sweden 14148
Drechsel, Helmut .....Box 498, Quakertown, PA 18951
Ducourneau, Robert .....13 Jay Ave., Ottawa, Ont., Can.
Dusek, Leo F. .....3425 Grantwood Dr., Cleveland, OH 44134
Dunne, W. R. .....11 Hidley Extension, Troy, NY 12180
Eberhart, W. R. .....1115 Jarvis St., Windsor, Ont., Can. N8P 1C8
Eckberg, Edwin (Ed) .....P. O. Box 5102, 5504 Currier Cir., Boise, ID 83705
Edwards, Robert V. .....308 E. Whitehall Rd., State College, PA 16801
Elsholz, William .....20656-N-Kennefick Rd., Acampo, CA 95220

Everingham, Kenneth J. ....251 J St., Carneys Point, NJ 08069
Faust, Gerald .....204 S. Hendricks Ave., Exton, PA 19341
Forgnoni, Robert .....Research Glass of N. J., N.W. Blvd., Vineland, NJ 08360
Frain, Chizuko .....Hillwood Place, Norwalk, CT 06850
Franer, William L. .....415 South Linden Ave., Miamisburg, OH 45342
Gasparik, Michael T. .....1177 McCarter Hyway, Newark, NJ 07104
Gerhart, Richard P. .....440 College Park Dr., Monroeville, PA 15146
Gilhooley, William A. .....1466 Van Curler Ave., Schenectady, NY 12308
Glover, John A. .....8225 Parkview, Munster, IN 46321
Good, Gordon .....15 Emerson Court, Amherst, MA 01002
Goodyear, Leon F. .....1713 Cottage Grove, Urbana, IL 61301
Gordon, Mel .....5951 NE 2nd Ave., Ft. Lauderdale, FL 33308
Grant, Richard A. .....3717 Wenbrook Dr., Kettering, OH 45429
Gray, Lou .....440 College Park Dr., Monroeville, PA 15146
Grout, John B. .....129 S. Granada, Alhambra, CA 91801
Gunther, Adolf .....3468 Sulin Court, Yorktown Heights, NY 10598
Haak, Werner H. .....4216 Trees Hill Dr., Lafayette, IN 47905
Hagedorn, James A. .....5946 Glenwood, Chicago, IL 60660
Haim, Walter A. .....5222 So. Nordica St., Chicago, IL 60638
Hapstack, Richard J. .....212 Marcella Rd., Parsippany, NJ 07054
Hawkins, Anthony J. .....P. O. Box 591, Alfred, NY 14802
Hernandez, Peter .....4 Tiverton, Ottawa, Ont., Can K2E 6L5
Higgins, Columb G. .....1875 Purdie Lane, Maple Glen, PA 19002
Hill, George H. .....144 Meaford Dr., Waterloo, Ont., Can. N2J 3T9
Horn, Francis .....39 E. Ridge Pike, Conshohocken, PA 19428
Hurff, Gerald M. .....10135 Lomond Dr., Manassas, VA 22110
Hydro, George .....13003 Twinbrook Parkway #204, Rockville, MD 20851
Hydro, John, Jr. .....3414 Randolph Rd., Wheaton, MD 02902
Hyland, Edwin J. .....436 Russell Rd., DeKalb, IL 60115
Jahn, Henry R. .....3932 Dyre Ave., New York, NY 10466
Keller, Raymond J., II .....RD #1, RTE 579, Ringoes, NJ 08859
Kern, Thomas E. .....424 Sunland Dr., Cincinnati, OH 45238
Kingsbury, Owen J., Jr. ....207 N. Eastern St., Greenville, NC 27834
Knisely, Samuel E. .....72 N. Main St., Mullica Hill, NJ 08062
Kolleck, Floyd W. .....368-55th St., Clarendon Hills, IL 60514

Kozak, Richard .....333 East Gate Dr., Algonquin, IL 60102
Kraus, John R. .....232 Mercury Rd., Newark, DE 19711
Kuehl, Keith A. .....17 Marilyn Dr., St. Joseph, IL 61873
Kresge, Benjamin .....1055 Esplanade, Bronx, NY 10461
Kummer, Fred H. .....5 Forsythe Dr., E. Northport, NY 11731
Kusel, George R. .....600 Lakevue Dr., Willow Grove, PA 19090
Kuznik, Rudolph .....7545 Hitchcock Rd., Youngstown, OH 44512
Langer, Manfred .....2000 Lehigh, Glenview, IL 60025
Langley, Russel W. .....2927 Marion St., Vineland, NJ 08360
Lanning, Charles P. .....340 Quince, Vineland, NJ 08360
Legge, John .....31 Nymark Ave., Willowdale, Ont., Can.
Legge, Ron .....36 Glynn Rd., Ajax, Ont., Can.
Lenzi, David J. .....416 Sycamore St., Elizabethtown, KY 42701
Leslie, Lorne F. .....3 Durie St., Toronto, Ont., Can. M6S 3E5
Leue, James .....7500 Brill Rd., Cincinnati, OH 45243
Lewandowski, Robert A. ....7418 Deep Spring, San Antonio, TX 78238
Lewis, Thomas L. .....5565 E. Evergreen Blvd., Vancouver, WA 98661
Leyden, John .....7506 1st Ave., North Bergen, NJ 07047
Lillie, Don .....495 Willowbrook, Smyrna, GA 30080
Logsdon, Edwin C. .....P. O. Box 803, Keystone Heights, FL 32656
Lord, Edward E. .....2302 McCormick St., Denton, TX 76201
Lorimer, Robert .....1034 W. Iowa, Sunnyvale, CA 94086
Losonsky, John .....2000 Lehigh, Glenview, IL 60025
Luertzing, Paul .....67 Howard St., Vineland, NJ 08360
Lundberg, Alf .....Chemical Center Lunds Univ., S-220 07 Lund, Sweden
Lutter, Eugene .....6243 Brigadoon Dr., Longmont, CO 80501
McCartney, James .....47 Lockwood Rd., Peekskill, NY 10566
McEwen, Frederick J. .....145 Robinson Ave., Newburgh, NY 12550
McKelvey, Thomas J. .....333 Sycamore Ave., Folsom, PA 19033
McQuillan, Robert .....405 West Virginia Ave., Glendora, CA 91740
McVey, Jackson E. .....5610 Doorn Lane, San Jose, CA 95118
Machek, Elco .....228 East 80th St., New York, NY 10021
Macur, Michael .....5 Davies Dr., Wappingers Falls, NY 12590
Maiolatesi, Elmo .....35 Henry Ave., Baltimore, MD 21236
Malloy, F. Joseph .....948 Harvard Rd., Monroeville, PA 15146
Mason, Austin .....6015 Milne Blvd., New Orleans, LA 70124
Medley, Robert G. .....4445 Northern Pike, Monroeville, PA 15146
Meldrum, W. H. .....19 Scriven Blvd., Port Hope, Ont., Can.

Messick, George E. .....370 Paper Mill Rd., Newark, DE 19711
Meyer, Frank J. .....1127 E. Waverly St., Tucson, AZ 85719
Mezynski, Stanley .....1209 N. King Charles Rd., Raleigh, NC 27610
Miller, Robert F. .....492 N. Washington St., Danville, IN 46122
Mistry, Keki P. .....1003 Bedford Rd., East Lansing, MI 48823
Morey, Frederick .....191 Grove St., Bangor, ME 04401
Morris, James F. .....716 Clinton, Evanston, IL 60201
Morgenfruh, Lothar F. .....159 Gary Dr., Trenton, NJ 08690
Motivala, Noshir H. .....10 Baker, Nashua, NH 03060
Nagle, Earl R. .....18 Sky View Dr., Cohoes, NY 12047
Nagy, Bela .....271 Main St., Gladstone, NJ 07934
Nash, Paul H. .....8765 Wakefield Ave., Baton Rouge, LA 70806
Nazzewski, Mathew .....9½ Richmond Lane, Adams, MA 01220
O'Brien, Donald M. .....400 Lone Beech, Monticello, IL 61856
Old, J. H. .....1431 Greenfield, Arcadia, CA 91006
Olson, Harry L. .....3 Crabapple Lane, Trenton, NJ 08638
Panczner, James E. .....1507 Hagley Rd., Toledo, OH 43612
Parillo, Edward .....1363 Dill Rd., South Euclid, OH 44121
Parks, George E. .....2227 Harn Blvd., Clearwater, FL 33516
Perry, David E. .....2904 Sangamon, Champaign, IL 61803
Pinkney, William A. .....Rt. 4 Box 643A, Edgewater, MD 21037
Powell, Edwin A. .....468 Fairway Court, Carneys Point, NJ 08069
Rak, Steve .....1042 West 51st Ave., Vancouver, B.C., Can. V6P 1C3
Reeves, Arthur G. .....7 Westborn Dr., Nashua, NH 03060
Rhoads, Charles .....143 Terwood Rd., Willow Grove, PA 19090
Roark, Roger R. .....594 Pine Dr., Pasadena, MD 21122
Roensch, Arno P. .....1511 Roma NE, Albuquerque, NM 87106
Roman, Paul W. .....57 Everett St., Patchogue, L.I., NY 11772
Russell, Robert L. .....5552 Madison Pike, Independence, KY 41051
Ryan, Lawrence W., Jr. ....155 Derby Rd., Melrose, MA 02176
Ryan, William C. .....NW 350 North St., Pullman, WA 99163
Safferling, Ottmar A. .....86-31 107th St., Richmond Hill, NY 11418
Sagimori, Kyoko H. (Mrs.) ..186 Buckthorn Way, Atherton, CA 94025
Sales, William A. .....2000 Lehigh, Glenview, IL 60025
Saydek, Michael C. .....234 Richards Ave., Piscataway, NJ 08854
Schipmann, Robert H. .....12 Kendall Rd., Kendall Park, NJ 08824
Schlott, Rudolf W. .....32 Highland Down, Shoreham, NY 11786
Schneider, Otto .....46 Kathleen Dr., E., Syosset, NY 11791

Schubert, George W. .....40 Holly Rd., Southampton, PA 18966
Schultz, Elwood C. .....Rt. 3 Box 264, Chapel Hill, NC 27514
Schulze, William J. .....203 Muehl Dr., Lockport, IL 60441
Scribner, Charles .....7 Fairfield Ave., Darien, CT 06820
Searle, David K. .....2012 Courser Dr., Sidney, B.C., Can.
Searle, Randolph H. .....136 Vivion Dr., Aiken, SC 29801
Severn, Peter J. .....221 Jefferson, Chelsea, MI 48118
Shidner, Terry R. .....705 Nokomis Dr., San Jose, CA 95111
Shoup, William H. .....116 West Park Dr., Charlottesville, VA 22901
Sierodzinski, Stanley F. ....7 Royal Cr., Newburgh, NY 12550
Siladi, Louis, Jr. .....23 Chelene Rd., Norwalk, CT 06851
Sites, George A. .....309 Georgetown Ave., Wilmington, DE 19809
Smart, David R. .....420 English St., Greensboro, NC 27401
Smith, Gordon A. .....402 N.E. 31st St., #215, Rochester, MN 55901
Smith, M. Howe .....88 William St., Norwalk, CT 06851
Socolowski, Robert .....11 JFK Circle, North Providence, RI 02904
Sorensen, David J. .....18419 N.W. 41st Ave., Ridgefield, VA 98642
Spaan, Andre W. .....103 Spitbrook Rd. C4, Nashua, NH 03060
Spessard, Lewis C. .....7855 Heritage Dr., Annandale, VA 22003
Squeo, Guy J. .....ONO68 Robbins, Winfield, IL 60190
Stanley, Russell .....130 Acorn Dr., Warminster, PA 18974
Steed, Michael E. .....P. O. Box 301, Statham, GA 30666
Stolp, Siegfried .....51 Quail Rd., Rocky Point, NY 11778
Stone, Charles L. .....P. O. Box 73, Carrboro, NC 27510
Stoop, Donald C. .....9 New Hillcrest Ave., Trenton, NJ 08638
Surdam, Walter E. .....Strawberry Ave., Vineland, NJ 08360
Sward, John A. .....RD7 Box 379A, Carlisle, PA 17013
Swopes, Chester A. .....P. O. Box 115, North Chicago, IL 60064
Teige, Roy W. .....3345 Moraine Dr., Hubertus, WI 53033
Thomas, Paul P. .....1302 Sage Loop, Los Alamos, NM 87544
Tobin, Robert B. .....4229 Sherrod St., Pittsburgh, PA 15201
Trechel, Gene .....32 Pine Brook Rd., Towaco, NJ 07082
Vezina, Leon J., Jr. .....11 Dartmouth St., Nashua, NH 03060
Volk, Rudolph .....10 Greenwich St., Bergenfield, NJ 07621
Wagner, Earl C. .....21 Erie Pl., Nutley, NJ 07110
Walthall, Harry G. .....Box 451, Gloucester Point, VA 23062
Watson, Merrill B. .....10 Woodland Park, Tuscaloosa, AL 35401
Wesanko, J. G. .....Box 22 RR#1, Deep River, Ont., Can.
Willey, Martin J. .....RD#3 Box 377, Schenectady, NY 12306

Williams, Larry R. .....Box 125, Montague, MA 01351
Williams, Vincent G. .....305 E. Maple St., Wenonah, NJ 08090
Wilson, Charles H., Jr. .....P. O. Box 26583, Richmond, VA 23261
Wilson, Robert R. .....6292 Tylersville Rd., West Chester, OH 45069

NON-MEMBERS

Beckett, Ross T. .....7890 Tannenbaum Rd., Colorado Springs, CO 80908
Berlund, Wilton A. .....6415 Oakwood Dr., Oakland, CA 94602
Bonham, Raymond L. .....10051 Lenor Dr., Affton, MO 63123
Cosgrove, George D. .....6768 Hickory Lane, Dublin, CA 94566
Cottee, Roy D. .....667 Portage Ave., Ottawa, Can. K1G 1T2
Holle, Robert .....1708 Maywood, South Euclid, OH 44121
Fukiharu, Shiuichi .....Tokuyama Nikaki Seisakusho, 366-4, Kushigahama-shioda, Tokuyama-shi, Yamaguchi-ken, Japan
Gorman, Robert J., Jr. .....406 Winslow Rd., Oxon Hill, MD 20021
Grant, Leroy .....I.B.M. Corp., 9500 Godwin Dr., Manassas, VA 22110
Kump, William R. .....5908 Coral Sea, Bethesba, MD 20851
Ikeda, Yutaka .....Asahi Seisakusho, 17, Ryugose-machi, Omuta-shi, Fukuoka-ken, Japan
Iwashima, Naoichi .....Nigorikawa Rikakogyo Co., Ltd., 23-14, Sebu-cho, Asahi-ku, Yokohama-shi, Kanagawa-ken, Japan
Kinoshita, Yoshio .....Kinoshita Rikakogyo Co. Ltd., 2-9-14, Mukogaoka, Bunkyo-ku, Tokyo, Japan
Motyka, Phillip .....Vitro Dynamics, Inc., P. O. Box 285, Rockaway, NJ 07866
Payne, James L. .....815 Maude, Mt. View, CA 94041
Richter, Robert J. .....Box 269 Harbison Rd., Valencia, PA 16059
Rodda, Edwin W. .....132 Station St., Herndon, VA 22070
Rutan, Hartford L. .....1719 West St., Concord, CA 94520
Sakai, Ryoji .....Sakai Rikagaku Glass Mfg., 3-26-2, Minamikoiwa, Edogawa-ku, Tokyo, Japan
Schoke, J. A. .....Voltarc Tubes, Inc., 102 Linwood Ave., Fairfield, CT 06430
Schwartz, William C. .....540 E. Laguna Dr., Tempe, AZ 85282
Shimoshita, Takatoshi .....Nichiden Rikagaras Kogyo Co., Ltd., 1874-50, Arakamiyama, Sumiyoshi Higashi-Nada-ku, Kobe-shi Hyogo-ken, Japan
Snidow, Ronald D. .....12025 Donna Ct., NE., Albuquerque, NM 87112
Strohschein, Rudy .....Rt. 1, Box 83, Micanopy, FL 32667
Sugai, Giichi .....Aoba Rikenkogyo Co., Ltd., 1-3-3, Komegabukuro, Sendai-shi, Miyagi-ken, Japan
Takizawa, Hirokatsu .....Asahi Shiyoko Co. Ltd., 5-7-1, Arakawa, Arakawa-ku, Tokyo, Japan