

Proceedings

THE SIXTEENTH SYMPOSIUM
ON THE
ART OF GLASSBLOWING

1971

THE

AMERICAN SCIENTIFIC GLASSBLOWERS SOCIETY

Proceedings
THE SIXTEENTH SYMPOSIUM
ON THE
ART OF GLASSBLOWING

Sponsored by

THE AMERICAN SCIENTIFIC
GLASSBLOWERS SOCIETY

THE PFISTER HOTEL
MILWAUKEE, WISCONSIN

June 30, July 1, 2, 1971

Copyright 1972

THE AMERICAN SCIENTIFIC GLASSBLOWERS SOCIETY
309 Georgetown Avenue, Gwinhurst
Wilmington, Delaware 19809

OFFICERS AND DIRECTORS
OF THE
AMERICAN SCIENTIFIC GLASSBLOWERS SOCIETY
1970-1971

OFFICERS

- President*—JONATHAN W. SECKMAN
Lockheed Missiles & Space Company, Palo Alto, California
- President-Elect*—WILLIAM A. GILHOOLEY
General Electric Research Laboratories, Schenectady, New York
- Secretary*—EARL R. NAGLE
State University of New York, Albany, New York
- Treasurer*—KARL H. WALTHER
Brookhaven National Laboratory, Upton, L. I., New York

SECTIONAL DIRECTORS

J. ALLEN ALEXANDER
CHESTER E. STELMACH
BILLIE E. PAHL
J. H. OLD
ELMO MAIOLATESI
CHARLES E. LANGBEIN
OWEN J. KINGSBURY
ALFRED H. WALROD
GORDON GOOD
JOSEPH WEST
ROLAND WANSER
THEODORE W. BOLAN
HOMER C. S. LAST
CZESLAW DEMINET
HOMER L. HOYT

PAST PRESIDENTS

J. ALLEN ALEXANDER
President Emeritus

KARL H. WALTHER	RICHARD W. POOLE
ARTHUR DOLENGA	WILLIAM E. BARR
ALFRED H. WALROD	CHARLES J. CASSIDY

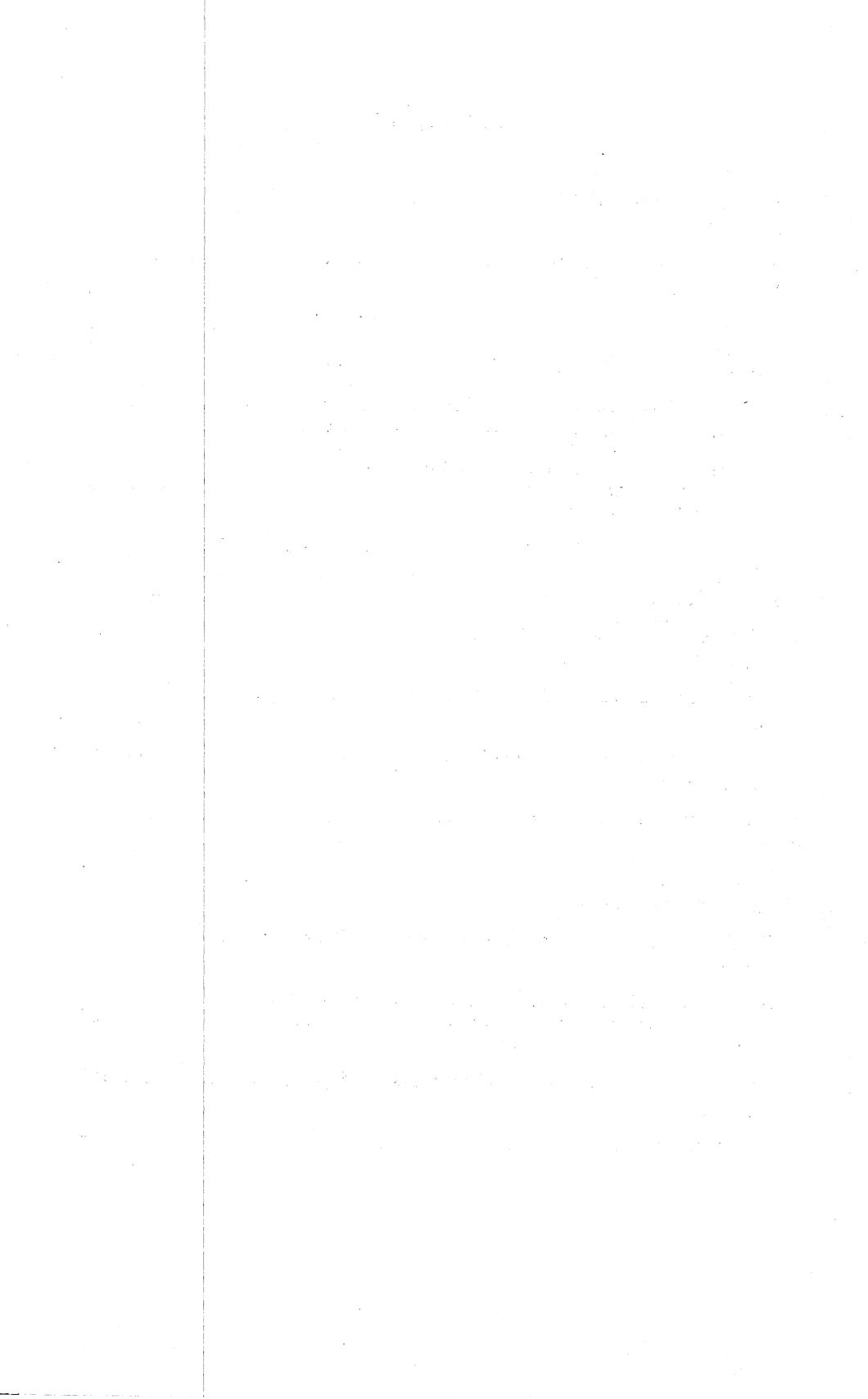
EXECUTIVE SECRETARY

GEORGE A. SITES

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

CONTENTS

By the Sweat of Your Brow	9
Dr. Peter Wargo	
Vitreous Aluminosilicates	12
Joseph W. Malmendier	
The Use and Limitations of Plastics and Elastomers in Glass Laboratory Apparatus	21
D. F. Shriver	
Glass CO ₂ Laser Qualified for Titan IIIc Launch Environment	29
George J. Bergen, Robert F. Harris and Harold E. Walker	
Fabrication of a Micro Columnated Source in a Conductive Coated Lens	42
Dane H. Anderberg	
The Glass High-gain Light Integrating Sphere for Laboratory Investigations of Chemiluminescence	48
Edwin E. Eckberg	
A Neophyte's Novelty Notes	58
Morris Liebson	
Gadgets and Tools for the Glassblower	66
James Morris	
Safety in the Glass Equipment Shop	71
John M. Reynolds	
Vycor and Improvements	80
Carlton T. Bedsole	
Glass in X-Ray Tubes	83
Edward E. Hafkemeyer	
The Engineering Function in Glass-working Operations	92
George H. Batley	
Insulator and Heat Exchanger for Ultra High Vacuum, High Voltage Discharge Chamber	97
W. J. Wheeler	
Thermal Expansion Mismatch and Stress in Seals	107
J. E. Hagy	
In Attendance	118



SYMPOSIUM COMMITTEE

ALFRED H. WALROD, Varian Associates

EDWARD W. METZ, Owens-Illinois, Inc.

WILLIAM A. GILHOOLEY, General Electric Research Labs

BILLIE E. PAHL, The Dow Chemical Company

PROGRAM MODERATORS

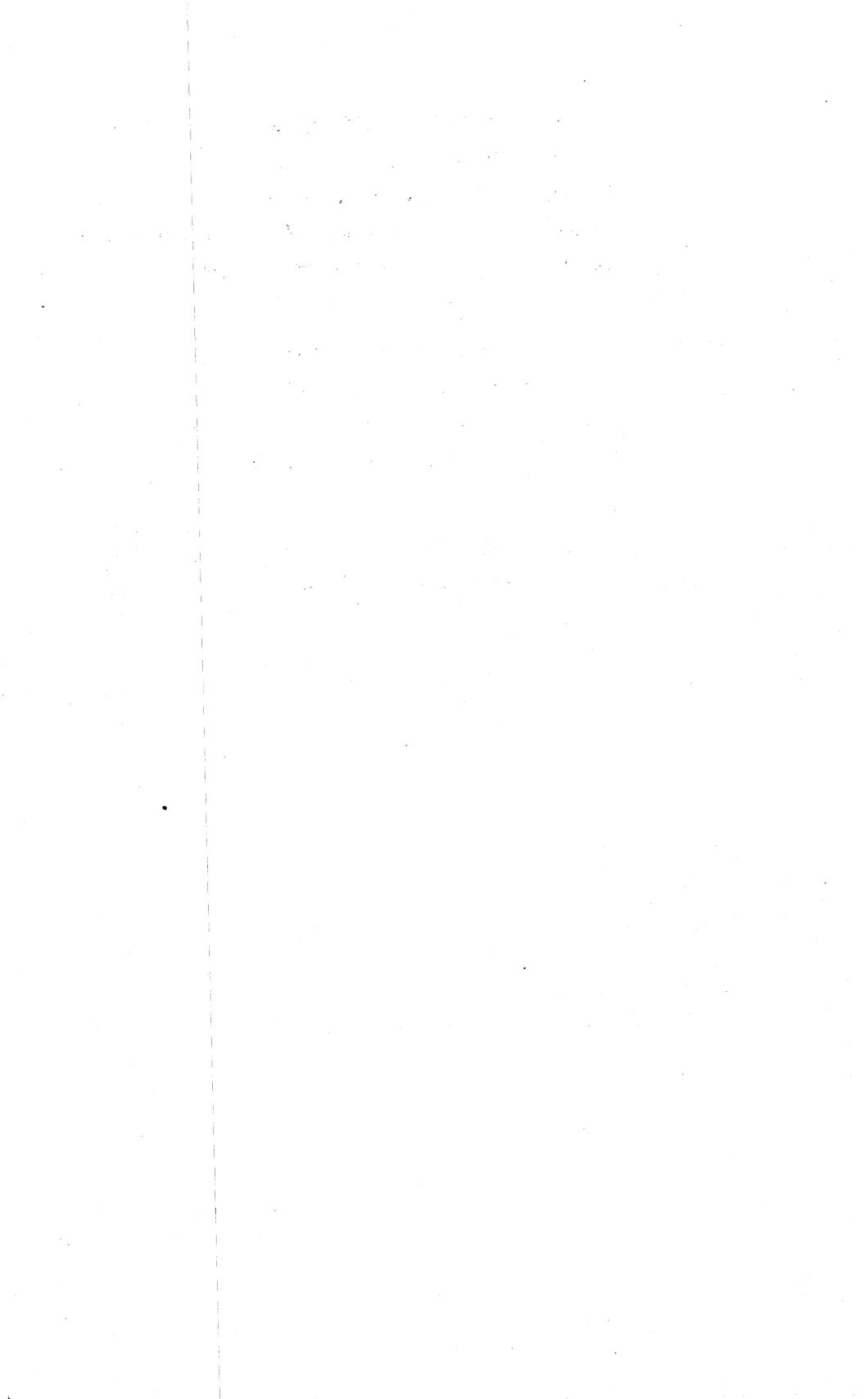
M. HOWE SMITH, Daran Products Inc.

JAMES A. HAGEDORN, University of Illinois

THOMAS J. DOODY, Argonne National Laboratory

REGISTRAR

DOLores I. SITES, The ASGS



BY THE SWEAT OF YOUR BROW

DR. PETER WARGO

Group Executive & Vice President

Management Services Group

Allis-Chalmers Corporation

There seems to be a feeling — an attitude — nowadays that would have one believe the day of the craftsman is long-passed. Don't you believe it. Unless you are willing to admit that the legendary slogan of a Chinese laundry has some merit. That slogan you will remember goes something like this —

“Don't rip your laundry in machines. Let us do it for you by hand.”

In all seriousness, on the surface I am willing to admit that it might seem to be true that craftsmen like glassblowers belong to a rapidly disappearing tribe. For example, the job title of “glassblower” does not appear among the six hundred sixty-seven assignments in the United States Army that the Military feels can lead to civilian careers. Nor for that matter are glassblowers included in Clarence E. Lovejoy's list of one thousand five hundred and fifty occupations he used in his book, “Career and Vocational School Guide.” And even the Bureau of Labor Statistics does not consider glassblowers as a job category. The truth is — there just are not enough glassblowers. The ratio I roughly calculate must be something like one glassblower to every ten thousand people.

Facing the facts squarely tells us that this situation is largely due to a decrease in the need for hand-crafted glass — a decrease that has occurred since the development of machines capable of producing glassware on a mass scale — machines like the Corning ribbon machine. And, of course, glassblowers are not the only ones feeling that type of squeeze.

The emphasis on mechanization has resulted in a decline in the need for many skilled crafts, and as the need declined, very few glassblowers received training. In the United States particularly, almost none of our high schools or vocational schools offer courses in glassblowing. However, I recently found out that at the university level here in Wisconsin, they think it's important enough to offer a course.

Perhaps it is really better that way because it is a demanding art. The techniques that are used, even today, were painstakingly developed by a succession of nameless unsung craftsmen. And this intricate, demanding art was passed down in a very personal way from father to son and from friend to friend. We should never fail on occasions when we get together to pay tribute to them in recognition of their hard-won accomplishments that have made this important work of today possible. And, gentlemen, while some may criticize the effect mechanization has had on craftsmen and on our society, the fact of the matter is that our ability to produce goods and services for each individual in this country is greater now than at any other time in history. Paradoxically, it is this very scientific and technological progress that has given the professional scientific glass-

blower an ever increasing, ever widening perspective and in effect, a new lease on life. Reflect for a moment that next to the transistor, no other scientific development in the past two decades has fired the imagination more than the laser. Initially, you will recall it was thought that the laser would have few practical uses, but today, it is being used in the fields of communications, medicine, surveying, photography, computers, metal-working, testing, tunneling, range finding, and catalytic action, to name a few. As Dr. Thomas Meloy puts it, "The light fantastic has been born!" And — my glassblowing friends — be sure to point out to everyone that scientific glassblowing is an integral part of this "light fantastic" — for glassblowing craftsmanship is absolutely necessary for the laser tube.

Mark Twain once had occasion to write, "The reports of my death are greatly exaggerated". It is my opinion that likewise any forecast predicting the demise of the scientific glassblowing profession is greatly exaggerated.

Not only is the scientific glassblowing profession a vital element in our economic system — an element that is pulling its own oar — but its need to survive as an important contributor to tomorrow's world signals a message that is extremely relevant to today's contemporary culture. The profession of scientific glassblowing demonstrates that a person *can* make it — can achieve a satisfying, worthwhile existence in our present technologically oriented society without the benefit of a college diploma. Please do not misunderstand what I am saying. I am not standing here telling you that a college education is not worthwhile. I just cannot help but suspect that too many people believe that you *must have* some kind of diploma if you are going to get and hold onto a decent job.

This way of thinking has been called, "The Sheepskin Psychosis" by writer, John Keats, and this label seems pretty accurate when you consider all of the parental anxiety, not over education, but primarily concerning jobs. It is this sheepskin psychosis that seems to have permeated our lifestyles, from those who are parents to our Federal Government, and, I believe, that it often leads us to rather ridiculous extremes.

Let me give you an example that comes immediately to mind. One Ohio businessman has stated in writing that he will only hire as drivers of his trucks, applicants that are Ohio State graduates! Now it may be that he is a trustee of the school and wishes to help guarantee jobs, but I submit to you, gentlemen, that if we carry this psychosis to such extremes — that we can, in all seriousness, demand a college diploma for a man to be a truck driver — we are pushing our youth toward a lifestyle that is a practical impossibility.

On the other hand, the fact that a craftsman does not need a Master's or a Doctorate to be an excellent glassblower does not belittle your profession in the least. As one of your group has so well said—

"Glassblowing is an art, and mastery of an art needs courage and an adventurous spirit."

Art also demands from those who take it up a measure of humility and dedication. Moreover, the most spiritually satisfying of all work is handcraft, work done by the hands under the direction of the mind. It is only the craftsman who can enjoy the challenge of his objectives, and the actual task he accomplishes is almost invariably good because he is directing the total formation of his creation. The craftsman does his work in his own way, on his own time, and subject to his own conditions. He alone decides the time it will take to complete the work — and the series of judgments involved in the execution of the work are his and his alone.

Earlier, I compared mechanization and its affect on craftsmen and on glassblowers in particular. There are questions, very important questions, that anyone even remotely concerned with the effects of mechanization must answer. Is man a cog in a machine? Or is he a product produced by machines? Is he subject to forces completely beyond his control? Where is man's freedom? Where is his identity? Will man become nothing but a machine in a physical environment of machines?

It should be perfectly clear to all of us that mass production is here to stay, and mass production means mechanization. So, we must answer these questions. And if we are to answer them cogently, then mechanization must be mastered by human values, instead of mastering human life by traditions which keep us from using the machines we build to dare to do what has never been done before.

Perhaps no other group represents a vocation that demands such mastery and such daring. And there is no doubt in my mind that you realize your social duties and obligations in your contributions to scientific progress.

It has been said, and I think it applies equally well to the glassblower's art, that the drafts of the plowman, the mason, the carpenter, and the smith are among the manual arts that are most closely connected with the life of man.

While the soldier may degenerate into a highwayman and the merchant into an adventurer and the sailor into a pirate, the manual artisan simply cannot change because the task of bringing the laws of nature into line with man's needs, first, of survival and, secondly, of enrichment, is a way of life that brings man very close to happiness.

VITREOUS ALUMINOSILICATES

JOSEPH W. MALMENDIER

Corning Glass Works

Research and Development Laboratories

Corning, New York 14830

ABSTRACT

Generally, aluminosilicate glasses are considered to be those which contain about 15 weight percent or more of Al_2O_3 . Commercial glasses of this type are Corning Codes 1717, 1720, 1723 and 9753 glasses. Schott Supremax, Philips 18, and Kimble EE-2 are other examples. Usually, aluminosilicate glasses of commercial importance also contain alkaline-earth oxides, such as MgO and/or CaO, and small amounts of B_2O_3 . These glasses tend to be more difficult to melt and lampwork than the more familiar borosilicate glasses, of which Corning Code 7740 glass is an example.

This paper will review the applications and advantages of aluminosilicate glasses. The properties of these glasses will be discussed with respect to their compositions and what is known of their structures. Excellent papers dealing with aluminosilicate have been presented at past symposia of this society. They will be summarized, along with more recent information about this useful family of glasses.

INTRODUCTION

Probably man's first contact with aluminosilicate glasses came when he produced fired clay objects. A vitreous aluminosilicate is the bonding agent in such articles and also in porcelain, which was a later development. Early porcelains were composed of clay, sand and feldspar, which is an alkali aluminosilicate mineral.

Alumina was inadvertently added to many antique glasses as a batch impurity or through the process of melting in clay crucibles. Glasses made in Thüringen, Germany, were well known for their superior workability and resistance to devitrification. These properties were imparted by the feldspar contained in the sands of the region that were used to make the glass.¹ The first glass scientist to conduct a systematic search for a means to improve the chemical durability of optical glasses concluded that the alumina contamination from his clay crucibles improved the durability more than any changes in composition that he had made.²

Modern aluminosilicate glasses are generally known for these favorable characteristics, too. Glasses of this type which are of commercial importance are usually more viscous (thermally harder), have higher electrical resistivities, and are less permeable to gases than the more familiar soda-lime, lead and borosilicate glasses. Most of their thermal expansions allow them to be sealed to molybdenum or tungsten. In many cases these glasses were developed for and have found their greatest application in electronic devices.

The following deals with the composition and properties of some commercial vitreous aluminosilicates related to their supposed structures. For comparison, similar information is provided on more familiar glasses.

COMPOSITIONS

Table I gives the approximate composition on a weight percent basis for several Corning glasses. The four glasses to the left of the dashed line, or others of very similar composition, are commonly used by lampworkers. Those glasses to the right of the dashed line are vitreous aluminosilicates. Code 1720 and 1723 glasses may be familiar to lampworkers who have fabricated apparatus for electronic and high-vacuum uses. Code 1710 glass is another Corning glass that has been used in such applications. It is almost identical to Code 1720 glass in composition and properties, therefore it will not be discussed here.

Table I

CORNING CODE		APPROXIMATE COMPOSITIONS							
		0800	0120	7740	7900	1717	1720	1723	9753
WT%	SiO ₂	73	56	81	96	66	59	57	30
	Al ₂ O ₃	1	2	2	0.3	19	19	15	38
	B ₂ O ₃			13	3		5	5	
	Na ₂ O	17	4	4			1		
	K ₂ O		9						
	CaO	5				7	7	10	32
	MgO	4					9	7	
	BaO					8		6	
	PbO		29						

Code 0080 glass is a soda lime glass with fair durability and low cost; it seals to Code 0120 glass and Dumet wire. The principal products manufactured from this glass are fluorescent lamp tubing, incandescent lamp bulbs and vacuum bottles.

Code 0120 glass is a potash soda lead glass noted for its low softening point and long working range. It has high electrical resistivity, and it seals to Dumet alloy and Code 0080 glass. Principal products are electronic bulbs, tubing, lamp and tube stems, and faceplates.

Code 7740 glass is a soda borosilicate glass. It is known for its low thermal expansion and good chemical durability, and is readily lamp-worked. Principal products are chemical ware, oven ware, drain lines, tubing and refractors.

Code 7900 glass is a 96% silica glass. It has excellent heat-shock resistance because of its low thermal expansion. Its high deformation temperature makes it difficult to lampwork. Its main use is for chemical ware. This glass is not melted directly, as are the others in Table I. A glass similar in composition to Code 7740 glass is melted and formed to the desired shape. The product is then heat-treated to cause phase separation. The sodium borate phase is leached out by acid leaving the silica-rich skeletal body intact. The porous body is then consolidated by firing at an elevated temperature. The final product is about 35% smaller by volume than the original article.

Code 1717 glass is an alkaline-earth aluminosilicate that was developed as a lower-cost substitute for high-silica glasses used in various discharge and halogen cycle lamps. Its major features are extremely low alkali content, high strain point, good electrical characteristics, and a thermal expansion in the range of tungsten and molybdenum.

Code 1720 glass is a lime-magnesia aluminosilicate characterized by a high strain point, low expansion, thermal shock resistance, and a high service temperature. The composition is used for the manufacture of stove-top cooking ware and ignition tubes for chemical analyses.

Code 1723 glass is an alkaline-earth aluminosilicate very similar in properties to *Code 1720* glass. However, since it is essentially alkali-free, it has better electrical properties. This has led to its use for resistor cane and electronic tubes. It has also been used for space and aircraft windows.

Code 9753 lime aluminosilicate glass, trademarked *Cortran*[®], is an infrared-transmitting glass. Products are formed by pressing, since the glass crystallizes readily. It is unlikely that a lampworker would be asked to fabricate articles from this aluminosilicate glass.

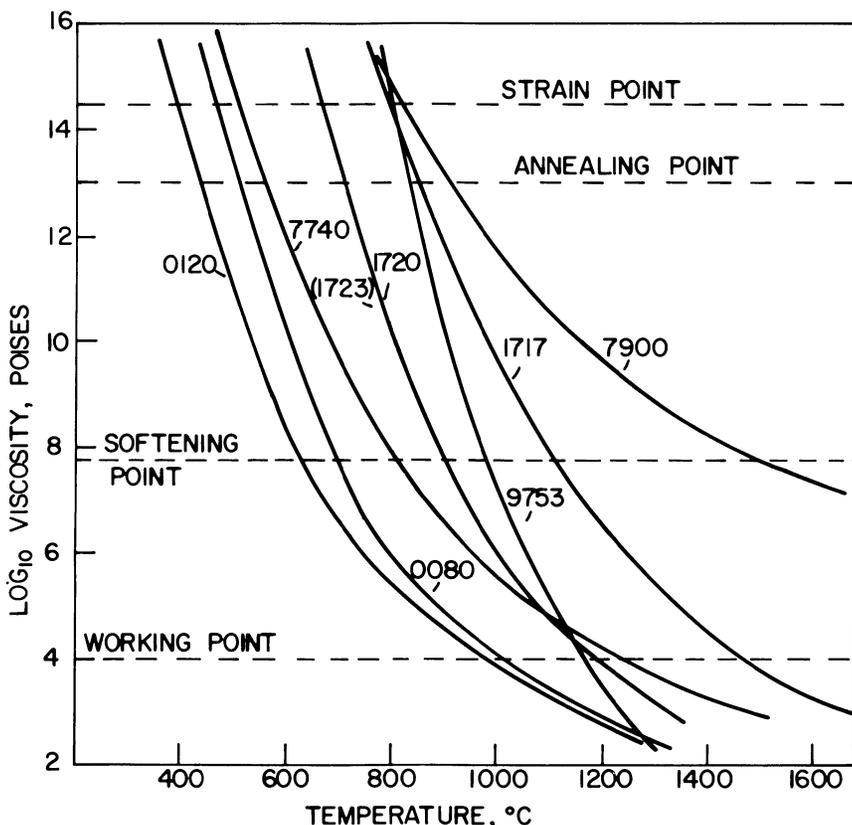
PROPERTIES

Table II lists properties for the eight glasses discussed in this paper. Certain characteristics of the commercial aluminosilicates are apparent when their properties are compared with those of the other glasses in the table. Generally their thermal expansions are near those of molybdenum and tungsten, their softening, annealing and strain points are higher (except compared to *Code 7900* glass), and their elastic (Young's) moduli are higher. The generally better electrical properties possessed by these aluminosilicates are due mainly to the alkaline-earth oxides present. The densities, Poisson's ratios, and the refractive indices show no significant differences between the two groups of glasses.

Table II
PROPERTIES

<i>CORNING CODE</i>	0800	0120	7740	7900	1717	1720	1723	9753
Exp. Coeff., X10 ⁷ /°C (0-300°C)	92	89	33	8	38	42	46	59.5
Softening Pt., °C	695	630	820	1500	~1130	915	908	981
Annealing Pt., °C	510	435	565	>910	854	712	710	832
Strain Pt., °C	470	395	515	>820	799	667	665	800
Density, g/cm ³	2.47	3.05	2.23	2.18	0.22	0.24	0.24	0.28
Young's Modulus X10 ⁶ PSI	10.0	8.6	9.1	10.0	12.0	12.7	12.5	14.3
Poisson's Ratio	0.24	0.22	0.20	0.19	0.22	0.24	0.24	0.28
Log Resistivity (ohm-cm), 350°C	5.1	8.0	6.6	8.1	10.1	9.4	11.3	15.0
Dielectric Constant 1 MHz, 25°C	7.2	6.7	5.1	3.8	6.2	7.2	6.3	8.9
Loss Factor, Percent	6.5	0.8	2.6	0.2	1.2	2.7	1.0	2.2
Refractive Index	1.512	1.560	1.474	1.458	1.524	1.53	1.545	1.602

The first three figures show the manner in which viscosity, resistivity and gas permeability vary with the temperature for the glasses under discussion. These properties are the most important in the applications for which modern aluminosilicates are used.

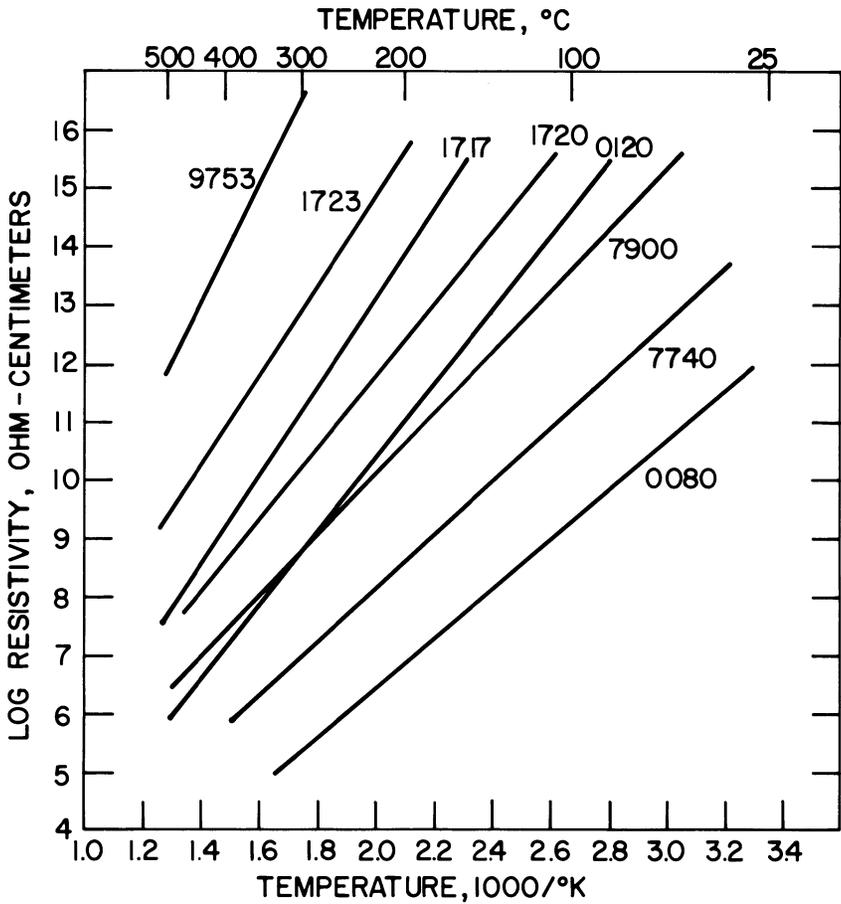


VISCOSITY VERSUS TEMPERATURE

Figure 1

Viscosity Versus Temperature

The viscosity versus temperature curve for Code 1723 glass is approximately identical to that for Code 1720, but a few degrees lower, as indicated by the viscosity temperatures given in Table II. At this point it should be noted that the slope of the viscosity-temperature curves for the aluminosilicate glasses are steeper. This is of great practical advantage in the manufacture of glasses. For even though aluminosilicates have annealing and strain points near those of high-silica glasses, they can be melted as easily as borosilicate glasses like Code 7740 glass. This is especially evident in the case of Code 9753 glass.



DC VOLUME RESISTIVITY VERSUS TEMPERATURE

Figure 2

DC Volume Resistivity Versus Temperature

Referring now to Figure 2, showing the resistivity vs temperature curves, it can be seen that the aluminosilicates have resistivities greater than that of Code 7900 glass. Code 7740 and 0800 glasses contain mobile alkali cations, which results in their resistivities being less than that of the high-silica Code 7900 glass. Code 0120 glass also contains substantial amounts of alkali, but their effect is offset by the large amount of highly immobile lead cations.

Alkali cations such as sodium and potassium possess a single positive charge, while cations such as lead and the alkaline-earth cations magnesium, calcium and barium are all doubly charged. These charges are

balanced by the negative charges of oxygen anions that are part of the glass network. Cations of silicon, boron and sometimes aluminum are also network members and are separated from one another by oxygen anions.

Glass volume consists essentially of oxygen anions and interstitial voids. Usually singly and doubly charged cations are located in the voids. Sodium and potassium are respectively about the same sizes as calcium and barium. However, they can move between voids much more readily under the influence of an electrical potential than the corresponding alkaline-earth cations, since they are only bound in the glass structure by a single charge. This explains the fact that Code 7740 and 0080 glasses have lower dc volume resistivities than that of Code 790 glass, which is essentially free of interstitial cations, and of the other glasses that contain mainly interstitial cations which are doubly charged.

Table III gives the glass compositions on a molecular basis, which can provide a better understanding of the relative numbers of the various cations present in a glass. For example, Code 0120 glass contains network member cations and interstitial modifier cations in the ratio of 79:36, with 10 of the modifier cations being doubly charged. Of course, these cationic ratios can be converted to cation percentages, which permits easier comparisons between glasses.

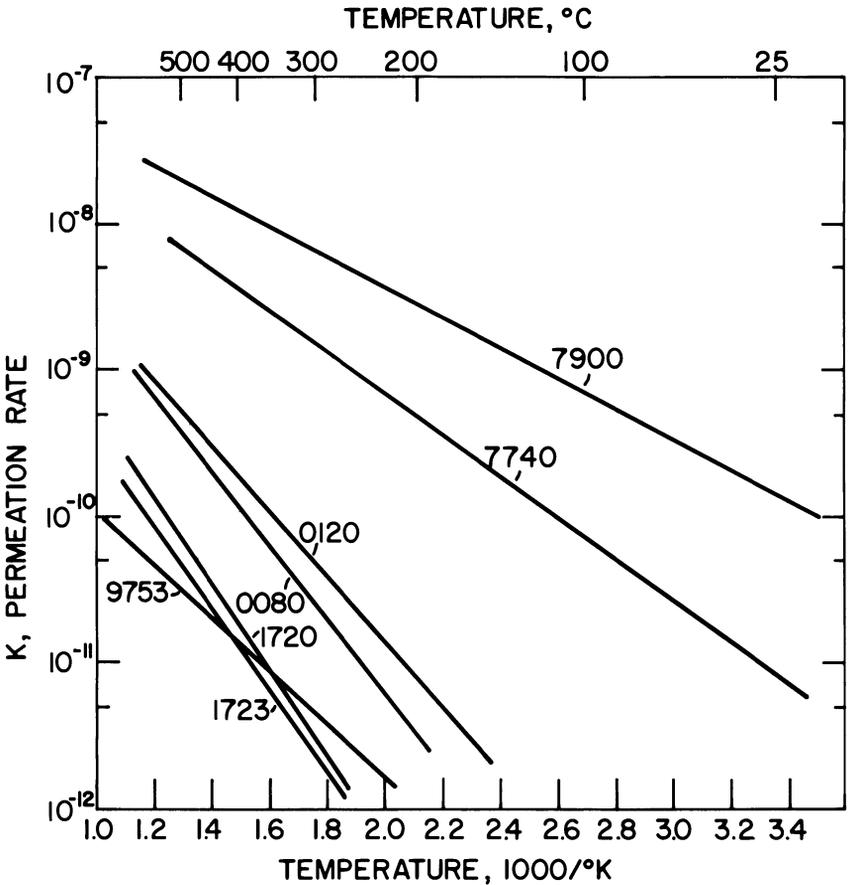
Table III
APPROXIMATE COMPOSITIONS — MOLECULAR PERCENT

<i>CORNING CODE</i>		0800	0120	7740	7900	1717	1720	1723	9753
Network Members	{ SiO ₂	72	75	83	97	75	61	61	35
	{ Al ₂ O ₃	1	2	1	0.2	13	12	9	26
	{ B ₂ O ₃			12	3		4	5	
1+ Cations	{ Na ₂ O	16	5	4			1		
	{ K ₂ O		8						
2+ Cations	{ CaO	5				9	8	11	39
	{ MgO	6					14	11	
	{ BaO					3		3	
	{ PbO		10						

The last property-versus-temperature plot, Figure 3, shows the helium permeability of the various glasses, except for Code 1717 glass, which has not been measured. Code 7900 glass has the highest permeability because it has no interstitial cations to block the passage of gas. The network cation-to-modifier cation ratios of the other nonaluminosilicate glasses decrease in the order: Code 7740, Code 0120, Code 0080. This is reflected in the relative decreases in permeability of these glasses as the network voids are filled with modifier cations.

The alkaline-earth-containing aluminosilicate glasses have more complex structures. Table III lists aluminum as a network member, but this is not always true. When aluminum does participate in the network, it is surrounded by four oxygen anions, and when it does not, it is usually

surrounded by six oxygens. Both types of aluminum coordinations can be found in a single glass. The relative amounts of each type are dependent upon the number and nature of the modifier cations also present. Generally, as the fraction of alkali and alkaline-earth modifiers increases, the number of four-coordinated aluminum cations also increases.



HELIUM PERMEATION VERSUS TEMPERATURE

Figure 3

Helium Permeation Versus Temperature

On the other hand, with increasing amounts of modifier cations, the boron which is also present in some of the glasses either increases its oxygen coordination from three to four, as shown in Figure 4, or remains coordinated to three oxygens. At least one of the three oxygens associated

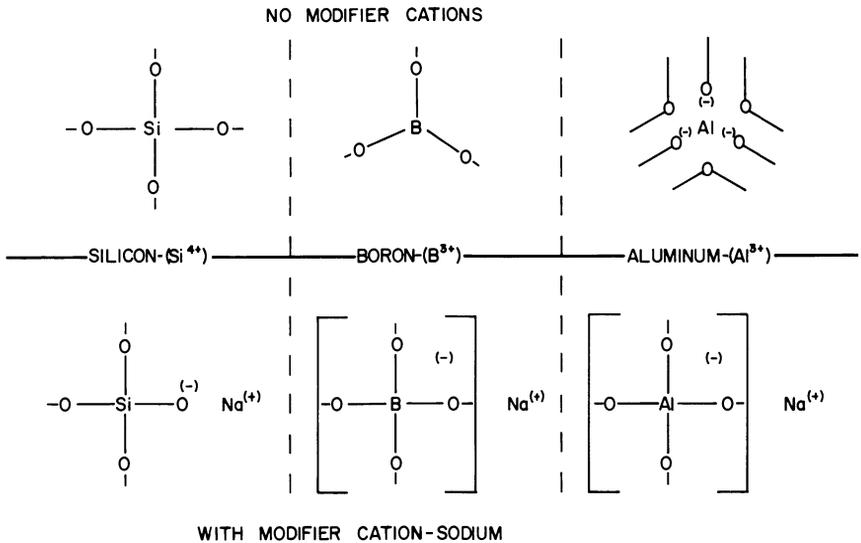


Figure 4

Two-Dimensional Schematic Structures

with such a modifier-influenced boron would not be shared by another network cation. It would be similar to the negatively charged oxygen shown in modified silicon structure. The exact nature and preferential order of coordination changes of the network-dwelling cations is the subject of much research today. However, it is quite evident from the helium permeability measurements that the alkaline-earth-modified aluminosilicate glasses of commercial importance have rather compact structures.

RELATED LITERATURE

Many excellent papers dealing with vitreous aluminosilicates have been given at previous symposia of this society. Most of the above helium permeability data was presented in 1962 by V. O. Altemose of Corning Glass Works.³ His more recent measurement of Code 9753 glass is also given herein with his kind permission. At the 5th symposium, Dietz⁴ described the development of the Kimble aluminosilicate glass EE-2. The following year, Bishop⁵ compared EE-2 to other Kimble glasses in much the same manner as this paper compares various Corning glasses. Elmer and Nordberg⁶ of Corning Glass Works discussed aluminosilicates as part of their paper on high-temperature glasses in 1961.

DeMaria⁷ also discussed high-temperature glasses, including aluminosilicates. His emphasis was on lampworking of such glasses. Four papers that dealt specifically with the sealing and/or lampworking of aluminosilicates were presented by Barbour, et al., Doule⁹, Mara¹⁰, and DnNah¹¹.

All of these papers mention that cleanliness is of utmost importance when working with aluminosilicates. Barbour, et al.⁸ gave the most detailed information on working with such glasses. After careful cleaning, they recommend preheating with gas-air and lampworking with hydrogen-oxygen. Aluminosilicates have a tendency to reboil in an oxygen-lean flame. However, Corning Code 1723 and Kimble EE-2 glasses are susceptible to reduction, so the mixture of hydrogen and oxygen can be quite critical.

Recently Yoldas¹² published a paper on the coexistence of four- and six-coordinated aluminum cations in glass. Other descriptions of glass structures, compositions, and properties can be found in excellent books by Rawson¹³ and by Bartenev¹⁴. The most informative source about commercial glasses is Volf's¹⁵ book.

REFERENCES

1. Weyl, W. A., Marboe, E. C., *The Constitution of Glasses, A Dynamic Interpretation*, Vol II, Part I, pp. 508-509, (John Wiley & Sons, New York).
2. Fraunhofer, J. Von, *Gesammelte Schriften*, Edited by E. Lommel, Munchen, 1888.
3. Altemose, V. O., "Gas Permeation Through Glass", Proc. Symp. Am. Sci. Glassblowers Soc., (7th) 1962.
4. Dietz, E. D., "Development of a High Temperature Electronic Glass", *ibid.* (5th) 1960.
5. Bishop, F. L., "Aluminosilicate Glasses", *ibid.* (6th) 1961.
6. Elmer, T. H., Nordberg, M. E., "High Temperature Glasses", *ibid.* (6th) 1961.
7. DeMaria, V. C., "High Temperature Glasses in Electronics", *ibid.* (10th) 1965.
8. Barbour, J. F., et al., "Techniques for Laboratory Working of Aluminosilicate Glasses", *ibid.* (6th) 1961.
9. Doyle, E. M., "The Use of Aluminosilicate Glass in Microwave Devices", *ibid.* (8th) 1963.
10. Mara, J., "Aluminosilicate Glasses in Electronics", *ibid.* (10th) 1965.
11. Dunah, R. E., "Modular Vacuum Tube Envelope Fabrication", *ibid.* (10th) 1965.
12. Yoldas, B. E., "The Nature of the Coexistence of Four and Six Coordinated Al³⁺ in Glass", *Phys. and Chem. of Glasses*, 12 (1), pp. 28-32, (1971).
13. Rawson, H., *Inorganic Glass Forming Systems*, (Academic Press, London and New York) 1967.
14. Bartenev, G. M., *The Structure and Mechanical Properties of Inorganic Glasses*, (Wolters-Noordhoff, Groningen), 1970.
15. Volf, M. B., *Technical Glasses*, (Pitman, London) 1961.

THE USE AND LIMITATIONS OF PLASTICS AND ELASTOMERS IN GLASS LABORATORY APPARATUS

D. F. SHRIVER

Department of Chemistry, and
Materials Research Center, Evanston, Illinois 60201

INTRODUCTION

Recently a variety of new materials, mostly plastics and elastomers, have been widely incorporated into laboratory ware. In some cases plastics have completely displaced glass, but most applications involve versatile combinations of glass with the new materials. The temperature and chemical resistance of most plastics and elastomers are more limited than those of glass. As a result, it is necessary to consider these properties in the selection of materials for an apparatus. To aid in this selection, this paper outlines the properties of some commonly encountered plastics and elastomers and illustrates their incorporation into apparatus for high-vacuum and inert-atmosphere work.

PROPERTIES OF MATERIALS

Solvent Resistance. One of the principal advantages of o-ring equipped joints and valves over the older greased variety, is their relative freedom from attack by solvents. Even though elastomers are generally less sensitive to organic liquids than are stopcock greases, the elastomers do exhibit some sensitivity which is dependent on the nature of the elastomer and the liquid. Therefore, it is necessary to choose the correct o-ring material for each application. Unfortunately, many equipment manufacturers offer only one type of o-ring material (usually Viton®) with the result that many o-ring failures occur in the laboratory which could have been easily avoided. For example Viton® o-rings disintegrate when exposed for a few days to common polar organic liquids such as acetone, pyridine and ether.

In contact with an organic liquid, most elastomers will swell due to absorption of the liquid. An acceptable o-ring material will swell only a few percent in which case its function is usually not impaired. However, a material which swells greatly is generally not satisfactory. Swelling not only causes failure due to the dimensional change but as shown in Figure 1, a large degree of swelling generally leads to a drastic decrease in strength. It is not generally meaningful to place exact numbers on the extent of swelling for a given elastomer because swelling is a function of the degree of cross linking, the nature of other materials in the rubber, and the temperature. In general, swelling increases with increased temperature during the exposure and with decreased cross linking in the elastomer. Fortunately, in most laboratory applications a qualitative assessment of the degree of swelling such as is given in Table I is all that is required to establish the best o-ring material.

It will be noted in Table I that two o-ring materials, Viton® and ethylene propylene rubber (EPR), have quite different solvent sensitivity.

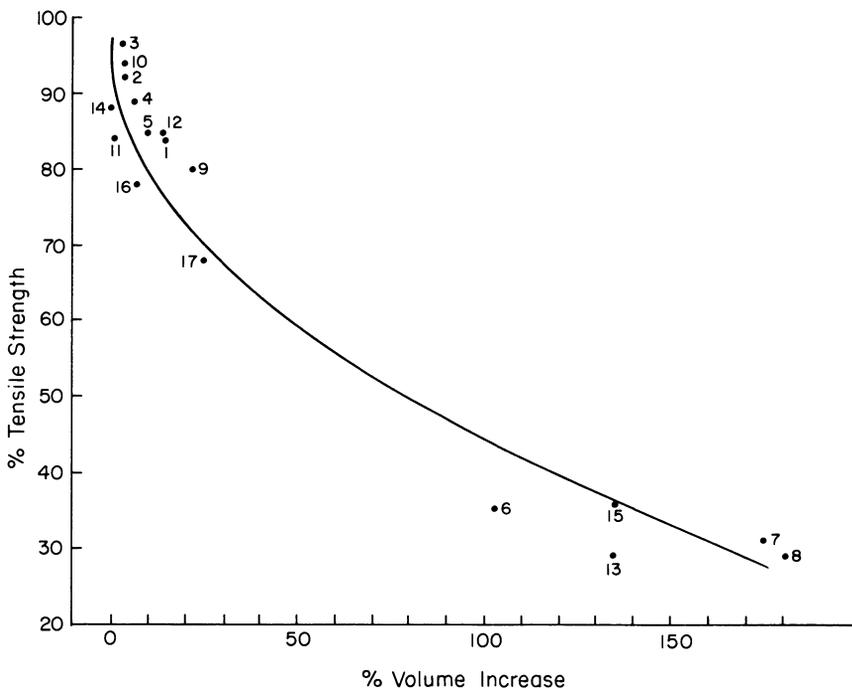


Figure 1

Percent tensile strength retained vs. percent volume increase for ethylene propylene rubber exposed for 28 days at 24°C to the following liquids: 1 glacial acetic acid, 2 acetone, 3 ammonia (30% in water), 4 aniline, 5 benzaldehyde, 6 benzene, 7 carbon tetrachloride, 8 cyclohexane, 9 cyclohexanone, 10 dibutyl phthalate, 11 dimethyl formamide, 12 dioxane, 13 o-dichlorobenzene, 14 ethyl alcohol, 15 n-hexane, 16 hydrochloric acid (37%), 17 sulfuric acid (conc.). Data from "Nordel An Engineering Profile," E. I. du Pont de Nemours and Co.

EPR is resistant to polar oxygen and nitrogen containing organic compounds such as pyridine, acetone and nitromethane. However, EPR swells excessively in contact with nonpolar organic solvents such as carbon tetrachloride and benzene. By contrast, Viton® is generally satisfactory in contact with nonpolar and slightly-polar organic liquids but as mentioned earlier it is usually destroyed by polar oxygen and nitrogen containing organic compounds. The good-fair-poor ratings given in Table I do not fully illustrate the extreme sensitivity of Viton® to the polar organics. Even though many of the other rubbers are given poor ratings, unlike Viton®, they may be used with the polar organics. For example, ethylene propylene rubber and silicone rubber are usually serviceable with ether. Thus, a stock of Viton® and EPR o-rings will meet most laboratory needs. Other combinations of o-rings might be considered for specific applications. In any case, it cannot be stressed too strongly that there is at present no universal elastomer for use in laboratory ware.

Table I
 PROPERTIES OF SOME ELASTOMERS^{1, 2}

Elastomers	Solvent Sensitivity				Chemical Reactivity				Approximate ³ Useable Temp. Range, °C
	Hydrocarbons	Polar Organics	Alcohols	Chlorinated Hydrocarbons	Strong Oxidants	Strong Reductants	Covalent Halides	Conc. Sulfuric	
Butyl Rubber	P, P	F,G,P,F,F	G, F	P	P, F	—	G, P	F	-54, 150°
Buna N	G, P	P,P,P,P,P	G, G	F to P	P, P	—	G, P	P	-54, 120°
EPR	P, P	F,G,P,F,F	G, F	P	P, F	G	G, P	F	-54, 150°
Chloroprene	F, P	P,P,F,P,P	G, F	P	P, P	—	P,P	P	-54, 150°
Silicone	P, P	P,P,P,F,P	F, F	P	P, F	—	F, -	P	-80, 230°
Viton®	G, G	P,P,P,P,P	G, G	G	G, G	P	G, G	G	-50, 300°

n-hexane
benzene

pyridine
acetone
diethyl ether
ethyl acetate
nitro methane

methyl alcohol
n-hexyl alcohol

bromine
50% chromic acid

Na in liq. NH₃

stannic chloride
sulfur chloride

1. G = Good, F = Fair, P = Poor All ratings are for room temperature
2. Adapted in part from data supplied by E. I. du Pont and by Parker Seal Co.
3. For the dry o-ring in air. The low temperature limits are only attainable with special formulations.

Plastics vary widely in their sensitivity to solvents. In the most favorable cases, the tendency to swell is much less than for elastomers. Many of the plastics commonly used in the laboratory (Teflon[®], Kel-F[®], polyethylene and polypropylene) are composed of polymeric molecules with no cross-linkings. These thermoplastic materials exhibit well defined melting ranges and except for Teflon[®], may be dissolved in solvents at elevated temperatures. Teflon[®] has exceptionally good resistance to solvents. Another polyhalogenated hydrocarbon plastic, Kel-F[®], is somewhat more affected by solvents, particularly halogenated hydrocarbons at elevated temperatures. Polyethylene and polypropylene swell appreciably in the presence of many organic solvents at or slightly above room temperature.

Resistance to Chemical Attack. In general, the halogenated hydrocarbon plastics and elastomers (Teflon[®], Kel-F[®], and Viton[®]) are resistant to strong oxidizing agents and strong acids, such as nitric acid. However, very strong reducing agents such as sodium solutions in liquid ammonia or organometallics attack the halogenated materials. The opposite order of reactivity is found for the hydrocarbon materials, polyethylene, polypropylene, and EPR which are attacked by strong oxidizing agents but resistant to strong reducing agents. As with solvent sensitivity, Viton[®] and EPR have quite different chemical reactivity which makes it advantageous to have available in the laboratory o-rings made from both types of rubber.

Resistance to Low and High Temperatures. At low temperatures most elastomers and plastics become brittle and are not serviceable. For an elastomer, the brittleness temperature decreases with a decreased degree of cross-linking and it may also be influenced by other substances used in compounding the finished product. Similarly, plasticizers which are sometimes added to thermoplastic materials will generally reduce the brittleness temperature. Teflon[®] is unique in not exhibiting the brittleness phenomenon. The upper temperature limit may be influenced by many factors such as, air oxidation, loss of strength and/or melting, and volatilization. Even before the loss of strength is appreciable the material is often subject to deform under a constant load at elevated temperatures. When this happens with an o-ring, failure of the seal may result. An approximate indication of temperature limitations is given in Table I. Teflon[®] is the most heat resistant of the commonly used plastics while Viton[®] and silicon rubber are the most heat resistant of the common elastomers.

EQUIPMENT DESIGN

O-Ring Sealed Joints. We may divide the o-ring sealed joints into two categories: (1) the flange-sealed type, Figure 2, where the primary seal occurs by compressing an o-ring between two flanges, and (2) the annular-sealed type, Figure 3, for which the primary seal is made by compressing an o-ring around the outside of a tube. The flange type is most familiar, however the annular type finds application when freedom of rotation is desired as with the vacuum line filtration apparatus illustrated in Figure

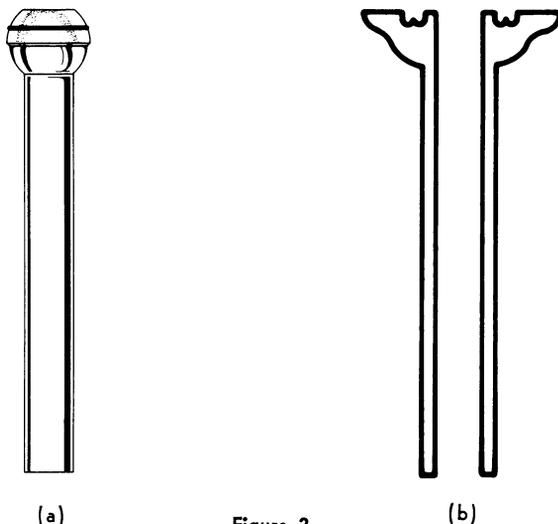


Figure 2

FLANGE SEALED O-RING JOINTS

(a) O-ring in ball joint. This design affords flexibility in the assembly of equipment. (Reproduced by permission of the copyright owner, Kontes Glass Co.)

(b) Urry's variation on the standard o-ring joint. The ridge in the o-ring groove assures a vacuum tight seal. (Reproduced by permission of the copyright owner McGraw Hill Book Co. From D. F. Shriver, "The Manipulation of Air-Sensitive Compounds," McGraw Hill Book Co., New York, N. Y. 1969.)

4. Another appealing feature of the annular sealed o-joint is that it may be used on practically any type of tubing of the correct diameter. Thus the union of dissimilar materials is easily accomplished. The recently introduced Kontes vacuum connector, Figure 3b, makes it possible to attach an nmr photochemical reactor tube to a vacuum system without blowing a joint on the tube. Closely related to these annular-sealed o-joints is the swage type fitting, such as Swagelok®, which may be used on glass tubing of the proper diameter providing a Teflon® front ferrule is employed.

Stopcocks and Valves. The Teflon® plug stopcock has become a standard laboratory item for non-vacuum applications. When high-vacuum or air-free performance is required, the standard stopcock design is not adequate. However, Teflon®-in-glass valves have been developed for these applications. The design and performance of these valves vary considerably. The valve design illustrated in Figure 5 gives a very good high vacuum performance and by means of a Teflon® wiper on the stem, the o-ring is protected from solvents. With all Teflon®-in-glass valves the Teflon® and/or o-ring seal on the stem is major source of leakage and valve failure. This factor is important in choosing the best orientation for the valve. For example, the visible-uv cell shown in Figure 6 has the seat of the valve oriented toward the cell, making long term observation of the cell contents

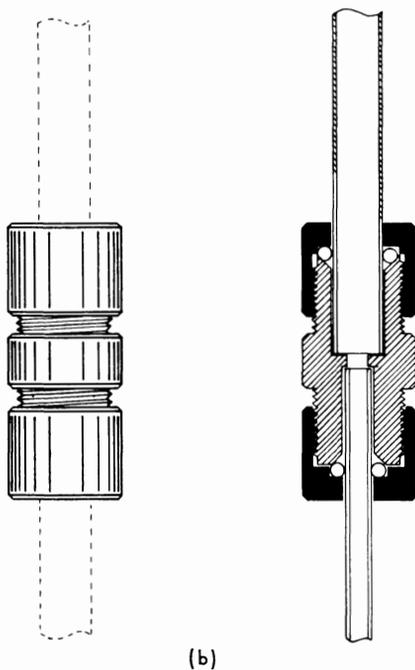
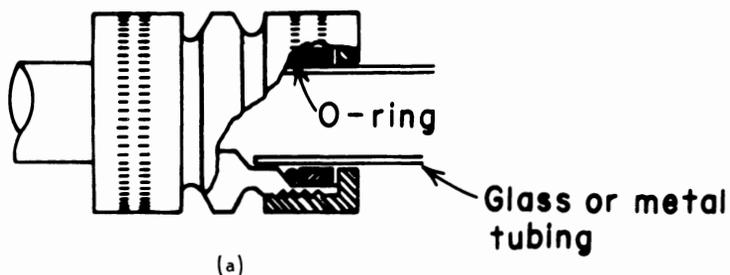


Figure 3

ANNULAR SEALED O-JOINTS

(a) Cajon® Joint. This union is constructed of metal. (Reproduced by permission of the copyright owner McGraw Hill Book Co. From D. F. Shriver, "The Manipulation of Air-Sensitive Compounds," McGraw Hill Book Co., New York, N. Y. 1969.)

(b) Kontes Vacuum Connector. This item is constructed from plastic. (Reproduced by permission of the copyright owner, Kontes Glass Co.)

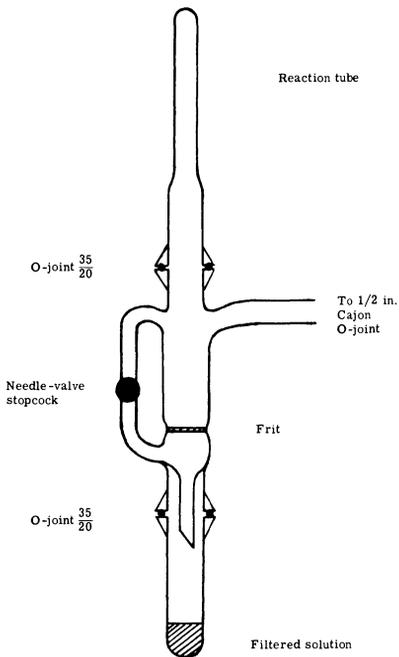


Figure 4

VACUUM LINE FILTRATION APPARATUS

The Cajon[®] joint allows rotation of the apparatus so that a filtration may be carried out following a reaction. This is a grease-free version of an apparatus described by Parry, Schultz and Gerardo, *J. Am. Chem. Soc.*, 80, 1 (1958). (From G. Kubas, Ph.D. Thesis, Northwestern University, 1971).

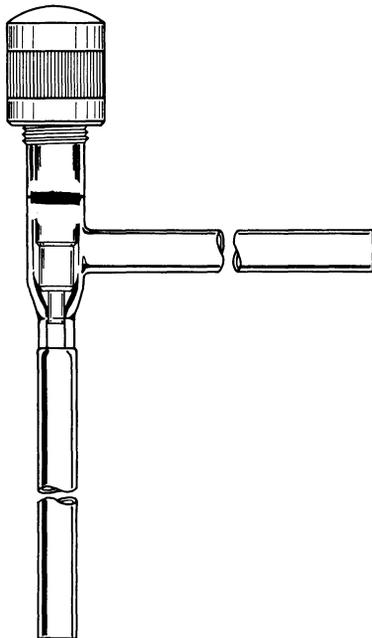


Figure 5

KONTES TEFLON[®]-IN-GLASS VALVE

The o-ring is protected from solvents by an adjacent Teflon[®] wiper machined on the stem. (Reproduced by permission of the copyright owner, Kontes Glass Co.)

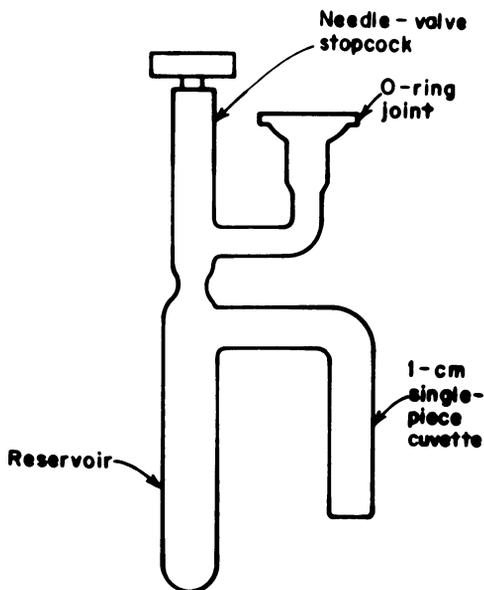


Figure 6

VACUUM LINE VISIBLE-UV CELL

(Reproduced with permission of the copyright owner, McGraw Hill Book Co. From D. F. Shriver, "The Manipulation of Air-Sensitive Compounds," McGraw Hill Book Co., New York, N. Y. 1969.)

possible with little chance of contamination by air. Swelling and potential breakdown of the o-ring is also minimized by this particular orientation of the valve.

CONCLUSION

Considerable improvement in laboratory glassware has been made in recent years by extensive use of plastics and elastomers in joints and valves. In general, the elastomer-to-glass or plastic-to-glass seal is much more resistant to solvent and heat than the standard greased joint. However, solvent sensitivity and chemical reactivity of polymeric materials do place significant limitations on their use and it is necessary to match the elastomer or plastic to the chemicals being handled. Frequently it is also possible to design the apparatus so that failure due to leakage past o-rings and exposure of o-rings to solvents is minimized.

GLASS CO₂ LASER QUALIFIED FOR TITAN IIIc LAUNCH ENVIRONMENT

GEORGE J. BERGEN*

ROBERT F. HARRIS

HAROLD E. WALKER

Goddard Space Flight Center
Greenbelt, Maryland

ABSTRACT

Carbon dioxide laser communication systems appear to be an attractive alternate to microwave and millimeter wave systems for a number of space applications.¹

With this application as a goal, I shall describe techniques that have been developed at GSFC and applied in the fabrication of a space flight qualified glass CO₂ gas laser tube. The space flight environment requires that particular attention be given to: dissipation of thermal energy; ruggedness for survival of the launch environment; packaging constraints and of course, operational life time.

It is recognized that other laser tube materials, such as beryllium oxide, are suitable alternatives to glass. However, space qualified glass tubes have been constructed which successfully survived launch² environmental tests. It is important therefore, to address this area of technology because unique material processing facilities or techniques are not required.

This paper details techniques used to fabricate a glass CO₂ laser tubes qualified for launch² environment tests.

INTRODUCTION

First, let us address the problem of dissipating thermal energy. The low thermal conductivity of glass is a definite draw back in this area. The carbon dioxide laser under consideration requires approximately 25 watts of input power and yields one watt of power in the coherent output. Therefore, approximately 24 watts of thermal energy must be efficiently channeled from the laser plasma through the glass laser wall into a suitable heat sink.

Secondly, the problem of designing a glass laser tube capable of surviving a launch environment is examined. The problem addressed here involved holding the tube in a manner such that it would survive the launch environment but at the same time the thermal path interface meets the above requirements. Also, interfaces intrinsic to the tube itself must be solved. Of critical importance is the electrode to glass interface and the associated graded seal to the laser tube.

THERMAL AND VIBRATIONAL MODEL

The thermal problem was examined in the following manner. A thermal model of the laser tube was designed and fabricated using "pyrex" (7740) glass for preliminary cooling and vibration tests (Fig. 1).

*Presented by George Bergen

THERMAL AND VIBRATIONAL MODEL

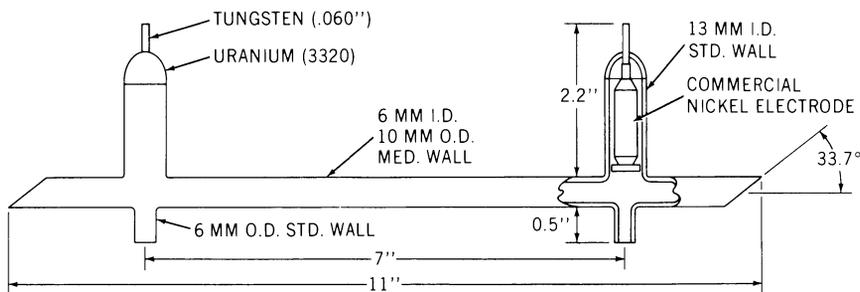


Figure 1

Thermal and Vibrational Model

The tube consists of two commercial nickel electrodes sealed onto a 5MM I.D. heavy wall tube body. A 33.7° angle was ground at both ends to accommodate two sodium chloride windows. Inlet and outlet ports were provided so the tube could be attached to a flow system in these early vacuum processing tests.

A metal heat sink was then fabricated to accommodate the completed glass laser. The spacecraft is designed with provisions for a heat sink which maintains a 20°C temperature $\pm 15^\circ$. The method of cooling the laser simulates this condition. Using water as a coolant, on a spacecraft, is impractical because of the Zero-G environment, complications arising by using anti-freeze and a variety of other difficulties. Efficient heat dissipation using conductive cooling can only be accomplished by intimate contact of the two bodies. Indium wire was wrapped around the laser body in order to achieve this contact between the tube bore and the metal heat sink. Thermal contact is achieved by compressing the indium between the matching configurations of the heat sink. (Fig. 2, Fig. 3).

The thermal model was then processed to ensure that it was a functioning laser tube. In order to simulate the spacecraft heat sink, a temperature controlled water bath was circulated through the metal housing for cooling purposes. The results of these tests indicated that the pyrex-indium-metal heat sink interface provides an adequate thermal conduction path.

The laser package was ready for vibration tests. These tests are necessary for qualification of hardware and/or scientific instruments for survival of the launch environment. Details of the vibration specifications are given in Tables I and II. The laser must be capable of satisfactory performance in accordance with the detailed specification while being subjected to sinusoidal sweeps at test level III as specified in Table I. The laser must also be capable of satisfactory operational performance after being subjected to sinusoidal sweeps and random vibration at test level

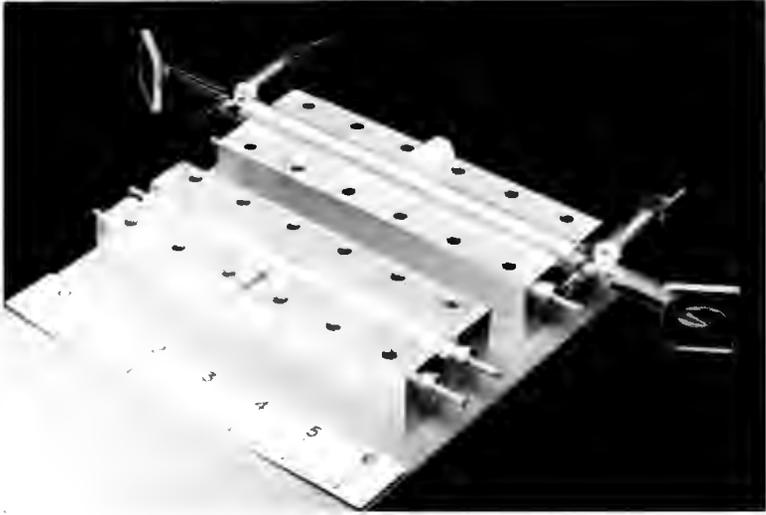


Figure 2
Model in Split Heat Sink

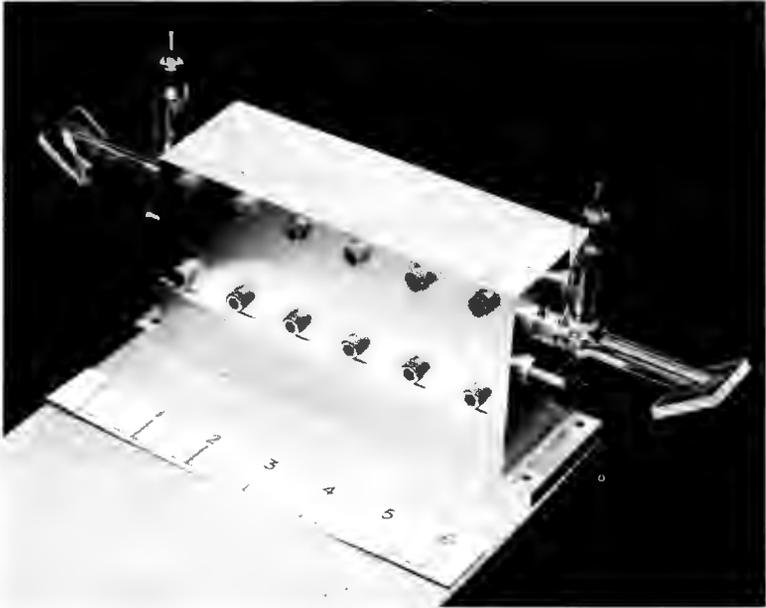


Figure 3
Model in Assembled Heat Sink

Table I
SINUSOIDAL VIBRATION

<u>Test Level</u>	<u>Axis</u>	<u>Frequency (Hz)</u>	<u>Input Level</u>	<u>Sweep Rate</u>	<u>Test Time</u>
Design Quali- fication Test I (Equipment Nonoperating)	Thrust and Lateral (Z-Z, X-X, and Y-Y)	5 to 22	0.5-inch double amplitude constant displacement	2 octaves/ minute	13.0 minutes (total)
		22 to 200	12.0 g (0-to-peak)		
		200 to 2000	5.0 g (0-to-peak)		
Flight & Ground Acceptance Test II (Equipment Nonoperating)	Thrust and Lateral (Z-Z, X-X, and Y-Y)	5 to 18	0.5-inch double amplitude constant displacement	4 octaves/ minute	6.5 minutes (total)
		18 to 200	8.0 g (0-to-peak)		
		200 to 2000	3.3 g (0-to-peak)		
Flight & Ground Acceptance Test III (Equipment Operating)	Thrust and Lateral (Z-Z, X-X, and Y-Y)		Velocity (cm/sec,rms)		
		20	3.9×10^{-2}		
		100	7.8×10^{-3}		
		200	3.9×10^{-3}		
		400	2.0×10^{-3}		
		600	1.3×10^{-3}		
800	1.0×10^{-3}				
1000	7.8×10^{-4}				

I or II as specified in Table I and II. The vibration is applied in each direction along the three orthogonal axes, with one direction being parallel to the thrust axis.

The thermal model passed these vibration tests. The program proceeded toward the development of a flight package based on the fact that

Table II
RANDOM VIBRATION

<u>Test Level</u>	<u>Axis</u>	<u>Frequency (Hz)</u>	<u>PSD Level (g²/Hz)</u>	<u>Overall Acceleration (g-rms)</u>	<u>Test Time</u>
Design Quali- fication Test I (Equipment Nonoperating)	Thrust and Lateral (Z-Z, X-X, and Y-Y)	20 to 250 250 to 2000	0.0010 to 0.16 increas- ing from 20 Hz at the rate of 6 db/octave 0.16	17.0	4.0 minutes each axis (Total 12 minutes)
Flight & Ground Acceptance Test II (Equipment Nonoperating)	Thrust and Lateral (Z-Z, X-X, and Y-Y)	20 to 250 250 to 2000	0.00046 to 0.07 increas- ing from 20 Hz at the rate of 6 db/octave 0.07	11.3	2.0 minutes each axis (Total 6 minutes)

the thermal interface was also suitable as a holding technique for the environment of launch provided precision jugging arrangements are applied throughout the laser tube build-up.

The choice of indium wire as a thermal bond was abandoned because stock glass tubing does not provide the precision tolerance necessary to ensure intimate contact between the heat sink and laser tube. Also, if the selection of glass was not precise, the compression necessary for thermal

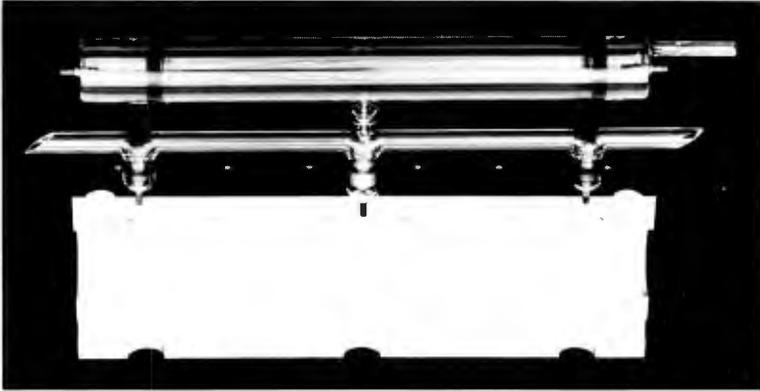


Figure 4
Single Reservoir Proto-Type

contact would result in a fractured laser tube. Solithane®, a poly-urethane material manufactured by Thiokol Chemical Corporation, was used between the tube and heat sink. This material was selected because it is a space qualified potting material with low outgassing characteristics. The addition of aluminum powder ensures adequate thermal contact and conductivity.

A variety of proto-type tubes were fabricated and tested, but because of electrode configuration or improper glass design, were rejected. For example, the tube pictured in Fig. 4 was unable to withstand the vibration tests because of the confinement of the graded seals around the electrodes. This particular tube was also deemed undesirable because of the high silhouette and its single gas reservoir.

In the design and fabrication of the final flight package, several criteria were established as a result of the previous failures. These were; simplification of the tube design and electrode configuration; ruggedness and a low silhouette to accommodate a compact space package.

FLIGHT TUBE CONFIGURATION

The entire tube is constructed of heavy wall (2MM) pyrex tubing. Uranium (3320) sealing glass is used to eliminate multi-grading glass sealing.

Tungsten was selected for the anode material because of its favorable expansion properties when sealed into uranium glass. A radius is ground on the end of the tungsten to insure a uniform discharge and elimination of hot spots. (Fig. 5).

The cathode is a combination of two metals, as illustrated in Fig. 6. The rod used in the sealing area is tungsten and the cathode configuration is pure nickle because of its favorable electrical characteristics. The cath-

ANODE ASSEMBLY

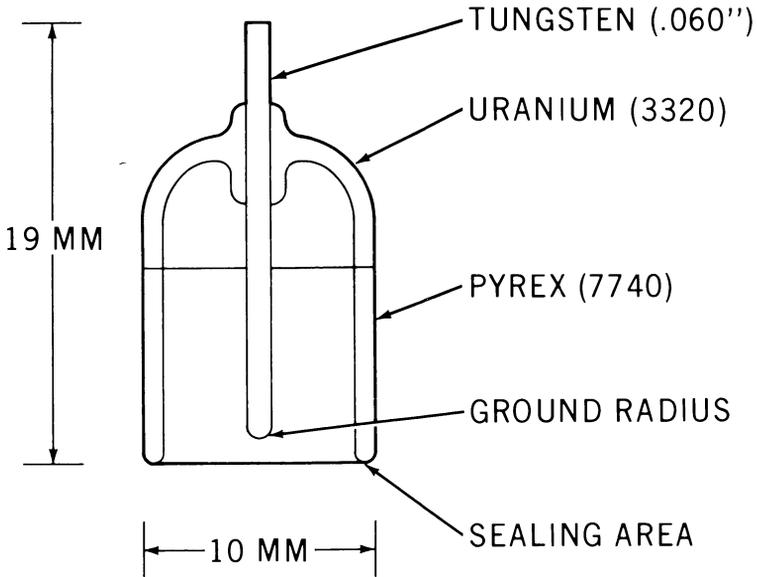


Figure 5
Anode Assembly

ode is designed in a spherical shape so as to confine sputtering products. The glass envelope includes a re-entrant ring seal to keep the plasma discharge confined to the inside of the cathode.

The tube is provided with two gas reservoirs connected by a cross section and sealed to the tube body directly under the cathode assembly. This is advantageous because it increases the gas capacity of the laser by a factor of eight and provides a low silhouette to the entire assembly.

A metal holder was designed for alignment of the cathode to the anodes and sealing of the reservoirs to the tube body. (Fig. 7).

The tube is oven annealed at a temperature of 560°C. The tungsten anodes were cleaned electrolytically in a ten percent (10%) solution of sodium hydroxide. The oxide in the nickel cathode assembly was removed in a hot solution of twenty percent (20%) hydrochloric acid. It is rinsed with a solution of ammonium carbonate to neutralize any remaining acid and flushed several times with distilled water.

The tube is then placed back into the alignment fixture and a metal plate attached to the upper portion (Fig. 8) in preparation for grinding the angles.

CATHODE ASSEMBLY

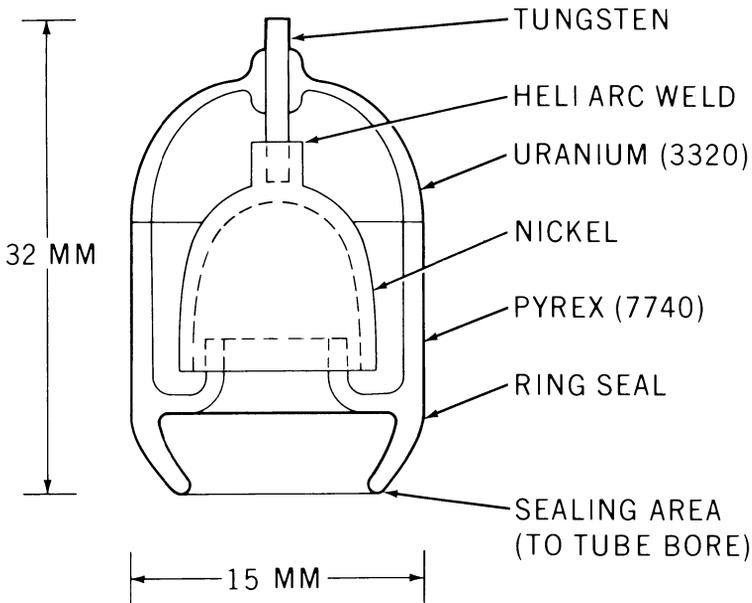


Figure 6
Cathode Assembly

Sealing wax is added between the electrode area and the metal holder to ensure against any vibration breakage during the grinding of the angles. The angles could be ground parallel to each other on pigeon-toed to any given degree by the addition of the top plate to the fixture. It was discovered by using this precision holder that the electrode balance of the two anodes with respect to the cathode was within one percent (Fig. 9).

The metal heat sink was re-designed to accommodate the new laser tube profile. It also provided an easy attachment to the communication package (Fig. 10, Fig 11).

The assembly pictured in (Fig. 12) successfully survived the sinusoidal and random vibration tests and was deemed qualified for Titan III C launch environment. A mold release agent was added to the heat sink wall for removal of the glass components for critical examination. Upon careful scrutiny, the glass laser was found to be free from any damage.

The laser package was reassembled and mounted on the communication flight unit as shown. (Fig 13, Fig. 14).

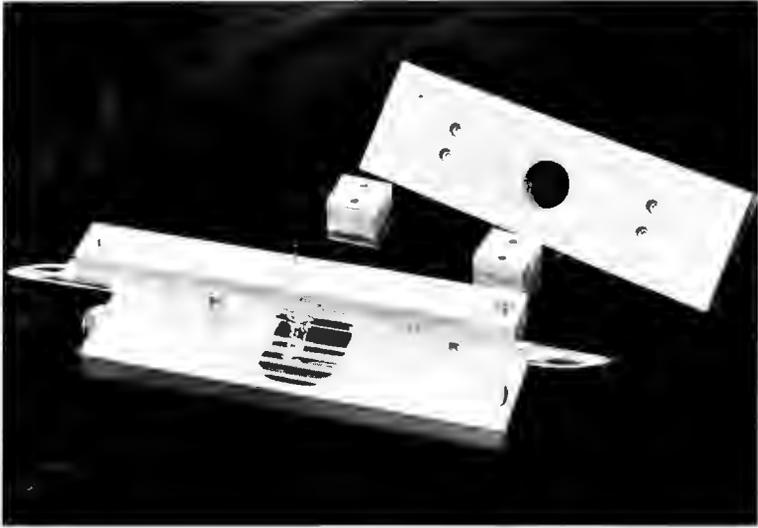


Figure 7
Alignment and Sealing Fixture



Figure 8
Fixture with Angle Grinding Plates



Figure 9
Laser Tube

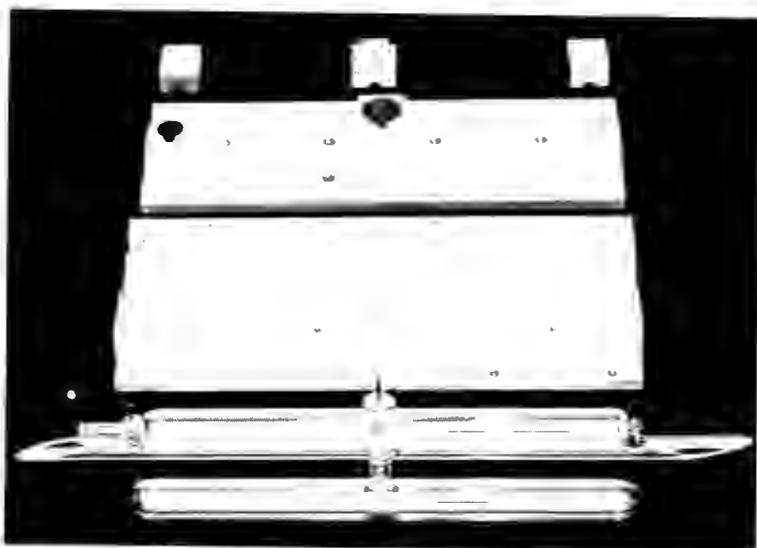


Figure 10
Laser and Heat Sink

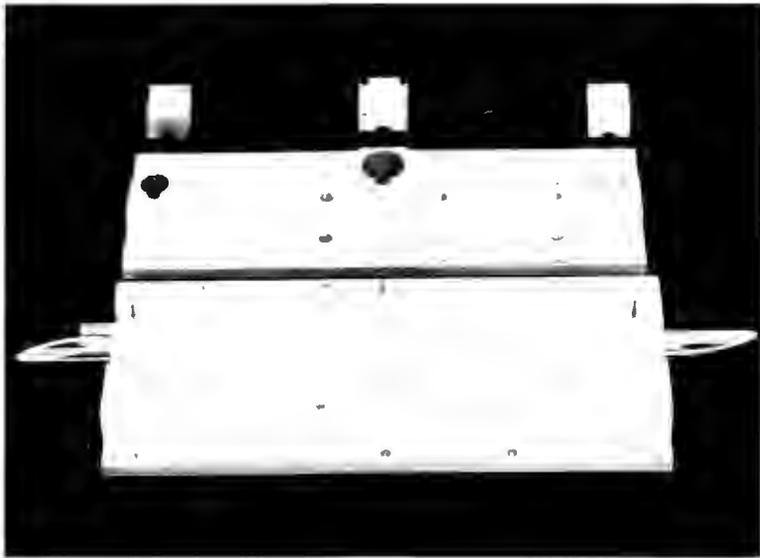


Figure 11
Laser Inside Heat Sink

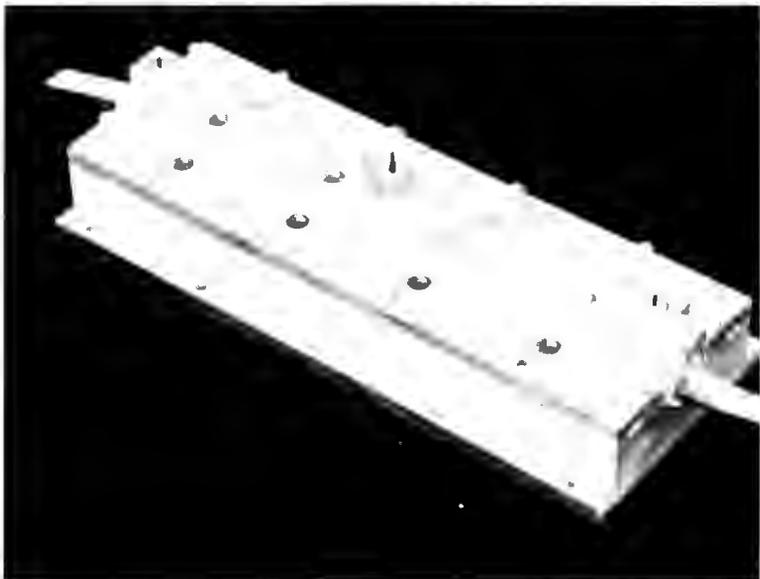


Figure 12
Launch Environment Test Package



Figure 13
Communication Package



Figure 14
Communication Package

CONCLUSIONS

Techniques have been developed which demonstrated the feasibility of fabricating passively (conduction) cooled glass carbon dioxide laser tubes which will survive launch² environmental tests. The success of these techniques relies on the use of precision alinement fixtures throughout the laser tube build up. Reproducibility of the requisite laser dimensional accuracies assures repeatability in the mechanical mounting of the tube as well as the thermal interface. Additional benefits are realized in terms of electrical balance between the electrodes and versatility in grinding precision Brewster angles. It is also noted that multi-graded seals should be avoided.

At this time, these tubes are undergoing life tests to evaluate vacuum processing procedures and window sealing techniques.

Acknowledgements

The authors wish to express their sincere appreciation to Woodrow W. Poland, Ernest C. Woodhouse, Howard N. Murray, and Anthony M. Votta for their helpful assistance in completing this experiment. Thanks are also due to John H. McElroy for his advice and suggestions.

REFERENCES

1. Carbon Dioxide Laser Systems for Space Communications. John H. McElroy. Presented at 1970 International Conference on Communications, San Francisco, Calif. — June 8-10, 1970.
2. Launch environment tests refers to the Titan III C specifications as outlined in Tables I and II.

FABRICATION OF A MICRO COLUMNATED SOURCE IN A CONDUCTIVE COATED LENS

DANE H. ANDERBERG
University of California
Lawrence Radiation Laboratory
Berkeley, California

Dr. Luis Alvarez, leader of Group "A" physics, predicted a potential usefulness of this tool in many phases of research which prompted the presentation of this paper. Hopefully, by reviewing briefly our earlier attempts, some of you may be able to avoid a few of the negative attempts made by us. In this case, fabrication difficulties were inversely proportional to the size of the research package.

In a study of the space resolution obtainable with a liquid xenon wire chamber, it was necessary to fabricate a well-columnated source of alpha particles. The single-wire chamber served its purpose of proving the feasibility of getting a readable signal of a particle passing through the liquid gas ionizing medium. The next problem was to duplicate this phenomenon with multiple wires or conductors in very close proximity to each other still getting individual wire read-out.

In low energy spark chambers for the purpose of tracking particles, this is possible as the conductors can be far enough apart to prevent high voltage breakdown as the path of the particles deviates enough that it is easily discernible. In the case of high energy particles, the path deviates very little so the conductors must be very closely positioned to detect any change.

To prove this step, conductors as small as one tenth micron positioned parallel with one another, spaced at 0.001" center to center was needed. This has been accomplished by mounting them on a suitable substraight. To prove resolution between wires it was necessary to have a micro source narrow enough to manually scan the conductors without overlapping. The lens which also served as the cathode of the liquid xenon test chamber. visually the position of the source in relation to the conductors and correlate this with the events taking place electronically.

The source holder was to be the flat surface of a microscope objective lens which also served as the cathode of the liquid xenon test chamber. Figure 1 shows the lens in shape and size. The size of the slot and type of coating will be covered later. Fabrication consisted of four basic steps: (1) Selecting optically inactive quartz and grinding and polishing it into a $\frac{3}{8}$ " diameter sphere with a facet on one side. (2) Coating the faceted half of the sphere with a conductive coating. (3) Grinding a slot in the center of the facet. (4) Preparing a radioactive source and securing the source into the slot.

The quartz sphere was ground and polished to a tolerance of within 0.0001" of the stated size with the facet leaving a lens thickness of 0.305" which is the focal point.

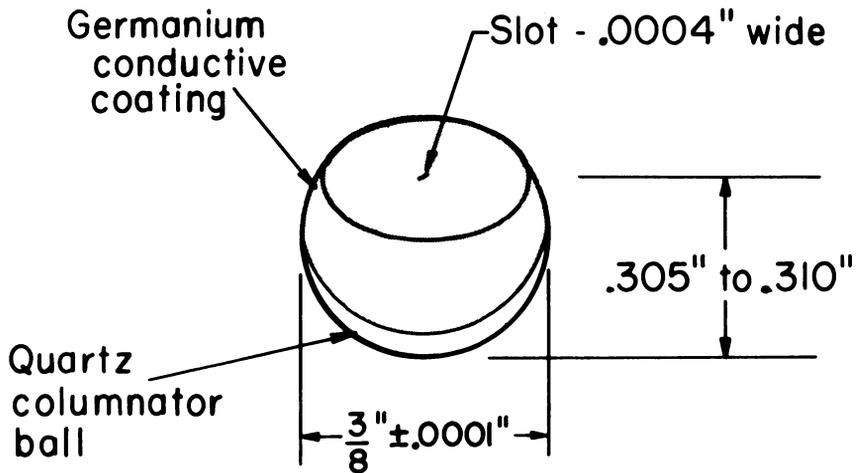


FIGURE 1

Next step was to coat the faceted half of the ball with the conductive film. To accomplish this, a half-sphere depression was cut in a graphite plate that would act as a holder and mask for the ball during the coating operation. The ball was then heated to a temperature of approximately 500°C in a furnace and stannous chloride vapor blown onto it leaving a coating of 500 to 1,000 ohms per square. Thus far the job is straight forward and mechanical in approach.

The ultra-sonic drill seemed to possess the qualities required to produce the slot or recepticle in the facet of the sphere. The initial shape of the desired slot was one of 0.015 inch long, 0.001 inch wide and a uniform depth of 0.001 inch . A razor blade of proper length was prepared as the forming tool and with diamond lapping compound as the abrasive, a skilled operator was able to produce several slots successfully.

We then vacuum deposited silver onto the bottom of the slot and put polonium 210 on top of the silver using aqueous polonium salt, but were not able to eliminate polonium contamination of the surface of the objective because it is highly reactive with the tin oxide coating and could not be removed without also removing the coating itself, so a solid metal wire was chosen as a new radioactive source carrier.

This meant a deeper slot whose sides would be able to columnate the emission, and of a shape that would secure the wire by wedging. The configuration of this slot allowed some extra length inasmuch that the emission length would now be controlled by the length of the "hot" wire, but a width of 0.015 inch to 0.002 inch and a depth of 0.003 inch to 0.004 inch were still quite critical to the success of the idea. In addition to size change, we discovered that the ultra-sonic drilled slots would not retain a wedged wire so a new approach to slot forming was in order.

A new cutter was made with a very thin cutting edge with the angles as acute as possible. A hard steel disc with the edge honed to a knife edge and the physical strength of the steel would provide the strength for the



FIGURE 2

Quartz columnator ball

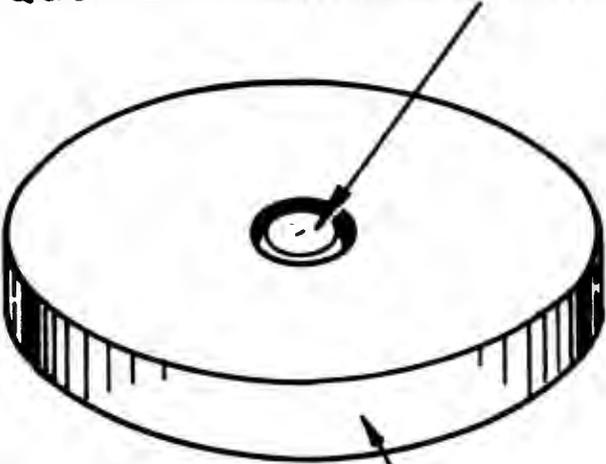


FIGURE 3

Steel holder

edge of the tool. Number 8,000 grit diamond grinding compound was applied to the wheel and slots were quickly formed but considerable chipping occurred due to a slight wobble and uneven thickness of the cutting edge. Re-evaluation was again in order.

Finally it was discovered that brass possessed the strength requirements. This proved to be an easy material to machine to the shape profile of a 15° included angle. When machined in place, this technique provided the accuracy of the cutting tool required and a simple base on which to mount a pivoted plate for the glass ball holder, along with a dial indicator to judge the depth of tool penetration. The 8,000 grit was used on this wheel also, but with the imbedding qualities for grit in brass the cutting action was clean and well defined. We are convinced that a wide selection of slot sizes and shapes can be predictably made. Figure 2 shows one of our first slots cut in this manner.

We left the ball mounted in the holding device as a means for easier handling during the hot wire imbedding process as shown in Figure 3. From previous experiences we learned that it's wise to mask any exposed area of the ball that we did not want radioactive contamination on. The exposed area of the ball was completely masked leaving only the slot that would receive the wire. Gummed plastic tape was used as the masking material. Available commercial tapes were all too thick so we made the masking tape using half-mil Saranwrap and deposited gum on it by dissolving the gum from cellophane tape in xylene and pouring the mixture onto the plastic leaving a very thin film.

The wire was prepared by plating it with Americium repeatedly, followed each time with a high-temperature bake. The wire was 0.001" diameter platinum, coated about ten mm in length until the total count reached 3×10^5 cpm. Ball, wire, microscope and manipulation tools were then placed in an open front hood and personnel were protected with gloves, apron and respirator for possible contamination control.

A portion of the hot wire approximately $1/32$ " long was cut off and carefully moved to the ball. Plastic tipped tools were used to manipulate the wire into the slot. The wire was then pressed into the slot with a dulled razor blade until it had wedged securely. This entire last operation was done with the aid of the microscope. Figure 4 shows the simple wedging assembly using magnets as a guide. Figure 5 shows the wire as it was handled in the most recent efforts by sticking it to the masking tape prior to cutting and then using the tape to move and place the wire. Bottom view indicates the wire in wedged position.

The ball and holder at this point were turned over and tapped firmly on a hard surface, blown with an air nozzle at 100 lbs. pressure and cooled repeatedly to liquid air temperature to make sure the wire was securely lodged in place. The masking was then removed from the face of the ball and the ball removed from the holder by heating carefully on a hot plate until the bonding wax had softened. Remaining wax was removed from the ball with solvents taking care that no foreign material would get in the slot and cover the wire.

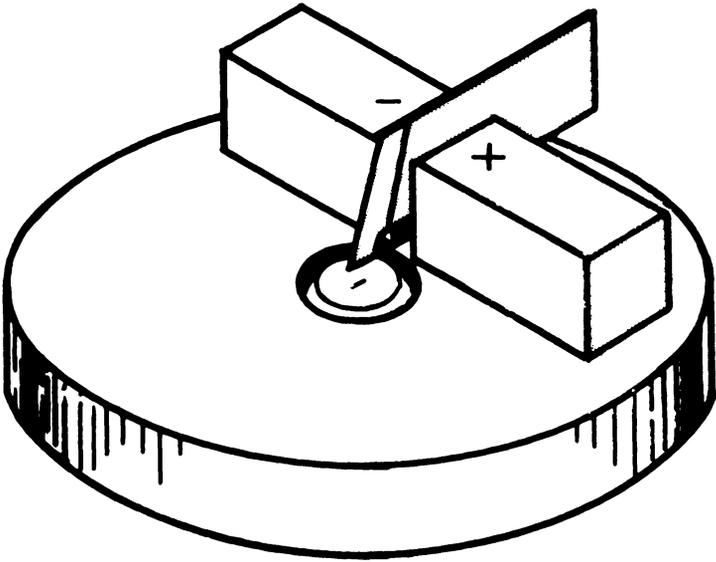


FIGURE 4

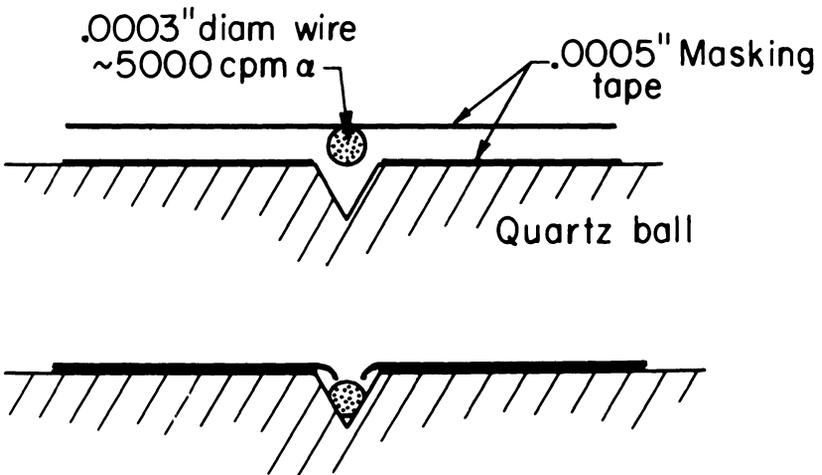


FIGURE 5

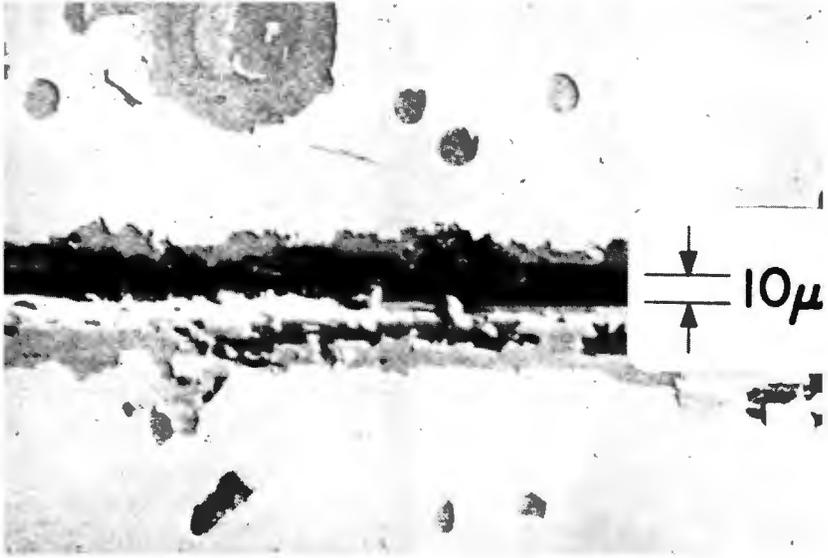


FIGURE 6

Our feeling of accomplishment turned out to be rather short lived. Although the physicists were delighted, they said now that we have proven the possibility, they wanted another source half that size, which, incidentally has been done. Figure 6 shows a slot with the wire in place. The poor appearing surface indicates the abuse on the coating in spite of the masking.

The same basic plan was followed for a lens with a 0.004" wide slot with a 0.003" diameter wire lodged into it. The only difference is the difficulty in manipulating the wire. It is highly subject to static charge which makes it do tricks such as standing on end and clinging to anything that touches it. It was at this point that the thin masking became important as the wire could become hopelessly entangled in the mass of gum protruding from the edge of the 0.001" mylar tape originally used for masking. This smaller source when finished yielded about 5,000 counts per minute of alpha.

Currently we are experimenting with different depth slots as to their effects on columnating the alpha, and have changed the conductive coating to germanium deposited by vacuum evaporation. The germanium is also a very soft film and makes masking even more imperative.

Present interest in this tool is not restricted to the research of high-energy particle tracking, but has interested medical research as a micro-scanning X-Ray type of tool. Potentially the source can be mounted in many different ways to adapt to direct computer read-out scanning operations.

THE GLASS HIGH-GAIN LIGHT INTEGRATING SPHERE FOR LABORATORY INVESTIGATIONS OF CHEMILUMINESCENCE

EDWIN E. ECKBERG

Air Force Cambridge Research Laboratories
L. G. Hanscom Field
Bedford, Massachusetts

ABSTRACT

The development of glass high-gain light integrating spheres for the study of chemiluminescence is described in this brief paper. Their purpose and usefulness in the research of chemiluminescence is covered, in part. However, emphasis is given to the glassblowing problems involved, and especially to the scientific glassblower's technique in the actual fabrication of the glass integrating sphere. The very critical importance of a constant monitoring of zonal glass temperatures during the glasswork is pointed out. The time periods involve many long hours. The internal specular thin-film deposition is summarized since it is a vital requirement in order to attain the optical "high-gain" desired. One successful method of providing special "windows" is included. The window materials are selected for their particular wavelength range of transmission for the chemiluminescent light signal output under study. Obviously, this transmission is extended beyond that of the parent glass of the light integrating sphere; or, beyond the visible spectrum. (e.g. either the ultra-violet and/or the infrared, inclusive of the visible.)

INTRODUCTION

This paper presents several matters related to high-gain light integrating spheres, L I S. The intent is to be informative. It appears convenient that the entire subject be sub-divided into several parts as follows:

- Part I. Definitions as applicable to the L I S.
- Part II. Purpose of the L I S.
- Part III. Glassblowing problems.
- Part IV. The scientific glassblower's technique.
- Part V. Specular thin-film deposition in the L I S.
- Part VI. Window provisions and window materials.

PART I. DEFINITIONS.

(a) Chemiluminescence. In any chemical reaction at relatively low temperature if light be emitted it is termed chemiluminescence.

(b) High-Gain (in Optics). The "gain" of an integrating passive surface may be defined. It is the ratio of the luminous flux incident upon a normal unit area surface, using the integrating surface, to the luminous flux incident upon the same normal unit area surface without using the

integrating surface; while maintaining a fixed distance between the normal unit area surface and the source.

(c) Light Integrating Sphere. The LIS is not a new device. Usually, it is a thin walled hollow sphere of a known inside diameter. They have been made of metal, glass, and plastics and have been in use for several years, being developed and adopted as a means of accurately measuring the full 4 Pi steradian output of unknown light sources. Results obtained with the original form of the LIS were found to be more useful than were possible using any prior design of photometer. The early forms of the LIS had smooth internal surfaces, and were coated with some metallic oxide (usually white) to furnish a uniform and diffuse surface. At least one or more ports or window holes were provided. The unknown light source was then positioned at the center inside the LIS . . . hence, all light emitted from this source acted quite orderly from the geometric-optical point-of-view. However, the signal output as detected through the port or window left something to be desired. The positioning of the device at the port was found to be most critical, and non-linear results were evidenced. This, in turn, required the use of the calculus for any accurate evaluation of the light source that was being measured. On the other hand it is true that the original LIS was a real improvement over earlier methods of instrumentation. It made possible rather absolute measurements, whereas the previous photometric method furnished only relative measurements.

About five years ago it was suggested that were the entire surface of the LIS dented, then rendered specular such as to provide multiple lenticles, that the output of the LIS may be expected to be nearly independent of the source position within the sphere. Numerous configurations were tried. The results were a positive improvement, and the so-called "high-gain" was realized.

PART II. PURPOSE OF THE LIS.

The purpose and use of the LIS when completed and checked for its gain factor is to make possible laboratory studies of certain chemiluminescent reactions, . . . especially those which have been observed in the upper atmosphere of the earth. (e.g. Auroras, Airglows, etc.) In the Laboratory these chemiluminescent reactions occur within the LIS. In effect the LIS becomes the reaction cell, or chamber. Vacuum paraphernalia with gas feeds and controls are accessory equipment. Highly sophisticated instrumentation detects, records, and evaluates the signal output of the reaction taking place in the LIS. This data, in turn, then is furnished to the scientist, who will rule on its validity.

PART III. THE GLASSBLOWER'S PROBLEMS.

Let us now assume that any glassblower, whether a Member of our Society or not were to be given the task of making one of these LIS. Let the simple original smooth surfaced type be called out. A standard Corning (Pyrex No. 7740) RBVM boiling flask and a Coroflo reducer

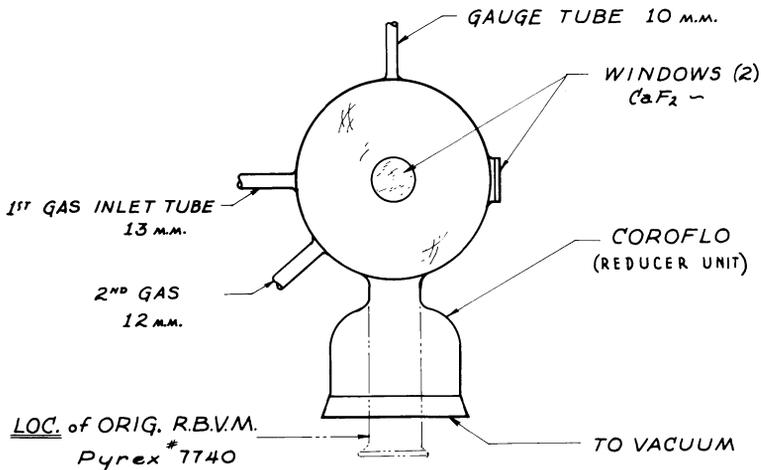


FIGURE 1.

Two Litre Smooth Surface Light Integrating Sphere.

unit are required items. He will note on the sketch that the finished device is to be used on a vacuum system. Several port tubes and two windows of special material are also specified. This glassblower's problems will reside in how well he may plan his work sequence, how closely he follows the rules of his craft with respect to both glass-strain and glass-annealing; and, his self confidence in his own capabilities with respect to all of the glassblowing operations involved. It is not at all improbable that he can and will get a sphere made. It may, of course, involve more than the one attempt.

PART IV. THE SCIENTIFIC GLASSBLOWER'S TECHNIQUE

The glassblower just mentioned may now be invited to join with all scientific glassblowers. The task, in this instance however, will be the development of a two litre L I S, the drawing of which calls for the dented or lenticular surface. Otherwise it is quite similar to the first sphere. A non-dimensional working drawing is furnished. The components listed on the sketch will govern all glassblowing operations as to size. In other words there are no close tolerances demanded.

Before we cover the step-by-step sequence of one successful method, it is best to mention the fact that in addition to the glassblower's lathe there is one other capital item required. This is a furnace of portable design. The one which the author used was home-made. The schematic (sectional) drawing shows that this particular model was heated by both electricity, and air-gas fuel. The furnace must be capable of prompt installation upon the ways of the lathe. A photograph of the furnace bears out the "home made" quality of the unit. In addition to the furnace we will require a large single-frame blast burner. Some may still call this a

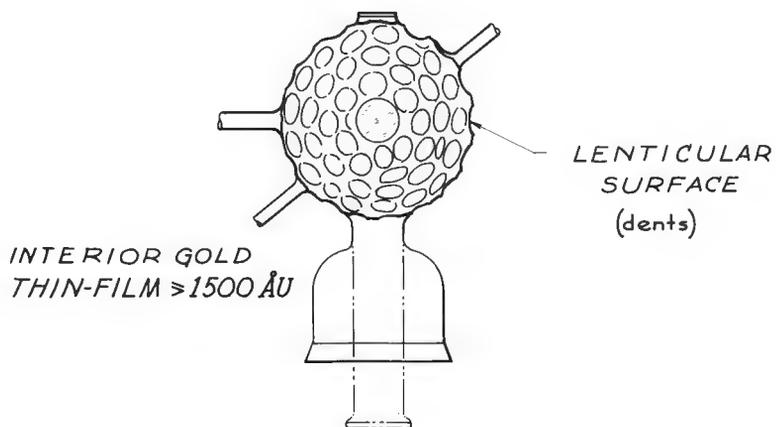
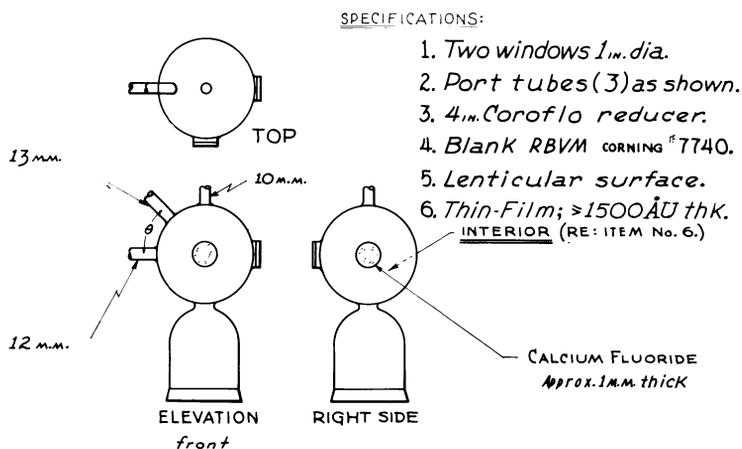


FIGURE 2.
High-Gain Light Integrating Sphere, Two Litre.



FIGURE 3.
High-Gain Light Integrating Sphere, Two Litre.

cannon torch. This burner must be air-gas-oxygen. It is essential that quick control be provided, which can be a foot operated economizer - with a pilot light at the head of the burner. Finally, besides the usual set of glassblower's tools, inclusive of the oxy-gas hand torch, a special denting tool should be made. I have used both carbon and metal heads. The metal does seem to stand up for longer use; but, in either case, the old-fashioned "Bee's-wax" dip is used, as applied to the preheated head of the denting tool before each series of dent forming.



A rough sketch of the setup used by the author is shown in Fig. 8. We should keep this figure in mind as we go through the sequence of operations, as follows:

Step One (Without Furnace on Glass Lathe)

Chuck the flask in the lathe and tubulate it with a piece of 20MM at a location diametrically opposite to the chucked long vial neck of the flask.

Step Two (Without Furnace on Glass Lathe)

Using the tubulation just provided, rechunk the flask and remove the long vial neck portion of the original flask, shaping so as to match the diameter of the Coroflo reducer unit. Then mount the Coroflo reducer in the other lathe head and seal the two units together. The precaution of slow and deliberate heat-up at the vial neck portion of the flask cannot be overemphasized. The glass can be very heavy at this location. The same precaution also applies to the Coroflo unit.

Step Three (Without Furnace on Glass Lathe)

Now remove the 20 MM tubulation. Since, in some cases, we are

(COAXIAL TO LATHE)

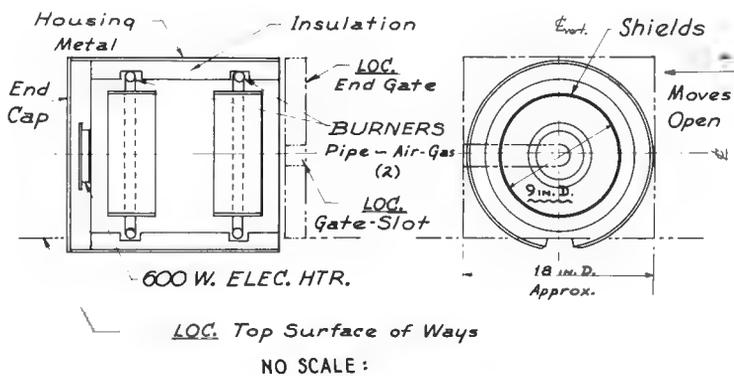


FIGURE 5.

Portable Furnace. Schematic/Sectional.

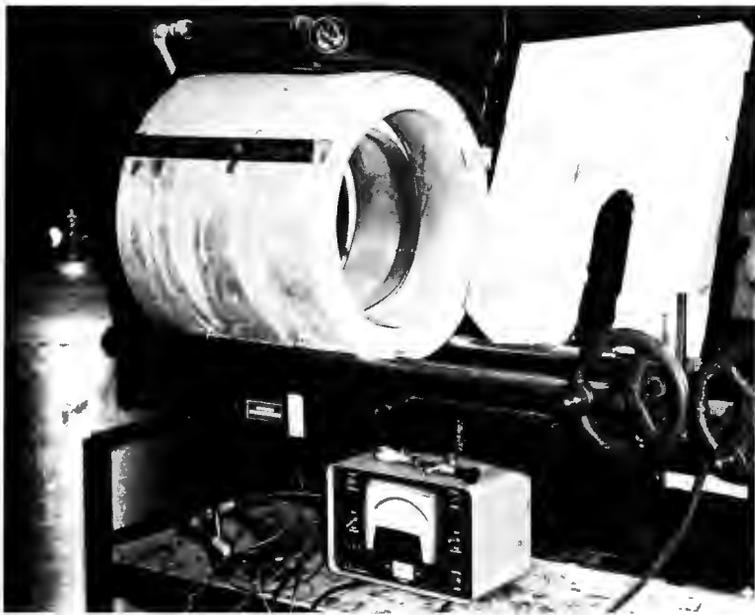


FIGURE 6.

Portable Furnace.

to use this very location, later, for a window port hole, it is advisable to allow a slight protrusion to remain, say about a quarter-of-an-inch.

Step Four (With Furnace)

Now install the furnace. Adjust and check all connections. Heat the unit up to 550° Cent. and hold. (NOTE: There are three thermocouple junctions. Each lead, respectively, to a three-station pickup alarm-type meter. The meter has been previously calibrated. One of the thermocouple junctions (TC) is centered inside the spherical portion of the work piece. The other two TCs are located in the furnace. The drawing also shows one blow-hose arrangement. A large-sized swivel, with a tee modification, permit both TC pickup and blowing during rotation, as may be required.)

Step Five (With Furnace)

While we await the temperature rise of the furnace unit, we can rotate the work piece and preheat it with the use of the large blast burner. Alternately, we can actually move the flask portion of the work piece into the furnace heat region and, closing the gate over the open end of the furnace, heat up both the furnace and the work piece together. The gate is provided with a slot which allows the free rotation of the work piece within the furnace. The rate of rotation is quite slow . . . about 15 or 20 rpm is adequate. As the temperature reaches the 550° Cent. level, the positioning of the large single-flame burner is checked so as to be at the right location to properly apply heat to the flask (when required) when the latter is removed from the furnace for denting.

Step Six (With Furnace)

When the temperature of the work piece has reached and been held at 550° Cent. (for 5 or 10 minutes) it can now be moved out of the furnace heat region and the denting operation may be started.

VERY IMPORTANT: Only two or three minutes is considered a safe average maximum time period to be allowed for denting. It is most obvious that the flask is rapidly cooling down. TC monitoring can help, and 510° Cent. can be the "rule-of-thumb" MINIMUM temperature. The blast burner can be used with a large bushy flame to promptly recover the 550° Cent. level. Also, the furnace can be used.

The hand torch is used for the denting operation. It promptly heats the area of glass (up to 820°+ Cent.) the accepted "Softening Point." When the glass has been softened the denting tool is now used. When the press against the hot softened glass is made, with a definite control and firmness, the blowhose is given a sufficient pressure to maintain the softened glass to be formed and or pressed outwardly against the denting tool's face. This pressure can actually be felt, or sensed, as the act is performed. This is why I have used the terms "control and firmness." At first, perhaps, only one or two dents can be made during the allotted time limit. But after some practice and the familiarity which accompanies same, as many as eight or ten dents can be formed during this very short period of time. Obviously, the rotation of the work piece is stopped during the denting operation. But during either method of heat up, the work piece

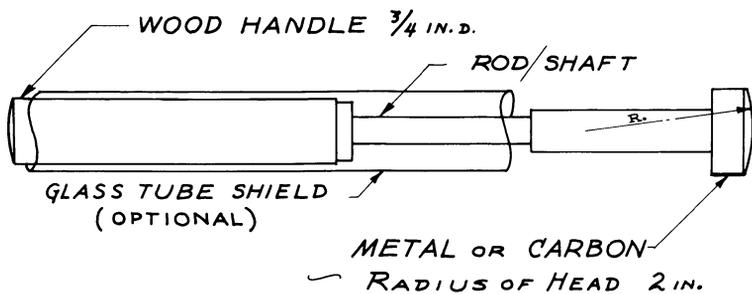


FIGURE 7.

Denting Tool.

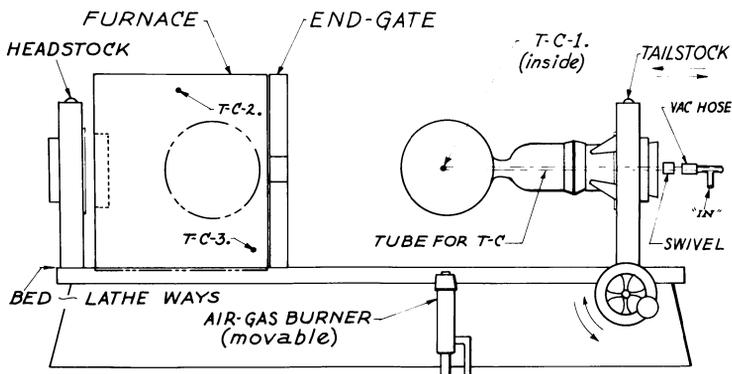


FIGURE 8.

Setup, a Sketch Drawing.

must be rotating. Whenever the glassblower might require a break, then the furnace, which is always riding at the 550° Cent. ("SAFE") temperature, can be used for indefinite periods of time. However, it is well that it be mentioned that prolonged use of the furnace (in my case) resulted in an appreciable percentage of monoxide (CO)! But I was prepared for this very possible occurrence. We have a meter which very accurately reads out the percentages of CO, and when it approached the danger level, I would move the work piece into the furnace, closing its gate, and start up an exhaust fan at one of the opened windows. Too, I'd leave the room, closing its door.

On the other hand, prolonged use of the blast burner produced uncomfortable room temperatures.

Eventually, the entire surface of the spherical portion of the work piece will be dented.

Step Seven

When denting is complete, the gauge and the gas port tubes are provided. Also the window port-holes. Each is done individually. The off-hand method is used with the hand torch. The window ports are best obtained by blowing relatively thin-walled bubbles of suitable diameter. These bubbles may then be either cut off, with the use of the wet glass-cut-off wheel, or they may be broken with the file; the latter method requiring fire-polish and possible reaming. Too, the brim will have to be hand-ground to provide the flat seat for the window disc. The glass (mechanical) cut-off wheel precludes this manual grinding. But either method has been used successfully.

PART V. SPECULAR THIN-FILM IN L I S.

When all of the glasswork has been completed, and this includes a thorough annealing cycle, the next step is to provide the highly reflective, or specular thin-film of metal within the sphere. Those of you who are familiar with this process may know it best by the terms "Thin-Film Depositioning." Too, those of you who have the proper facilities to carry forward the work can perform this without delay. For those who are unversed in the subject, the thin-film depositioning is accomplished in an ultra-high vacuum bell-jar system, and the desired metal is vaporized and recondenses upon the walls of the glass. In this particular instance, the heater which vaporizes the metal must be inside of the spherical portion of our L I S. This, of course, requires some special jigs and fixtures. In my own cases, since we were interested in the infrared end of the spectrum, I used pure gold, as vaporized from a tungsten heater filament. The vacuum system, in the case of using gold, must be of excellent performance. 5×10^{-7} Torr, or millimeters of Hg is the recommended "Blank-off Pressure." Otherwise the gold will be dross, or contain impurities — even tungsten. The thickness of the gold film approximates 1500 to 2000 Angstrom Units. (e.g. This is so thin that it would require about 200,000 times its thickness to add up to one inch of gold!) The gold is uniformly deposited. The deposition requires several days for so-called molecular readjustment.

PART VI. THE GAIN MEASUREMENT.

Modern and conventional photometric measurements are made after the film has been properly aged. When deposition conditions are good, and the gold is of excellent purity, the "gain" factor can be as high as X 15. X 16 gain is about the theoretical limit. When the source lamp, a small pulsed incandescent bulb having very low power is moved about the entire space within the integrating sphere, there should be no variation of the output signal. This is an important characteristic since it simplifies the mathematics, as well as having increased the output signal — as compared to that of the original smooth surfaced L I S. The gain of the latter rarely exceeded X 3 or X 4.

PART VII. WINDOW MATERIALS AND ATTACHMENT TO L I S.

Several infrared transmitting materials are available, and used. Even rock-salt crystal. Others are: calcium fluoride, barium fluoride, sapphire, and quartz. Occasionally silicon and germanium. The choice of material depends entirely upon the wavelength of the signal being investigated. Quartz, is comparatively limited for far infrared work, yet it is the only material which can be fused to the parent flask glass, by properly grading the seal. Therefore, we avail ourselves of the use of some form of adhesive material. The following have been used with complete success: Dow-Corning's "SILASTIC RTV." Hysol's "EPOXY." Curtis Associates' "SILVAC." In each case, the manufacturer's cure cycle should be followed carefully.

CONCLUSION:

The method outlined and described in this paper may appear to have some drawbacks, or defects. Please bear in mind, however, that it is based entirely on the fact that this worker was but one man, and worked alone on the task at all times. He frankly admits that with the aid of at least one additional qualified man, the time and quality would benefit. Be that as it may, success is a good and fine experience.

ACKNOWLEDGEMENTS

The author is grateful to the following personnel at the Radiation Effects Branch (O P R) of the U. S. Air Force Cambridge Research Laboratories:

Dr. A. T. Stair, Monitor OPR.

Dr. Randall E. Murphy, Physicist.

Marshall H. Bruce, Theoretical Physicist.

E. Ray Huppi, Chief Engineer, Airborne Instrumentation Upper Atmos.

John W. LaSpina, Technician, Airborne Instrumentation Upper Atmos.

A NEOPHYTE'S NOVELTY NOTES

MORRIS LIEBSON

Department of Physics

West Point, New York

Although associated with glasswork for many years, I never had any success with novelty work until some two years ago, on discovering Hammerfahr's book. Setting up shop in my basement as shown in Figure 1, I went through the recommended exercises, step by step. In so doing, I found that some glasses worked differently than others. Not just the difference between soft and hard glass, but the soft glasses themselves seemed to vary considerably in hardness (or softness). On taking notes of these effects, it occurred to me that members of this society might be interested in my findings.

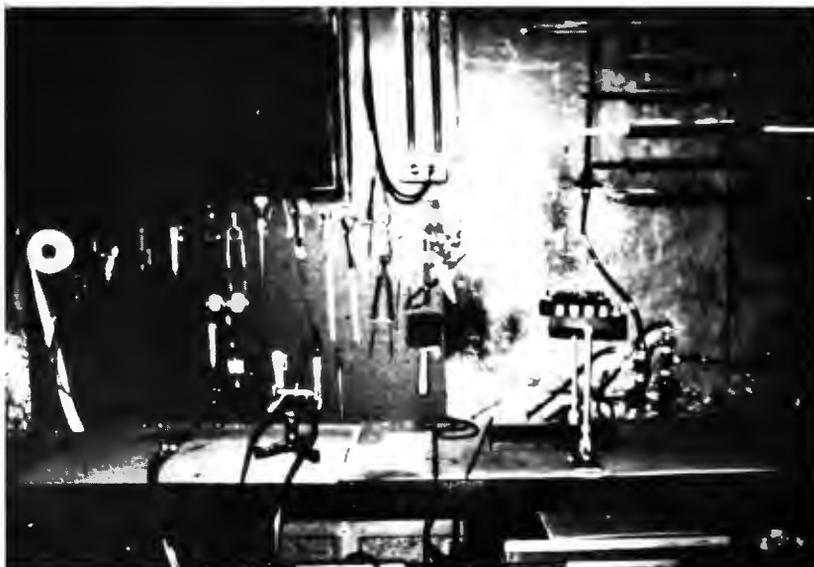


Figure 1

For comparison's sake each glass was checked by first blowing a small sphere or bubble using a small diameter iron tube (Fig. 2), drawing a fiber of the material, and finally burning off a small section of rod. One end of the rod was burned off in the cross fires and the other with a blast burner. This was all alone with gas and air. A very few exceptions were checked with gas and oxygen.

The idea of blowing a bubble of the glass was to check the softness. For very soft glasses I could blow a comparatively large bubble, i.e. up

to two inches in diameter (Figs. 3, 4, 5). For a comparatively hard type of soft glass I was lucky to get a one half inch ($\frac{1}{2}$ ") bubble. Burning off a section of rod gave me an indication of blackening or discoloration (Fig. 6). If the glass does not discolor in the blast burner, it is quite stable and can take much abuse in the cross fires.

By far the greatest number of glasses checked were those obtained from Techno-Scientific Supply Co.¹ These were all soft glasses in an interesting variety of colors. Glass was also obtained from G. Finkenbeiner,² and Houde Glass Co.³ A sample of rod and tubing was obtained from the British Co. of Plowden and Thompson Ltd.⁴ However, because of the mail strike in that country communications were so delayed that I could not work their material into this investigation. However, pictures of their samples are included (Fig. 7).

For an amateur with limited means and equipment, the "alabaster"¹ glasses are excellent. These are the softest of those tested and because of this softness it is possible to work much larger masses of this glass with a small cross fire, than any other type tested. It was quite easy to blow a two inch (2") diameter bubble of this material. Attempts to blow such larger bubbles with harder glasses caused discoloration or blackening. The Finkenbeiner kit consisted of black, blue, red and white rod; and amber, blue, green, red, and white tubing. Although comparatively hard, this glass did not blacken or discolor even when worked in the blast burner. Glass samples obtained from Plowden and Thompson came in a very large selection of colors. There were twenty-six different colors of rod and three

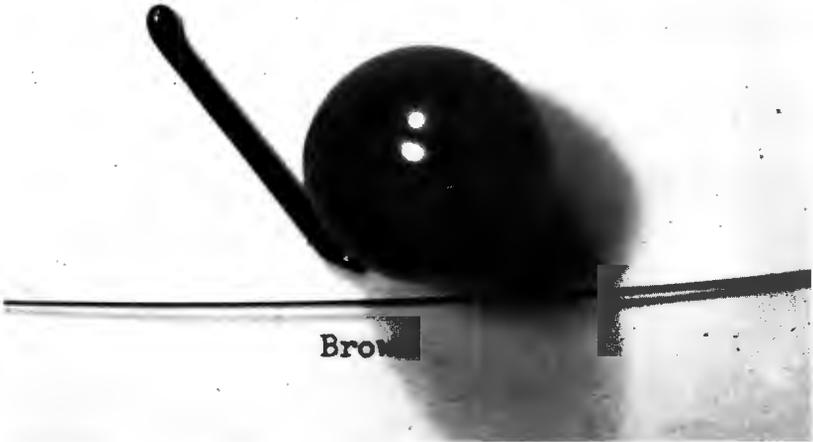


Figure 2



Alabaster
Rose

Figure 3



Brown

Figure 4

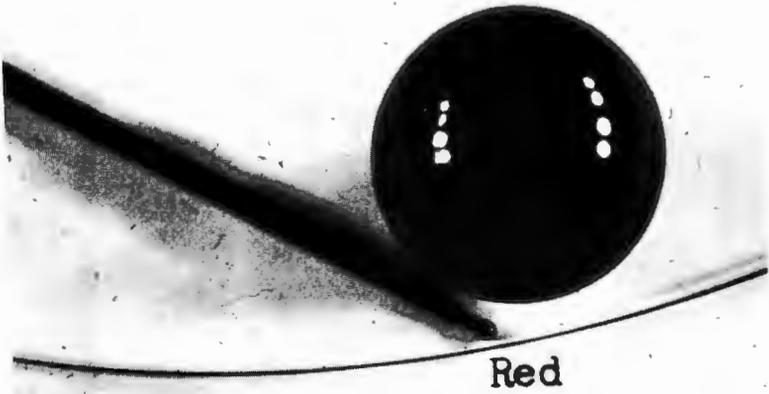


Figure 5



Figure 6



Figure 7

of tubing, all clearly labeled. The manufacturer's literature, which came with this glass, describes it as lead-free soda. The hardest of these soft glasses tested was the sel-ruby. In the cross fires I could barely make a $\frac{1}{2}$ " bubble (Fig. 8), however, it is available in tubing, and the tubing can be used to make larger, hollow items. It may be of interest to note that uranium glass, Corning No. 3320, could be worked in my gas-air cross fires, quite slowly, of course, compared to the gas-oxygen flame normally used on this material. This is quite desirable, for novelty items made of it will glow when subjected to ultra-violet light giving a most spectacular effect. It is interesting to note that among the samples sent by Plowden and Thompson was a small piece of uranium soda-glass which fluoresces just as strongly under ultra-violet radiation, as does the Corning 3320.

Table No. 1 shows the relative softness of some of the glasses tested. The names of the colors are those used by Techno-Scientific except for the Corning glasses.

Much of the colored rod received was either old or under strain for it shattered even when heated very slowly. Flame annealing helped considerably. I would imagine oven annealing to be much better.

The various slides speak for themselves. All were photographed in color at the same distance with a thirty-five millimeter camera, permitting comparison as to size, color and blackening.

Samples of some pulled points and finished novelties are shown in Figures 9, 10, and 11.

Table 1

Very soft —	alabaster blue alabaster rose alabaster green	
Soft —	<u>Transparent</u> amethyst blue green rose pink yellow	<u>Opaque</u> black baby blue robin's egg blue brown gray jade opal orange yellow
Medium —	<u>Transparent</u> amber aquamarine	<u>Opaque</u> red turquoise
Hard —	<u>Transparent</u> sel-ruby crystal Corning 0120	<u>Opaque</u> chalk white
Borosilicate —	black crystal Corning No. 3320 Corning No. 7740	



Figure 8

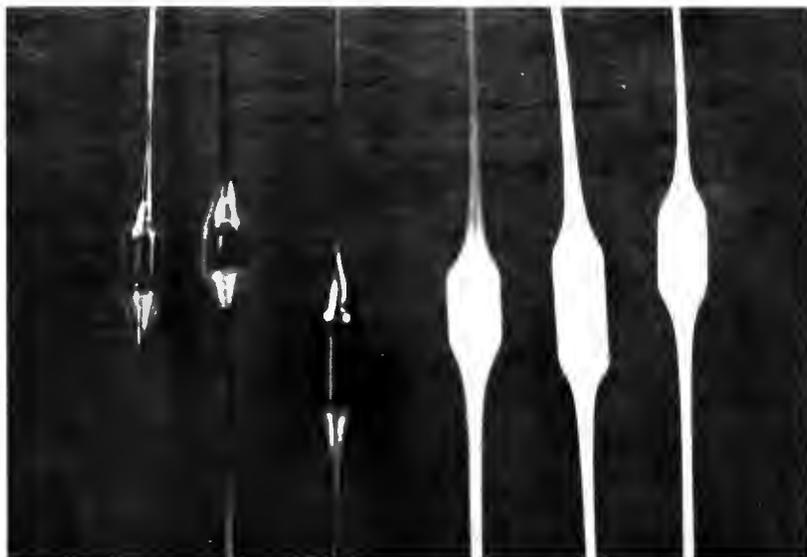


Figure 9



Figure 10



Figure 11

1. Techno-Scientific Supply Co., Inc., P. O. Box 191, Baldwin, NY 11510
2. G. Finkenbeiner, Inc., 36 Rumford Avenue, Waltham, MA 02154
3. Houde Glass Co., 1195 McCarter Hwy., Newark, NJ 17104
4. Plowden and Thompson, Ltd., Dial Glass Works, Stourbridge, Worcs., Great Britain

GADGETS AND TOOLS FOR THE GLASSBLOWER

JAMES MORRIS

Northwestern University

Evanston, Illinois

Carbon tapers, glass scoring knives, and polariscopes are used by most glassblowers. These tools are available commercially, but the cost may be more than one can afford. Presented in this paper, therefore, are plans and costs for assembling these instruments.

The carbon taper is the easiest to make. The taper requires a three inch piece of five eighth inch hexagonal steel bar drilled and threaded with a one half inch thirteen tap. A piece of one half inch graphite rod is screwed into the hexagonal bar and cut so the graphite extending from the end of the steel is ten times as long as the largest diameter on the hexagonal jig. This will make a one to ten taper on the graphite. Using the flat side of the hexagonal jig as a guide, the graphite is held against the rotating flat plate grinder plate and is turned every few seconds to the next flat on the guide. Grinding and turning and checking (to be sure it is grinding evenly) are continued until the end of the graphite forms a point. When the taper is shaped to satisfaction, it is removed from the jig. The handle part is wrapped with two layers of one sixteenth asbestos string. By wetting and then rolling the handle, the handle will be a heat resistant holder when it is dry (figure 1).

CARBON HEX TAPER

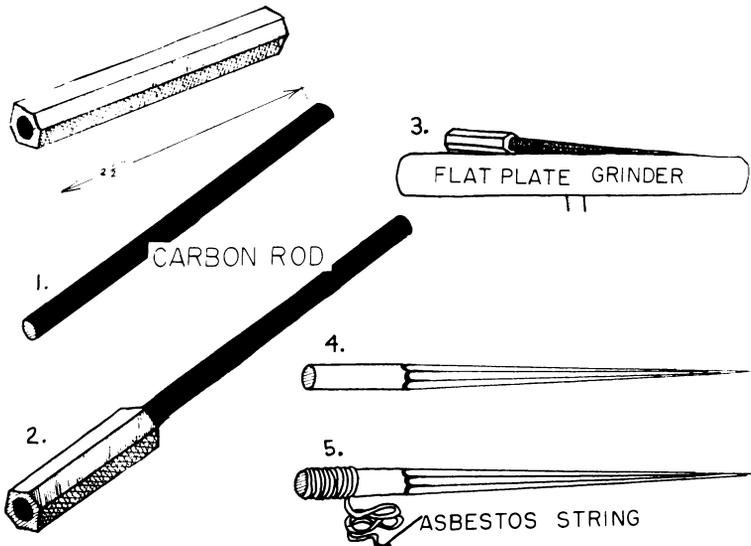


FIGURE 1

The carbaloy glass scoring knife is made from a piece of tungsten carbide one eighth inch by one fourth inch by one and one half inches, a number fourteen one and one half inches wood screw, and a number two file handle. A three inch piece of tungsten carbide is notched in the middle using a Carborundum or diamond saw and is broken into two one and one half inch pieces. The head of the wood screw is cut off and a notch one eighth inch wide and a little less than one fourth inch deep is cut into the top of the shank of the screw. The tungsten carbide is fitted and silver soldered into the notch. The wood screw now attached to the tungsten carbide is screwed into the file handle (figure 2). The knife can be sharpened easily on a grinding wheel (green stone) designed for sharpening tungsten carbide tools used in a machine shop.

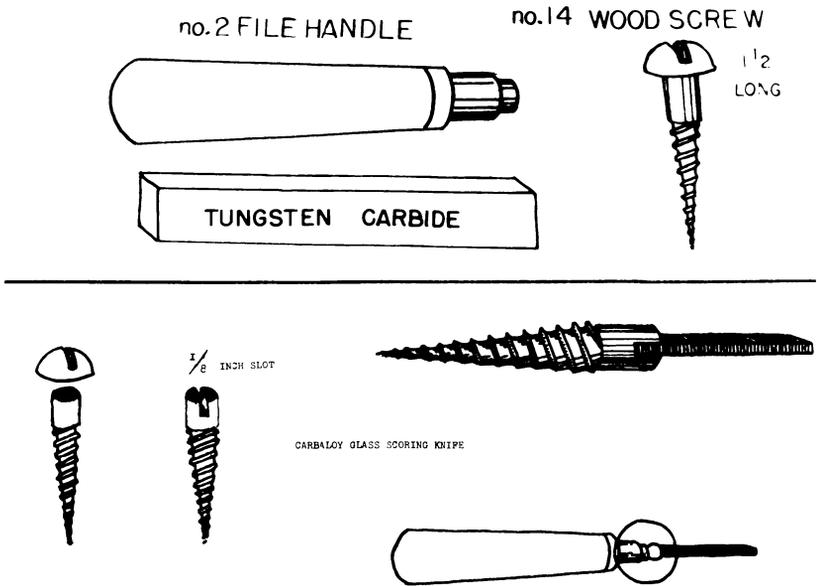


FIGURE 2

The polariscope consists of two parts. The first, a box, a light bulb, a polarizer, frosted glass, a metal mask, and glass discs. The front opening of the box is twelve inches by twelve inches with a channel wide enough to hold a twelve by twelve inch frosted glass plate, two glass discs twelve inches in diameter, a linear polarizer cut into a twelve inch circle, and a metal mask twelve by twelve inches with the center cut to a circle about eleven and one half inches in diameter. The center of the top edge of the linear polarizer is marked so the center top can be easily identified after it has been cut into a circle. The polarizer is sandwiched between the two glass discs, and electricians' black plastic tape is applied around

the edges to bind the discs together. All of the parts are mounted in the channel frame (the identifying mark about forty-five degrees from the top) with the frosted glass next to the bulb and the metal mask in front of the linear polarizer (figure 3).

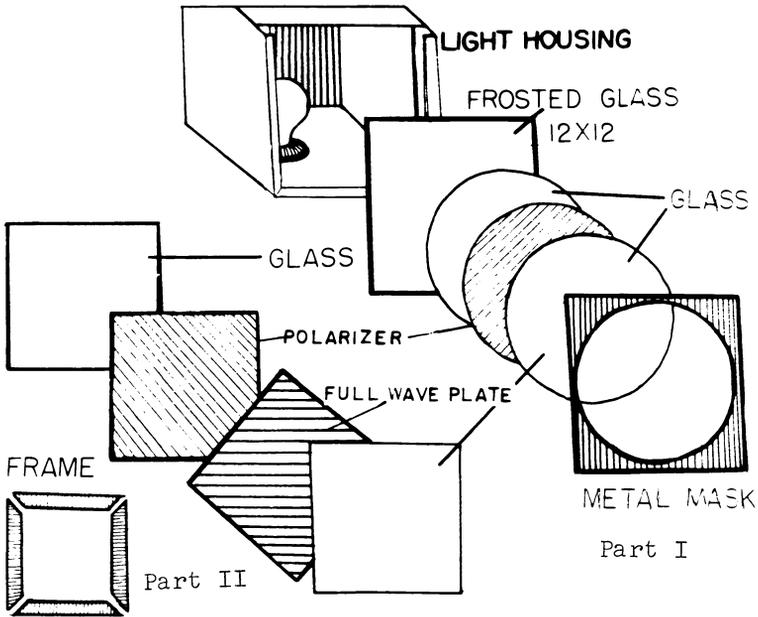


FIGURE 3

The second part of the polariscope is assembled in the following order: a six inch square of glass, a six inch square of linear polarizer, a six inch full wave plate turned forty-five degrees and the corners cut to fit inside the six inch square, and a six inch square of glass. These are mounted into a sturdy wooden frame (figure 3). The full wave plate is important as it gives color and makes the strain in glass more visible.

The manometer jig, one of the gadgets for the glassblower, is a piece of wood with grooves cut into the edges; when tubes are placed in the grooves, the tubes are spaced so a meter stick will fit between them (figure 4a).

Another gadget is a simple centering device for a Litton H.S. Lathe. A piece of tubing three and one half inches outside diameter, three inches inside diameter, and one and one half inches long is threaded to fit on the back of the lathe chuck. Three holes, evenly spaced around the outside, are drilled and tapped for one fourth inch bolts. The bolts are used to center the glass (figure 4b).

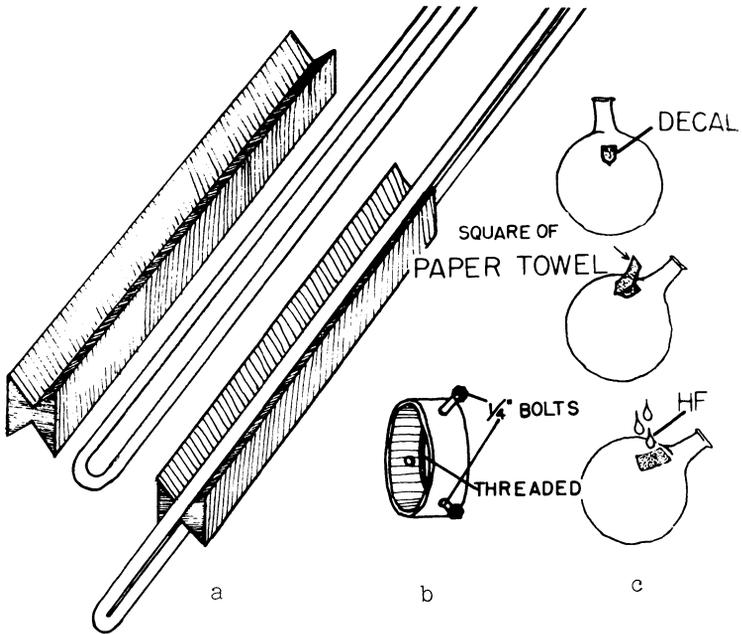


FIGURE 4

To remove a decal from glass, a piece of paper toweling is cut large enough to cover the decal. One corner of the towel is moistened with water to hold the paper in position over the decal. Hydrofluoric acid is then dropped onto the paper until the paper is saturated. After one minute, the decal will wash away in water (figure 4c).

WHERE TO BUY PARTS LIST

I. Carbon Hex Tapers:

Graphite Rods — A.G.S.R. Grade

1/2" Diam. 24" Long, 44¢ each — minimum order \$25.00

(Other Graphite Sizes can be included to make up the order, such as a graphite plate 1/2 x 6 x 24 1/2" — \$2.71 each)

For other information or to order, contact:

Union Carbide Corp.
Carbon Products Division
120 South Riverside Plaza
Chicago, Illinois 60606

II. Tungsten Carbide Glass Scoring Knife:

Tungsten Carbide — Grade 883

Choice of

<u>Cat. No.</u>		<u>1-9 pieces</u>	<u>10-24 pieces</u>
STB48B	$\frac{1}{8}'' \times \frac{1}{4}'' \times 1\frac{1}{4}''$	\$3.78 each	\$1.53 each
STB48C	$\frac{1}{8}'' \times \frac{1}{4}'' \times 2\frac{1}{4}''$	\$4.53 each	\$2.28 each
STB48D	$\frac{1}{8}'' \times \frac{1}{4}'' \times 3''$	\$4.93 each	\$2.68 each

There is a 40% discount to educational institutions.

Information and order from:

General Electric Co.
 Metallurgical Products Division
 P. O. Box 237
 Detroit, Michigan 48232

III. *Polariscope:*

<u>Number Needed</u>	<u>Cat. No.</u>		
1	31-52-62-26	Linear Polarizer 6" x 6" Plastic Laminated	\$ 5.50
1	31-52-62-28	Linear Polarizer 12" x 12" Plastic Laminated	\$20.50
1	31-52-62-56	Full Wave Plate 6" x 6" Plastic Laminated	\$ 6.30

Made by: Bausch and Lomb
 Rochester, New York 14602

Purchase from your lab supply dealer.

SAFETY IN THE GLASS EQUIPMENT SHOP

JOHN M. REYNOLDS

General Electric Company
Schenectady, N. Y.

INTRODUCTION

Safety considerations vary depending on the kinds of everyday activity in any given workplace. Unlike most shop operations which involve mostly mechanical and electrical hazards, the activity in glass fabrication shops include hazards of toxic emissions and exposure to physical agents. Foremen and supervisors should be familiar with their responsibilities and liabilities, particularly in view of the new Occupational Safety and Health Act which was effective on April 28, 1971.

TOXIC EMISSIONS

1. *Silica*

When quartzware is heated to its working temperature, an invisible emission occurs. This emission is characterized by the familiar cobweb effect (Figure 1) that occurs at the heating station. The air contaminant that is produced is silica; and the occupational disease associated with silica is silicosis. The limit of exposure to concentrations of silica as established by the ACGIH¹ is 250 million particles per cubic foot of air (depends on the percent crystalline silica present). This limit is based



Figure 1

Cobweb effect of vaporized silica

¹American Conference of Governmental Industrial Hygienists

on an 8 hour/day concentration. Excursions beyond the limit are acceptable provided that they are appropriately time (exposure) compensated. It is expected that this threshold limit value will be standardized under the new law; and employers will be required to prove that these concentrations are not exceeded.

The most effective control measure is to provide local ventilation at the source of emission. If this proves impossible, respiratory protection must be provided for the employee.

2. Mercury

Mercury has been an occupational hazard since its discovery in ancient times. It is a unique metal, since it is liquid at ambient temperatures. The hazards associated with mercury generally are attributed to its vapor pressure. Even at room temperature, vapor will emanate from open pools or spills of mercury that are capable of producing a hazardous concentration in air; and when mercury is heated, the concentration in air will increase (Figure 2). There is virtually no warning properties because there is no characteristic odor.



Figure 2

Bean of Mercury — size of pin head

The small drop of mercury exhibited in this figure — if totally vaporized and distributed in a room 100,000 cubic feet in volume, will reach a concentration equivalent to the threshold limit value for an 8 hour a day — 40 hour week exposure.

Glass technicians must have adequate training in regard to the handling of metallic mercury, since they often work with the substance during the fabrication of diffusion pumps, manometers, McCloed gauges and other equipment.

3. *Cleaning Chemicals*

Many organic solvents, acids and mixtures of chemicals are used to clean glassware. The hazards involved are obvious and include toxic effects from inhalation, skin irritation, corrosiveness, flammability and unexpected adverse reactions.

Protective equipment (Figure 3) such as face shields, protective clothing and respirators can be used to protect the operator, however, these do not afford complete protection. The foreman or supervisor must understand the hazards peculiar to a specific substance or he must consult with an authority on the subject. Then it is his responsibility to provide adequate measures of control.



Figure 3
Protective equipment

PHYSICAL AGENTS

1. *Infrared and Ultraviolet Radiation*

The intensity of heat required to reach the melting point of quartz is such that infrared and ultraviolet radiation can be emitted. During these operations the technician must be equipped with protective eyewear (Figure 4). Depending on the intensity of the flame used, it is desirable to wear the #5 shaded lens which is capable of removing 98% of the

infrared and 99½% of the ultraviolet radiation; and nearly all glare. Some commercially available goggles are capable of giving the protection of a #8 — #10 shaded lens (nearly opaque to infrared and ultraviolet) while still allowing the visual transmittance of #4 lens.



Figure 4

Eye protection for IR and UV

Glass containing sodium will, upon heating, emit hazardous sodium glare, special eye protection lenses are commercially available that filter out the sodium spectra. The most common is didymium.

2. Radio Frequency Heating

The major sources of injury in the operation of high frequency equipment are electrical shock and burns to operating, testing and maintenance personnel. These injuries can be attributed to poor location, lack of proper grounding, inadequate or inoperative interlocks, insufficient shielding, unsafe practices (reaching into enclosures to adjust or extract pieces) and faulty maintenance practices.

The most effective means to protect an operator is to totally enclose the heating coils and electrically interlock the enclosure (Figures 5A and 5B).

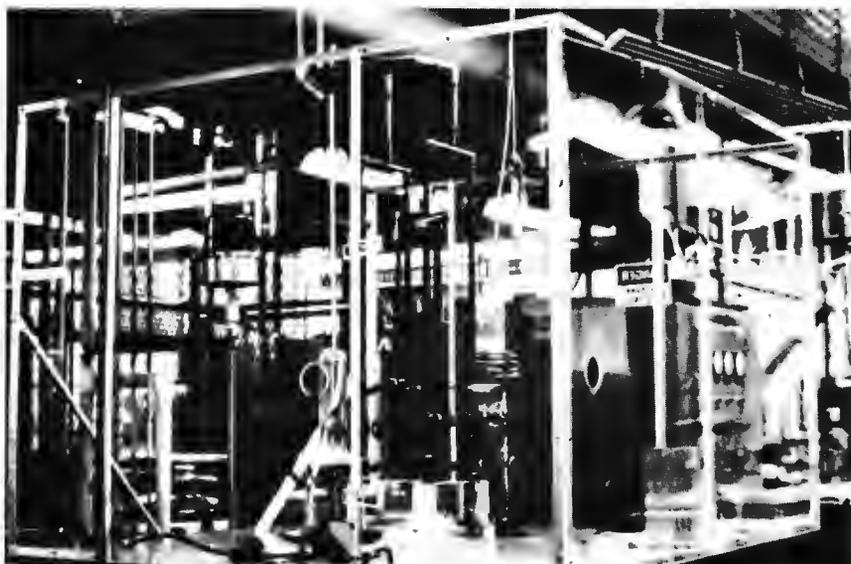


Figure 5A
RF enclosure



Figure 5B
RF enclosure inter-lock

3. Noise Exposure

The Federal law regarding noise relates the level of noise to exposure time. The criteria for permissible noise exposure is as shown in

Table 1

<u>Duration per Day, Hours</u>	<u>Sound Level (dB-A) Slow Response</u>
8	90
6	92
4	95
3	97
2	100
1½	102
1	105
½	110
¼ or less	115

The decibel notation (dB) is a simple logarithmic device used to express the ratio of two like quantities proportional to the power or energy of a noise. The "A" designation is frequency-response network which compensates for variations in human response.

Noisy operations must be surveyed by a qualified expert; and engineering methods should be incorporated to reduce levels to within acceptable limits. Otherwise, hearing protection must be supplied to the employee. In addition, the area should be properly identified.

MECHANICAL HAZARDS

1. *Machines*

Injuries from the operation of machines such as, drill presses, lathes, cutoff saws and grinders and others are caused by:

- A. Contact with moving machine parts or tools.
- B. Being caught between work and machine.
- C. Flying chips entering the eyes.
- D. Material falling on fingers and toes.

These injuries can only be reduced by properly guarding moving parts (Figure 6), providing dead-man switches (Figures 7A and 7B) and mandatory procedures for the wearing of personal protective equipment (safety glasses, goggles, gloves, face shields, etc.). Most of all, it is important that the operator is fully trained in regard to the operation of equipment.



Figure 6
Cut-off wheel guard



Figure 7A
Using drill press with dead-man switch



Figure 7B
Close-up of dead-man switch

2. High Pressure and Low Pressure Equipment

High pressure gas cylinders must be properly secured at all times. Even small cylinders constitute a hazard. If a defect occurs that releases the gas instantaneously, the resultant projectile produced, can be devastating to equipment and personnel.

Glass equipment under vacuum should be shielded to protect against flying glass in the event of an explosion.

ELECTRICAL HAZARDS

1. One of the most overlooked and most serious hazards is that from electrical shock. It is absolutely necessary to have a good electrical maintenance program to eliminate the possibility of shock hazards.

Most of the electrical fatalities (Table II) that occur each year in the U. S. are due to ungrounded, portable electrical equipment. The equipment often becomes energized when the metal framework makes contact with broken, or exposed wires inside the tool, or if normally insulating surfaces become conducting.

Table II

PHYSIOLOGICAL EFFECT OF CURRENT

Threshold of Sensation	1 ma
Painful	10 ma
Freezing Current	10 — 25 ma (Holds victim fast to circuit)
Current Producing Unconsciousness	15 — 100 ma
Ventricular Fibrillation	100 ma to 2 or 3 amps

A separate low resistance ground wire must be provided (Figure 8), attached to the framework of the equipment and to a good ground. This will provide an electrical path if a defect should develop. In addition to grounding, it is extremely important that the power cable is free of defects (Figure 9), with regard to its insulating cover. Bodily contact with a defective cable and to a low resistance object would bypass the protective ground circuit and the operator would consequently receive a serious electrical shock.



Figure 8
Polarized plug



Figure 9
Faulty leads

VYCOR AND IMPROVEMENTS

CARLTON T. BEDSOLE

Corning Glass Works Plant
Danville, Virginia

This paper presents an overview of the VYCOR® process and states some of the most recent improvements.

VYCOR® is a glass of 96-98% silica that approximates fused quartz in many of its properties. This glass has the same general field of application as fused quartz. Its softening temperature is somewhat lower and expansion slightly higher. Greater ease of fabrication extends its uses materially beyond those of fused quartz.

A family of VYCOR® brand glasses can be made by varying the process conditions or subjecting the glass to additional processing steps. In its porous state the structure can be impregnated with various oxides to generate a range of end products. At present the process yields some twenty (20) glasses with properties such as infrared transmission, controlled water content, UV transmission and controlled UV cutoff.

Because of the physical properties, optical properties, and high resistivity, VYCOR® finds basic use in laboratory glassware, ground joints, one-piece graded seals, missile domes, chemical trays and jars, heat sheath, germicidal lamps, photochemical lamps, sun lamps and many others.

Simply stated, the manufacturing process could be reduced to forming the raw glass, leaching out unwanted elements, drying, and firing to consolidate the porous structure.

VYCOR® is made by an ingenious process whereby a composition relatively high in fluxing oxides is melted and formed to the desired shape, but somewhat oversized. After heat treatment, these fluxes are practically all removed by acid leaching. Following the drying operation, the article is fired in a vacuum at high temperature to consolidate the remaining 96-98% silica structure. During the firing operation, the article shrinks to its finished size.

VYCOR® has been marketed for over thirty-five years. There have been numerous developments and improvements over the years. I will point out some of the most recent improvements in the manufacturing of Vycor® tubing of the standard brand 7913 glass.

Concentrated efforts in the forming process have improved dimensional and visual quality. Through changes in the forming process, outside diameter, wall, and out of round dimensions have been improved. Outside dimension range is now 40% tighter around nominal than before the process modifications. Wall thickness range has been improved by 15%. These improvements yield a more uniform product in all aspects of dimensional quality.

A major change to the beginning of the drawing process has reduced surface imperfections and markings to the lowest level ever obtained in

VYCOR® tubing. This improvement yields a product with projection quality. Projection quality is defined as distortion free sections when projecting light from a 25 watt arc lamp through the tube onto a screen.

In addition to dimensional and visual improvements mentioned above, the physical properties of the 96-98% final structure have been improved through adding new improved equipment. The annealing point has been raised from 1,000°C. to 1,050°C. New vacuum units with uniform temperature control and a higher vacuum capability allows a more uniform consolidation, during which more water vapor is extracted. The average water content $\beta(\text{OH})$, mm^{-1} has been reduced from a level of 0.4 to less than 0.3. Furthermore, a unique process to firepolish the consolidated tubing was developed in 1970. These automatic units provide a more uniform firepolished surface.

Some of the special features and applications of various VYCOR® brand glasses are given below. For convenience sake, the glasses are described in numerically increasing order.

CODE 7905—This glass is controlled for high infrared transmission below about 3 microns wavelength. An 0.125 inch thickness of Code 7905 glass will transmit a minimum of 80% at 2.72 microns. It is available in sheets to 4 mm thickness, pressed ware and other forms. Code 7905 glass domes are used in heat sensing missiles which “see” or “home on” infrared waves.

CODE 7906—Same as 7905 for sheet thickness up to one-half inch.

CODE 7910—It is an ultraviolet transmitting glass specially prepared to transmit at least 70% at 254 millimicrons for 2 mm thickness. Its annealing point is 950°C. The glass has wide applications as envelopes for low pressure mercury vapor lamps.

CODE 7911—This is an ultraviolet transmitting glass with low gas content. It has high electrical resistance and low power loss. It is somewhat harder than 7910 glass and can therefore be used at higher temperatures without deformation. Its ultraviolet transmittance is controlled to at least 85% at 302 millimicrons through one wall.

CODE 7912—This glass has the same overall properties as Code 7910 but is more transparent to wavelengths below 254 millimicrons. Its minimum transmittance at 185 millimicrons is 2% for a thickness of one millimeter. It is available only in tube form and is chiefly used in low pressure mercury arc, germicidal, and ozone producing lamps.

CODE 7913—This glass has a low gas content. It has no special restrictions on transmission. Because of its excellent heat resistance it is used as containers for sintering, calcining, high temperature fusion, and chemical reaction at elevated temperatures. In tube form, it is used as thermocouple protecting tubes and tips, heat sheath tubing, hot-furnace sampling tubes, etc. There are many more applications for this glass. It is available in tubing, rod, sheet, pressed or blown or fabricated ware.

CODE 7913—OPTICAL GRADE—It is made from optical quality base glass and is available in three optical grades. In Schlieren quality, the maximum gradient of the average index of refraction through the blank, measured perpendicular through the window, is held to a minimum of 5×10^{-6} per inch. The optical grade glasses are used in windows for mapping, for wind tunnel applications, in space capsules, or where high internal quality is required for use at elevated temperatures. Maximum size is 20-inch square or 20-inch diameter. Maximum thickness is $\frac{3}{4}$ inch ground.

CODE 7915—This glass will not darken or solarize. It is nearly 98% silica and is controlled to contain less than 0.002% Na_2O and 0.003% water (determined by infrared adsorption data) to minimize devitrification and outgassing in service. The annealing point is about $1,060^\circ\text{C}$. and Code 7915 glass is least likely to deform at high temperatures. It is available in thin wall tubing.

CODE 7930—This glass is usually referred to as “porous glass” or “thirsty” glass. Code 7930 is the intermediate form of porous glass obtained in leaching the base glass used in manufacture of the final fired VYCOR® brand glass. It has an average pore diameter of 40 angstrom units. Its chief commercial application is as moisture getter in electronic components.

CODE 7931—This number is used to designate clear graded seals fabricated from porous glass tubing. The seals are useful for sealing 96% silica glasses or fused quartz to PYREX® brand Code 7740 glass. The soft end may be colored blue for identification. Graded seals are available in sizes up to 115 mm O.D. These seals are finding increased applications in both industry and laboratory. They are currently also used in the manufacture of more sensitive photomultiplier tubes.

CODE 7950—This is a red-stained reconstructed glass which absorbs most of the visible light from a tungsten filament (2700°K .) but effectively transmits infrared. It is available in tubing, sheet, blown and pressed ware. In tubing form it is finding increasingly wide application as an envelope for radiant heaters.

CODE 7953—This glass is black and transmits less than 0.1% in UV, visible, and IR for a 5 mm thickness.

GLASS IN X-RAY TUBES

EDWARD E. HAFKEMEYER
General Electric Company
Medical Systems Division
Milwaukee, Wisconsin

ABSTRACT

This historical development of the use of glass in x-ray tubes is traced from early tubes with soft glasses, through the introduction of borosilicate glasses, to the development of specialized glasses in modern x-ray tubes. The properties of an ideal glass for x-ray tube use are presented. The metal alloys commonly used with x-ray tube glasses and the types of seals used are also described. Examples of modern multi-seal x-ray tubes are presented.

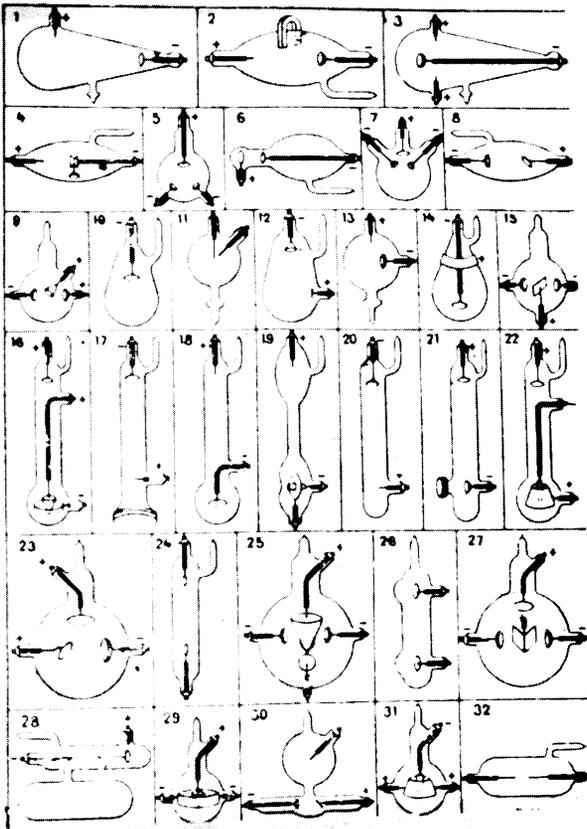


FIGURE I

It is indeed amazing that, within months after the discovery of x-rays by Roentgen — on December 28, 1895, or January 23, 1896, — frantic efforts were made to produce x-rays. It follows that there was an increased need for competent glass-blowers. Figures 1 and 2 show some of the early

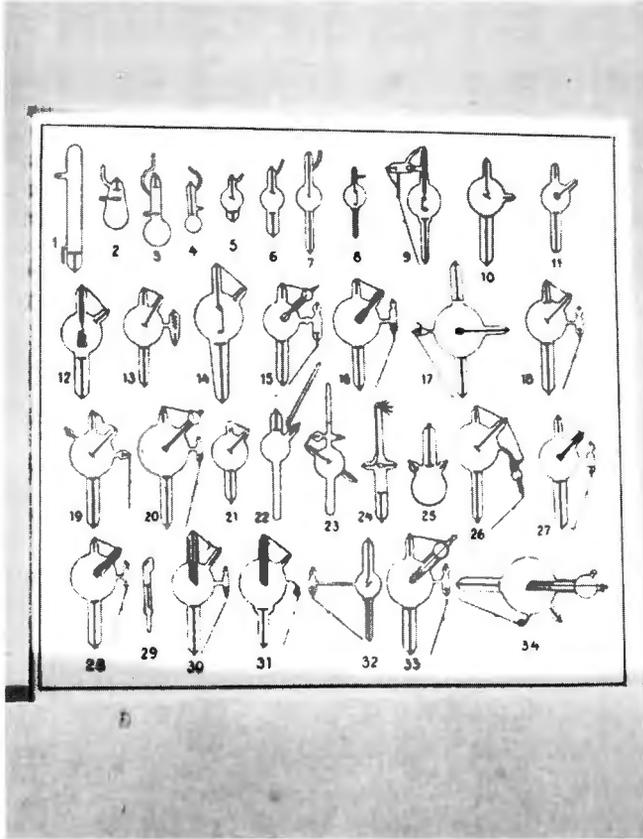


FIGURE 2

x-ray tubes. These were truly a challenge to these artisans. The tubes shown in Figure 2 were made by Muller of Hamburg who also made tubes for Roentgen. These early tubes were cold-cathode, gas type units. Some of the appendages on the tubes were ingenious schemes to help control the gas pressure in the tube, and the voltage applied to it determined the tube current. It is safe to say that standard radiological techniques would be almost impossible to obtain under these circumstances. As can be imagined, the tubes of that day were made from lime glass with platinum seals. Some tubes were made from lead glass with lime glass x-ray windows. This construction provided inherent radiation shielding.

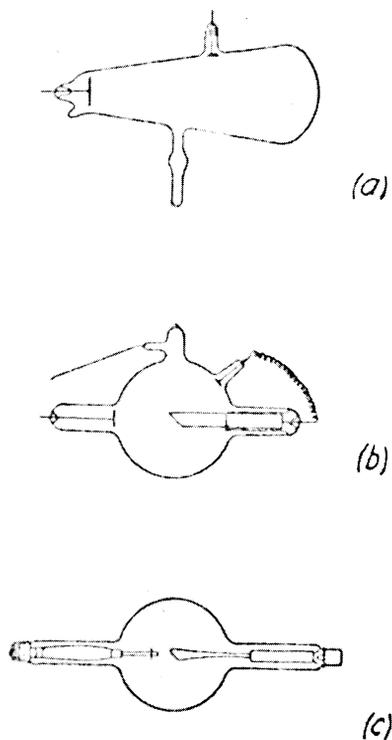


FIGURE 3

In 1913, when Dr. Coolidge discovered the hot cathode x-ray tube, many changes occurred. At this time, the tubes were highly evacuated because the x-ray tube current could be adjusted by controlling the filament temperature. Along with this came increased x-ray output which required increased electrical power input. When it is realized that an x-ray tube is less than one percent efficient, it is apparent that the heating effects within the tube became a problem. That is still true today! The rating of an x-ray tube depends principally upon the melting point of the target. This is the reason the targets are made from the refractory metals, tungsten, rhenium and molybdenum.

Figure 3 shows vividly the changes in tube design. Figure 3(c) shows the familiar "Coolidge tube." The spherical center bulge was needed to keep the glass from softening and sucking-in.

In the early nineteen thirties, hard or borosilicate glasses were developed. With these came the now familiar iron-nickel-cobalt alloys Fernico and Kovar. Nিকেlex (42% Nickel-Steel) was also developed during this time. It was an economy grade Kovar for less than critical application. Tungsten was limited to lead-in use. Figure 4 shows the improved



(a)



(b)



(c)

FIGURE 4

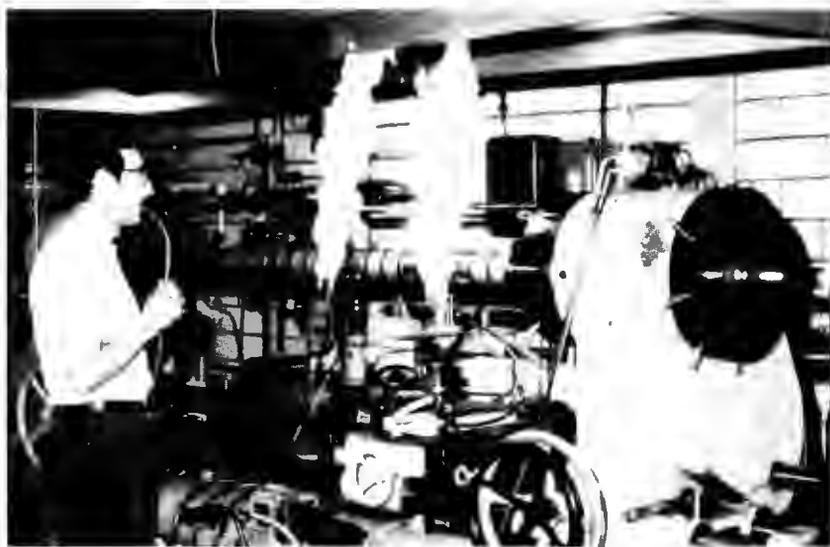


FIGURE 5

designs utilizing cups, sleeves and rings. The multi-section tubes we now make contain 23 glass sections and are about 11 feet long. Figure 5 shows a glassblower making such an assembly on a glass lathe designed and built by GE.

Figure 6 shows a rotating target, x-ray tube used in diagnostic medical radiology. The bulb is Pyrex and the end seals are 7050 (705 AJ) glass with Kovar seals.

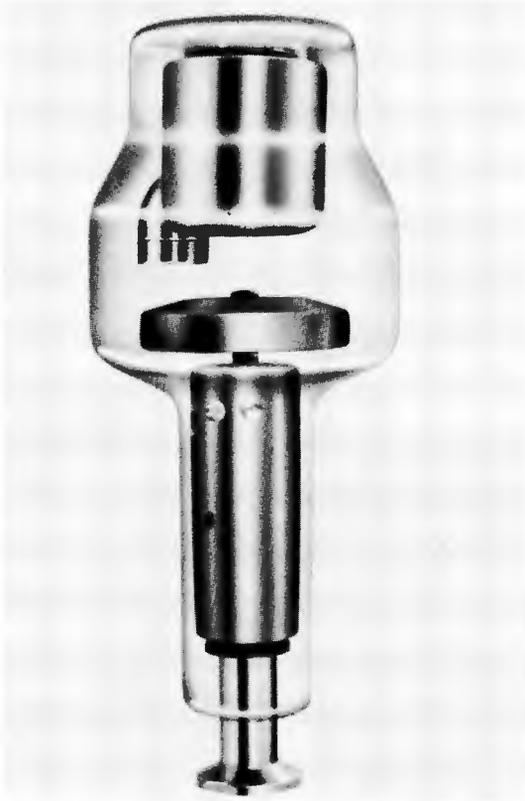


FIGURE 6

Back in these halcyon days despite the often personalized service, the quality of x-ray tubes varied widely even within the production of one manufacturer. There was then a standard joke among salesmen, offered whenever a customer voiced too many complaints — against one particular tube. The suggestion for “curing that particular tube was to wrap it in a towel, place it for 15 minutes in a hot oven, then remove it and finally hit it sharply with a hammer.” This quote is from an amazing book “The Trail of the Invisible Light” by E. R. N. Grigg, M.D. and published in 1965 by Charles C. Thomas, Springfield, Illinois.

X-RAY TUBE GLASS REQUIREMENTS

The hard glasses available in the early thirties were Corning No. 7050 (705 AJ), 7740 (Pyrex) and 7720 (Nonex). These glasses had certain disadvantages:

1. The 7050 glass had very high resistivity which prevented the uniform discharging of electrical charges built up on the glass walls.
2. The 7050 glass suffered from "weathering." It had to be stored in a dry environment and had to be worked very carefully to avoid hazing or "clouding-up."
3. The 7050 glass had non-uniform expansion characteristics from melt to melt.
4. The 7740 provided a poor match to Kovar unless 3320 (Uranium) glass was used as a seal-grading glass.
5. The 7740 also had very high resistivity.
6. The Nonex glass was objectionable because of its high lead content. Remember, in x-ray tubes, the output is too low already and the lead in the glass would attenuate the output markedly — especially in the long wavelength portion of the x-ray spectrum. One solution was to provide x-ray windows by thinning the glass wall in the exit port area or providing a beryllium window.

A NEW TYPE OF GLASS

Ultimately, thru the joint cooperation of the glass companies, a general purpose x-ray tube glass was developed. These glasses are:

Corning Glass Works — 8830
Libbey Glass Company — K650
McKee Glass Company — 402

Table I shows a compilation of the characteristics of a number of hard glasses. Figure 7 shows a plot of volume resistivity as a function of temperature. To better appreciate the relative resistivity between 705 AJ and K650, the resistivity of the former is about 6 times the latter.

Figure 8 shows a plot of linear expansion coefficients of Kovar and certain glasses as a function of temperature. Noteworthy is the fact that the curves are not linear. This makes matching glass and metal much more difficult. Figure 9 shows an excellent collection of glass to metal seals. Of principal interest are:

Seal 6C The edge of the cup or eyelet is beveled — about 30° — rounded and polished. The beading glass is applied — in a manner known only to you artisans. Then the rest of the glass structure is added. Sometimes the "plunge seal" is made wherein the metal and glass are "properly" heated and the metal is merely plunged into the hot glass. We use the former method in almost every instance.

Table I
GLASS CHARACTERISTICS AFFECTING X-RAY TUBE
MANUFACTURE & PERFORMANCE

Glass Type	7050 (705AJ)	7720 Nonex	7740 (Pyrex brand)	3320 Uranium	K650	8830
Softening Point °C	703	755	819	784	711	716
Annealing Point °C . . .	496	518	553	535	503	513
Strain Point °C	461	484	510	497	473	483
Resistivity Log R						
@ 350°C	6.77	7.20	6.68		6.35	6.24
250°C	8.31	8.82	8.15		7.99	7.68
Dielectric Strength						
Volts/Mil. 200°C	80	80	100		80	153
25°C	330	270	400		350	
Coefficient of Expansion						
x 10 ⁻⁷ in./in./°C	46	36	33	40	47	48

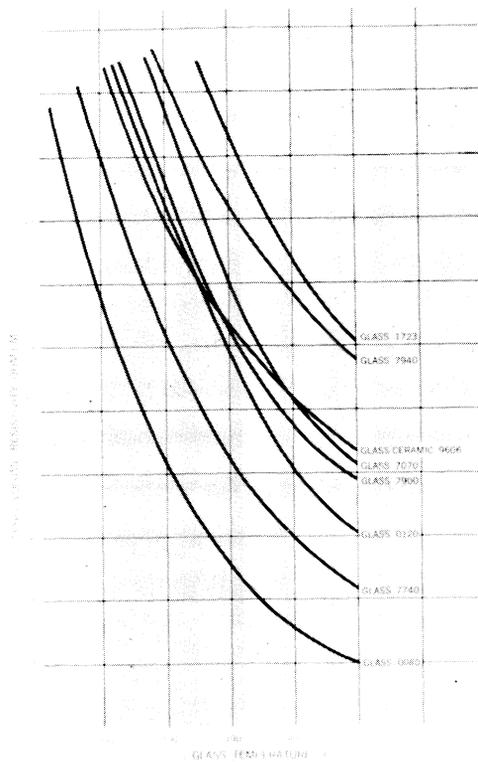


FIGURE 7

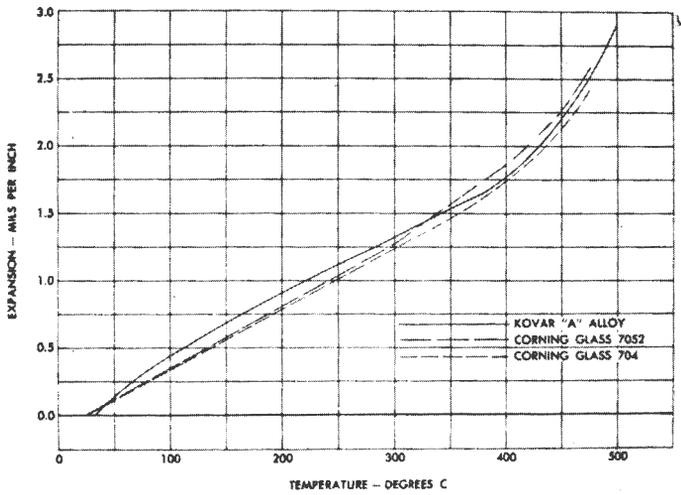


FIGURE 8

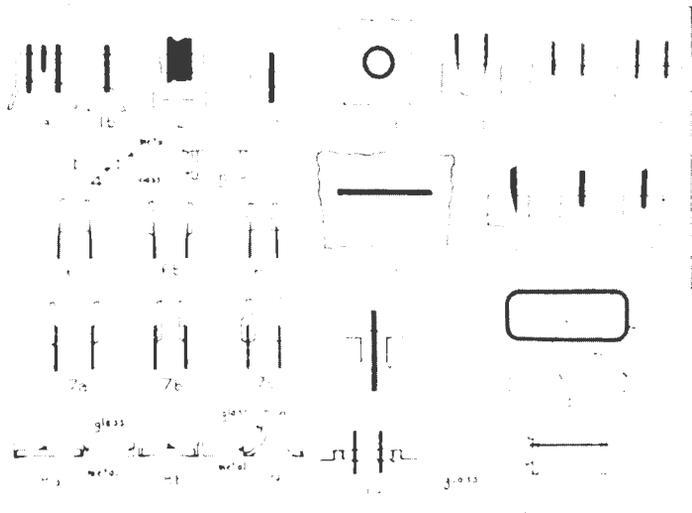


FIGURE 9

Seal 12 Where a lead goes thru an eyelet, we usually insert a beaded lead into the eyelet and achieve sort of a "plunge-seal" at the tips of the eyelet.

GENERAL COMMENTS

The types of seals are legion in number. It is surprising that "new" types of seals are being re-born frequently. All one has to do is go back far enough and you'll find that the new concept was merely an old Lazarus given new life. After all, the old artisans were quite ingenious — also.

REFERENCES

1. "The Trail of the Invisible Light," E. R. N. Grigg, M.D., published by Charles C. Thomas, Springfield, Ill., 1965, pg. 41.
2. Op. Cit. pg. 43
3. "Glass Selection and Production Techniques for X-Ray and Other Tubes," M. J. Zunick and J. B. Gosling. Proceedings of the National Electronics Conference, 1950.
4. Op. Cit.
5. G. E. Medical Systems Employee Orientation Course.
6. G. E. Medical Systems (artist's rendition).
7. "Engineering with Glass," Corning Glass Works, 1962.
8. "Kovar Alloy," The Carborundum Company, Form No. 5134.
9. "Glass-to-Metal Seals in Electronic Components and Applications," A. J. Monack — Electrical Manufacturing, 1947.

THE ENGINEERING FUNCTION IN GLASS-WORKING OPERATIONS

GEORGE H. BATLEY

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

THE GLASSBLOWER

A glassblower is many things, but probably first and foremost, he is an artist. He employs glass and fire in much the same way that a sculptor uses stone and chisel, and, like the sculptor, results usually reflect his own particular individuality.

But, he is also a technician. He must be able to interpret blueprints and other engineering specifications. He also must be able to use the common tools of measurement in order to check his dimensions and dimensional tolerances. And, he must be sufficiently conversant with the chemistry and physics of his materials, including fuels — so that his finished article is not only to specification, but also of high quality workmanship.

He is a mechanic to the extent that he has to operate machinery, usually several different types of glass-working lathes or forming machines, but often some rather complex auxiliary equipment as well.

He does some clerical work, or even accounting, depending on what records he must keep and to what extent these records are used in arriving at a cost-pricing structure for the product he makes.

In a small shop, the glassblower is often a general handyman. He may have complete responsibility for ordering or procuring his raw materials, tools, or other equipment. He usually does all associated glass operations such as cutting, grinding, polishing, cleaning, or annealing. He might be his own maintenance man, depending on how well he keeps his equipment in proper working condition, and his work area in a reasonable semblance of cleanliness and order. He might even have to package and ship the finished product.

At Fischer & Porter every effort is made to relieve the glassblower of as much secondary activity as possible, other than his primary basic function: that of working glass.

This isn't necessarily as simple as it may sound at first. On the one hand, there is a natural desire to achieve, and maintain, a high degree of productivity at predictable and profitable levels. On the other hand, the artistic temperament of the well trained and highly skilled technician must be remembered, and taken into consideration. He cannot be made to feel regimented into an assembly-line classification. There has to be leeway to permit self-expression and the psychological lift derived from doing something a little challenging, a little different than the usual, and doing it well — the building of a little "self" into every product made.

THE FISCHER & PORTER ORGANIZATION

To implement this philosophy into actual practice requires a delicate balance, and it would probably be impossible to achieve if it weren't for some important associated factors: namely, a good manufacturing facility, sound company policies, and pleasant working conditions.

As an example, the Fischer & Porter Glass Division originated as a shop for manufacturing glass components for the Company's Instrument Division, principally precision-bore Rotameters. It was expanded to produce a line of Laboratory and Pharmaceutical apparatus ware, best categorized as catalog items: and then further expanded to include various lines of Specialty Products. It is today, a wholly-owned subsidiary corporation of the parent company, with glass shops in Los Angeles and Vineland, N. J. in addition to the Main Plant in Warminster, Pa. Over the years, no expense was spared to develop the Glass House into one of the most modern and best equipped facilities of its kind. Much of the actual glass-working equipment was fabricated in-house through the efforts of a capable Machine Design and Development group, and their associated shops. The original instrument components are still produced today, as well as similar products for OEM accounts, by high-speed, high-volume, almost completely automatic procedures. The glass-worker in this area is more of a fire-setter, rather than a glassblower. However, successful operation still depends in large measure on the experience and know-how of the operator, and, the area is still a valuable training ground for prospective glassblowers.

Most Laboratory Apparatus-type catalog items are also still produced by semi-automatic methods, or by bench workers using manual techniques. This offers an additional area for training of prospective glassblowers, and the techniques are a little closer to those required for scientific glassblowing work.

Most specialty product items are fabricated in somewhat isolated areas that are laid out to comply with Government Security Regulations, since a percentage of this type activity is sub-contract work that comes under such restrictions. These areas are self-sufficient insofar as any glass-working operations are involved, and are fully equipped even to facilities for glass preparation and inspection. However, this does not limit the group from access to, or use of, all plant facilities, as required, just as long as security measures are not violated: such as the Testing Laboratories, the Glass Finishing Departments, the Quality Control facilities, the Machine Shops, and so on. The work in the Specialty Product area is highly dependent on the skills of the scientific Glassblower.

OPERATIONS

The physical existence of a well equipped and well laid out manufacturing facility has obvious advantages, but it is not sufficient in itself. The work load must flow through the facility in orderly and well planned fashion with a minimum of disruptions. Equipment must be kept in proper

operating condition, adequate utilities must be readily available at all times, and all materials required for a specific job must be also readily available.

At Fischer & Porter, materials are stocked under a computerized inventory control system, with provisions to include in-process material inventory as well as raw material stocks, and also with provisions for automatic reordering to maintain a required minimal supply. For glass alone, the raw material inventory includes several hundred different items in more than two dozen common compositions; for quartz, agreements with a major supplier, now permits as complete a stock of raw materials as is available anywhere.

Incidentally, if any of you should have need for odd size special glass and can't procure it through normal channels, try us. Give us a call. We have helped many customers in the past. Although we are not in the resale business as such, we are glad to lend a hand where needed. It's worth a try to call on us — we will be glad to help if we can.

All production orders are processed through the shops by Process Sheet procedure, which is also adapted to a computerized control system. The process sheet is primarily a list of the Standard Operating Procedures for each step in the sequence of manufacturing operations, but it does also include the Production Planning scheduling and routing, provision for recording and classifying in-process spoilage for Cost Accounting and Quality Assurance purposes, and also specific instructions, such as tooling fixtures to be used; or cleaning procedures that must be followed, etc. Where applicable, a blueprint accompanies every Work Order, with all the pertinent engineering information also included.

For an operation of this nature, and of this magnitude, the Organization, as a whole, is generally Sales-oriented. It has to be. However, the manufacturing facility is generally Engineering-oriented. And again, it almost has to be. The glassblower is depending on the services of several other people, some of whom he may never actually meet or know personally, but whose efforts, nevertheless, are very important in helping to produce a smooth and efficient operation in his own work area.

There are two people that he does come into personal contact with, though, almost daily. The one is his immediate supervisor, whose functions are pretty well known and whose primary responsibility is to operate a good shop: scheduling and maintaining satisfactory production levels; keeping good working conditions and orderly flow of materials — in and out of the department; taking care of a myriad of little personal problems.

The other is the Product Engineer for the particular product or product line that involves the glassblower. Here the functions are generally not quite as familiar.

THE ENGINEERING FUNCTION IN GLASSWORKING OPERATIONS

In a large glass manufacturing division, the engineer, like the glassblower, is many things, too, and also like the glassblower, he usually reflects his own particular individuality.

Probably first, and foremost, he is a technologist: his educational background or his plant training and experience are meaningless unless they can be converted into actual application. For new products, or for major revisions to existing products, it is highly advantageous for preliminary development work and evaluation to be conducted at a laboratory or pilot plant level. Some minor development work has to be performed on the actual production lines, and in any case, pilot plant development has to be eventually phased out of the laboratory and into regular production channels. Desirable progress through these various stages necessitates very close cooperation between the Engineering, the Supervisory, the Production, and the Quality Control groups involved. At Fischer & Porter, like most large companies, this is best effected by having an experienced glassblower actually assigned to the Engineering staff as a consultant, and the laboratory or pilot plant facility also under engineering supervision. Such a facility can double as a production unit, especially for miscellaneous small-volume orders, and it is a valuable training ground for glassworkers who will eventually be making the product, particularly if new techniques or procedures and materials are involved.

The engineer's responsibility in this activity is chiefly of planner, Coordinator and Reporter. Although some experimentation is unquestionably still performed on a trial-and-error basis, and probably always will be, experience has shown that developmental results are best achieved, more efficiently, quicker and much more economically, if performed on a systematic step-by-step approach which also permits proper evaluation at each step of the investigation. This requires planning: it is not enough just to collect a lot of data and then try to make something out of it. The proper information has to be obtained, in a form that permits desired evaluation, preferably in accordance with one of the statistical techniques designed for just this purpose, experimentation in industry on a production-line basis.

Long before activities reach this stage, however, a great deal of planning and evaluation has already been expanded. Any order originates from an initial customer-vendor contact, either as a formal quotation request, or an invitation to bid, or from some informal discussion. Ability to make a product is not enough: it has to be made with high quality workmanship, at competitive prices, and in quantities and times that permit a committed delivery schedule. It is imperative that there be an up-to-the-minute record of all in-house orders. Whether in an in-process status, or as an order backlog item: also an up-to-the-minute record of the production planning demands on each department, both equipment-wise and personnel-wise: and further, what flexibility exists such as a need for additional help, transfer of personnel, a second shift, additional equipment or equipment modification, etc. This obviously, involves a liaison between Sales, the Order Entry and Production Planning elements and direct Manufacturing groups. The coordination of the activities however, is an engineering function: with direct responsibility for furnishing manufacturing costs to permit proper pricing: for supplying Process Sheet detail and manufacturing drawings to comply with customer specifications: for providing any tooling or fixturing that might be required in the manu-

facture: and, for recommending optimum quantities and times for an individual job order to permit proper interdepartmental flow of material and the routing and scheduling required to meet delivery commitments.

For many scientific applications, it is also important that there be an Engineering-to-Engineering liaison between customer and vendor, over and above the usual Sales-to-Purchasing contacts, or arrangements at other levels. Although it would be highly desirable to have every individual product exactly like each of its individual brother and sister units, and every lot exactly like every preceding and following lot, these ideal conditions are never actually achieved. There is a continuing pattern of change, subtle in most cases, but changes nevertheless: such as a different lot of some raw material, slightly different equipment settings, the variation in the off-hand glassworking, etc. The important feature is not that each piece be exactly alike: but, that the differences stay within a family of characteristics which differentiate that particular family from some other. Whereas actual compliance with specifications is a Quality Control or a Quality Assurance function, these variations have to be constantly evaluated on the production lines, both in Vendor and in Customer plants, and this function is an Engineering one, particularly if some corrective action is necessary. The Customer-Vendor liaison at this level is highly beneficial for both parties.

In these respects, the Engineer is a combined policeman and fireman: he must pound a production line beat, not only to keep up-to-date on current production, but also to assure that certain Standardized Procedures that require Certification are actually being complied with: and in case of trouble, he must be immediately available to answer the fire call.

He may have to act as a Referee, if rejectable material is merely sloppy workmanship that should be repaired or salvaged, or if some major change has occurred that requires a corrective action.

The Engineer has several secondary responsibilities. He has to meet periodically with the sales representatives of Suppliers and Sub-contractor, as well as the door-to-door peddlers that sometimes actually do have a better mousetrap: a new material, a special burner or other piece of equipment, a better process. He also has to sift through several trade journals constantly, to keep abreast of new developments in the field. He sometimes feels that he is buried under a mountain of paperwork.

However, when a particularly challenging job or development reaches a satisfactory conclusion and the glassblower swells a little with pride from the psychological lift of hearing "Good job — well done," some of this rubs off on the Engineer, too, and he also gets a lift. These results can only be achieved from a real team effort, and the principal ingredient of this teamwork is a mutual respect for one another. At Fischer & Porter, we strive for this goal.

INSULATOR AND HEAT EXCHANGER FOR ULTRA HIGH VACUUM, HIGH VOLTAGE DISCHARGE CHAMBER*

W. J. WHEELER

Glass Technician

University of Wisconsin

Madison, Wisconsin

ABSTRACT

This paper contains information and construction details for a ten foot long, eighteen inch diameter glass tube, and heat exchanger of twenty one, 1" I.D. by 10½ foot long tubes, joined at both ends to loops 16½" O.D. of the same tubing size. Equipment used and sealing techniques are described and photographs are included.

INTRODUCTION

The construction of a high vacuum discharge chamber required glass assemblies with the following characteristics:

1. Made of Pyrex® Glass, heavy walled and strain free.
2. One tube, eighteen inches in diameter, ten foot in length, both ends open.
3. One assembly of twenty-one parallel tubes, 1" I.D., 10½ feet long, joined to a glass ring of the same size tubing at both ends, with an entrance and an exit tube incorporated in their design. To be vacuum tight. (Vacuum outside and 14 psi. inside.)
4. All assemblies to be able to withstand temperature ranges from room temperature, bake out at 450°C., and operate at liquid nitrogen temperature at a pressure of 10⁻¹¹ Torr.
5. All assemblies to be installed in a 24" diameter by 14 foot long stainless steel chamber with removable end bells.

FABRICATION OF 18" TUBE

Blown cylinder blanks in 18 inch diameter by 4 foot long sections were available¹, so it was decided to build a machine to join two four foot lengths to one two foot length. The design of the machine is as follows: the 18 inch tube was supported internally with a specially designed chucking assembly on a central 6 inch, 14 foot long steel pipe, set on two "A" frames, with roller bearings at each end, and rotated by our Litton HSA lathe through a belt drive (Figure 1A).

This gave us a support on which the 10 foot length of Pyrex glass could be joined and a means of variable speed rotation at a minimum of

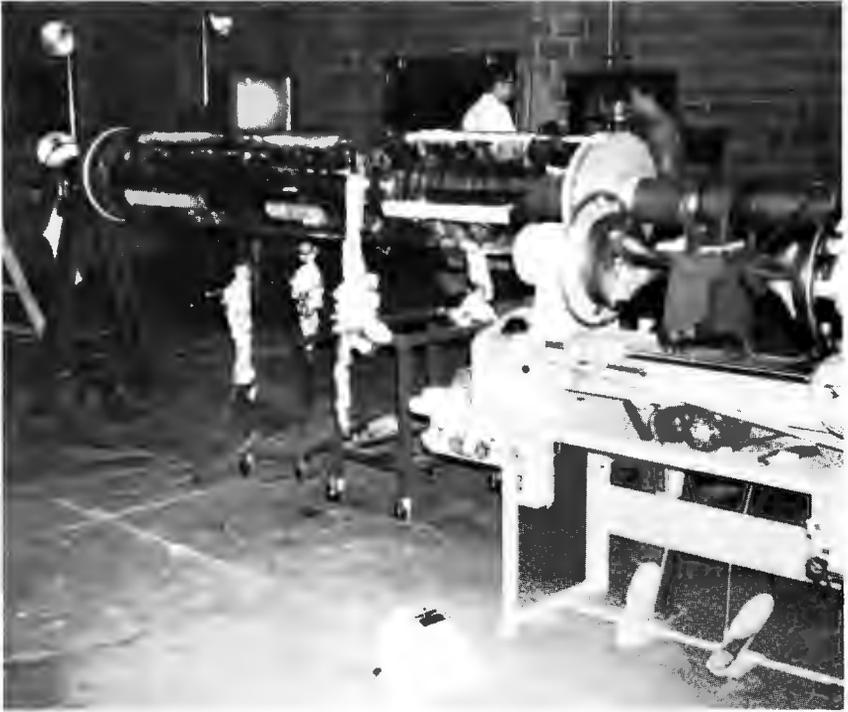


Figure I-A



Figure I-B

cost. The chucks were designed and built in the Chemistry Department Instrument Shop, and an overhead chain hoist was installed in a sub-basement area providing plenty of floor and overhead space.

It was decided to seal the 4 foot length to the 2 foot section, then to remove this assembly from the lathe, to anneal it, then rechuck it and seal it to the remaining 4 foot section. As the 6 foot length of tubing was quite light, two men could handle it easily in the chucking operations. Prior to the sealing operations, a preheat cycle of two to three hours was used, gradually increasing air flow to the two Litton hand annealing burners until a good strong bushy flame burned off the carbon layer deposited in the early stages of heating. A reversal of the preheating cycle was used to cool the tube down to room temperature after the sealing operations.

Sealing the 18 inch tubes was accomplished with two Litton 10 fire 7 jet Lathe Burners, set vertically on a movable cart, surrounding the 18 inch tube except at top and bottom. Each burner was fed with its own oxygen and hydrogen cylinder. A spare cylinder of hydrogen and oxygen was "T"ed into each burner, in the event the original supply ran out. With this method of supply, we were assured 20 minutes of maximum heat per burner (Figure 2).

While induction heating would have been the optimum sealing method, the requirement for only two seals and the financial constraints, dictated the choice of oxygen and hydrogen heating. This method of heating proved satisfactory on tubing of this size.

MOVABLE CHUCK DETAILS

In order to close the space between the heated ends of the 18 inch tube, a movable chuck, working through the center of the central pipe was designed. Two rings of 6 inch I.D. by 7 inch O.D. by 3 inch long steel tubing were used as sliding surfaces and supports for chuck jaws. A lateral motion was transmitted by a hand crank wheel at the extreme end of the 6 inch pipe, rotating a threaded shaft, engaging a lever connected to one of the sliding rings in the chuck. An in and out motion can be transmitted to the entire chuck, and the tubing on the chuck as well in this manner (Figure 1B).

Half an inch either way was sufficient. The hand wheel rotated along with the central pipe, the chucks, the tubing, and the covers for the ends of the tubing, and was rotated faster by hand to impart motion to the glass in one direction, and held counter to the rotation to reverse direction of chuck travel. It was found that this method gave us very sensitive control over movement of the glass during sealing, even though done remotely through commands and hand signals to an assistant.

The chuck jaws were 1" by 2" steel, held on lengths of 1" by 2" channel iron by 1" threaded stock. Rotating the perforated adjusting nuts on the threaded stock gave us a lateral motion to center up the tubing relative to the axis of the central 6" tube. A tool was made of 1/2 inch steel rod having a hinged end turned down to fit holes in adjusting nuts, the handle



Figure 2



Figure 3

being 6 feet long. At one point the adjusting nuts are over 5 feet from glass tube open ends. Insertion of the tool into one of the holes in the nuts and either pushing or pulling gave us the motion on the threaded stock to tighten the chucks on the inside surfaces of the glass tubing.

On chucking the 18 inch tubes, about a 5 mm gap was left between the two cylinders to be sealed, and a 2 inch diameter pad of asbestos tubing, 2 mm thick cushioned the interior of the glass tubing from the chuck jaws.

A 3:1 pulley ratio between the central shaft of the machine and a pulley held in the tailstock jaws of the Litton lathe gave us about the desired speed for sealing, 20 rpm. At this step up ratio, we found that the Litton lathe develops enough torque to overcome any imbalance and consequent erratic rotation of the 18 inch tube. Only on the first sealing operation was it necessary to balance the machine to overcome weight differences due to different wall thicknesses per section. A steel bar was clamped to chuck jaws well back from the heated area. This balanced the entire rotating portion before seals were made.

The glass tubing varied in outside diameter by as much as 1 cm., so centering the glass on the chucks was done by measuring up from the top surface of the central pipe to the outside edge of the glass tubing at four places 90° apart along the circumference. A compromise is affected between the high sides of one tube and the low sides of the other by rotation of the tubing on the chucks before final tightening of chuck jaws.

To mount the sections of glass tubing on the chucks we lifted and held the central 6 inch steel tube with the overhead crane, removed one "A" frame, slid the glass tubing over the open end, then replaced the "A" frame. The ends of the tubing were sealed with galvanized metal covers held in place with heat resistant tape to make the assembly air tight. Access to the interior for blowing was made through the shaft of the handwheel and holes were drilled in the 6 inch steel pipe to admit pressure of blowing to inside of glass assembly.

After sufficient preheating, the oxygen hydrogen burners were wheeled into place, lit, and adjusted for maximum heat (Figure 3). At the point that the heated ends began to bead up and get noticeably heavier, the movable chuck was engaged, closing the gap between the ends of the glass tubing. Heating was continued until a smooth seal was accomplished. Although provision was made to blow into the center area, it was not necessary, as the speed of rotation (20 rpm) kept the glass from either spinning out or shrinking in. Sealing time was about 15 minutes per seal (Figure 4). Only in the later stages of sealing was it found necessary to paddle the seal with a graphite tool to maintain roundness, this due largely to uneven wall thickness in the glass cylinder.

Glassblowers were protected from excessive heat during sealing operations with aluminum coated full length shop coats, and welders face masks. The use of these safety devices allowed us to move at will as close as we wished to the heated zone.



Figure 4

ANNEALING OVEN MODIFICATIONS

A 4 foot deep, 3 foot high and 2 foot wide Lydon annealing oven is currently in use in the glass shop facilities, and it was decided that if a "snorkel" were built to accommodate the additional length of tubing, a suitable annealing operation could be performed. An 8 foot by 2 foot steel table was constructed, set on casters, and an extension, 2 foot square and 8 foot long was constructed of 2 inch thick Thermobestos Insulation, and set on this table to retain the heat of the oven. The distance from either end of the 18 inch tube to the sealed area was 4 feet, and all seals on the heat exchanger were at the ends, therefore no additional heating was used in the "snorkel". It merely served to seal the oven door and extend the heated space (Figure 5).

Measurements of the temperature showed the range to be 375°C . at the outboard end, and 555°C . in the oven proper. The tubes were held at this temperature for one hour and subsequent viewing with polarized light showed the seals to be strain free.



Figure 5

FABRICATING THE 21 TUBE HEAT EXCHANGER

Rings were made by sealing sections of an arc of 1 inch I.D. heavy walled tubing, generating a glass ring $16\frac{1}{2}$ inches O.D. (Fig. 6). A fixture to hold short sections of 1 inch I.D. tubing was made, and twenty-one 3 inch long sections of 1 inch I.D. tubing were "T"ed into the rings, seals made with a hand torch, and annealed (Figure 7).

The ends of the 3 inch glass sections were cut off evenly with a hot wire and the whole assembly annealed. Then, ten foot long sections of 1 inch I.D. tubing were strapped to Transite forms with bands of stainless steel. Spacing of the ten foot tubes was determined by the spacing of the "T"s in the end rings. Closed circuit seals were then made between the parallel tubes and the end rings.

Three Transite spool forms were used, supported by a central 3 inch steel pipe, giving us a means of transporting the assembly to the anneal-



Figure 6

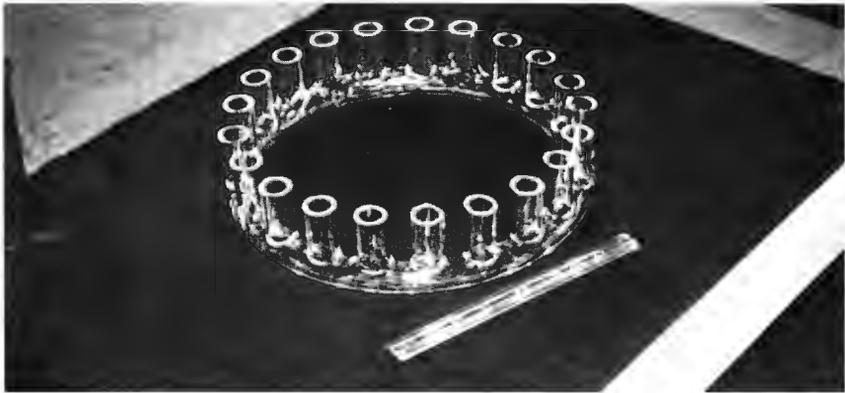


Figure 7

ing oven. The Transite spools were designed so that they could be dismantled and drawn out from between the parallel tubes at the completion of all sealing operations (Figure 8).

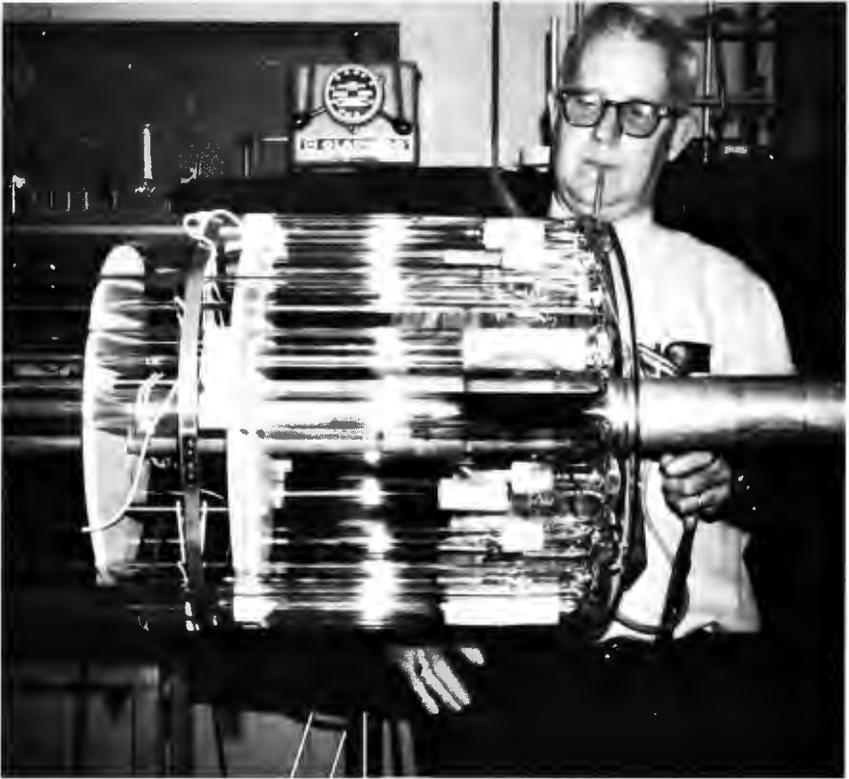


Figure 8

A "blind seal" was made at one end of one 10 foot, 1 inch I.D. and one, 1 inch I.D. tube sealed to the outboard end of one ring to affect a filling tube and an exit tube for liquid nitrogen. These tubes will be connected to a metal bellows and then to a Kovar to Pyrex seal through the stainless steel shell for filling with liquid nitrogen in a flow system. From a reservoir outside the vacuum chamber, cold nitrogen gas is forced into the heat exchanger from one end, then around one ring, then through twenty parallel tubes, to the other ring, then out through the exit tube. The cold gas will be sent through the heat exchanger until the point where the temperature is lowered sufficiently to retain liquid nitrogen. In this application both entry and exit tubes for the liquid nitrogen are at the same ends of the heat exchanger. It will hold about 35 liters of liquid nitrogen when in use.

Handling this assembly was done by lifting only at the ends of the protruding 3 inch steel pipe, and it was supported in the annealing oven at one end with a trolley, allowing us to insert one end, then roll assembly to back of oven. With one end positioned at the back wall of the oven, the other end supported from above, the oven extension "snorkel" was rolled

into place to support the other end. A cover of the Thermobestos Insulation was then put into place, sealing the opening in the annealing oven and the extension as well.

Some sagging was detected after a number of annealing cycles, but rotation of the annealing position minimized this tendency. The Transite forms, the 3 inch pipe and straps brought the entire weight up to about 210 pounds, so the support from the inside through the ends of the 3 inch pipe was very necessary.

The 18 inch tube was transported by as many as 6 men, using 3 inch canvas straps and a "T" bar support at 3 places along its length. Elevators were large enough to accommodate it, but because of the protruding ends of the 3 inch pipe on the heat exchanger, it will have to be swung into a fourth floor window with a crane. A skid or bed will be constructed at that time to protect it in transit.

It is well to note that by increasing the length of the central 6 inch steel tube, a continuous section of practically any length could have been constructed at no appreciable increase in the cost of the machine. The techniques described herein could also be adapted to different diameters and lengths as well.

The heat exchanger has to withstand a pressure differential of 1 atmosphere, and be high vacuum tight, so was pumped by glass shop personnel, then leak chased with a Tesla coil and a helium leak detector. The 18 inch tube defines the area of the glow discharge, and insulates the discharge from the stainless tube when the apparatus is in operation.

CONCLUSION

The completion of this project demonstrates the fact that while it was initially felt that the construction of this apparatus might be beyond the capabilities of the Chemistry Department Glass Shop, through the splendid cooperation of Dr. Claude Woods, Russell Riley and Michael Wheeler and others on the staff of the Chemistry Department, the assemblies were made with a minimum of effort and cost, using, to a large extent, existing equipment and facilities.

¹Corning Glass Works, Corning, New York.

THERMAL EXPANSION MISMATCH AND STRESS IN SEALS

H. E. HAGY

Corning Glass Works
Research and Development Laboratory
Corning, New York 14830

ABSTRACT

A basic discussion is presented on thermal expansion mismatch, stress-strain relationships, and allowable stress level criteria to establish a better understanding and working knowledge of the Thermal Expansion Differential Tables published by Corning. The nature and behavior of the expansion curve of glass is discussed with particular attention to the setting point concept for predicting mismatch. A simple explanation on how and why stresses develop in seals is coupled with an approximation formula that can be easily and practically used in the shop. The strength of glass is touched upon briefly to give an understanding of permissible stress levels generally quoted for annealed glass. Principles covered apply to glass-to-glass, glass-to-metal, and glass-to-ceramic seals.

1. INTRODUCTION

For many years Corning Glass Works has published an aid to those involved in glass sealing applications known as the Thermal Expansion Differential Tables. These are distributed on a hand-out basis mostly by sales and engineering representatives of Corning. Upgrading is planned every several years to introduce new compositions and delete obsolete glasses. A new version is probably past-due, as the last revision was in 1962.

The purpose of this paper is to give those who find the tables useful a better insight into how the tables are derived, what significance the numbers have, and the basis for establishing practical limits. To do this, four principal physical properties of glass must be discussed: elasticity, strength, viscosity, and thermal expansion. Finally, approximation formulae are given with which stresses can be estimated from the differentials.

2. THE TABLES — GENERAL EXPLANATION

The 1962 revision of the tables is reproduced in Figures 1 and 2 with the thought that, although many readers have copies, by being included in this paper they might be a more useful and permanent reference. The explanation which accompanies copies of the tables is repeated here in the following paragraphs.

“Tabular values are expansion differentials between possible sealing pairs for glass-to-glass, glass-to-metal, and glass-to-ceramic seals. The following explains tabular notation and the basis on which these differentials were determined.

1. Differentials represent the difference between the two expansion curves in question at the setting point of the glass, in the case of a glass-to-metal seal, or at the setting point of the softer glass, in the case of a glass-to-glass seal. Values given are in parts per million.

2. The setting point has been arbitrarily defined as 5°C above the strain point of a glass.

3. A number with a negative sign directly above it indicates that the material listed in that row has a lower effective expansion than the material listed in that column. The omission of a negative sign signifies a positive value.

4. Differentials in excess of 1000 parts per million are indicated by either a large negative or positive sign, depending on the direction of the mismatch.

5. Metal-to-metal and metal-to-ceramic combinations are of no interest in glass sealing and are indicated by a large X.

Caution should be observed in attempting to predict residual stress in a seal from the tabulated differentials, since in any particular seal the setting point is a function of:

A. *The geometry of the seal.* The use of strain point plus 5°C for the revised tables is a departure from previous editions, in which the midpoint between the annealing and strain points was used. The work of Hagy and Ritland⁽¹⁾ has shown that the setting point can be either above or below the strain point, depending on geometry. The new definition is applicable for a symmetrical parallel-plate seal and seems to be a good compromise for the range of setting points observed. For the most part, only minor changes in the differentials are brought about by this redefinition.

B. *The cooling rate* used in annealing the seal. Both viscosity⁽¹⁾ and thermal expansion⁽²⁾ of a glass are rate dependent, thus affecting the setting point and the differential. A cooling rate of 1°C per minute was used in the determination of the expansion curves (actually contraction curves) from which the differentials were calculated.

C. *The difference in annealing ranges*, in the case of a glass-to-glass seal. A seal between glasses having similar annealing ranges will have, in general, a lower setting point than normal.

Despite these reservations, the tables should prove to be useful guides in making decisions in sealing problems. As a general rule, any differential less than 100 parts per million can be interpreted as a very good sealing condition. Good seals can be made between materials with differentials ranging from 100 to 500 parts per million. However if two materials having a differential at the higher end of this range are chosen for a sealing application, the risk of failure is greater and, therefore, more care should be exercised in making the seal. Materials with differentials from 500 to 1000 parts per million can be used for progressively smaller and thinner seals."

3. ELASTICITY — BRITTLINESS

It is frequently stated that glass is brittle; but, although this is a fact, many people do not have an exact idea of what brittleness means. The essence of brittleness lies in elastic behavior, and thus it is necessary to briefly review stress and strain.

If forces are applied to a body, it deforms. The amount of deformation is a measure of the strain which the body experiences. For instance, imagine a bar exactly ten inches in length which is to be subjected to axial tensile forces. Suppose that careful gaging shows that when the forces are applied the length increases by 0.0100 inch. The imposed strain is the change in length (ΔL) divided by the original length (L), or $\Delta L/L$, and in this case is $0.0100 \div 10.000$ inches or 0.00100 in/in. Note that the strain is really unitless. Therefore the in/in can be dropped and other ways of expressing the same number would be 0.100% or 1000 parts per million (ppm).

Furthermore, the bonding forces within the body resist this deformation or strain. This brings up the notion of stress. As the internal bonds

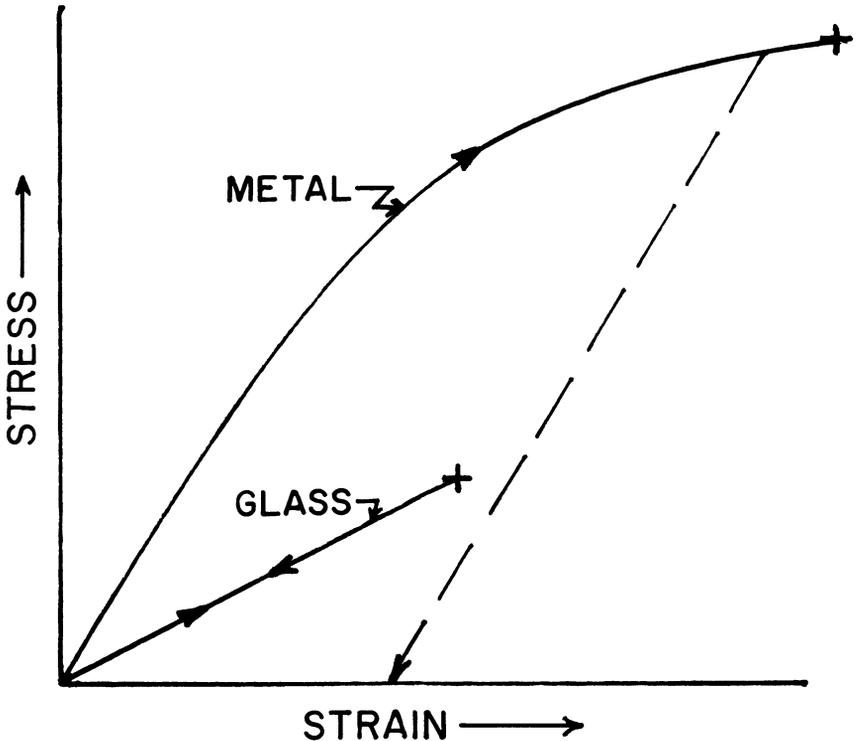


Figure 3
Stress-Strain Curves for Glass and Metal

are stretched, internal forces are created and the stress is the average force divided by the cross sectional area being strained. Stress has the units therefore of force per unit area and typical units generally expressed are pounds per square inch (psi), kilograms per square millimeter, etc. A balance exists between the internal and external forces. Hence the stress can also be calculated from the imposed force and the cross sectional area.

A stress vs. strain plot as shown in Figure 3 aids in furthering our understanding of elastic behavior and brittleness. Here we see quite different curves generated for glass and metal. The curve for glass is a straight line to the stress where fracture occurs, shown by a (+) sign. If stressing were to be reversed to zero before failure occurred, the strain would also return to zero. In other words, no permanent distortion has resulted. This is perfect elastic behavior—the criterion for brittleness. The slope of the stress-strain curve is known as the elastic modulus and this ideal proportionality between stress and strain is Hooke's law.

On the other hand, notice the behavior of the metal. As the stress is increased, more strain develops for each interval of stress. This is due to the fact that permanent distortion or creep is occurring. In fact, if the stressing were to be reversed to zero before fracture, a permanent strain would result as shown in the figure.

4. STRENGTH — STRAIN TOLERANCE

We are told that the theoretical strength of glass is in the order of one to four million pounds per square inch, and yet, for safety, tensile stresses of 1000 psi should not be exceeded. How can one explain this large discrepancy?

The major factor that accounts for this is surface damage. Handling, even in moderate ways, can cause surface flaws which, even though minute, produce severe stress concentrations when the glass is subjected to a tensile stress. Consider a flaw in the form of a notch as shown in Figure 4. With a tensile stress applied as shown by the large arrows, a

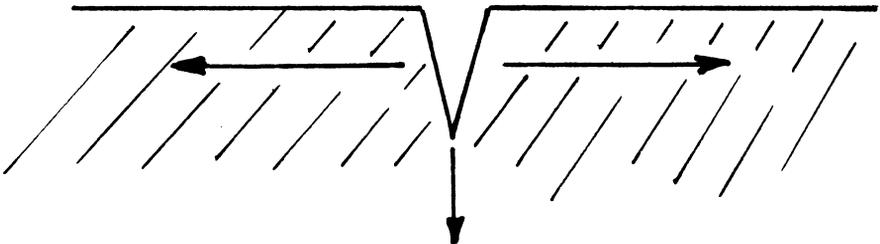


Figure 4

Stress Magnification by Surface Flaw

tremendous magnification of this stress occurs at the tip of the flaw. Estimates of the stress concentration factor have been made as high as several hundred fold. Thus an applied stress of 10,000 psi can be magnified by the flaw geometry to a magnitude of 2-million psi. Therefore, if fracture occurs in such a situation, the glass is really breaking at the theoretical level.

But where does the 1000 psi design stress limit come from? Moderate abrasion of annealed glass surfaces, as by sandblasting for instance, lowers the strength to about 8000 psi. More severe surface abrasion can reduce the strength to even lower values — even to half this value. However, taking the 8000 psi. as a starting point we immediately lower this to 4000 psi. for long times under stress. This is because the 8000 psi. figure is arrived at with short duration (~ 1 minute) loading conditions. If a stress of 4000 psi were applied to the same sandblasted glass specimens, failure would eventually occur, although it may take several months. This is called the stress-time or static fatigue effect and it is apparently due to stress-induced corrosion at the tip of the flow due to water vapor.

Realistically, we can never really be sure about the severity of the flaws in a particular piece of glass earmarked for a long-term application. Therefore, to surely be on the safe side, a safety factor of four is applied which then brings the design level down to 1000 psi. This is the limit generally regarded as safe for large pieces of glass. There is another factor involved in the strength of glass that should be briefly discussed and this is the size or area effect. This factor simply and logically comes about from the fact that a small piece of glass will have less chance to have a serious flaw from a probability standpoint. Furthermore, the smaller mass involved in a small article of glass argues for less severe flaws from impact, scuffing, or simply resting on an abrasive grit. Thus, we are able to allow higher working stresses for smaller and smaller articles.

In the previous section we talked about the elastic modulus as being the ratio of stress to strain:

$$\text{elastic modulus} = \frac{\text{stress}}{\text{strain}}$$

and

$$\text{stress} = \frac{\text{stress}}{\text{elastic modulus}} \cdot$$

The elastic modulus falls between 8 million and 12 million psi for most commercial glasses. If the elastic modulus for a particular glass is not known, an assumed value of 10 million psi will probably lead to an error of less than 20 percent. Taking this value as an average modulus for glass and using the design stress of 1000 psi for larger articles, we get

$$\text{strain} = \frac{1000 \text{ psi}}{10 \times 10^6 \text{ psi}} = 100 \times 10^{-6} \text{ or } 100 \text{ ppm.}$$

In the explanation of the tables quoted in Section 2 it is emphasized that good seals can be made when the expansion differential is 100 ppm

or less. The reader can now appreciate the origin of this limit and also understand why progressively smaller seals can tolerate higher values — although a greater risk is still present.

5. THE SETTING POINT

Ordinary commercial glasses have no melting point and no associated abrupt change in viscosity like metals have when they change state on freezing or melting. The viscosity-temperature curve for glass is a smoothly changing function with this property decreasing some twenty orders of magnitude as the temperature is increased from room temperature to above 1200°C.

With this in mind, it is obvious that in making a seal it is necessary to heat to a sufficiently high temperature to reduce the viscosity to a point where the glass will flow to conform to the geometry of its sealing mate and make a proper sealing joint. Then it is necessary to cool the seal at a slow rate through the annealing range to avoid excessive thermal residual stresses in the glass.

What happens during cooling as the glass and its sealing mate want to contract at different rates due to expansion differences? Take the case of a glass and a metal as shown in Figure 5.

In the upper part of the figure the contraction curves are shown for both the glass and the metal. These are adjusted a proper amount on the expansion scale to illustrate a point. The lower portion of the figure presents two stress-temperature curves. One of these is physically realistic and the other is fictitious, but both produce the same resulting stress at room temperature. The dashed line in the lower portion is the difference between the two contraction curves plotted with a different scale factor. Thus when the glass is rigid and elastic, the dashed line will fall on the stress curve (when stress is proportional to strain).

Consider the realistic curve which follows the course A→C→D. As the seal is cooling, the viscosity of the glass increases to the point where it will not completely release the stress tending to develop because of expansion differences. Thus, at point A we see stress start to develop in the glass and follow the curve shown with a cross-over in sign a little above room temperature (point D). From point A to point C the glass viscosity has been increasing smoothly from about 10^{11} poises to 10^{16} poises.

The fictitious curve A→B→C→D produces the same stress at room temperature and, in fact, is identical below point C. This curve would be produced if the viscosity of the glass were zero above point B and infinite below point B. Point B is therefore the setting point temperature. This gives the essence of the idea of the setting point of glass — “that temperature where the glass *effectively* transforms from a flowing body to a rigid elastic body.” This happens near the strain point for all glasses and the actual temperature in relation to the strain point depends on the seal geometry. A good “average” value is 5°C above the strain point — the value used in making the tables.

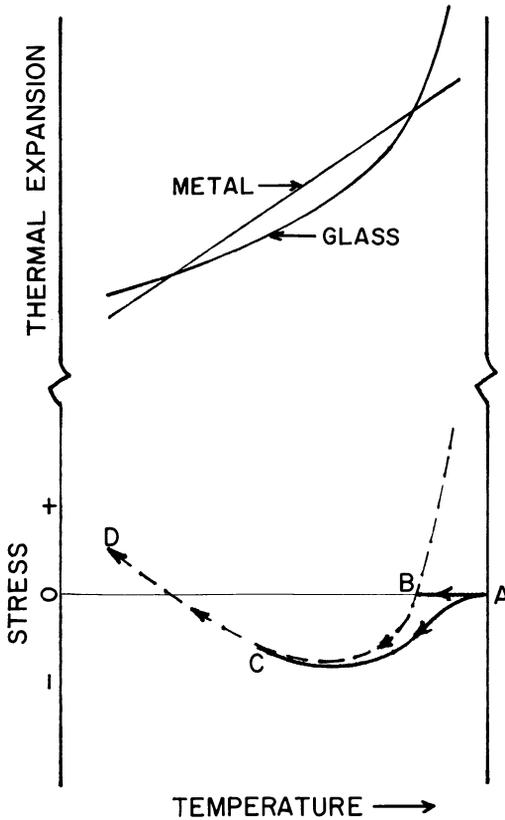


Figure 5

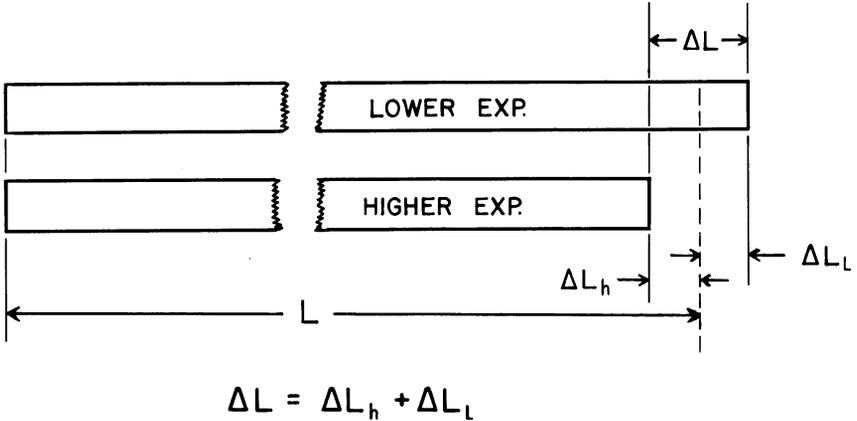
Expansion Differences between Metal and Glass and Stress Development in a Seal as a Function of Temperature

As the tables' explanation points out, when two glasses are sealed together, the glass with the lower strain point determines the setting point.

6. DIVISION OF MISMATCH — STRESS ESTIMATION

Consider two materials sealed together that have differing expansions. At the setting point the dimensions of the two bodies at the seal plane are equal by virtue of viscous flow in the glass. However, on cooling to room temperature, the higher expansion body will want to assume a smaller dimension than the lower expansion member. To visualize what happens in this physical disagreement, refer to Figure 6. Here we have divorced the two bodies and show the free contraction length at room temperature. But in the actual case when they are joined they must assume the same dimension (dashed line) if the seal is to stay intact. Thus the higher expansion material is forced to a longer dimension and experiences tension and the lower expansion material is forced to a smaller

dimension and experiences compression. The writer recognized that such a seal will bend, but, for simplicity, we can ignore this. The figure shows how the expansion differential or mismatch is divided so that the sum of the two strains equals the differential. The differential $\Delta L/L$ is the number given in the tables for a given sealing combination.



$$\text{EXP. MISMATCH} = \frac{\Delta L}{L} = \frac{\Delta L_h}{L} + \frac{\Delta L_L}{L} \quad (\text{STRAIN UNITS})$$

Figure 6

Illustration of How the Expansion Differential is Divided between Two Materials in a Seal

The following equation can be used to approximate strain distribution and stresses in seals:

$$\frac{\Delta L_\ell}{L} = \frac{\Delta L/L}{\left| 1 + \left(\frac{t_\ell}{t_h} \right) \cdot \left(\frac{E_\ell}{E_h} \right) \cdot \left(\frac{1 - \gamma_h}{1 - \gamma_\ell} \right) \right|},$$

where E_ℓ and E_h are the elastic moduli of the two materials, t and t_h are the thickness at the joint, γ_h and γ_ℓ are the Poisson ratios.

As an example, take the case where

$$\begin{aligned} \Delta L/L &= 200 \text{ ppm} \\ E_\ell &= 9,000,000 \text{ psi (glass)}, \gamma_\ell = 0.20 \\ E_h &= 27,000,000 \text{ psi (metal)}, \gamma_h = 0.30 \\ t_\ell &= 0.10'' \\ t_h &= 0.20'' \end{aligned}$$

Therefore: $\left(\frac{E_\ell}{E_h}\right) = 1/3; \left(\frac{t_\ell}{t_h}\right) = 1/2; \left(\frac{1 - \gamma_h}{1 - \gamma_\ell}\right) = 7/8$

$$\frac{\Delta L_\ell}{L} = \left| \frac{200 \text{ ppm}}{1 + (1/2)(1/3)(7/8)} \right| = \frac{200}{1.146} = 175 \text{ ppm}$$

$$\frac{\Delta L_h}{L} = 25 \text{ ppm (by difference)}$$

To calculate the stress:

$$\text{Stress} = \frac{\text{Strain} \times E}{(1 - \gamma)}$$

$$\text{Stress (glass)} = \frac{175 \times 10^{-6} \times 9 \times 10^6}{0.80} = 1960 \text{ psi (compression)}$$

$$\text{Stress (metal)} = \frac{25 \times 10^{-6} \times 27 \times 10^6}{0.70} = 980 \text{ psi (tension)}$$

7. SUMMARY

The writer has presented a more extensive explanation for the Corning Glass Works Expansion Differential Tables. The interrelationships of expansion, elastic behavior, and strength were discussed to give a better understanding of glass strain tolerance.

A simple formula is suggested for estimating seal stresses, and a practical example given. It is emphasized that the formula is for estimation purposes only and not for rigorous stress analyses.

REFERENCES

1. Hagy, H. E. and Ritland, H. N., "Viscous Flow in Glass-to-Metal Seals," J. Am. Ceram. Soc., **40** (2) 58-62, 1957.
2. Hagy, H. E. and Ritland, H. N., "Effect of Thermal History on Glass Expansion Characteristics," J. Am. Ceram. Soc., **40** (12) 436-442, 1957.

IN ATTENDANCE

The following are on record as having attended the Sixteenth Symposium on the Art of Glassblowing held at the Pfister Hotel, Milwaukee, Wisconsin, June 30th, July 1st, 2nd, 1971. As a fully paid registered participant, these persons are entitled to a copy of the Proceedings.

REGISTRATION LIST — MEMBERS

Adkins, M. Wayne Okla. State Univ., Stillwater, OK
Agam, Benjamin Tel-Aviv University, Dept. of Chemistry,
Glassblower Lab., Tel-Aviv, Israel
Albert, Ferenc J. I.B.M., E. Fishkill, NY
Alexander, J. Allen 20 Merwood Dr., Upper Darby, PA 19082
Alexander, Peter University of Alberta, Physics Dept., Ed-
monton, Alberta, Canada
Alexandre, Norman University of Colorado, Boulder, CO
Amling, Charles M. University of Notre Dame, Notre Dame, IN
Anderberg, Dane H. Lawrence Radiation Lab., Berkeley, CA
94720
Andrews, Frank Wheaton Glass Co., 3rd & G Sts., Millville,
NJ
Arias, Arthur Hycel, Inc., P.O. Box 36329, Houston, TX
Armstrong, Gilbert Armstrong Art Glass, 5N432 Neva Ter.
Itasca, IL 60143
Ball, William B. Eastman Kodak Co., Rochester, NY
Barr, William E., Consultant . 1620 Powers Run Rd., Pittsburgh, PA 15238
Becke, Helmut Bell-Northern Research, Ottawa, Ont., Can.
Benner, Wayne H. Miami University, Oxford, OH 45056
Betchner, Neil J. L. G. Nester Co., P.O. Box #666, Millville,
NJ 08332
Bishop, Jim Battelle, 505 King Ave., Columbus, OH
43201
Bivins, John H. Philip Morris, Inc., P.O. Box 26583, Rich-
mond, VA 23261
Black, Gerald G. Hewlett Packard Co., 5301 Stevens Creek
Blvd., Santa Clara, CA 95050
Blessing, David University of Notre Dame, Notre Dame, IN
46556
Blomquist, Theodore V. Harry Diamond Labs., Washington, DC
Bolan, Theodore W. Philips Labs., 345 Scarborough Rd., Briar-
cliff Manor, NY 10510
Boussert, Christian State Univ. of New York, Chemistry Dept.,
Stony Brook, NY 11730
Brandler, Frank Hoffman-LaRoche Inc., Nutley, NJ 07110

Brereton, Walter Univ. of New Brunswick, Fredericton, N.B.,
 Canada
 Brosious, Edward C. Yale University, 217 Prospect St., New
 Haven, CT 06520
 Burt, Stewart W. Xerox Corp., Xerox Square, Rochester NY
 14604
 Buttino, Albert L. Albo Associates, 13 Miles Standish Rd.,
 Schenectady, NY 12306
 Cabaniss, Robert Univ. of Missouri, St. Louis, MO
 Campbell, Clair Battelle, 505 King Ave., Columbus, OH
 43201
 Campbell, Robert G. Trent University, Peterborough, Ont., Can.
 Capurso, Maurice ITEK Corp., 10 Maguire Rd., Lexington,
 MA
 Carter, Frank B. M.I.T. Lincoln Lab., Wood St., Lexington,
 MA 02173
 Caselli, Michael J. Argonne National Lab., 9700 Cass, Argonne,
 IL
 Cassidy, C. J. Westinghouse R & D Center, Beulah Rd.,
 Churchill Boro, Pittsburgh, PA
 Cavanagh, John R. RR#3, Box 11322, Brownsburg, IN 46112
 Chandler, David Univ. of Waterloo, Chem. Ens., Waterloo,
 Ont., Canada
 Chatas, Lawrence Chatas Glass Co., Vineland, NJ 08360
 Christie, Henry L. Carleton University, Ottawa, Ont., Canada
 Cicero, Philip Cicero Scientific Glass, 308 W. Empire Ave.,
 Benton Harbor, MI 49022
 Clements, Edwin H. Owens-Illinois, 1700 N. Westwood, Toledo,
 OH
 Cosgrove, George D. Sandia Corp., Livermore, CA
 Cristaudo, Lorenzo Southern Illinois Univ., Carbondale, IL
 62901
 DeAngelis, William Technicon, Tarrytown, NY
 Deery, Edward J. Heights Lab. Glass Inc., Box 604, Ardsley,
 NY 10502
 DeFelice, A. S. Owens-Illinois, Inc., Levis Devel. Pk., 25875
 U.S. Rt. 25, Perrysburg, OH 43551
 Deminet, Ches Boeing Company, P.O. Box 3981, Seattle,
 WN 93124
 DeWolff, William N. The Upjohn Co., 301 Henrietta St., Kala-
 mazoo, MI 49001
 Dolenga, Arthur General Motors Res. Lab., 12 Mile and
 Mound, Warren, MI
 Dollé, André Rauland Co., 5600 Jarvis, Niles, IL 60648

Dombi, Geza Natl. Ctr. for Atmospheric Res., 1850 Table
 Mesa Dr., Boulder, CO 80302
 Dorsi, David C. Bell Telephone Labs., Murray Hill, NJ
 Doody, Thomas J. Argonne National Lab., 9700 S. Cass, Ar-
 gonne, IL
 Derbick, S. C. G.E., X-Ray, Milwaukee, WI
 Dehn, William R. National Institutes of Health, Spencerville,
 MD
 Dougherty, Richard E. University of Arkansas, Rm. 104 S.E. Bldg.,
 Fayetteville, Ark. 72701
 Eckberg, Edwin E. AirForce Cambridge Res. Labs., Hanscom
 Fld., Bedford, MA
 Eisenberg, Arnold University of Victoria, Victoria, B.C., Can.
 Elsholz, William E. Univ. of California, Lawrence Rad. Lab.,
 P.O. Box 808, Livermore, CA
 Epperson, Howard L. University of Georgia, Athens, GA
 Frain, Chizuko GlassMasters, Norwalk, CT
 Fox, Joseph J. National Institutes of Health, Bethesda, MD
 Frederiksen, Chris 7415 W. Madison, Forest Park, IL 60130
 Frejosky, Andrew Lewis Research N.A.S.A., 21000 Brookpark
 Rd., Cleveland, OH
 Gass, G. J. Bell Telephone Lab., Whippany, NJ
 Gilhooley, William A. General Electric Corp., Res. & Dev. Ctr.,
 Schenectady, NY
 Glover, John A. Atlantic Richfield Co., Harvey, IL
 Goldman, David Polytechnic Inst. of Brooklyn, 333 Jay St.,
 Brooklyn, NY
 Good, Gordon Univ. of Mass., Goessmann Lab., Amherst,
 MA
 Goodman, William D. Olin Corp.—Res. Ctr., 275 Winchester
 Ave., New Haven, CT
 Gordon, Mel Coulter Electric, 590 W. 20th St., Hialeah,
 FL
 Gorham, Jack S. Columbia Univ.—Rad. Lab., 538 W. 120
 St., New York, NY 10027
 Graul, William C. Dow Chemical Co., Midland, MI
 Green, Harry Univ. of California, Lawrence Radiation
 Lab., Livermore, CA
 Gregar, Joseph S. 3235 N. Summit, Milwaukee, WI 53211
 Grout, John B. Calif. State College at L.A., 5151 State
 College Dr., Los Angeles, CA 90032
 Greiner, Siegfried Rauland, Div. of Zenith, 5600 W. Jarvis
 Ave., Niles, IL 60648
 Gutilla, Sam J. 9681 Allen Ave., Rosemont, IL 60618

Haak, W. H. R.R. 12 Box 395, Lafayette, IN 47905
 Hagedorn, James A. Univ. of Illinois, Chicago Campus Box 4348,
 Chicago, IL 60680
 Halmbacher, Paul Pope Scientific Inc., P.O. Box 495, Meno-
 monee Falls, WI 53051
 Henson, Thomas A. Duke University, Dept. of Physics, Dur-
 ham, NC
 Hernandez, Peter Energy Mines & Resources, 568 Booth St.,
 Ottawa, Ont., Canada
 Hill, George H. Univ. of Waterloo, Waterloo, Ont., Canada
 Hill, Wallace G. Voltarc Tubes, Inc., 102 Linwood Ave.,
 Fairfield, CT 06430
 Hostetter, T. A. (Miss) Univ. of Guelph, Guelph, Ont., Canada
 Huth, Harry J. Washington Univ. School of Med., St.
 Louis, MO
 Hydro, George WRAIR-Walter Reed Army Med. Ctr.,
 Washington, DC
 Hyland, Edwin J. Northern Illinois Univ., DeKalb, IL
 Jackson, Edmund J. Studio of Glass Blowing, 9 Fifth St., Dravos-
 burg, PA 15034
 Jackson, Thomas W. American University, Mass. & Neb. Aves.,
 N.W., Washington, DC 20016
 Jacobs, Susan M. (Miss) Decalcraft Limited, 10 Queen Elizabeth
 Blvd., Toronto, 18, Ont., Canada
 Jones, Keith A. Atlas Chemical Ind. Inc., Concord Pike &
 Murphy Rds., Wilmington, DE
 Jubera, Andrew M. 4400 Fifth Ave., Pittsburgh, PA 15213
 Kalbin, Alex J. IBM Corp., Monterey & Cottle Rds., San
 Jose, CA 95114
 Kern, Thomas Procter & Gamble Co., Ivory Dale, OH
 Ketchum, Richard A. Bendix Res. Labs., Bendix Ctr., Southfield,
 MI 48076
 Kingsbury, Owen 207 N. Eastern St., Greenville, NC 27834
 Kleinert, Richard A. I.B.M., Kingston, NY
 Knisely, Samuel E. Mobil Oil Res. & Dev., Paulsboro, NJ
 Koch, Edward Sterling Glass Labs. Inc., 267 Main Ave.,
 Stirling, NJ 07980
 Kocsi, Andrew Route #138, West Kingston, RI 02892
 Korosi, Michael State Univ. of New York at Buffalo, Buf-
 falo, NY
 Kowalski, Edward Sandoz-Wander, Inc., Route 10, East Han-
 over, NJ 07936
 Kraus, John R. DuPont Experimental Station, Wilmington,
 DE

Kresge, BenjNational Vacuum Labs., 1125 Longwood
 Ave., New York, NY
 Kummer, Egon M.Daran Products Inc., 227 Westport Ave.,
 Norwalk, CT
 Kummer, Fred H.Brookhaven Nat. Labs., Upton, Long Is-
 land, NY
 Langer, ManfredKontes-Martin, 1917 Greenleaf Ave., Evan-
 ston, IL
 Langbein, C. E.Gulf Research & Dev. Co., P.O. Drawer
 2038, Pittsburgh, PA 15230
 Langley, Russel W.Continental Glass Co., Richland, NJ
 Last, C. S.National Res. Council of Can., Ottawa,
 Ont., Can. K1A OR9
 Legge, J. E.University of Toronto, Dept. of Physics,
 Toronto, Ont., Can.
 Lenzi, David J.U.S. Army Med. Res. Lab., Fort Knox, KY
 Leo, Salvatore R.Gulf Research & Dev. Co., Harmarville, PA
 Leslie, FredUniversity of Toronto, 200 College St., Tor-
 onto, Ont., Canada
 Lewis, Thomas L.Crown Zellerbach Corp., Central Res. Dept.,
 Camas, WA 98607
 Liebson, MorrisU.S.M.A.—Physics Dept., West Point NY
 10996
 Lillie, Don1165 Oakdale Dr., Smyrna, GA 30080
 Litton, Charles V.Litton Eng. Labs., Dayton, NV
 Loguidice, John B.9681 Allen, Bosesmont, IL 60018
 Lord, Edward E.Texas Woman's Univ., Denton, TX
 Lysandrou, Lysander4306 N. Whipple, Chicago, IL 60618
 McKisick, Robert V.National Bureau of Standards, Boulder, CO
 80302
 Mason, Austin6015 Milne Blvd., New Orleans, LA 70124
 Mateyka, Wilbur C.Univ. of Ky., Chem. Dept., Lexington, KY
 40506
 Mehta, S. L.Howard Univ., Dept. of Chem., Washington,
 DC 20001
 Merriam, Donald R.Procter & Gamble Co., Miami Valley Labs.,
 Cincinnati, OH 45239
 Mezynski, StanleyN.C. State Univ., Physics Dept., Raleigh,
 NC 27607
 Michiel, William G.66 Rochester, Carleton Place, Ont., Canada
 Mikols, Stanley J.Dow Chemical Co., Midland, MI 48640
 Miller, Robert F.Eli Lilly & Co., P.O. Box 618, Indianapolis,
 IN
 Mistry, KekiMich. State Univ., Chem. Dept., E. Lans-
 ing, MI 48823

- Moegenbier, Josef Rauland Div. of Zenith Radio, 5600 W. Jarvis, Niles, IL
- Moffitt, Agnes E. Bell & Howell, Pasadena, CA
- Morgenfruh, Lothar F. Mobil R & D Corp., Inc., P.O. Box 1025, Princeton, NJ 08540
- Morris, James F. Northwestern Univ., Chem. Dept., Evanston, IL 60201
- Nagle, Earl R. State Univ. of NY, 1400 Washington Ave., Albany, NY
- Nash, Paul H. Ethyl Corp., Baton Rouge, LA
- Nazzewski, Mathew Sprague Electric Co., Marshall St., N. Adams, MA 01247
- Oatley, Donald W. I.B.M. Corp., P.O. Box A, Essex Junction, VT 05452
- O'Brien, Donald M. Univ. of Illinois, Urbana, IL 61803
- Old, J. H. Univ. of Southern Calif., Los Angeles, CA
- Pahl, Billie E. Dow Chemical Co., Midland, MI
- Page, J. A. L. Natl. Res. Council Div. of Biology, Ottawa, Ont., K1A, OR6, Can.
- Palmer, Robert Hooker Chemical Corp., Niagara Falls, NY
- Panzner, James E. Owens-Illinois, Inc., 1700 N. Westwood, Toledo, OH 43607
- Parillo, Edward V. General Electric Co., HITOL Dept. #931-Nela Park, Noble Rd., Cleveland, OH 44112
- Parks, George E. Neutron Devices Dept., General Electric Co., P.O. Box 11508, St. Petersburg, FL
- Perry, David E. Univ. Illinois, Chem. Eng., Urban, IL
- Pinkney, William A. Howard University, 2355 6th St., Washington, DC 20001
- Pollock, J. J. The Dow Chemical Co., Midland, MI
- Pye, A. W. National Res. Council, Radio Div., Montreal Rd., Ottawa, 7, Can.
- Rak, Steve Univ. British Columbia, Chem Dept., Vancouver, 8, B.C., Canada
- Reeves, Arthur G. 9310 SW 83, Miami, FL 33143
- Reinhardt, Karl E. R. Squibb & Sons, New Brunswick, NJ 08902
- Remkus, Stanley F. 858 Bank St., Painesville, OH 44077
- Rishel, James W. Univ. of North Carolina, Chapel Hill, NC
- Roark, Roger R. BIR Glass Inc., P.O. Box 1040, Pasadena, MD 21122
- Robertshaw, George I.C.I. Fibres Ltd., Hookstone Rd., Harrogate, Yorkshire, England

Roensch, Arno P. Los Alamos Scientific Lab., P.O. Box 1663,
 Los Alamos, NM
 Roman, Paul W. Brookhaven National Lab., Upton, L.I., NY
 Russell, Robert I. Proctor & Gamble, Cincinnati, OH
 Rothfels, John Deseret Test Center, Dugway, UT 84022
 Ryan, Lawrence W. Mass. Inst. of Technology, Cambridge, MA
 Safferling, Ottmar A. Brooklyn College of the City Univ. of New
 York, Brooklyn, NY
 Sales, William A. Box 158, Deerfield, IL 60015
 Sandlin, Wendell I. Environmental Protection Agency, F.W.A.C.
 College Station Rd., Athens, GA 30601
 Saoner, Joseph L. Daran Products Inc., Norwalk, CT
 Saulsbury, Claude Pfeiffer Glass Inc., 140 Bennington Dr.,
 Rochester, NY 19616
 Schipmann, Robert H. 12 Kendall Rd., Kendall Park, NJ 08824
 Schneider, Otto Eck & Krebs, Inc., 27-09 40 Ave., L.I. City,
 NY 11101
 Schulze, William Argonne National Lab., 9700 S. Cass Ave.,
 Argonne, IL 60439
 Schwartz, William C., Jr. Motorola Inc., 52nd St., Phoenix, AZ
 Schumann, Karl Columbia Univ., Chem. Dept., New York,
 NY
 Scott, Thomas S. Canadian Industries Ltd., Central Res. Lab.,
 McMasterville, Quebec, Canada
 Searle, Randolph H. E. I. du Pont de Nemours & Co., Inc., Savan-
 nah River Lab., Aiken, SC
 Seckman, Jon W. Lockheed Res. Lab., Palo Alto, CA 94304
 Severn, Peter J. University of Michigan, Ann Arbor, MI
 48118
 Sheward, Orus A. 2318 Amelia Circle, Tallahassee, FL
 Simon, Richard H. S. C. Johnson & Son, Inc., 1525 Howe St.,
 Racine, WI
 Sites, George A. Houdry Labs., Air Products & Chems. Co.,
 Linwood, PA
 Smart, David R. Lorillard Res. Ctr., 420 English St., Greens-
 boro, NC
 Stelmach, Chester E. Univ. Oil Prods. Co., 30 Algonquin Rd.,
 Des Plaines, IL 60016
 Smith, Howe Daran Products Inc., 227 Westport Ave.,
 Norwalk, CT
 Squeo, Guy J. American Oil Co., Whiting, IN
 Steed, Michael E. Univ. of Georgia, Athens, GA 30601
 Stone, Charles L. Univ. of North Carolina, Raleigh, NC
 Swopes, Chester A. Abbott Lab., 1400 Shordan Rd., N. Chicago,
 IL 60064

Szalkowski, Bruno Brigham Young Univ., Provo, UT 84601
 Tardif, Ralph Holobeam, Inc., 560 Winters Ave., Paramus,
 NJ 07652
 Teige, Roy W. 3345 Moraine Dr., Hubertus, WI 63033
 Thompson, Jr. Matt L. Univ. Res. Glassware, Box 51, Carrboro, NC
 27510
 van den Hoff, J. National Res. Council of Can., Div. of
 Chem., Sussex Dr., Ottawa, Ont., K1A
 OR6, Canada
 van Hespén, C. C. Univ. of Chicago, Dept. of Chem., Chicago,
 IL
 Volk, Rudolph Heights Lab. Glass, Yonkers, NY
 Wade, Melvin J. U.S. Army ECOM, Ft. Monmouth, NJ
 Walas, Joseph W., Jr. Univ. of Mass. (Goessmann Lab.), Amherst,
 MA 01002
 Walrod, Alfred H. 70 Alviso St., Santa Clara, CA 95050
 Walther, Karl Miller Place Rd., Middle Island, NY
 Ward, I. A. Atomic Energy of Canada Ltd., Pinawa,
 Manitoba, Can.
 Watson, Merrill B. Univ. of Alabama, University, AL
 Weiss, Gunther UML, 7323 NE 96th Ave., Portland, OR
 97208
 Weppner, Richard T. Natl. Bur. of Stds. LAD-274.00, Boulder CO
 80302
 Wesanko, J. G. Atomic Energy of Canada Ltd., Chalk
 River, Ont., Canada
 West, Joseph Roswell Park Mem. Inst., 6660 Elm St.,
 Buffalo, NY
 Wheeler, Mike Univ. of Wisconsin, Madison, WI
 Willey, Martin Corporate R&D Center, G.E. Co., P.O. Box
 1088, Schenectady, NY
 Williams, C. L. University of Hawaii, Honolulu, Hawaii
 William, Larry R. Univ. of Mass., Amherst, MA
 Wilson, Robert R. National Lead Co. of Ohio, P.O. Box 39158,
 Cincinnati, OH
 Zurek, Tony A. 3400 Bryn Mawr Dr., St. Charles, MO 63301

NON-MEMBERS

- Campbell, David R. Univ. of Wisconsin-Milwaukee, 3201 N. Maryland Ave., Milwaukee, WI
- Evans, Brin National Res. Council, 100 Sussex Dr., Room 2055, Ottawa, Can. K1A OR6
- Green, Richard Dr. Concordia College, Moorhead, MN 56500
- Hamon, Richard E. General Electric Co., Electronics Park — Bldg. 6, Syracuse, NY 13201
- Iovine, Michael Louisiana State University, New Orleans, LA
- Kuehl, Keith A. Univ. of Illinois, Urbana, IL
- Langley, Russel E. Continental Glass Co., Richland, NJ 08350
- Merritt, James Univ. of Minnesota, Glass Technology Services, S-146 Chem Annex Bldg., Minneapolis, MN
- McGirr, J. P. General Electric Co., P.O. Box 11508, St. Petersburg, FL 33733
- Opsahl, Kenneth L. MMM Company, St. Paul, MN
- Osty, Julius Argonne Natl. Lab., 9700 S. Cass Ave., Argonne, IL 60439
- Rice, George A. General Motors, Warren, MI
- Rieker, John A. Starlight Industries, Rosemont, PA 19010
- Sampson, Walter F. MMM Co., St. Paul, MN 55101
- Vermillion, Bill Vanderbilt University, Box 1521, Sta. B, Nashville, TN 37203

