Proceedings

THE TWENTY-FIRST SYMPOSIUM
ON THE
ART OF GLASSBLOWING

1976

THE

AMERICAN SCIENTIFIC GLASSBLOWERS SOCIETY

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THE TWENTY-FIRST SYMPOSIUM
ON THE
ART OF GLASSBLOWING

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OPTICAL RADIATION HAZARDS IN GLASSBLOWING*

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ABSTRACT

Heated glass can emit significant amounts of electromagnetic energy in the infrared portion of the optical spectrum, and this radiation can cause damage to the human eye. An evaluation of the hazard potential shows that many glassblowing operations can produce corneal infrared exposure intensities in excess of 0.01 W-cm^{-2}, which has been proposed elsewhere as a safe limit for chronic exposure. Recommended guidelines for the selection of protective eyewear are presented. Included is a study of the infrared transmission characteristics of several common types of filters and spectacle lenses.

INTRODUCTION

Damage to the lens of the eye from exposure to hot surfaces was first described in 1786. Since that time this occupational disease, sometimes called "glassworker's cataract," has been the subject of much discussion and investigation. It has been known for many years that the disease is caused by optical radiation from hot surfaces, and more recently the damage mechanism has come to be fairly well understood. The combined effects of automation and increased concern for worker health and safety have caused a reduction in the number of workers affected; this, coupled with the fact that it normally takes many years of exposure to produce noticeable damage, has resulted in a lack of quantitative human exposure data.

In this paper we will examine the optical radiation hazard to glassblowers, estimate the degree of the hazard, and discuss the effectiveness of protective equipment.

THE PHYSICS OF OPTICAL RADIATION

The electromagnetic (EM) spectrum is diagrammed in Figure 1. It can be seen that radio waves, microwaves, infrared, visible light, ultraviolet, x-rays, and gamma rays all are manifestations of the same phenomenon: electromagnetic radiation. Only the wavelengths are different. For the purposes of this discussion, we will define optical radiation as that portion of the EM spectrum in which the waves may be brought to a focus by a more-or-less conventional lens. These wavelengths include infrared (IR), visible light, and ultraviolet (UV), and may be most conveniently discussed in terms of nanometers (nm), when 1 nm = 10^{-9} meter (0.000000001 meter). Figure 2 shows this portion of the EM spectrum.

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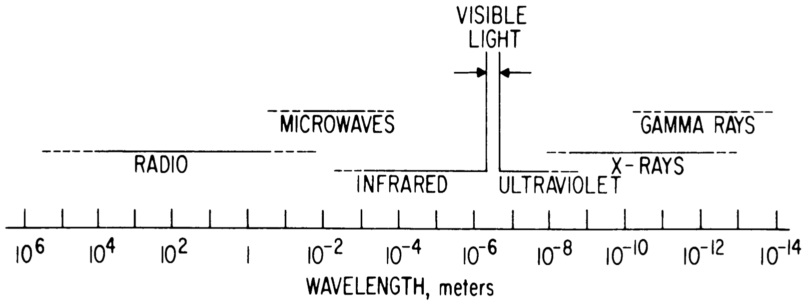
*Based on work performed under the auspices of the U. S. Energy Research and Development Administration



THE ELECTROMAGNETIC SPECTRUM

Figure 1

The electromagnetic spectrum (note logarithmic scale).

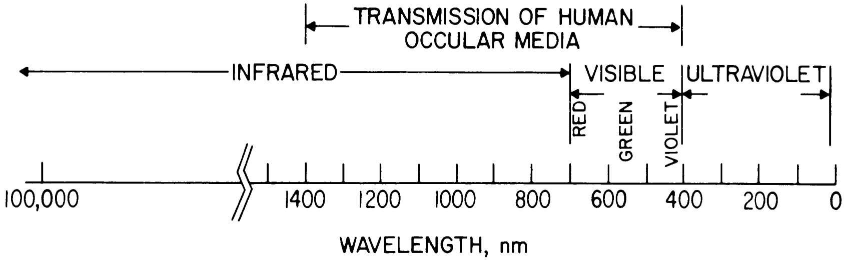


Figure 2

The infrared, visible, and ultraviolet portions of the electromagnetic spectrum.

Optical radiation is produced when an electron in an excited atom falls to a lower energy state. In a heated solid such as glass, atoms become excited as a result of their thermal motion; their electrons spontaneously fall to lower energy states (emitting EM radiation in the process), only to be raised back to an excited state by the input of more thermal energy. Thus, the process of excitation and emission is continuous, as long as thermal energy is supplied. In fact, all materials emit EM radiation at temperatures above absolute zero (—273°C); as the temperature is raised, the amount and wavelength distribution of the EM radiation is changed.

At very low temperatures, only a very small amount of EM energy is radiated, and this is at very long wavelengths in the far IR. As the temperature is raised, the amount of EM radiation is increased and the wavelength region in which the maximum amount of energy is emitted becomes shorter. A perfect radiator of EM energy in the optical region is called a "blackbody." Figure 3 shows the amounts of energy radiated at various wavelengths by a blackbody at several temperatures. It can be seen that the wavelength peak shifts to shorter wavelengths as the temperature is raised. Note that even at high temperatures, most of the EM energy is radiated in the IR, with only relatively small amounts in the visible.

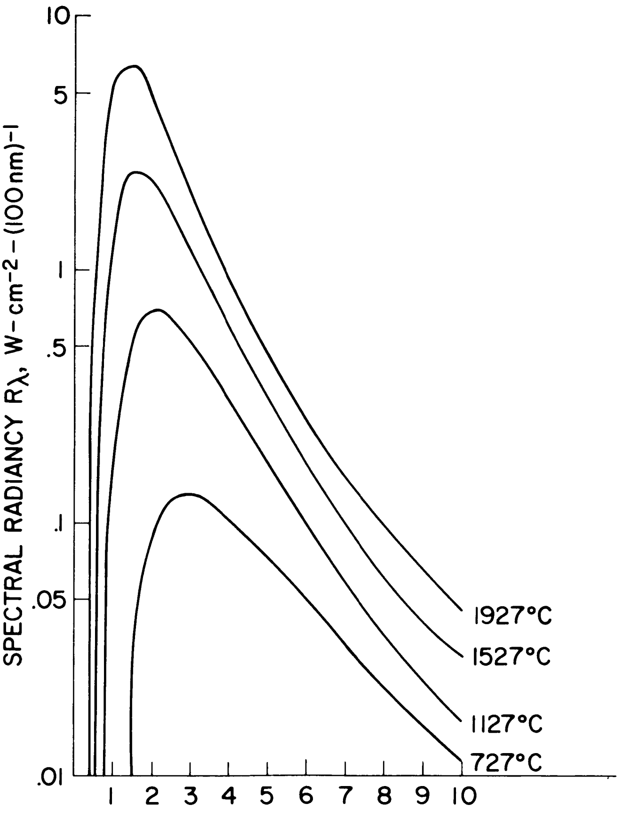


Figure 3

Blackbody radiation (note logarithmic scale).

HAZARDS

A horizontal section of the human eye is shown in Figure 4. The main refractive element of the eye is the cornea, and fine focussing (accommodation) is performed by the lens. These optical elements function together to bring visible light to a sharp focus on the retina, where the visual sensation is produced and transmitted to the brain. The space between the cornea and lens is filled with a liquid known as the aqueous humor, while the interior of the eye is filled with the jelly-like vitreous humor. These four components of the eye, cornea, aqueous humor, lens, and vitreous humor, are known collectively as the "ocular media."

In a normal, healthy eye, the ocular media are clear and colorless, meaning that essentially no visible light is scattered or absorbed. On the other hand, *in*visible light is absorbed at most wavelengths. All light of wavelength shorter than about 380 nm is absorbed by the ocular media.

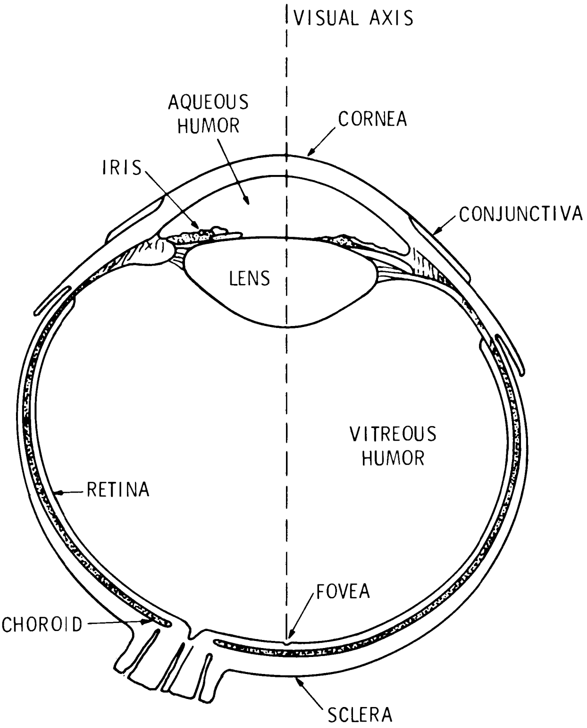


Figure 4
The human eye, in horizontal section.

The retina will respond to "near UV," but we can't see it because it is absorbed by the lens. UV of shorter wavelength is absorbed by the cornea. The situation is reversed in the IR: the retina will not respond to wavelengths longer than 700 nm-750 nm, but the ocular media are transparent in varying degree out to about 1400 nm, as can be seen by referring back to Figure 2.

It is the EM energy which is absorbed in the ocular media that is of concern in the context of this discussion. Absorbed energy is converted instantly to heat, and the lens of the eye is poorly protected against overheating. The lens may be overheated either directly from energy absorbed within it, or indirectly from energy absorbed elsewhere in the ocular media. Continual or repeated overheating of the lens can cause it to become cloudy or opaque, a condition known as cataract.

A reexamination of Figure 3 shows that UV is of no concern to us, as only negligible amounts are produced at glassworking temperatures. It is the IR that produces the potential hazard, at wavelengths greater than about 1000 nm. Glass is not a blackbody. Its total emissivity is approximately 0.8 at 500°C(1), meaning that it radiates at 80% efficiency compared to a blackbody. In order to simplify this discussion, we will make the following assumptions: (1) at all temperatures, glass behaves like a blackbody having 80% efficiency; and (2) all energy is radiated in the IR. The first assumption will not produce a large error. The second assumption will produce a significant error only at high temperatures, and this error will be in the direction of overestimation of the hazard (less than a factor of two, however). The total amount of EM power radiated by a hot glass surface can then be estimated by the Stefan-Boltzmann Law(2). Figure 5 shows the results of this calculation.

Matelsky(3) has stated that a significant number of employees in various glass- and steel-producing plants have exhibited anomalies of the eye lens after 10-15 years of exposure to radiant energies from white hot surfaces (1450-1650°C); these hot surfaces produced corneal exposure

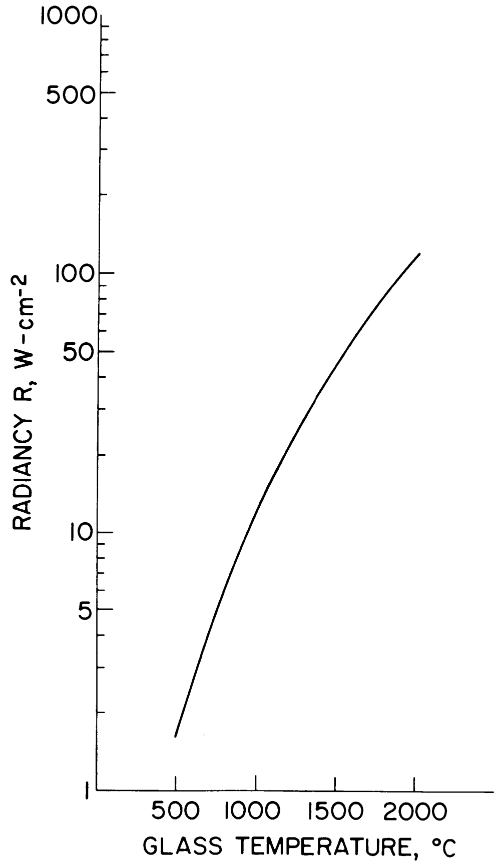


Figure 5

Electromagnetic radiation from hot glass, assuming a constant emissivity of 0.8. (note logarithmic scale).

intensities of 0.08-0.4 W-cm^{-2}. American National Standard ANSI Z136.1-1973(4) limits permissible corneal IR exposure intensity to 0.1 W-cm^{-2} for long exposures to IR wavelengths greater than 1400 nm, but in light of the above this may not provide a sufficient safety factor for all IR wavelengths. Sliney and Freasier(5) have suggested that 0.01 W-cm^{-2} be used as a safe limit for all IR wavelengths if exposures are prolonged and repeated. Following their lead, we will assume that 0.01 W-cm^{-2} at the cornea represents a safe long-term exposure limit for IR radiations produced at glassworking temperatures.

Corneal exposure intensities for glassblowers can be estimated from the following approximate relation:

$$I_c \cong R \frac{A}{2\pi r^2} \qquad (1)$$

where I_c is the corneal exposure intensity in W-cm^{-2}, R is the radiant power density in W-cm^{-2} at the hot glass surface (from Figure 5), A is the cross-sectional area of the heated zone in cm^2, and r is the distance between the glassblower's eyes and the workpiece in cm. Using this relation, we find that a 5 cm diameter Pyrex tube with a 10 cm. wide hot zone (1000°C) will produce a corneal exposure intensity of 0.06 W-cm^{-2} at the eyes of a glassblower 40 cm (16 inches) away. If the workpiece were fused silica instead of Pyrex, the hot zone would be near 2000°C, and the corneal exposure intensity would be 6.0 W-cm^{-2}.

PROTECTION

From the above it is obvious that protection of the eyes from IR is desirable in many glassblowing situations. Given the wide variation in workpiece sizes, working temperatures, and individual work habits, it might seem that a determination of the amount of protection necessary in a scientific glass shop would be impossibly difficult. Our task is made easier, however, by the fact that IR exposure intensities of less than 1 W-cm^{-2} on exposed skin will cause severe pain; therefore, if the glassblower's face is exposed, eye protection which transmits 1% of the IR will provide adequate protection, since he will not be able to tolerate skin exposures in excess of 1 W-cm^{-2} for more than a few seconds.

The IR transmission of several common types of spectacle lenses was measured in the wavelength range 800 nm-14,000 nm with Beckman DK-2A and Perkin-Elmer 700 spectrophotometers. Table I summarizes the results of these measurements. It can be seen that of the lenses measured, only Filterweld and Noviweld-didymium have IR transmission of 1% or less throughout the wavelength region of concern.

Didymium is of particular interest because of its wide use in glassblowing. The transmission spectrum of didymium has several peaks in the wavelength region 1000 nm-3000 nm, as is shown in Figure 6; these peaks account for the apparent high transmission shown in Table I. If one performs a detailed calculation, considering all transmission and absorption peaks, and the spectral distribution of blackbody radiation at ~1000°C, it is found that didymium offers reasonably good protection for much Pyrex work. Using our previous Pyrex example (5 cm diameter

TABLE I

MEASURED IR TRANSMISSION OF VARIOUS TYPES OF SPECTACLE LENSES

Lens	Maximum Transmission (%) in Each Wavelength Band								
	1000 - 1500 nm	1500 - 2000 nm	2000 - 2500 nm	2500 - 3000 nm	3000 - 3500 nm	3500 - 4000 nm	4000 - 4500 nm	4500 - 5000 nm	> 5000 nm
Didymium	90	80	70	50	15	8	7	2	< 1
Filterweld* (Shade 4)	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1
Plastolite*	40	100	100	10	< 1	< 1	< 1	< 1	< 1
Therminon**	80	70	55	40	5	3	3	2	< 1
Calobar* (Medium Shade)	10	18	22	23	15	7	7	1	< 1
Noviweld-didymium* (Shade 5)	< 1	< 1	1	1	1	1	1	< 1	< 1
Ordinary Spectacle Crown	100	95	80	75	20	12	12	10	< 1
G20***	< 1	3	20	20	7	5	4	3	< 1

* American Optical Co., Southbridge, Mass.
** Therminon Lens Corp., Des Moines, Iowa
*** Bausch and Lomb, Rochester, New York

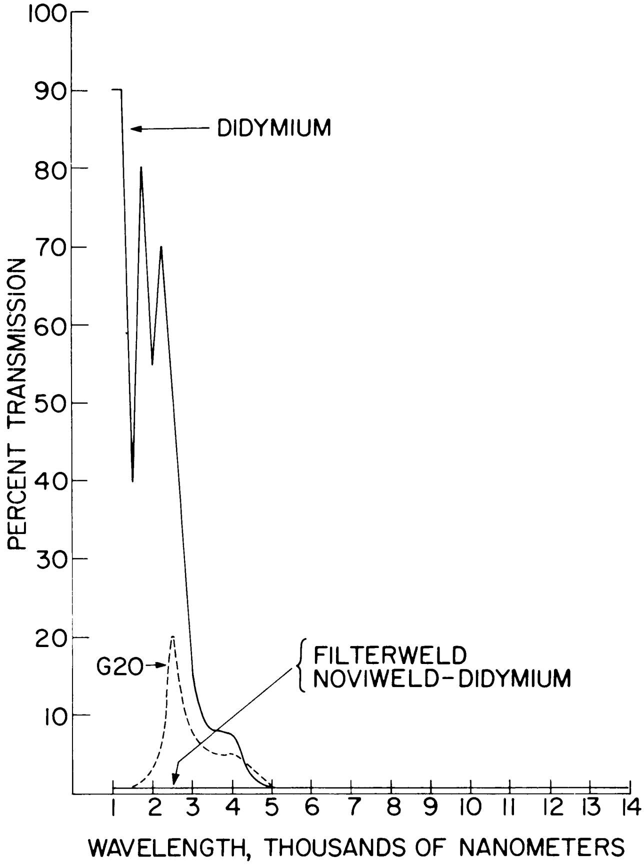


Figure 6
Infrared transmission of selected glasses.

tube with a 10 cm hot zone at 1000°C, glassblower's eyes 40 cm. away from work), we find that $I_c = 0.01$ W-cm^{-2} when standard 3 mm thick didymium lenses are worn.

G-20 glass, also shown in Figure 6, has somewhat better IR absorption than does didymium. A detailed calculation similar to that for didymium yields $I_c = 0.002$ W-cm^{-2} for a glassblower wearing 3 mm thick G-20 spectacles.

Sites(6) has reported that bifocal prescription didymium spectacles are available from Wale Apparatus Co., 400 Front Street, Hellertown, PA., 18505. A lengthy investigation by the author failed to uncover any sources for bifocal G-20, although there is in principle no reason why G-20 bifocals could not be made by the "bonded add" method discussed by Sites.

Work with high-temperature glasses such as Vycor and fused silica, and work with large pieces of Pyrex, requires protective filters with low overall IR transmission. Figure 3 shows that the IR emission peak for work with fused silica and Vycor will be in the wavelength range 1000-2000 nm, so it is particularly important that the protective filters used for work with these materials have low IR transmission in this region. Table I and Figure 6 show that Filterweld and Noviweld - didymium meet these requirements. It is not known whether these glasses can be obtained in prescription form.

Maximum IR transmissions for various shades of welding filters are shown in Table II. Welding filters made by some manufacturers (e.g., Filterweld) may exhibit significantly lower IR transmission. It can be seen that shade 6 or darker will provide reasonably good IR absorption. A problem arises when a face shield is used, however: the warning provided by skin pain is no longer functional, so it is possible for the glassblower to get close to the work and thereby increase his I_c to potentially

TABLE II

Maximum infrared (770 nm - 12,000 nm) transmittance of welding filters (from ANSI Z87.1 - 1968(7).)

Shade Number	Maximum IR Transmittance (%)
1.5	25
1.7	20
2.0	15
2.5	12
3.0	9
4.0	5
5.0	2.5
6.0	1.5
7.0	1.3
8.0	1.0
9.0	0.8
10.0	0.6
11.0	0.5
12.0	0.5
13.0	0.4
14.0	0.3

unsafe levels. A solution to this problem would be the wearing of IR-absorptive spectacles under the face shield; however, these spectacles should have high visible light transmission, because most of the visible light will have been absorbed by the welding filter.

Schott KG-3 glass seems almost perfect for this application. It transmits 85% of the visible light (it has a slight green tint), yet it is highly absorptive in the IR, as shown inFigure 7. With clip-on didymium filters, this glass would be excellent for work with large Pyrex pieces. Spectacles made of this glass are sold as laser protective eyewear by the Fred Reed Optical Co., Inc., P. O. Box 1336, Albuquerque, NM 87103. They are available in plano, single vision, and bifocal form, and all meet ANSI Z87.1 criteria for safety glasses.

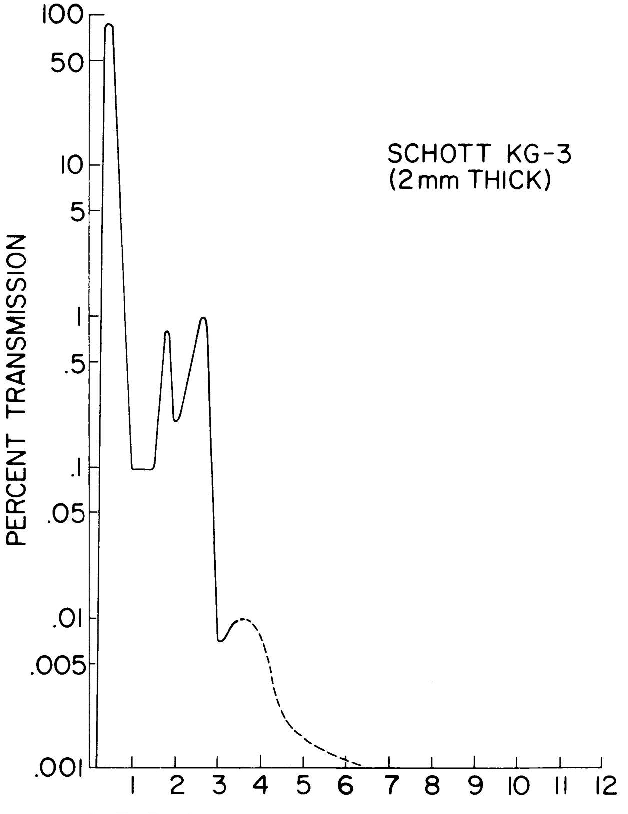


Figure 7

Infrared transmission of Schott KG-3 glass (manufacturer's data). Transmission data are for a 2 mm thick sample; standard 3 mm thick spectacle lenses would provide considerably lower IR transmission. Note logarithmic scale.

CONCLUSIONS

Glassblowing presents a potential hazard to the glassblower in the form of infrared radiation from the hot glass, which may cause damage to the lens of the eye after years of continual exposure. It is stressed that little quantitative data is available concerning the severity of exposure necessary to cause damage, and the "safe" limit of $I_c = 0.01$ W-cm^{-2} is based on estimates from other sources; furthermore, this limit is for continual exposure, and occasional brief overexposures are unlikely to cause harm.

A glassblower can reduce his IR exposure by wearing protective eyewear which has low IR transmission for wavelengths greater than 1000 nm. The widely-used didymium and G-20 spectacles should provide adequate protection for work with Pyrex, unless the workpiece is large. For work with large Pyrex pieces, additional IR filtering in the wavelength region 1000 nm-3000 nm is advised. For work with Vycor and fused silica, filters with IR transmission of 1% or less for all wavelengths greater than 1000 nm should be used: Filterweld, Noviweld-didymium, and all welding filters of shade 6 or greater should provide adequate protection. If the IR radiation is sufficiently intense to require the use of a face shield, the filtering ability of these glasses may not be adequate to protect the eyes from the IR. In such cases, IR-absorptive spectacles should be worn under the face shield. Spectacles made of Schott KG-3 glass appear to be nearly ideal for this purpose.

Although most glassblowers prefer not to use clip-on filters because of the added weight, KG-3 spectacles with clip-on filters are attractive from all other standpoints: the KG-3 would provide superb IR protection and the necessary vision correction, while the clip-on filter (didymium, welding shade, etc.) would provide the desired visible-light filtering.

ACKNOWLEDGEMENTS

I wish to thank T. L. Duffy for performing most of the IR transmission measurements, and W. Schulze for stimulating discussions and information about those phases of glassblowing with which I — as an amateur — was not familiar.

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NEW PRODUCTS IN FUSED QUARTZ

N. G. Grafton

General Electric Company
Cleveland, Ohio 44117

Type 214 is a new grade of clear fused quartz tubing—*and* in terms of *composition, starting material* and *physical properties* is similar to *Type 204* tubing—a product which may be familiar to you since it has been General Electric's standard tubing material for the past 15 years. *Type 214* is made from the same—domestically sourced—raw material. The primary difference between Type 214 and Type 204 is in its method of manufacture—rather than in end product characteristics. The process for manufacture of Type 214 grew out of a major long-term program aimed at finding a way to produce quality quartz tubing *more* uniformly and *more* economically on a volume basis— responsive to the growing needs of the major markets—lamp and semiconductor. *Type 214* is the result.

The process which is *new* in every respect, meets the objectives outlined for the program and *now* the position of 214 in most all applications has been firmly established. With the continuing development of larger and larger size capability, the material is also used in volume semiconductor applications. 214 can now be manufactured in sizes up to 70 mm diameter while 204 is available in sizes up to 150 mm.

Production facilities for 214 are located at both Willoughby, Ohio—where Type 204 is also made—and at Newark, Ohio. The Newark plant, a new addition, was specifically designed to accomodate the new process and optimize the advantages of the new development. We consider this facility a new direction in our quartz business—employing, as it does, the most up-to-date process control and manufacturing systems and signifying the departure from some traditional and longstanding methods of quartz processing.

So, we view 214 as a major step—one which we're hopeful will be tuned to *your* needs now and in the future. Now, let's look at some of the specific properties of Type 214.

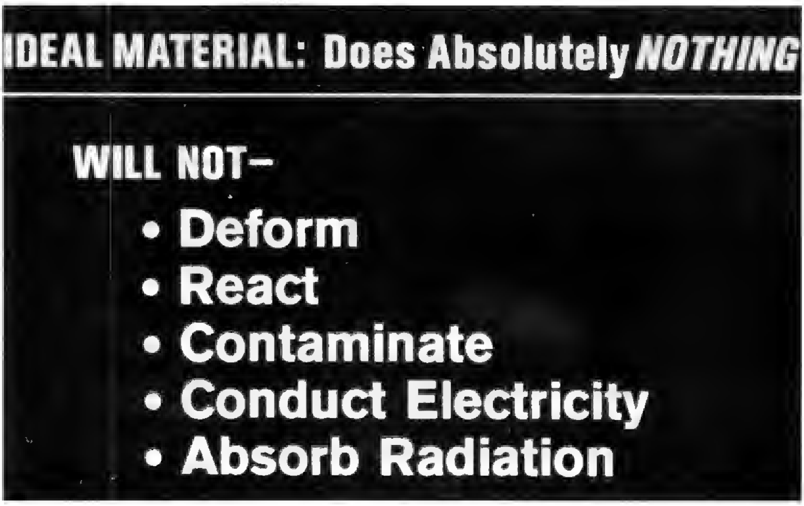
GENERAL PROPERTIES OF FUSED QUARTZ

Coefficient of Thermal Expansion	5.5 x 10^{-7} cm/cm·°C (20°C-320°C)
Fusion Temperature	1800°C
Softening Point	1670°C
Annealing Point	1140°C
Strain Point	1070°C
Density	2.2 gm/cm^3
Thermal Conductivity	3.3 x 10^{-3} gm cal·cm/cm^2·sec·°C
Specific Heat	.18 gm cal/gm
Electrical Resistivity	$10^{9.5}$ ohm·cm (350°C)
Dielectric Strength	410 volts/mil at 20°C, 1 MH_z
Dielectric Constant	3.75 at 20°C, 1 MH_z

In discussing the physical properties of 214, we'll try to concentrate on those *most* significant to *most* users. In general, these involve the degree to which the material can withstand the effects of very high temperatures—for example, sag or deformation, loss of mechanical strength, chemical reaction or contamination of other materials.

- **Sag or Deformation**
- **Loss of Mechanical Strength**
- **Chemical Reaction**
- **Contamination**

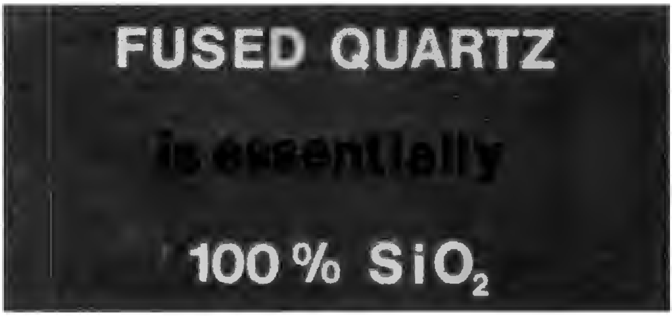
The major applications for quartz involve its use as a container whether it be as a diffusion furnace tube or epitaxial tube (in which case it contains silicon and gases), or as a lamp envelope (where it may contain a filament or mercury vapor). In a sense then, the ideal material is one which does absolutely nothing—it doesn't *deform, react, contaminate, conduct electricity* or *absorb radiation*—particularly infrared or ultraviolet, and it doesn't do these things under the most trying circumstances—temperatures up to 1400°C, in contact with some highly reactive substances, and subject to severe gradients of hot and cold. Fused quartz approaches the ideal—more so than other glasses and other materials—because its composition is almost 100% silica—SiO_2—a compound which is highly refractory—it fuses at about 1800°C and is quite viscous up to that point—is relatively inert, transmits light and doesn't transmit electricity. There are other materials, of course, which outdo silica in one or more respects—tungsten is much more refractory, for instance—but none combine all these properties to the same degree and at the same time can be



IDEAL MATERIAL: Does Absolutely *NOTHING*

WILL NOT–

- Deform
- React
- Contaminate
- Conduct Electricity
- Absorb Radiation



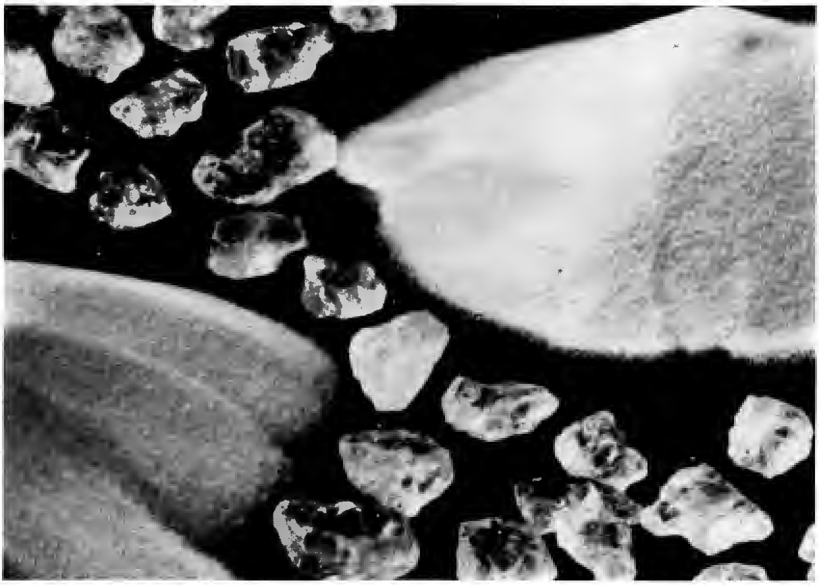
FUSED QUARTZ

is essentially

100 % SiO_2

economically fabricated to the shapes required by lamp and semiconductor processors.

An indication of how well or to what degree a particular quartz material will approach that ideal of doing *nothing,* is the degree to which the material really is 100% silica. For, as best we can tell, the addition of even very small quantities of other materials deteriorates those characteristics which are important, and quartz starts doing more of those things that it shouldn't. As a practical matter, no quartz product—at least none available today—is 100% silica. This is true whether it is Brazilian crystal or sand, either found in nature or made synthetically, or fused into a glass. And of course, even 100% silica will do *something* if certain temperatures are exceeded and it *certainly* will absorb radiation beyond certain wavelengths regardless of what is done. That's just characteristic of the material. Our *objective* in making a usable fused quartz glass is to come as near as we can to reaching those ultimate performance characteristics. We do this by upgrading and maintaining purity—within the bounds of economic reality—from the starting raw material through processing. Contamination—whether inherent in the raw material or introduced during manufacturing—invariably degrades one or more performance characteristics to some degree. Whether it be a *metal oxide, water,* or some other contamination, the temperature tolerance may decline, propensity for recrystallization—an important factor in retention of mechanical strength—increases, electrical resistivity deteriorates, and new absorption bands are created in the UV and infrared. Nothing good happens.



CONTAMINANTS

Metallic Trace Elements:
Water...or Any Property
Not 100% SiO_2

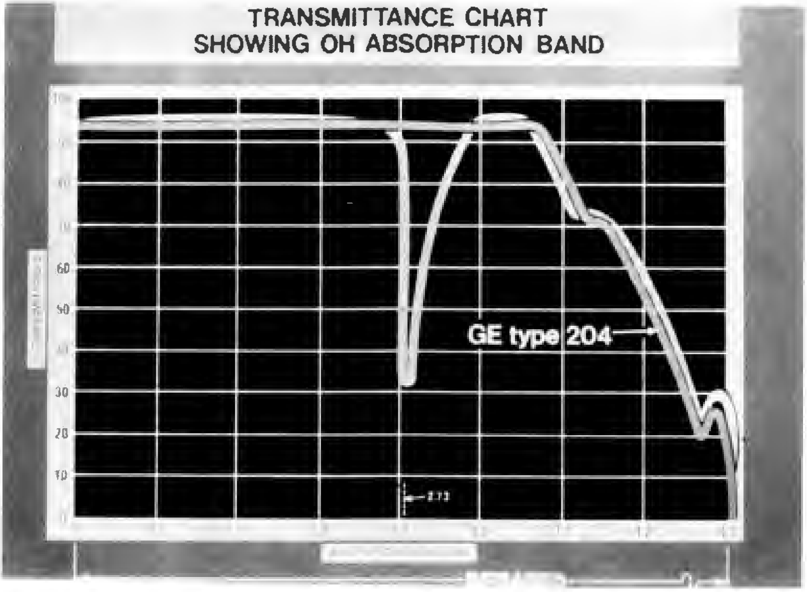
So, since it all starts with purity, let's look at the chemical analysis of Types 214 and 204. In every aspect, 214 is the same as 204.

TYPE 214 and 204

Al	26	Ti	2
Fe	3	Zr	2
Mg	1	B	<1
K	3	Cr	1
Na	3	Cu	1
Li	.5	Mn	2

Water—or "OH" radical content—is generally determined by the amount of absorption of radiation at the "water band"—or 2.73 microns in the infrared. The criteria for comparing water content is called Beta Factor and is the log of the ratio of transmission at 2.6 microns and 2.73

microns. Based on these measurements, water content of both 214 and 204 is very low and can be graphically shown when compared to fused quartz with a water content. The green line is Type 204 or 214 while the yellow line is fused quartz having a water content.



SAG

Because of the similarity in composition, 214 can be expected to perform in a manner comparable to Type 204. For example, resistance to sag or deformation. There are two types of sag—beam bending and out-of-round.

A sag test was performed on Types 214 and 204 with tubing 54 mm diameter at 1260°C. The tubes were placed on supports 24″ apart in the furnace and held at this temperature for eight hours. Both tubes had the same wall thickness. Both were then removed and measured for sag. The calculated log viscosity values were found to be 14.20 for Type 214 and 13.70 for 204. Since the sag rate is inversely proportional to the log of the viscosity, the higher the number, the more sag resistance—Type 214 was found to have sagged less than 204.

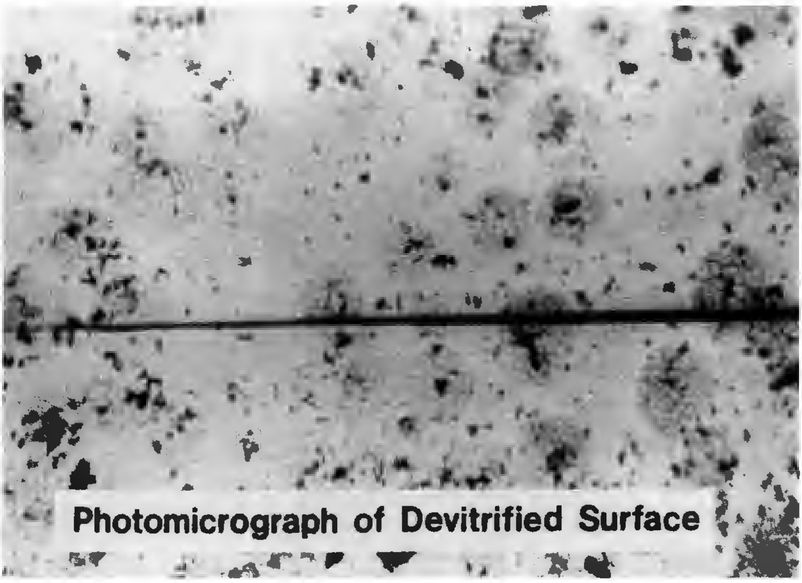
Almost any contaminant, including water, will lower melting temperatures. This slide depicts Type 204 or 214 tubes subjected to a time and temperature test versus material produced from Brazilian crystal which carries higher impurity levels.

TEST FOR SAG
(Log Viscosity Values)

Type 214 - 14.20

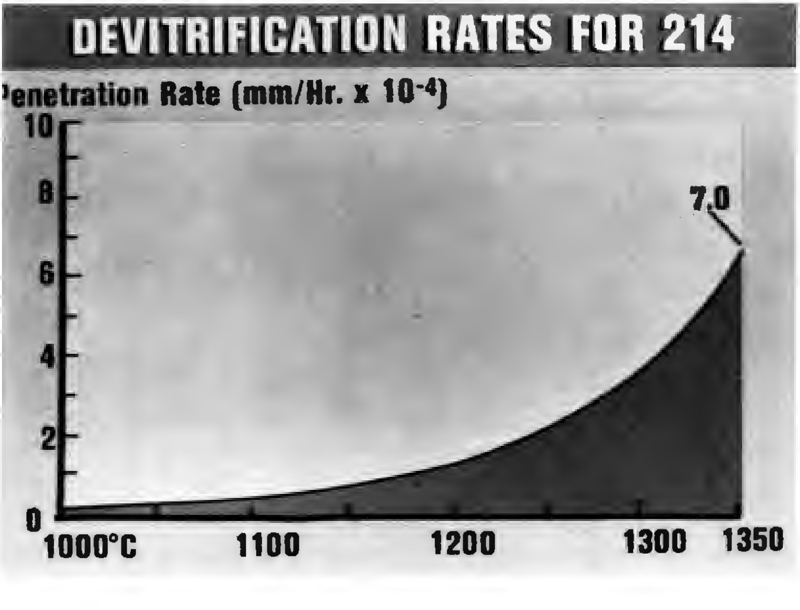
Type 204 - 13.70

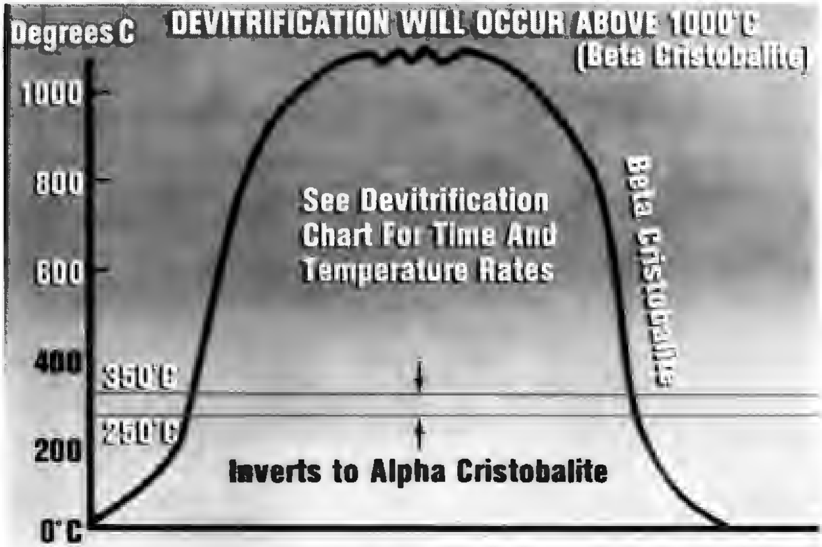
Another important consideration is the rate of devitrification or recrystallization of the material. When fused quartz is subjected to high temperatures, say 1000°C and over, but below its fusing point of 1800°C, it will tend to recrystallize. This phenomena starts at the surfaces and proceeds into the body of the material. This is a photomicrograph of a devitrified surface. The longer the time at elevated temperatures, the deeper the penetration and the larger the crystals formed. While at the high temperature—unless complete recrystallization has occurred—a rel-



Photomicrograph of Devitrified Surface

atively stable mechanical condition exists and there generally is no failure. The problem occurs when cooling takes place. The crystal structure goes through a series of inversions—or lattice size changes—as various lower temperatures are reached. The most significant of these occurs between 250-300°C when the material formed at high temperature transforms to the stable form at low temperature—crystoballite. This inversion





is very destructive to the mechanical integrity of a piece of quartz if any substantial amount of devitrification has occurred. For this reason, when possible, it's advisable to minimize cycling the material below 300°C.

Devitrification, if controlled, can have a beneficial effect. The crystal structure which forms on the outer surface of a diffusion tube, for example, has a higher viscosity than the fused material. Therefore, once formed, it imparts additional stiffness and resistance.

Mechanical properties of Type 214 are the same as Type 204 and are shown here. As already discussed, the most critical factor in retention

MECHANICAL PROPERTIES

BASIC MECHANICAL PROPERTIES OF SILICA GLASS

Density	2.2 g/cc clear
Hardness	4.9 (moh's)
Tensile Strength (modulus of rupture)	7,000 psi
Compressive Strength	160,000 psi
Bulk Modulus	Approx. 5.3×10^6 psi
Rigidity Modulus	4.5×10^6 psi
Young Modulus	10.4×10^6 psi
Poisson's Ratio	.16

of mechanical strength is the rate of devitrification and the effect of thermal cycling. Other considerations involve the freedom from flaws or defects in the structure.

Electrical properties for 214 show the same high resistivity as 204. In most general applications, the high resistance and low loss characteristics of fused quartz far exceed the electrical performance requirements.

ELECTRICAL PROPERTIES

Electrical Resistance: 9.5 $\log_{10}$ F for cm^3 at 350°C
Dielectric Strength Volts/mil:
Dielectric Loss Factor: less than .0004 at 20°C 1 Mc
Dielectric Constant: 3.75 at 20°C 1 Mc
Specific Resistivity OHMS/cm^3 min: 10^{18}
Dissipation Factor: less than .0001 at 20°C 1 Mc

Aside from the inherent physical properties of the basic material, the size reproducibility is important. The dimensional uniformity of Type 214 tubing is excellent. Of particular note is the reduction in variability of the wall tolerance—by about 50% compared with Type 204. Diameter tolerances are also much better.

Type 214 tubing has been proven in all volume lamp applications for quartz arc chambers, in mercury, metal halide lamps, and as filament tubes for quartzline types—both general lighting and photo. The superior dimension characteristics—particularly wall uniformity—have resulted in substantial savings in lamp manufacture—primarily as a result of reduced shrinkage in seal making. The transition to 214 was essentially "problem-free", demonstrating the retention of desired physical characteristics in Type 214.

Semiconductor applications rely heavily on the refractory, purity and inertness characteristics of fused quartz, and as has been discussed, 214 excels in these properties. While use of 214 for semiconductor processing has been more limited than for lamps—because of past size limitations—testing of the material in a variety of uses has—as anticipated—shown that 214 can be directly substituted for 204 in most all cases. So far the material has been successfully used for:

— Diffusion tubes
— Plug and capsule diffusions

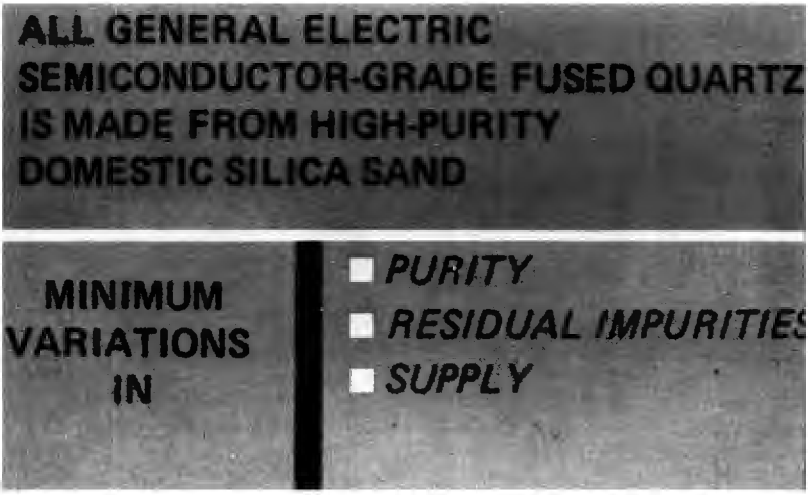
— GaAs processing
— Fabrications—boats, joints, hardware

Volume semiconductor applications require tubing in sizes from 30 mm to 150 mm diameter. Type 214 is currently available in sizes up to 70 mm and in standard lengths up to 8 feet.

Type 214 rod is also available in sizes up to and including 19 mm diameter. This rod has outstanding dimensional and visual quality—it being quite free of air lines and inclusions and, of course, having the same desirable properties as tubing.

We expect that Type 214 will replace Type 204 for all volume uses in sizes up to 70 mm—and we are working to extend our upper limit of diameter capability. This will result in a savings to you as 214 prices are considerably below 204 for the same size.

Semiconductor requirements now come in a matched set of consistant high-purity materials all made from domestic sand — solid material
— crucibles
— rod
— and tubing.



ALL GENERAL ELECTRIC SEMICONDUCTOR-GRADE FUSED QUARTZ IS MADE FROM HIGH-PURITY DOMESTIC SILICA SAND

MINIMUM VARIATIONS IN

☐ *PURITY*
☐ *RESIDUAL IMPURITIES*
☐ *SUPPLY*

By this time it is no secret that we see 214 as a major advance in quartz technology—one that responds to some critical and growing needs in major markets. We think the process which has been developed is one which will answer the call for more economical production of quartz materials.

NEW FLUORESCENT LIGHT BULB (LOW VOLTAGE IONIC MERCURY VAPOR)

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Ecklux R&D Vacuum Laboratory, Inc. (E.E.E.)
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ABSTRACT

A new fluorescent low voltage light bulb is discussed. The new bulb operates on standard low voltage line service, or 120 to 240 v.a.c. It is an ionic gas and mercury vapor discharge device. The outer envelope of the bulb is coated on its inside surface with a suitable phosphor. Within the envelope are contained a grouping of paired, matched, and polarized fused quartz discharge tubes; (UV). These UV tubes are the source of the necessary strong wavelength in the ultra violet required to produce the desired fluorescence of the phosphor. (e.g. 2536.52 ÅU.). When the phosphor used is modified magnesium-tungstate a bright snow white fluorescence is demonstrated. (6500° K.). At least six other colors are available using various combinations of the different phosphors, glass color, and fill gases. Each of the UV tubes are unidirectional with respect to the flow of its discharge current. Therefore, as paired and polarized they utilize the full wave of the input alternating current. A mechanism to reduce the starting voltage drop to a level well below that of the line voltage is also covered in this paper. The two electrodes used at the two ends of each UV source tube, respectively, are designed to function so that the one electrode at one end of the tube is an anode, and the other electrode at the other end of the same tube, respectively, functions as a low drop hollow cold cathode. The anode permits no reverse current flow. This anode to cathode design as connected in series-parallel-parallel will accomodate a wide range of operating pressures. Indeed a gas pressure as low as two Torr (hg) and one as high as twenty-five Torr can be used quite effectively. The small amount of fluid mercury that is added to attain the ultra violet discharge will of course reach a vapor pressure from the preliminary quick heatup of the gaseous discharge. This vapor pressure will be dependent upon the actual operating temperatures of the device. The mercury vapor discharge dominates the spectrum of the bulb's operation. The fill gas and the mercury vapor total pressure is common to the contents of the surrounding envelope. Hence, with this reserve volume of both gas and vapor the discharge is stable, and a long life expectancy can be predicted. Too, in view of the half-wave operation of each of the individual UV discharge source tubes, the life-expectancy (from the cold cathode point-of-view) can be assumed to be a double life span. It has been established that the life of the cold cathode fluorescent tube approximates 18,000 to 20,000 hours of useful lighting. From this it appears that the new fluorescent light bulb will have a life in excess of 35,000 hours. A screw-type medium sized base is attached. This base and some portion of the re-entrance glass stem are used to contain suitable resistance to limit the operating current of the bulb. This resistance is a non-inductive type of wire wound resistor. Thus, little or no power-

factor is involved. The glass envelope and the glass reentrance stem are, presently, made of borosilica glass. These items may later be modified to the use of soft glass. However, the UV source tubes will always be of quartz. Fabrication and the processing for proper purification, conversion, and evacuation will be mentioned, with references. Data on the luminosity will be presented. The latter will be based upon observations made of the bulb's output with the use of the well-known light integrating sphere. The bulb is an attractive device since it promises to give several times the amount of light as compared to an incandescent bulb of similar wattage.

NEW FLUORESCENT LIGHT BULB (LOW VOLTAGE IONIC DISCHARGE DEVICE)

Author's Notations:

> Back in the late '20s the late Raymond Machlett, then the President of Rainbow Light, Inc., invented the low-voltage neon lamp or bulb. It was made in productive quantity (more than 100,000) and used, mostly, in sign display installations. At first, the 110 volt was produced. It was costly (cesium metal was required—and was in very short supply), and the bulb was modified to take 220 volts A.C. I was Mr. Matchlett's assistant, during the experimental stage, and later was placed in charge of the processing. The Machlett lamp was a twin tube positive column discharge device, and had a starting strip.(1) The depression, or rather the stock market collapse, was cause for the abandonment of this item. Very few mercury type bulbs were made at that time. They were of the 120 v.a.c. vintage. Blue and green. Recently, I saw a few of these lamps in an older installation at Times Square, New York.
>
> The "crash" resulted in my reassociation with Miles Pennybacker, who reformed the VOLTARC TUBES, Inc. There, in Newark, N.J., I was able to successfully perfect the Unidirectional Gas Discharge System.(2) Also, for Mr. Pennybacker, I did the experimental and first production work on his cone shape hollow cold cathode design.(3) It is still widely used. In 1969, I obtained my "Gas Laser Tube System" (U.S. Patent).(4) I had presented on this subject, in Boston at the A.S.G.S. Society Symposium in June 1966. It mentioned an improvement over Machlett's low voltage lamp, and this is used in the present design. The present novelty is actually one that was possible many years ago. However, because we lacked fluorescent chemicals, and because of the economy status of the nation, it has never been manufactured since the Machlett low voltage bulb. The latter, as I have stated, was red neon. But it was a positive column discharge unit.

It appears that the pertinent considerations of this new low voltage fluorescent bulb has been fairly well covered in the abstract. A few remarks, however, will complete the matter.

FIG. 1. illustrates the general scheme of the fluorescent light bulb, of this present design. The drawing was made several weeks prior to actual experimental work. (Note: Due to cataract-eye.) Looking back, it was rather ambitious to assume that such a design could be made in one attempt. As a matter of fact, about twenty or more bulbs of various specific detailed design have been made. They all follow, in principle, the original design as shown in the drawing. There is one important change. The shape of the surrounding envelope is best when made similar to the source tubes. This, in geometric language calls for a tubular envelope—or, cylindrical.

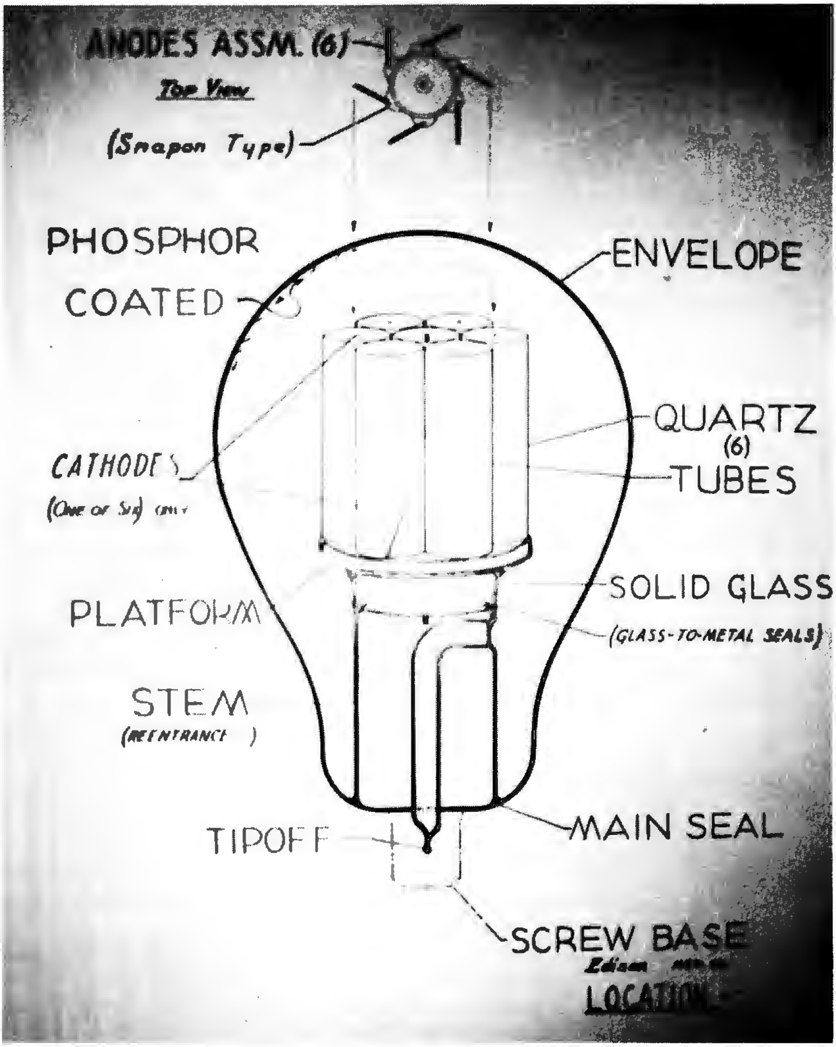
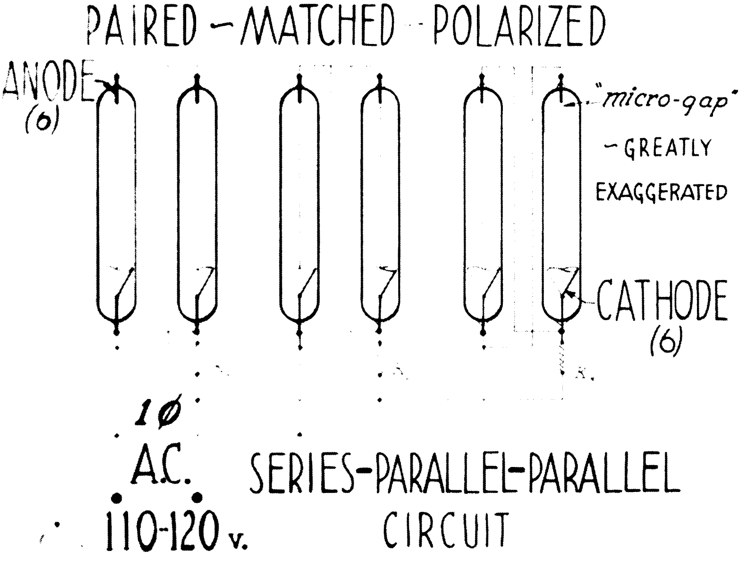


FIG. 2 presents the electrical schematics for this type of device. It does appear somewhat complicated, but (believe me) it is quite basic and simple.



Although all the test bulbs, to this date, have used the "CONEX"(5) hollow cold cathode, and the anode as referenced, (REF. 2), it has become evident that in order to attain the low voltage drop desired, these items require some modification. The cathode will be changed to a conical shaped wire-wound element, and the anode is to be improved by proper beading and insulation. The latter is a must in order to prevent reverse current flow. The materials are of great importance. Each that has been used has been given a fair but accelerated life test. Presently, the emissive coating on the cathode requires modification to make it suitable for mercury discharge conditions.

The method of pumping and filling the bulb is at present one which depends entirely upon internal bombardments, and ultra-high vacuum pumping. Also, some moderate external baking is used. However, on most devices of this type it is the usual practice to have a high frequency bombarding unit. The latter will be acquired. Many materials have been tested out. There remains much work to be done, here.

Finally, the glass costs are presently prohibitive. Soft glass can and should be used. It is standard lamp and bulb practice. The production in large quantities is a matter for machines. At least semi-automatic. Stem making machine. Sealing machine. And, as to pumping, which is termed "exhaust" in the lamp and bulb trade, it is at least advisable to install a "trolley system" doing approximate batch lots of sixteen or twenty at one time. Only the future can answer these many problems.

There is, however, no question at the present time that this type of fluorescent bulb can be made successfully. It would save an appreciable amount of energy, over time, as compared to the incandescent light bulb.

This brief paper was submitted in a most tardy manner. The writer is very grateful that it has been worked into the Program. It is planned to apply for new patent coverage on this invention.

In closing: After about fifty years of glass work and related activities it is but natural to get the feeling of "History Repeating Itself." This is exactly the feeling that I have had many times during the past three months of experiment and design effort. It is an interesting project, however, and one that will receive the best effort that I can continue to give to it.

Perhaps, in time, you will hear more of this fluorescent light bulb. That remains to be seen. Were I to have my say in the matter I should like to name it one of two Trade Mark (REGIS). Names:

The PENN-MACH VIII
or;
ECKSOL - II

Thank you very much for your attention. I appreciate it very much.

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CUSTOM BUILDING OF A TELEVISION BULB

SIEGFRIED GREINER

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Display Device R&D Laboratory
Niles, Illinois 60648

You may ask, "Why build your own TV bulb when they can be obtained from glass suppliers such as Corning and Kimble?" The answer is that they do not build TV bulbs of the size, shape and contour that we need. That is why we take existing TV glass, recut and reshape it to our particular requirements.

1. The project is begun with a 25″ panel which is scored with a glass cutter to approximately the correct dimensions. The panel is turned over and tapped on the scribe mark with a small hammer until a slight crack appears. Once this happens, the tapping is continued, ahead of the crack, until the panel is cracked all the way across. This is done on all four sides. At this time, the panel is spherically shaped so it must be flattened out. This is done by placing it in an annealing oven and running the temperature up to about 700°C. The sagging process is monitored periodically, and once the glass is flat, the oven is turned off and the panel is cooled overnight. This flat panel, or faceplate, is now ground and polished in the Polishing Department.

2. The faceplate now has a finely polished surface and from this point on, it must be handled very carefully to prevent any scratches from being introduced. (Incidentally, this is very important with all the glassware used in the manufacture of vacuum envelopes.) Now, one side of the glass faceplate is completely taped over with masking tape. With the use of a diamond saw, the plate is cut to the exact dimensions required. (It used to be standard procedure to tape both sides of the glass, but this caused problems with tape getting caught up in he cut, making the operation more difficult.) With the faceplate now cut to size, the masking tape is peeled off, and with the use of a sander belt, all the edges are beveled.

3. To get the proper distance from the faceplate to the neck, the funnel must be stretched; then a glass ring is fritted between tthe funnel and the faceplate. To do this, the glass panel is mounted vertically on the cut off saw using a big "C" clamp and a 90° angle plate. The panel is cut into on all four sides leaving approximately 2″ on each corner uncut. In order to stabilize the ring and the panel, the slots are filled with hot pitch. The piece is then returned to the vertical position on the saw, and very carefully, cut through the corners. The glass is then soaked in trichlorethylene to dissolve the pitch. When the ring is freed, it is ground on a flat grinding wheel using carborundum, first rough and then fine grit. Then, as with the faceplate, the edges are beveled on the sander belt.

4. As mentioned earlier, the funnel has to be stretched. The funnel is set on a matching steel plate. Then, a wet 2″ wide asbestos paper tape is wrapped around the plate and glass funnel several times, the asbestos

being pressed firmly against the glass. When a good seal is made, the unit is placed into an annealing oven, and the temperature is run up to 200°C for about an hour, until the asbestos is dry and hard. The oven temperature is then increased to 350°C to preheat the funnel. While the funnel is getting up to temperature, the glass lathe is prepared.

A vacuum chuck is on the left side; a carbon block on a dragbar arrangement, the correct distance from the vacuum chuck; and about 12″ of 1⅛″ glass is held in the right hand chuck.

When the lathe is prepared and the oven has reached 350°, the hot funnel is removed from the oven and held to the vacuum chuck. The pump is turned on, holding the funnel in place. At this point, it does not run true. A bushy gas flame is used to further heat the funnel. When it is well above the strain point, the funnel can be lined up to run true by stopping the lathe and pump and moving it to the center. This may take several attempts and is usually done by two men.

At this point, the neck portion is fire cut at the spliceline. This is done because it is 1-7/16″ diameter, and a piece of 1-1/8″ glass must be spliced in its place. While the splice is still soft and pliable, the carbon mold is brought down until it rides on the neck. Slowly, more of the funnel is heated up, with the operator puffing lightly through the blowhose, making the yoke area follow the contour of the carbon. All during this forming operation, a gas flame is directed on the rest of the funnel to keep it from cooling too much. (When working with this type of glass, you must keep in mind that it is a high expansion glass. If a crack should develop it is often impossible to save the device.)

When the funnel has the right length and contour, it is removed from the lathe and placed in a 400°C oven. The funnel is annealed by bringing the oven temperature to 500°C, holding it for a short time and then cooling it at about 2° per minute down past the strain point. The oven is then shut off and the funnel can be removed when room temperature is reached.

5. After the funnel is annealed and cooled to room temperature, a mixture of solder glass or frit is prepared. This is done by first mixing frit (which comes in powder form) with a vehicle such as amyl acetate to a 13:1 ratio, or about the consistency of ketchup.

A bead of frit is poured slowly on the edge of the glass ring. Then it is set aside to dry. An hour or so later, when it is dry, the ragged edges are cleaned up with a razor blade.

The glass ring is then set into a fritting oven, the funnel is set on top of the ring, and a weight is placed on top of the funnel.

The oven is then programmed to run through a fritting cycle, during which time the ring and the funnel will be hermetically sealed.

6. The next procedure is to frit the faceplate to the funnel-ring assembly. This is done by the same method as the ring to funnel seal. (You may ask what keeps the first frit from melting again. The answer is it takes a higher temperature to remelt a completed frit seal. This is

due to the thermo-setting solder glass which was especially developed for color TV picture tubes. So, if the same temperature is used during the second seal, the first will not remelt.) This bulb can be heated during evacuation to a temperature above 400°C without shifting of the faceplate.

7. At this point there is a complete bulb. It is now held in a four prong chuck in a lathe and aligned to run true. Then, part of the neck is cut off by first scribing the glass with a tungsten carbide tool and then holding a small gas oxygen flame to the scribe area while it is turning in the lathe. A new neck with a contact button is then sealed to the bulb.

The entire bulb is now ready to be gun sealed. The gun is inserted into the neck, properly aligned, then held in a three prong chuck. The press of the gun and neck are preheated using a radiant burner. (I realize these burners are not widely accepted in the glassblowing industry, but for this operation they are ideal. We used to preheat with just a gas flame but found a large number of our presses cracked during preheating. This is because a gas flame has a temperature of about 1000°C. To compensate for this high heat, we would hold the flame about 3″ below the glass; but every little breeze would blow the flame away from the glass, making it difficult to heat uniformly. With the radiant burner, we do not have this problem since there is no flame involved.)

The gun seal itself is made with a gas oxygen two-burner. The neck glass is heated to its softening point, then paddled down until it touches the press. This area is heated until a very even seal develops. During this time, nitrogen is flushed through the tubulation to prevent the gun parts from oxidizing. This seal procedure is done in a rather speedy fashion so the pins do not deform.

After the seal is completed, the area is annealed using a radiant burner. The radiant burner is used because it leaves much less residual stress in the glass.

The TV bulb is now completed. All that remains to be done is to evacuate and process it. This is also done in our Glass Shop, but I will not go into it at this time.

In conclusion, I would like to thank our Laboratory Director, Mr. James W. Schwartz, for encouraging me to give this paper, and also Messrs. Dennis Hoffman and John Guerrieri for their assistance in completing this project.

CONSTRUCTION OF A 12″ DIAMETER PYREX LIQUID SCINTILLATING CELL WITH AN OPTICALLY CLEAR WINDOW 3/16″ THICK

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The need for a larger scintillating cell for the study of neutron detection prompted the Applied Physics Division at Argonne to contact the Chemistry Division's glassblowers to inquire into the possibility of having such a cell fabricated of borosilicate glass and 12″ in diameter. This was quite a large cell so it did provide a challenge as to how we would proceed to fabricate such a cell.

The following description and slides will show the procedure used to complete the cell.

Using a 12″ O.D. x ½″ wall length of tubing and chucked into our large Litton lathe it was then cut down to form a flat bottom using a set of 12 seven jet cross-fires and a specially constructed hand torch consisting of 3 seven jet cross-fires and a 2″ x 24″ carbon paddle. After forming the flat bottom a hole was blown out near the edge of the flat bottom and a 3″ length of ½″ tubing sealed to the flat bottom and after hand annealing placed in the annealing furnace overnight.

Using a hot wire to cut off the flat bottom end at a length just over 1¼″ it was then sent to our optics department where under the supervision of Edward Nowicki it was ground down to 1¼″ and the edge highly polished to facilitate sealing the window to the cell.

Optics then cut the 12″ diameter window and beveled one edge of it at an angle of about 45° so when the window is placed upon the cell body it has an overlap of from .020″ to .030″ of an inch to ensure no problem in making a good seal.

The cell was placed on a turntable and the window placed upon it and mounted above it was a carbon disc 14″ in diameter x ¼″ thick, this disc was used to transmit heat to the surface of the window. On top of this carbon disc was placed a 12″ diameter hot plate and on top of the hot plate a tray 12″ in diameter and 2″ deep filled with mica and covered with aluminum foil to help contain the heat from the hot plate.

The variac was then turned on along with the twintable and heat slowly applied to the carbon disc above the window, the variac setting being raised periodically so as to add more heat to the carbon disc until a desired temperature is reached at which time two hand torches with #5 tips are then applied to the edge of the window and gradually increased in intensity from an all gas flame to a gas and oxygen flame. When the window is ready to be sealed to the cell one annealing torch was removed and two hydrogen and oxygen torches applied with #4 tips and at this

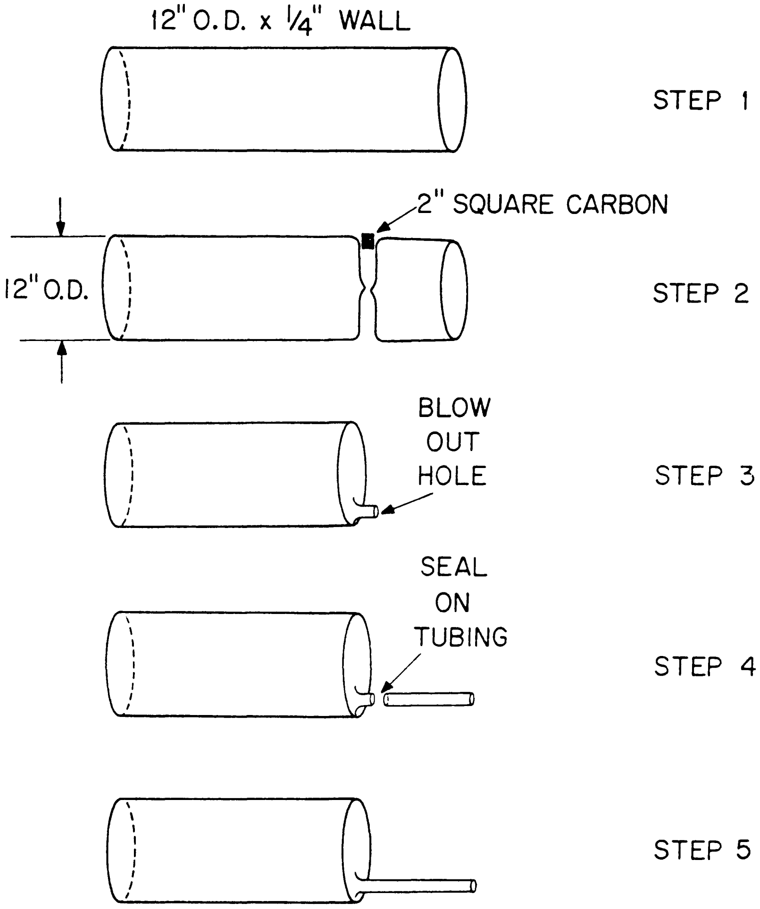

Figure 1

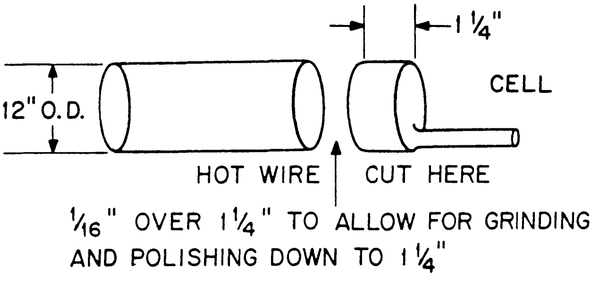

Figure 2

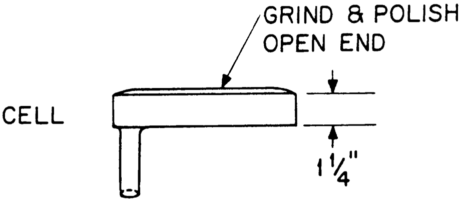

Figure 3

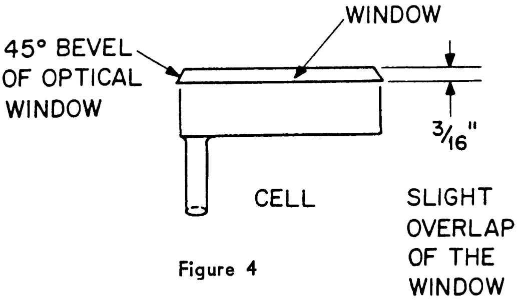

Figure 4

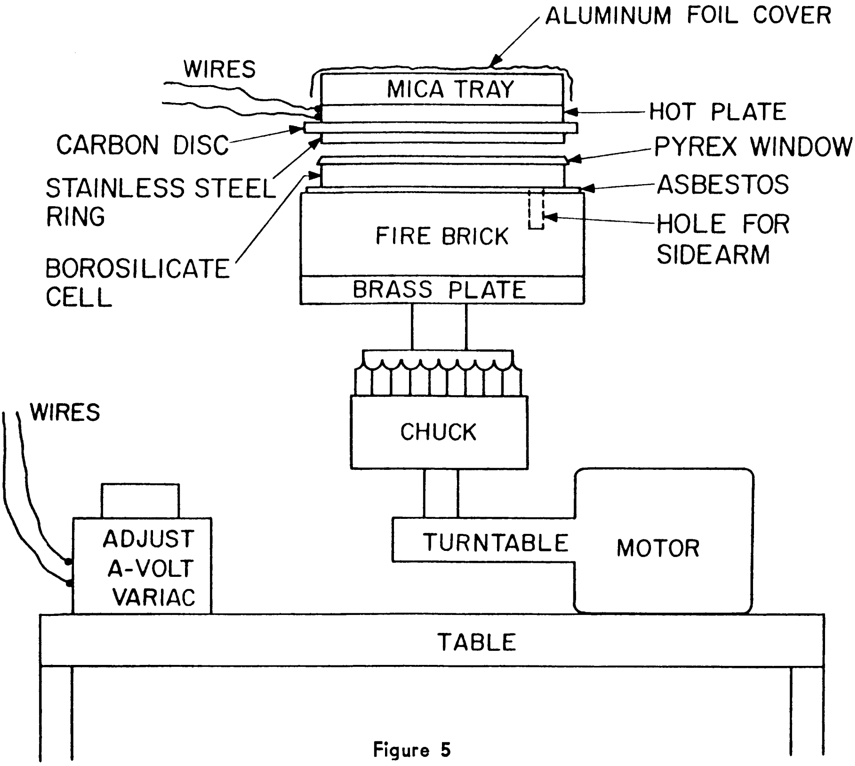

Figure 5

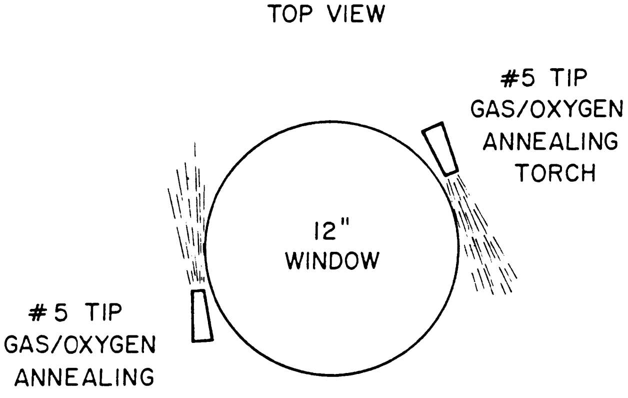

Figure 6

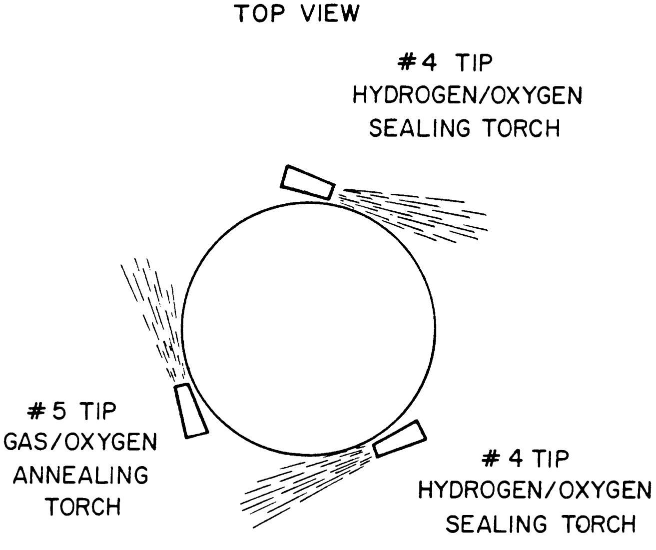

Figure 7

time the variac temperature setting is reduced so as not to allow the body of the cell to get so hot as to cause a bulging at the seal of the window and cell by the weight of the window.

Care must be exercised at the sealing of the window to ensure a good seal by concentrating the sealing torches at the very edge of the window and burning it in properly as the window is 3/16″ thick and the wall of the cell is 1/4″ and this type of seal requires a great amount of heat.

The cell with the window should always be kept moving during the sealing operation as not to concentrate too much heat from the torches in one area to avoid setting up excessive strains at these points.

After completing the sealing of the window to the cell the hydrogen and oxygen torches are removed and the two annealing torches applied again, this time to reduce somewhat the strains at the area of the seal and at the seal itself. Also at this point the temperature of the variac is reduced.

After sufficient annealing was completed the torches were then removed, the variac turned off, and the twintable stopped and the cell taken from the twintable and placed in a specially constructed container to transport it to the annealing furnace which had been preheated to 1040° and placed in the furnace for overnight annealing of the cell.

After removing the cell from the annealing furnace a 200 cc round bottom flask was sealed to the existing ½″ O.D. tubing that was previously sealed to the cell body. This seal was hand annealed so as not to have to place the completed cell back in the furnace again for further annealing.

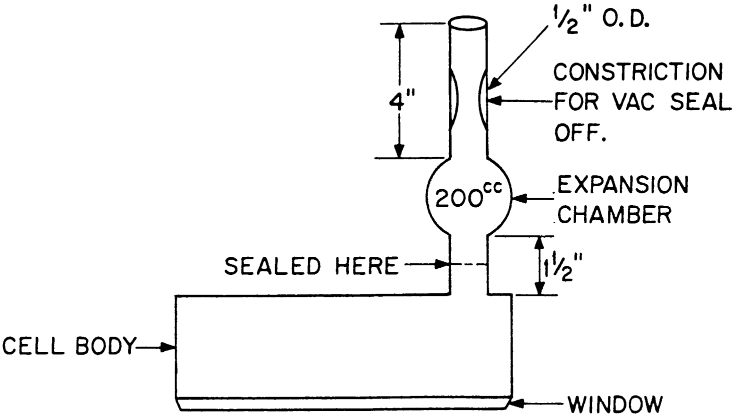

Figure 8

The cell now completed as far as the glassblowing was concerned, was then sent to the Applied Physics Division of Argonne where it was coated completely with a diffuse white reflector paint to enhance light efficiency except for the optically clear window which remained clear.

It was then filled with a Pilot B Solution and used very successfully in experiments conducted by Dr. Allen B. Smith and Alfred Engfer of the Applied Physics Division and in further experiments at the Fast Neutron Generator at Argonne.

After being used successfully for many months the cell was returned to the Chemistry Division and is now on display there.

CONSTRUCTION OF A THREE-COMPARTMENT CELL TO STUDY THE ELECTROLYTIC HYDROGEN-DEUTERIUM SEPARATION FACTOR

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INTRODUCTION

Construction techniques are described for a three-compartment electrolysis cell used in the General Chemistry Branch of the Chalk River Nuclear Laboratories to study the electrolytic hydrogen-deuterium separation factor. This cell (Fig. 1) consisting of anode, cathode and reference electrode compartments is the latest model in a series; data obtained with an earlier version have been described by Hammerli et al.(1)

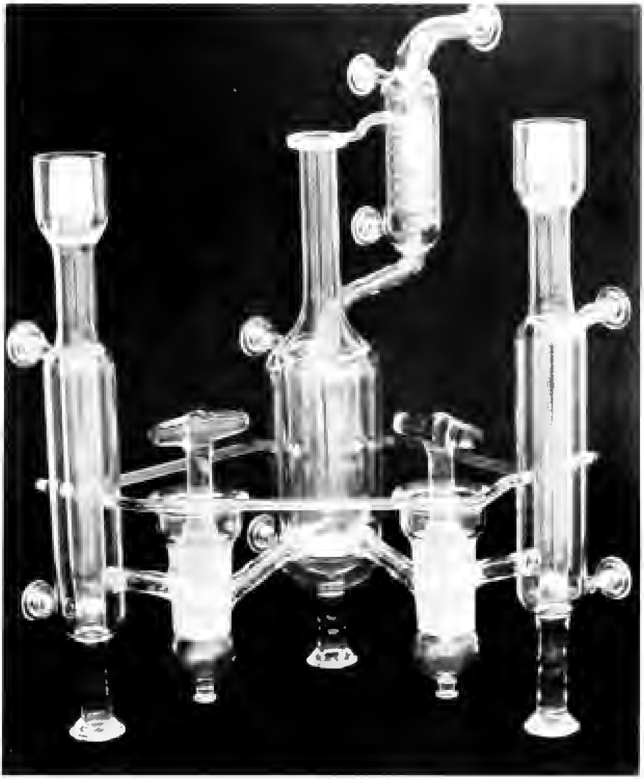

Figure 1

The completed cell. From left to right, the reference electrode compartment, the cathode compartment with condenser, and the anode compartment.

FUNNEL SELECTION

The inner part of the cathode compartment (Fig. 2) was made from a 30 ml., M porosity, no. 36060 Buchner type funnel with fritted disc. Uniformity across the frit was important. Over a period of time an electrolyte that even slightly attacks silica will eventually dissolve its way through a thin spot and ruin the cell. Even thickness and consistent porosity will give a uniform flow of gas across the frit when purging the electrolyte. Accordingly the funnels were checked by inserting a volume of water and flowing nitrogen through the fritted disc. Thin spots or lack of uniformity in the fritted discs usually showed up at 3 to 4 p.s.i. The two best of the twenty-four funnels tested were selected but the quality of the fritted discs was generally very good, and most of them would have done the job.

LUGGIN CAPILLARY

The reference electrode is connected to the cathode compartment by a Luggin capillary (Fig. 3).

A button was formed on a 9 mm piece of tubing and one end drawn down to 0.5 mm I.D. Experience has shown that the wall of this capillary should not be too thick as it will act as shielding. The drawn down capillary was sealed 2 cm from the button.

Some glass tubing was crushed in a mortar and pestle and separated with Tyler sieves. The 40-60 mesh fraction was washed first in distilled water and detergent, then in a cleaning solution of sodium bichromate dissolved in sulphuric acid, rinsed with distilled water and finally dried in an oven. The 40-60 mesh size was selected because it contained fragments small enough to enter the 0.5 mm capillary and yet large enough to form a sintered section without overly restricting the flow. Some of the ground glass was poured into the 0.5 mm capillary. The glass was held in an upright position and rotated in a relatively soft cool flame until it became apparent that the ground glass had formed a fritted section approximately 7 to 8 mm long. Excess glass was removed. The sealed end was then carefully ground off on a wet carborundum wheel so that the end was square and 1.9 cm long from the button.

CATHODE COMPARTMENT

The Buchner funnel was flame cut in the lathe and a piece of 3.8 cm tubing added. A button was formed 11.5 cm from the fritted glass disc. A piece of 5 cm quartz tubing was held in the tailstock of the lathe and slid over the funnel. A flame was applied to the revolving quartz envelope to warm the fritted glass disc gradually. When it appeared that the fritted disc was warm enough the quartz envelope was removed and the flame was applied directly to the funnel. A hole was blown directly above the fritted disc. The diameter of the hole corresponded with the button on the Luggin capillary. A 1 cm O.D. - 4 mm I.D. piece of tubing was joined on the same plane 45° away. The assembly (Fig. 4) was then annealed.

Figure 2
Inner part of the cathode compartment showing the fritted disc.

Figure 3
The Luggin capillary.

Figure 4
The Luggin capillary sealed into the cathode compartment.

The excess glass on the Luggin capillary and tubing were ground off on a wet carborundum wheel until they just slid into a 5.1 cm tube. The ground surfaces were polished on a Sundstrand Machine tool using a cork belt. The glass was then appropriately cleaned and dried. The funnel was held in the tail stock and a piece of flame cut 5.1 cm tubing in the head stock. A conventional ring seal was formed by tooling the edge of the 5.1 cm tubing to the top of the funnel assembly (Fig. 5). The excess glass was pulled away and a 8.5 cm, 10 ml pyrex syringe barrel (uniform bore) was blown onto the cathode compartment head. An 8 mm piece of tubing was joined near the ring seal to be joined eventually to a condenser. A no. 5 O-ring joint was joined as an outlet for the water jacket. (Prior to a series of runs, the cell was treated in a hot aqua regia then baked at

Figure 5
Sealing the jacket to the inner part of the cathode compartment.

525°C overnight. To facilitate the removal of the cell from the coolant no. 5 O-ring joints were used extensively as inlets and outlets in the water jacketed sections.)

The cathode assembly was then held in the head stock by the pyrex syringe and the outer jacket was collapsed onto the flared bottom of the funnel to form the ring seal. A 4.5 cm piece of no. 9 O-ring was joined to the bottom. The outer jacket was collapsed onto the Luggin capillary to form a ring seal and a piece of 1 cm O.D. - 4 mm I.D. tubing joined to it. The same procedure was used on the anode inlet. A no. 5, O-ring joint was joined as an inlet for the water jacket. Halfway up the compartment, and on opposite sides, two pieces of 5 mm rod, 4 cm long were joined to the water jacket to add strength and stability to the completed cell (see Fig. 1). The entire lower half of he cathode must be kept warm throughout this operation. The completed cathode compartment was then annealed.

ANODE AND REFERENCE ELECTRODE COMPARTMENTS

The inner part of the anode compartment (Fig. 6) was made from a 14 mm O.D. tube with a C porosity fritted disc. (Corning no. 39570). A piece of 2.2 cm tubing was joined above the fritted disc. A piece of 10 mm O.D. - 4 mm I.D. was sealed at this joint. A button was formed 12.5 cm above the fritted glass disc. The excess glass below the fritted disc was flame cut and flared. The side arm was cut and polished in the same manner as described for the cathode assembly to fit nicely into a 3 cm O.D. piece of tubing. This inner part was then washed and annealed.

A button was made on a 19/22 male ground glass joint 1 cm below the ground surface. A conventional ring seal was made in a 3 cm O.D. piece of tubing extending over the joint. The tubing was then cut 3.5 cm above the ring seal on a diamond saw and fire polished to form a cup around the joint.

Flame cut 3 cm O.D. tubing was then tooled onto the button of the anode. The 19/22 joint was joined to the anode with a distance of 4.5 cm between ring seals (see Fig. 7). A no. 5 O-ring joint was joined for the water outlet. The piece was then reversed. The outer tubing was collapsed

Figure 6
Inner part of the anode compartment.

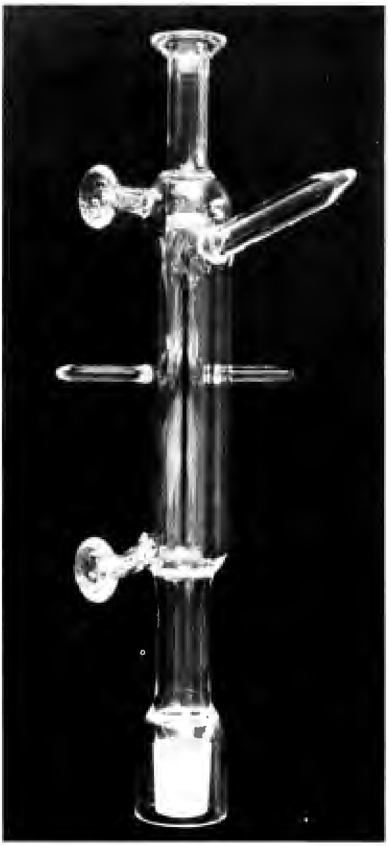

Figure 7
Completed anode compartment.

onto the flared inner piece, and a no. 9 O-ring joint 4.5 cm long was added. The side arm was sealed using the 1 cm heavy wall tubing. A no. 5 O-ring joint for the water inlet completed the anode. Two pieces of 5 mm rod, 4 cm long were added halfway up the water jacket eventually to be joined to the cathode and reference electrode compartments.

The anode compartment was duplicated, except for the different positions of the water inlet and outlet, to make the third part of the electrolysis cell, the reference electrode compartment.

CONDENSER

A condenser was made using a coil made from 8 mm tubing wound on a ½″ brass tube and sealed into a piece of 2.8 cm tubing. The distance between ring seals was 8.5 cm. A support rod was welded on the side and a no. 9, O-ring joint was sealed on the upper part and bent over (Fig. 8).

Figure 8
The condenser.

ASSEMBLY

All parts were annealed and assembled (see Fig. 1). The two stopcocks used were a special order from Eck & Krebs. They are a solid plug, oblique bore with liquid seal, high vacuum design, with the side arms coming out at a 70° angle. Extreme care was taken when joining stopcocks to the existing tubing. Uniformity in the bore was important, otherwise a bubble would form breaking the electrical circuit. A piece of rod was added to the bottom of the stopcocks for general stability when the cell was being washed or annealed. Supporting rod was joined between the three compartments for added strength. (The cups fabricated around the 19/22 joints allowed the joints as well as the stopcocks to be wetted and sealed by the electrolyte.)

ACKNOWLEDGMENTS

The author is indebted to the users of the cell, M. Hammerli and W. J. Olmstead, for helpful suggestions in its design and to Ken Gauthier for the photographs.

REFERENCE

1. Hammerli, M., Mislan, J. P., and Olmstead, W. J., J. Electrochem. Soc. *116* (1969) 779.

SELF-CENTERING BALL, SOCKET AND O-RING JOINT HOLDER

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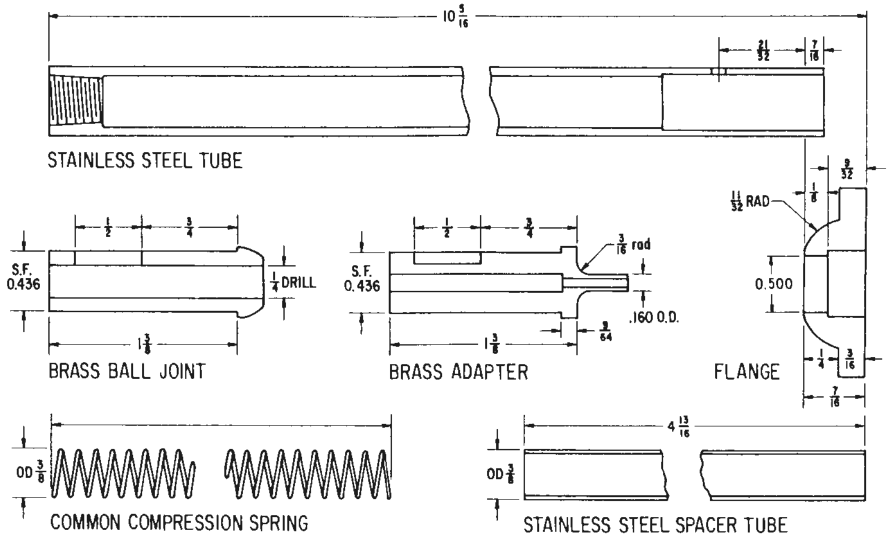
This metal holder was designed to hold various sizes of ball joints and flat O-ring joints in perfect alignment. This is accomplished by using a 12″ piece of stainless steel tubing ½″ O.D. with a hose connection at one end and a flange of 1⅜″ Dia. with a flat surface on the other.

This flange will hold and center either a socket or an O-ring joint being clamped together with a standard Thomas pinch clamp.

Inside the 12″ tube is a spring 4″ long and a metal sleeve 5″ long. This spring maintains a pressure on the male insert which centers the glass joints being held.

The male inserts are designed to conform to whatever type of joint you wish to hold. Also a slot is cut in the male insert so that a set screw

GLASS SHOP SPINNING ADAPTER



going through the wall of the 12″ x 12″ stainless steel tubing will keep the male insert from falling out.

Basically this holder is a sliding metal piston that will fit the I.D. of the joint to center it and a flat surface at right angle to the axis of the stainless steel holder so the joint will run true.

DETECTION AND EVALUATION OF STRAIN IN GLASS

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INTRODUCTION

The detection and evaluation of strain in glass can be done in many ways. It can be done mechanically, electrically, and also optically. For example, a study of the fracture markings of a broken glass specimen can tell much about the nature of the strain, its direction, relative magnitude, etc. Unfortunately, this is after the fact and we are interested in viable products not corpses.

Another mechanical method of analyzing strain is by means of applied coating. Here, the glass specimen is painted with a brittle lacquer. After the lacquer has dried hard, the specimen is stressed. From the network of cracks in the lacquer the nature of the strains that produced them can be inferred. However, this method is time consuming and not very accurate.

Electrical methods have also been used to measure strain in glass. Typically, a grid of fine wires having a relatively high coefficient of electrical resistance with respect to pressure is directionally oriented and cemented to selected areas of the glass specimen. The specimen is stressed and its change in ohmic resistance as measured by a sensitive bridge constitutes a measure of the strain magnitude and geometry. This is also a time consuming and not operationally practical method.

Finally, we have optical methods for strain analysis. One such group is by means of an interferometer. This is also overelaborate for our purpose. The method that has been found most applicable to the needs of the production shop and the glassblower turns out to be the one that uses polarized light. Four such polariscopic methods and their application for the detection and evaluation of strain in glass will be described.

ELASTICITY BACKGROUND

The study of strain by means of polarized light is aptly called photoelastic analysis. It involves both the elastic properties of the material being examined and the optical properties of light. So, before we take up the polariscopic analysis of strain let us briefly extract some of the relevent underlying background with respect to both elasticity and optics.

Let's start with strain first. Just what is it? How does it differ from stress? To begin with, strain is an elastic deformation, a change in size or shape produced by a stress. Thus a simple strain like one induced by a weight hanging from a suspended glass rod would principally be the small stretching in the vertical direction divided by the rod length. The stress essentially is equal to the force per unit area causing the strain. In this example it would be equal the weight attached to the rod divided by the cross sectional area of the rod.

The relationship between stress and strain was provided by Robert Hooke, a contemporary of Sir Isaac Newton, and also a leading member of the English Royal Society. Hooke found that stress was directly proportional to strain.

$\sigma = KS$ where K was the elastic constant. Hooke's Law defines elasticity as requiring proportionality between stress and strain and when stress is released, strain is released. Molten glass, liquids, and gases with a free boundary are not elastic because they will yield and flow when subjected to stress. Glass at ambient conditions and up to several hundred degrees Fahrenheit is almost perfectly elastic in that it follows Hooke's Law up to the fracture stress.

GENERATION OF STRAIN IN GLASS

As far as strain in glass is concerned a distinction is generally made between so called "temporary" and "permanent" strain. The former is applied to mechanical or low temperature thermal shock in which the strain disappears with the termination of the stress.

"Permanent" strain on the other hand is the kind generated when glass is cooled rapidly from a temperature above its so called strain point. It is caused by the outer layer of the glass cooling first and becoming "rigid". As the inner layer follows it in cooling, it cannot undergo the normal thermal contractions or shrinkage associated with the lower temperature because it is cemented to the rigid outer layer. Consequently strain develops as the inner layer is stretched and the outer layer compressed.

To release this type of strain, an annealing treatment is required in which the glass is heated above its strain point until the strain relaxes by semi-plastic flow. The glass must then be cooled sufficiently slowly so that no excessive thermal expansivity gradients develop between adjacent layers.

It is this "permanent" strain with which the glass worker is primarily concerned and for which polariscopic aids are used. To describe these polariscopic aids a review of some of the involved optical principles is in order.

OPTICAL BACKGROUND

Light is an electromagnetic wave travelling with a velocity of 3×10^{10} cm/second. The wave length of light is what determines its color. Visible light ranges in wavelength from approximately 390 millimicrons or nanometers in the violet to 770 nanometers in the red. Typical wave length values for the familiar colors are listed:

COLOR	WAVE LENGTH, NANOMETERS
violet	410
indigo	445
blue	480
green	535
yellow	580
orange	620
red	710

When a light ray encounters a refractive interface as in Fig. 1 it is generally both refracted and reflected. The direction of the refracted ray is given by

$$N \sin \theta = N' \sin \theta' \qquad \text{(Snell's Law)}$$

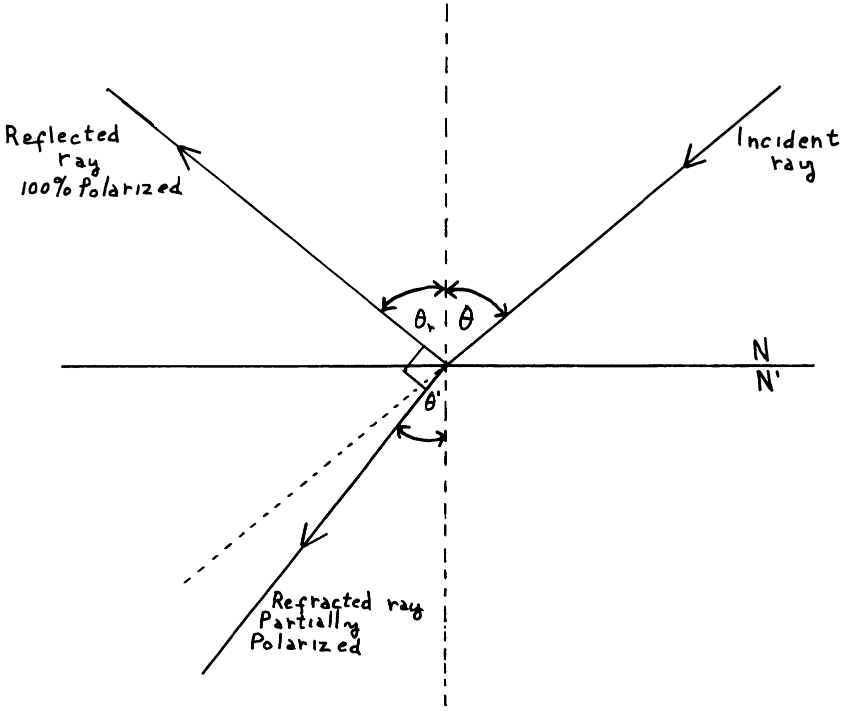

Figure I
Light Ray at Refractive Interface

It might be noted here that optical convention divides space into two kinds, image and object space. For object space all parameters like lengths, angles, indices of refraction, etc., are indicated by unprimed designations like L, θ, N, while for image space these parameters are primed

L', θ', N'. In both object and image space angles are all measured from the normal to the surface.

The absolute index of refraction is defined as the ratio of the velocity of light in vacuum to that in the given medium. For practical work the index of refraction relative to air rather than water is generally used because most measurements are usually made in air. Since the velocity of light in a medium like water or glass is less than in air, we find that the indices of refraction are greater than one. For water it is 1.33 and for glass it varies from about 1.4 to about 2. for very dense optical glass.

As far as polarization is concerned it should be remembered that light is a transverse electric wave. That is it vibrated in a direction at right angles to its direction of travel like a wave on the surface of a body of water. A beam of light consists of millions of such waves each vibrating in different random directions. Hence it is unpolarized.

Linear polarized light on the other hand is different in that it consists of light vibrating in a single plane. Polarized light can be produced in many ways. For example, when the two refractive media of Fig. 1 are air and glass respectively, and the angle between the refracted and reflected ray is 90°, the reflected ray becomes 100% polarized. The polarizing or Brewster angle is given by

$$\text{Tan}\ \theta = N' \qquad \text{(Brewster's Law)}$$

For soda lime glass with an index of 1.52 this would require an incident angle of 53°. Alternatively the refracted ray which is partially polarized may be utilized after enhancing through the use of multiple plates. Traditionally, specially cut prisms like the Nicol and Glen Thompson have been among the most efficient means of producing polarized light but these generally have given way to the use of Polaroid material which is very efficient, can be made in large sizes and is inexpensive.

The effectiveness of polarized light in detecting and evaluating first birefringence and then strain, stems from the work of crystallographers. As early as 1814 David Brewster commented upon the similarity in optical behavior of a glass specimen under stress to a uniaxial crystal. The correlation between stress and birefringence was discovered by Brewster. Birefringence was found to be directly proportional to the stress upon the glass.

Since as Hooke had previously pointed out stress was proportional to strain, in effect a glass specimen having a "permanent" strain could also be evaluated by its birefringence. The basic photo-elastic equation deriving from Brewster was (R), the optical retardation due to birefringence was equal to the principal stress difference (P — Q) multiplied by the thickness of the specimen (T) and its stress optical coefficient (C).

or
$$R = C\,T \quad (P - Q)$$

$$(P - Q) = \frac{R}{CT}$$

The unit for the stress optical coefficient has since been named the Brewster. One Brewster = 10^{-13} sq. cm. per dyne. For borosilicate glass it has a value of about 3.9.

Insofar as birefringence plays an important role let us take a closer look at the connection between strain and birefringence. Annealed glass and crystals that are isometric (having perpendicular and equal axes) are isotropic. This means that their properties including the velocity of light through the glass or isometric crystal is constant regardless of direction of the light path or vibration direction. Accordingly the index of refraction is constant. Strained glass and most crystals however are anisotropic. Thus, properties including the velocity of light through those media are different for different directions of the light path or different vibration directions. The net result of birefringence is that one component of the light ray is retarded with respect to the other, and this situation can, under conditions to be described, produce optical interference.

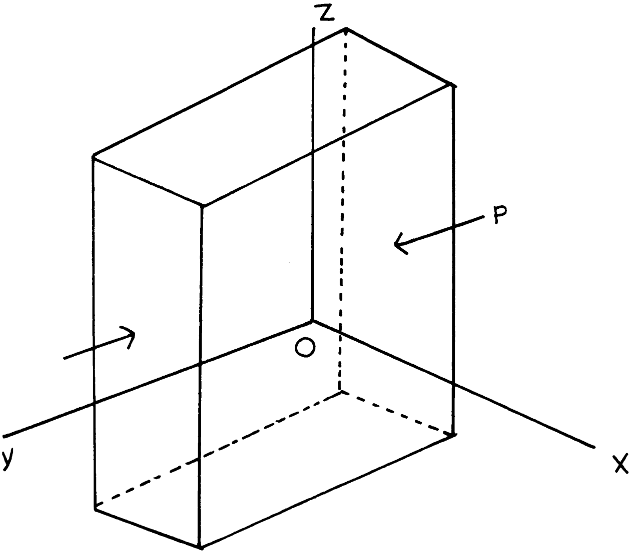

Figure 2
Glass Plate Under Compressive Thrust

In Fig. 2 we have a rectangular annealed glass plate represented with edges parallel to the X, Y, and Z axes. Unstrained the plate is isotropic and the index of refraction constant. Under the action of a compressive thrust P parallel to OY the plate behaves like a uniaxial crystal with the direction P representing the optic axis. The glass will be compressed in the direction OY and expanded equally in the other directions.

Along the direction P of the strained glass plate and along the optic axis of a crystal no birefringence is evident because the velocity of light will be the same for all vibration directions. At a direction at right angles to P, or the optic axis, the birefringence will be a maximum and for immediate positions, intermediate values of birefringence will be found. For maximum strain the position of maximum birefringence has to be employed. In a strained glass specimen or crystal, the birefringence is numerically equal to the difference between the maximum index of refraction corresponding to the minimum light velocity direction, and the minimum index of refraction corresponding to the maximum light velocity direction.

The effect of the polariscope upon the strained glass is to make the optical retardation due to the strain birefringence evident to the eye by means of light or darkness or color. Also by evaluating such effects the strain can be measured. Let us illustrate this by means of the basic polariscope.

THE BLACK AND WHITE POLARISCOPE

In Fig. 3 we have a representation of the simplest type of polariscope which is known as the linear or black and white polariscope. It consists of a light source, diffuser, polarizer (usually a polaroid sheet or laminate) and an analyzer (usually a polaroid with a vibration direction at right

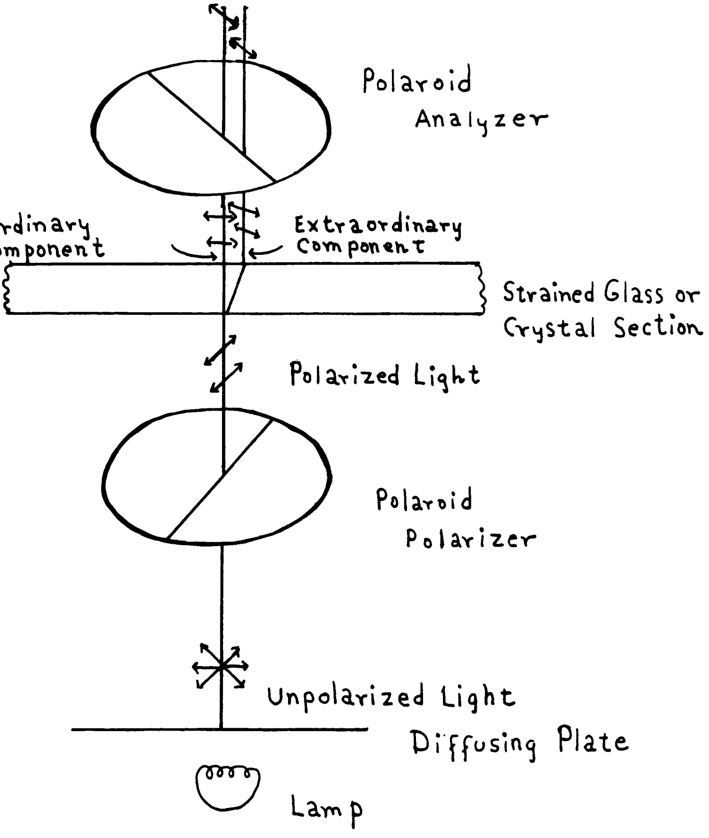

Figure 3
Linear Polariscope

angles to the polarizer). When viewed through the analyzer, the field appears dark or black. If a piece of annealed glass is placed between the polarizer and analyzer, the field still appears black no matter how we orient the specimen. The effects of internal reflections may be removed by immersion in a matching index liquid cell.

However, if we place a strained glass sample in the conventional linear polariscope the situation changes. As we view the sample with the axis in a horizontal position, we observe light thru the sample. As we rotate the sample 360° along an axis parallel to the line of sight we note that it lights up and then darkens four times. It is at its brightest in its horizontal and vertical positions and is dark at 45° to this position. The intensity of light that one views as the specimen is rotated is a function of the angle and in practice the axis of the specimen should be horizontal or vertical for maximum effect.

One of the shortcomings of the black and white polariscope is that it does not give a sensitive picture to the eye. As the strain in the glass is increased, the appearance is from black to dark gray to medium gray, to light gray, without any conspicuous color being evident until a large amount of strain, almost 300 nanometers, is present.

THE FIRST ORDER RED POLARISCOPE

In the color or 1st order red polariscope the situation is improved by placing a full wave plate retarder with the axis at 45° to the analyzer directly behind the analyzer. This serves to retard the light a full wave length to a first order red or pinkish lavender color.

It might at first appear strange that a combination of white light and neutral filters should yield a lavender color. The reason for this can be understood by reference to Fig. 4. Here the effect of optical retardation due to the birefringence of a quartz wedge in the field of what is essentially a black and white polariscope can be seen. When the apex of the

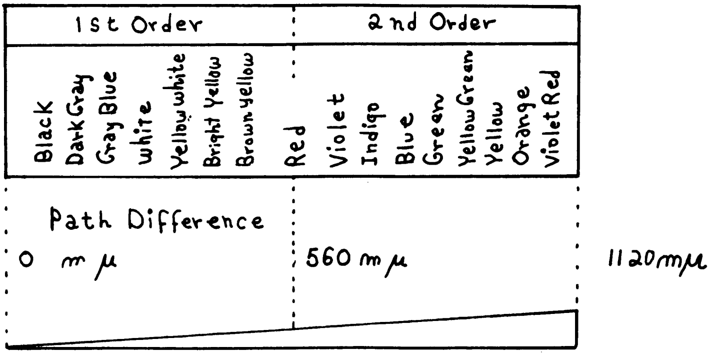

Figure 4
Quartz Wedge Birefringence Effects in Linear Polariscope

wedge with practically no thickness is viewed with this arrangement there is essentially zero retardation introduced because the retardation depends upon the thickness as well as the birefringence of the material. Consequently the field is black. As the thicker portions of the wedge are viewed, the color rises to gray, light gray, and when a thickness corresponding to a retardation or optical path difference of 560 nanometers is reached, we come to the end of the first order. The field is now lavender pink because the optical path difference is sufficient to cause destructive interference for the green, yellow, and orange part of the white light spectrum leaving violet and red to come through. This part of the spectrum comes through because the wave lengths of these colors are such as to produce retardations that yield constructive interference.

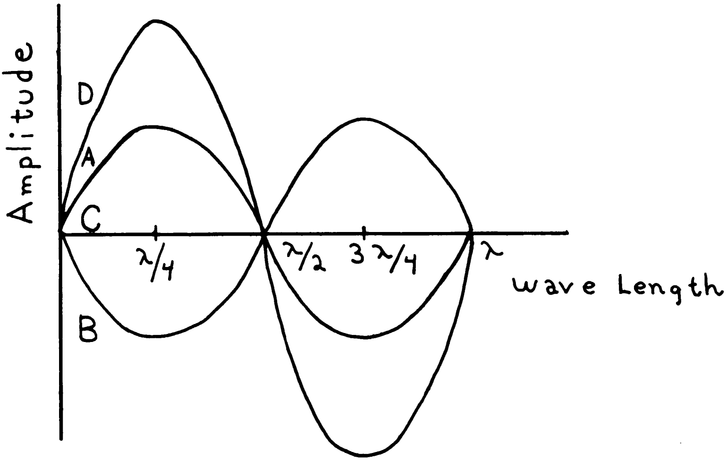

Figure 5
Destructive and Constructive Optical Interference

Typical optical interference is illustrated in Fig. 5. It can be destructive as in the case of A and B where the crest and trough of the wave are directly superposed (optical path difference equals $N\lambda/2$, where N is an odd integer and λ is equal to the wave length). In this case A and B are algebraically combined to produce zero amptitude or darkness. When the two waves are in the same phase (path difference equals $N\lambda/2$, where N is an even integer), the amptitudes add up algebraically to produce brighter light of amptitude D. For optical interference the two light waves must have a constant phase relationship and be of the same wave length. In the polariscope this is effected by the birefringent strained glass causing the same wave front to divide into two components at right angles to each other as illustrated in Fig. 3. When these components pass through the analyzer they emerge in the same plane and will undergo constructive or destructive interference as the path difference requirements are met for different wave lengths. Interestingly enough, in the case of the polariscope the vector sum of the projections of these components interchanges the path difference requirements for constructive and destructive interference with constructive interference occurring at odd integral values of $\lambda/2$ and destructive interference at even integral values of $\lambda/2$.

The advantage of the color polariscope is that even a small amount of strain manifests itself as a color change rather than a hard to assess light intensity change. While in the black and white polariscope distinct color did not appear until the retardation was almost 300 nanometers, in the first order red polariscope a retardation of even 20 nanometers will show a purplish cast in the specimen against the pink lavender color of the field. About 50 nanometers retardation will show a blue color.

In operation the procedure for determining strain is simple. However, some precautions with respect to orientation of the sample must be observed. This will be evident from an examination of Fig. 6. This rep-

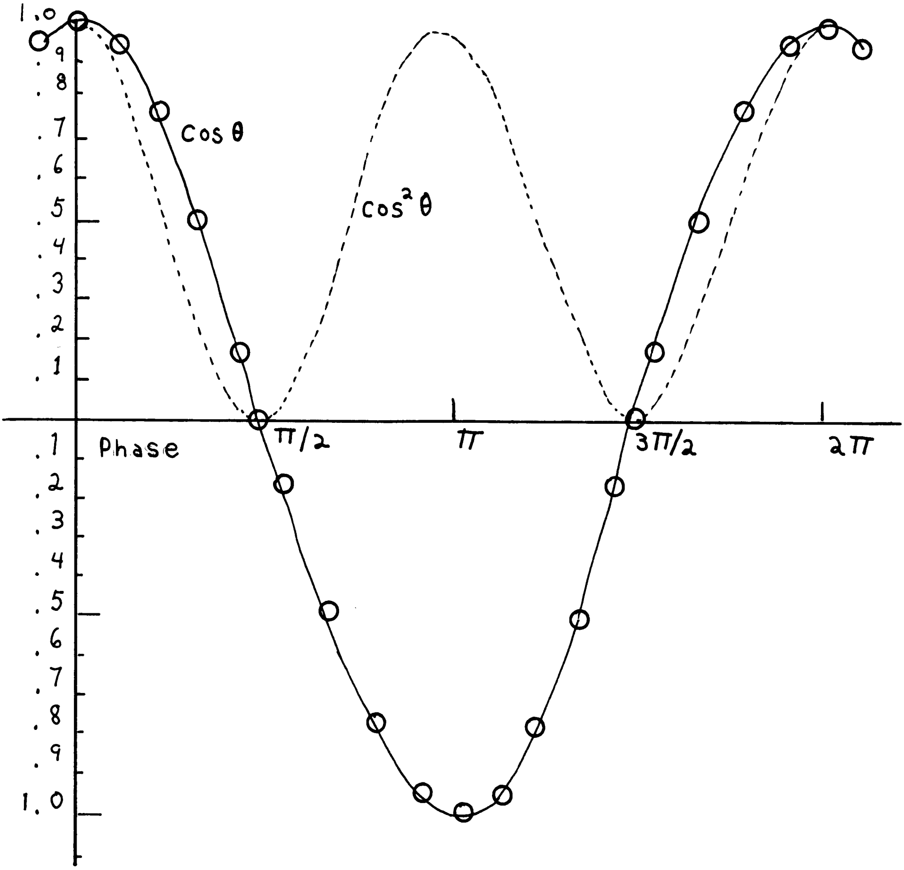

Figure 6
Variation of Amplitude with Rotation of Polaroids

resents the variation of the intensity of polarized light when viewed through two polaroids when one is rotated with respect to the other.

The intensity of the light as a function of the phase angle is given by the following equation:

$$I = I\ \text{max.}\ \text{COS}^2\theta \qquad \text{(Malus' Law)}$$

During the course of a complete cycle (2π or 360°) the COS^2 function undergoes two maxima and two minima. Physically we observe this as we rotate the polaroid as two maximum brightness positions and two maximum darkness positions. The polariscope also has 2 maxima and 2 minima positions. The polariscope is normally oriented so that the horizontal or vertical position of the glass strain axis is the maximum birefringence position, and the 45° position of the glass strain axis is the minimum or null position for the sample. Of course in the black and white polariscope a black field indicates the null condition while in the color polariscope the usual pink lavender field indicates the null condition.

If we were to examine a glass rod for strain we would position it horizontally in the polariscope since its strain axis is along its length. Now even a small amount of strain would manifest itself as a color change. If the sample is irregular it could be rotated somewhat in both directions until the maximum birefringence showed up, indicating the maximum strain.

A closer evaluation of strain with many types of polariscopes can be secured by calibrated strain discs which have 22.8 nanometers of strain per disc .25″ from the circumference. Since strain is algebraically additive in these discs, multiple stacked discs will yield multiple strain birefringence values. In practice (ASTM-C-148) one is added to N, the number of discs needed to make the closest color match just below the color of the strained sample. Multiplying N+1 by 22.8 nanometers yields the retardation estimate for the sample.

For a flat rectangular plate stressed at right angles to the line of sight the relationship between strain in pounds per square inch and birefringence can be expressed as

$$\sigma = \frac{R}{0.175\ cd}$$

where R = retardation in nanometers
c has a value of 3.9 for borosilicate glass, and
d is the thickness viewed

The color polariscope can also be used to determine the sign of the strain, that is whether it is tensile or compressive. Normally the color polariscope is arranged so that tension when the strain axis is horizontal is indicated by the color ascending, that is going from the pink lavender to purple, to blue to green etc., as the tension becomes greater. This can be confirmed by bending downward an annealed glass rod while it is held horizontally in the polariscope. The color in the upper or convex arc should follow this sequence during the bending while the color in the lower or concave arc should go from lavender to red to yellow.

QUARTER WAVE POLARISCOPE

In the quarter wave polariscope the arrangement of the optical elements is similar to that in the first order red polariscope. However, a quarter wave plate of about 141 nanometers retardation is used in place of the full wave. In the null position the field is black.

When a strained flat glass sample of low birefringence is introduced it produces a hazy dark cross which upon rotation of the analyzer separates into oppositely moving arcs. These are blue-gray on the concave portion of the arc and brownish on the convex portion. To make a strain measurement the analyzer is rotated until the blue gray color is displaced by the brown color at the desired location. The angular rotation then constitutes a measure of the strain in the sample. A rotation of one degree corresponds to a strain birefringence of 3.14 nanometers for white light.

In use with a ring sample a dark circular band is seen at the center. The analyzer is then rotated until the dark band displaces the light at the edge. The rotation to achieve this is then a measure of the strain birefringence.

The precision of this method is improved through the use of collimated monochromatic light like from a mercury or sodium lamp with appropriate filters. All elements must be accurately lined up with appropriate apertures to limit the beam. Magnification by means of a telescope, microscope, or projection may be employed.

PHOTOELASTIC POLARISCOPE

In the photo-elastic polariscope used for engineering studies of the effect of stress on plastic or glass models of complex shapes, two quarter wave plates are utilized. One is positioned behind and at a 45° angle to the polarizer and the other, similarly before the analyzer. Photographs are taken of the model and subject to mathematical analysis beyond the scope of this article to yield a complete strain picture.

CONCLUSION

In summary the detection and measurement of strain in glass crosses many disciplines, each of which have to be taken into account. Strain in glass unfortunately leaves no readily observable manifestation of its presence. However, through the mediacy of the polariscope, the birefringence of a strained glass specimen produces a retardation of one component of a light ray with respect to the other and upon recombination, optical interference effects occur. These effects are readily visible as patterns of brightness, darkness, or color, which can be interpreted in terms of strain.

An immediate answer can be provided by these means as to whether a glass article is strain free, whether it has been annealed or not and whether the grade of the annealing is comparable with the normal product. For simple geometries a direct measure of strain can be made. For complex geometries special techniques and extensive three dimensional photoelastic analyses are required.

Finally, defects such as stones, crystalites and striae can also be made very evident. This results from the strain due to differential expansion, which produces local birefringence.

FIBER OPTICS

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INTRODUCTION

Current prominence of fiber optics is due primarily to its applications in signal transmission. Light-weight glass optical fibers have been developed which allow for an efficient transmission of communication signals over large distances without amplification. A small number of optical fibers can transmit many voice or video channels of information reducing the weight and cost of cables. The transmission of signals through optical fibers is also immune to electromagnetic interference. All of the above features make the use of optical fibers in communications ever more attractive.

While the field of fiber optics in communications is about 10 years old, a somewhat earlier and still very significant application of optical fibers is in light and image transmission. The use of imaging fiber optic structures contributes to the design of a wide variety of inspection instruments with a special significance in the medical applications. This paper describes some of the techniques of design of such structures and their applications.

IMAGING FIBERS

The suitable transmission of image through flexible structures was made possible by the alignment of thin optical fibers made of glass core coated with a glass cladding, and was first reported in conjunction with its application to medicine.[1] When constructed of individual fibers about 10μm in diameter each. Fig. 1, the imaging bundle remains quite flexible

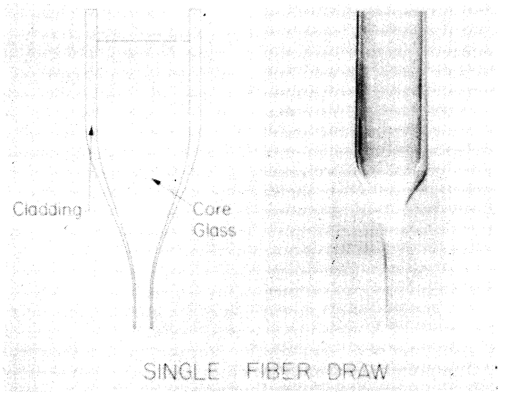

Figure I

allowing for small bending radii. The aligned individual fibers are tied together at their ends only while the rest of the fibers along the length of the bundle remain loose and therefore flexible.

The fabrication of flexible imaging fiber bundles involves precise winding of the optical fibers on a highly polished and uniform cylinder with the circumference of the latter determining the length of the imaging bundle, Fig. 2. The winding of aligned fibers can be obtained directly from the fiber-pulling furnace or from a separate spool of non-aligned fibers. When the entire imaging bundle is obtained in a single winding process, an overhang of fibers wound on the outer layers develops after the structure is removed from the cylinder. Such overhang can be eliminated by winding single or small number of fiber layers and then cutting them into strips to form the aligned fiber bundle, Fig. 3. The latter process although more laborious usually renders better uniformity in the aligned bundle.

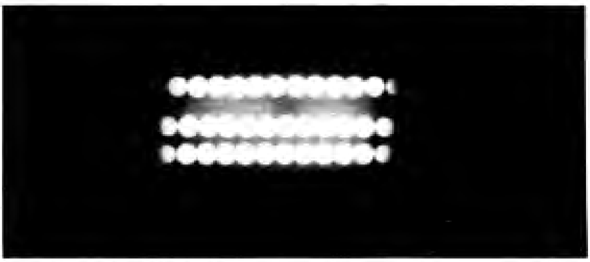

Figure 2

Figure 3

The resolution attainable with a perfectly aligned fiber optic imaging structure is determined by the fiber diameter when the fibers are singlecored and cylindrical. For hexagonal configuration of fibers, the resolution, in optical line pairs/mm is, at best, equal and usually slightly less than 1/(2d) where d is the fiber diameter in mm. A simple test of image quality may be obtained by viewing the transmission of a sharp edge such as a knife edge. A more quantitative measure of the edge response is the optical transfer function while the modulation transfer function provides a still more accurate means of determining the image quality transmission of optical fibers.[2]

In many cases it is more convenient to obtain highly aligned flexible imaging bundles by drawing in the vertical furnace a multiple fiber. Such a structure contains a number of small cross-section glass fibers of relatively high refractive index fused into a low-refractive index matrix. A multiple fiber of 50 to 100μm thickness can be handled in a manner similar to the single-core fiber, but has the image-conveying property of a number of single fibers of much smaller diameter. To realize the maximum resolution capacity of multiple fibers, they are shaped geometrically to facilitate close packing and so eliminate interstitial spaces. Thus, a square multiple fiber 60μm on a side containing 36 optically distinct 10μm cores arranged in a 6 x 6 array immersed in a low-refractive index coating material has a resolution capability of approximataely 40 line pairs/mm. Such multiple fibers although still thin enough to allow a reasonable degree of bending, are not adequate for the highly flexible medical instruments and are presently used primarily in the design of inspection fiberscopes for industrial applications. Figure 4 shows the cross-section of an 8 x 8, 60μm on the side multiple fiber made of flint glass core and soda-lime glass cladding.

Most commercially available flexible fiberscopes for medical applications utilize imaging bundles of 2 to 5mm in diameter. The smallest flexible imaging structure reported had a cross-section of 1mm^2 and was made of 9μm in diameter each, individually aligned optical fibers with a length of 70cm.[3] When used in most of the known applications of endoscopy

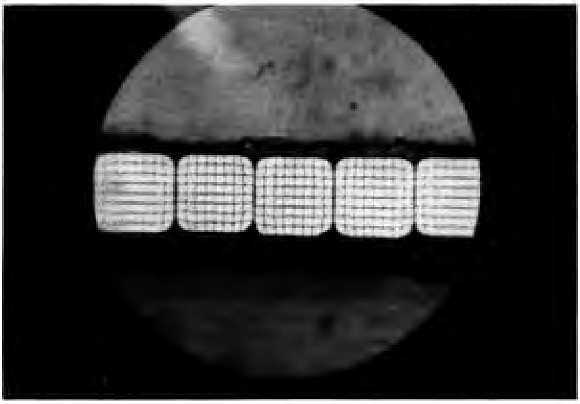

Figure 4

Figure 5

where the device is inserted through natural openings in the human body, the size of the above imaging bundles is quite adequate. However, when the endoscope is used percutaneously its size must be considerably reduced. On the other hand, a percutaneous fiberscope does not usually require a flexible structure.

A rigid multifiber can be obtained directly by an additional process of pulling a large number of optical fibers in a furnace, Fig. 5, in a manner that preserves their alignment similar to the fabrication of a 6 x 6 array of fibers, Fig. 6. A multifiber 0.5mm in diameter containing 11,000 fibers of 5μm diameter each was used in an early version of a fiber optic hypodermic probe designed to observe tissue microscopically, in vivo, at various depths under the skin.[4] The absence of lenses at the distal end of the hypodermic probe required that the end of the fiber bundle be placed in contact with the specimen being viewed, limiting the field of view to the small area of the imaging bundle. The subsequent development of 0.5mm diameter lens systems using plano-convex lenses resulted in the design of a hypodermic fiberscope with an imaging structure which provided a

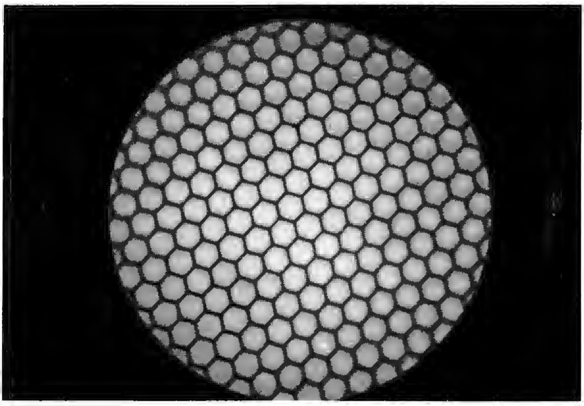

Figure 6

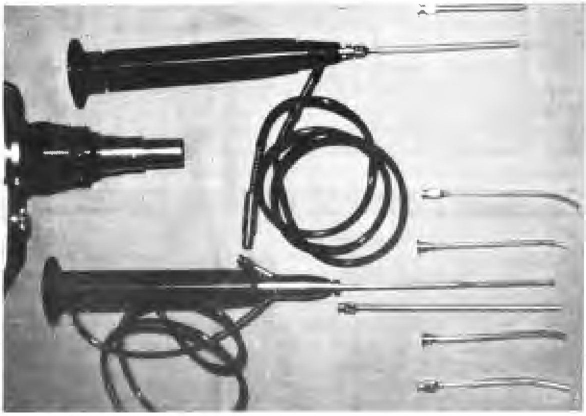

Figure 7

very large depth of field (3mm to infinity) and a field of view of over 40°.[5] It was first used in a percutaneous endoscope for the visualization of the spinal canal and cord, Fig. 7.[6]

The choice of glasses for core and cladding of the optical fiber requires that their respective indices of refraction be sufficiently different to render a fiber with a relatively large numerical aperture. A large number of glass materials may be found to satisfy this requirement. However, other requirements related to the fabrication process must be considered. The softening points of the two glasses and their expansion coefficients must be matched and the material must be obtained in a suitable form; e.g., the cladding is best utilized when it is in the form of a tubing. All of the above requirements greatly narrow the choice of available materials. Of the materials used most frequently are lanthanum crown and flint glass for the core with refractive indices of about 1.67 and 1.62, respectively, and soda-lime for the cladding with a refractive index of 1.52; the latter is also available in tubing form.

The fabrication of a rigid multifiber is further complicated by the additional heating process when the matrix of fibers is being produced in the furnace. The requirements of compatible expansion coefficients and softening points become yet more severe with the complications arising from non-uniform heating of the multifiber. As a result, it is extremely difficult to produce a perfect imaging multifiber, i.e., a structure free of broken individual fibers. Each such broken fiber appears as a black spot in the conveyed image. Nonetheless, the ability to produce an imaging multifiber with a very high resolution of $5\mu m$ per individual fiber diameter is attractive enough to employ it in the fabrication of flexible imaging bundles. This is obtained by producing a multifiber made of individual optical fibers each with a double coating where the outer cladding is chosen to be selectively etched afterwards. Up to the present this etching process of flexible imaging bundles could be employed only with structures containing individual fibers of no less than about $18\mu m$ in diameter which is considerably larger than in the currently available aligned imaging bundles. Of course, if successful, the etching process could reduce the cost of fabrication, and, in principle, should provide imaging structures of very high resolution.

All of the previously mentioned imaging structures utilize step-index optical fibers. Current developments in graded-index optical fibers have a great significance in the design of imaging structures for small percutaneous endoscopes. Developed by the Nippon Electric and the Nippon Sheet Glass Companies in Japan the new lens-like glass fiber is named SELFOC to indicate its focusing properties. The fiber guide has a parabolic distribution of the refractive index and is capable of image transmission through a single fiber of a specific length. Its resolving power, which depends on the field angle ranges from 200 to 400 line pairs/mm. The resolving power is affected by the irregularity of the parabolic refractive-index variations, flatness of the terminal surfaces and chromatic aberration.[7] Similar to the step-index fibers, an imaging fiber with a graded-index of refraction should have a large numerical aperture, i.e., a wider spread in the refractive index than is usually the case in optical fibers used in communications. At present the imaging graded-index fiber is fabricated in sizes ranging from 1 to 2mm in diameter and in lengths from 4 to 35 mm which provide both erect and inverted images. Depending on the length of the fiber, an object located at a distance from one end of the rod will form an image beyond its other end surface and thus will function as a biconvex lens. By combining several sections of the graded-index rod imaging structures up to 24cm long were constructed.

ILLUMINATION FIBERS

Optical fibers were originally and still are used to transmit light in a variety of instruments both in conjunction with fiber optic imaging structures or to provide "cold-light" illumination at the distal end of hollow or all-lens fiberscopes. The fabrication of illumination fibers is considerably simpler than in the case of its imaging counterpart in that no alignment or coherence in the fiber arrangement is necessary. Both glass and plastic fibers are used and the major concerns in their performance is flexibility and low transmission loss. Of course, since only short lengths of fibers below several meters are involved, the low attenuation required is far below that expected in the current low-loss fibers for communications. An important consideration in the choice and preparation of optical fibers for illumination in medical instruments is the spectral response of transmission of light. The faithful rendition of color in viewing and photography depends on the spectral content of the white-light illumination.

In order to increase the available light at the distal end of the fiberscope light sources of ever-increasing intensity have been developed. In recent years, powerful arc lamps of high intensity and small source size have become available. Special lens and reflector designs have been used to concentrate the light onto the bundle of illumination fibers. However, due to the incoherence of conventional light it is not possible to effectively capture and concentrate such light onto a very small area of the illumination fibers. On the other hand, laser light which is coherent can be focused down to an area limited only by diffraction or a spot of about $1\mu m$ in diameter. However, a typical laser produces monochromatic light which cannot be used to obtain color rendition of the illuminated object. Recent work in ion lasers indicated that it is possible to obtain efficient lasing at several wavelengths simultaneously.[8] Using a krypton-ion laser with a special broad-band output mirror it was possible to obtain a 1mm diameter beam of white light which was then focused on a single optical fiber of $85\mu m$ in diameter using a conventional lens of 38mm focal length.[9] By proper adjustment of gas pressure and discharge current a balanced output of three colors at 476.2nm, 568.2nm, and 647.1nm rendered white light. The 94cm long optical fiber was made of F2 flint glass core and R6 soda-lime cladding, and had a numerical aperture of 0.57.

Adequate illumination of $10\mu W/cm^2$ for viewing and color photography was obtained using 45mW of white light from the laser which was attenuated in the optical fiber to 15mW of radiant power. Computed fractional attenuation losses due to surface and internal transmission only are 0.32, 0.37, and 0.46 for the corresponding red, green and blue colors, while the measured values were 0.60, 0.60, and 0.75, respectively, the difference attributable to the internal reflection losses. In order to reduce the speckle effect due to the coherent source[10] a 0.25mm thick Teflon diffuser was used which further reduced the available radiance by a factor of five. Subsequently, a more efficient method of reducing the speckle was devised, wherein the optical fiber was being slightly vibrated to frustrate the coherence of the transmitted light. Maximum white light output from the above laser was 150mW, such power levels still being far below the power handling capability of glass optical fibers.[11]

In applications where black-and-white displays are adequate, a very inexpensive He-Ne laser can be utilized effectively to obtain TV or photographic displays. The red light from a 3mW He-Ne laser was transmitted through the same 94cm optical fiber and rendered a radiant power of 0.25 mW through a 0.25mm thick Teflon diffuser. The alignment of the laser beam with the optical fiber can be simplified by using a tapered structure of the latter. An optical fiber 75cm long made of the same material as described above was fabricated 5mm in diameter at one end tapered over a length of 10cm down to a $50\mu m$ diameter. The tapered optical fiber provided illumination similar to that obtained with a conventional fiber and a lens.

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SILOXANE POLYMERS FOR SEALING APPLICATIONS

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1. INTRODUCTION

You, the glassblowers assembled today in this room are used to handling glass. You are the experts in forming and shaping glass into many useful articles. How often in your daily work you wished that your masterpiece remained in its original form without breaking, that it could take stresses and flaws. Is it ever possible to synthesize glass like material which would combine all the good properties of glass (clarity, chemical resistance, heat stability, dielectric strength, nonflammable, etc.) with all the good characteristics of plastic (flexibility, resiliency under impact, low temperature cure and forming, etc.). Many experimental attempts were made in the past, and as of today, substantial progress was achieved in meeting this historical challenge.

Siloxane polymers are the best representative of a group of materials forming a bridge between glasses and plastics. The structures and properties of these polymers will be examined first, and then their sealing characteristics will be described, and finally, their present and future applications will be illustrated by examples.

2. SILOXANE POLYMERS

Siloxane polymers are materials containing silicon and oxygen groups. (name: SIL-OX-ANE). They are logically divided into mono-, di, and tri-siloxanes by the number of functional groups. Most of this presentation will center around the tri-siloxanes since they are approaching glasses in many respects.

The first slide will assist us in explaining this point by tabulating the structural features with some properties of silica glasses, siloxane resins, silicones, and mono-siloxanes. This table is arranged from the silica glasses, on the left, to mono-siloxanes, on the right. The silica glasses are well known to you, therefore, their structures are shown first. The left column of this table indicates the structural aspects and properties compared in this table; namely —state, number of oxygens per silicon, number of organic groups per silicon, function, type of structures, and finally, schematic representations of the structures.

All these materials have the same main structural feature. The *common* denominator is the-Si-O-bonds. They differ in the *state;* glasses are solids at room temperature. Siloxane resins are also solids at room temperature after cure. Silicones are viscous fluids or rubbery pastes. Finally, the simple mono-siloxanes are also fluids with lower viscosity than silicones.

NUMBER OF OXYGENS

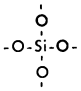
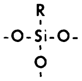
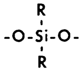
Glasses have 4 oxygens surrounding 1 silicon, arranged in the tetrahedra configuration.

Siloxane polymers have 3 oxygens around 1 silicon and 1 organic group replacing the 1 extra oxygen present in the silica glasses.

Silicones have 2 oxygens and 2 organic groups around their silicones.

Simple siloxanes have only 1 oxygens per silicon and 3 organic groups.

TABLE 1

	SILICA GLASSES	SILOXANE RESINS	SILICONES	SILOXANES
STATE	Solid	Solid	Viscous Fluids Rubbery Pastes	Fluids
NUMBER OF OXYGENS PER SILICON	4	3	2	1
ORGANIC GROUPS PER SILICON	0	1	2	3
FUNCTIONAL	TETRA -	TRI -	DI -	MONO -
STRUCTURE	3-Dimensional	3-Dimensional Cross-Linked	2-Dimensional Chains	End Groups
SCHEME	 $\begin{array}{c} \vert \\ O \\ \vert \\ -O-Si-O- \\ \vert \\ O \\ \vert \end{array}$	 $\begin{array}{c} R \\ \vert \\ -O-Si-O- \\ \vert \\ O \\ \vert \end{array}$	 $\begin{array}{c} R \\ \vert \\ -O-Si-O- \\ \vert \\ R \end{array}$	R_3-Si-OH

As indicated in this first table, the number of oxygens is decreasing from 4 to 1 (glasses to mono-siloxanes) and inversely the number of organic groups is increasing from 0 to 3 (glasses to simple siloxanes). The sum of oxygens and organic groups is equal to 4, which is the silicon coordination.

FUNCTIONAL

Glasses are tetra-functional
Siloxane Polymers are tri-functional
Silicones are di-functional
Simple Siloxanes are mono-functional

STRUCTURE

Glasses are 3-dimensional
Siloxane Polymers are also 3-dimensional
— cross-linked
Silicones are 2-dimensional chains
Simple Siloxanes are end group

SCHEME = Common -O-Si-O-

Glasses — two additional oxygens
Siloxane Resins — one additional oxygen
and one organic group (R)
Silicones — two organic groups (2 R's)
Simple Siloxane — three organic groups (3 R's)

This table strongly suggests that siloxane polymers are expected to behave like glasses and like silicones. Let us now examine the properties of siloxane polymers and see in what properties they are similar to glasses and what other characteristics they share with silicones.

3. PROPERTIES OF SILOXANE POLYMERS

The next slide (No. 2) compares the siloxane polymers with glasses and silicones in physical, thermal, optical and electrical properties.

— Properties of siloxane polymers between commercial soda-lime glasses and silicones:

Density, tensile, compressive, flexural strength, hardness, refractive index, dielectric strength, and maximum service temperature.

— Properties of siloxane resins approaching glasses:

Light transmission, light cut off, dielectric strength

— Properties of siloxane resins similar to silicones:

Thermal conductivity, dielectric constant

The siloxane polymers belong to the thermosetting resins. These are cured and hardened by heating. Properly cured siloxane resins are hard, transparent, chemical and heat resistant, and nonflammable.

TABLE 2

Properties of Siloxane Resins Compared to Properties of Glasses and Silicones

Physical	Glasses*	Siloxane Resins	Silicones
Density, GM/CC	2.47	1.3	1.1
Tensile Strength, × 1000 psi	200	3.5	1.0
Compressive Strength, × 10^3 psi	1000	30	16
Flexural Strength, × 10^3 psi	8	6	5
Hardness	500 K	120 R	89 M
Thermal			
Thermal Conductivity BTU/Hr./Ft.^{2}/°F/Ft	0.5	0.08	0.12
Thermal Expansion × 10^{-7} per °C	90	1300	600

* Glass - Commercial - Soda - Lime - Silica

Properties of Siloxane Resins Compared to Properties of Glasses and Silicones

Optical	Glasses*	Siloxane Resins	Silicones
Refractive Index	1.51	1.49	1.2
Light Transmission %	90-97	90-95	Opaque
Light Cut Off, micron	0.38	0.3	Opaque
Electrical			
Dielectric Strength, volts/mil	1200	900	180
Dielectric Constant, 10^6 cycles	7.2	3.2	3.2
Maximum Service Temperature °C	460	260	160

* Glass - Commercial - Soda - Lime - Silica

4. SEALING WITH SILOXANE RESINS

Siloxane resins are cured at relatively low temperatures (150°C-220°C), and consequently, some of the first applications of these materials were in sealing and coating a variety of materials. Generally, they can be used for any sealing applications where silicones or glasses are used.

4.1 *Metal Sealing Coating*

Metals have many excellent useful properties, but they have to be protected from oxidation to achieve some decent service life. Paints, galvanization (Zn-coatings) glass enamels, and also, silicones are used for metal coatings. Siloxane resins are used with many advantages for sealing and coating metals with relatively low melting points like aluminum, copper, and others. Silicones can be also used, but they are not hard enough to protect the metals from abrasion and they also are adversely affected by exposure to sun rays (UV).

Siloxane resins are applied *as thin coating* (0.5 mil) by spraying, brushing, dipping, or flow casting. Thickness is usually controlled by solid solvent ratios. Solids concentrations of about 25% will yield film 0.2 mil thick by dipping. For proper *cure, the temperature and time* should be well controlled; e.g., cure and temperature of 175°C will require 10 minutes cure time for 20 mil thick film of siloxane resins on aluminum. Only 5 minutes are required to cure the same film exposed to 200°C. The temperatures, as low as 75°C, can cure siloxane resin in about 24 hours. Properly cured coatings are hard and transparent like glass. *Pencil Hardness* of 7/H and above is normally expected for good films of siloxane resins. 0.5 mils thick film remains intact in flexing of the base metal, in as much as 10% of metal elongation (G.E. Test). *Dielectric strength* of these coatings is high. Some films measured as high as 3000 volts/mil.

Thin coatings are *stable in vacuum* with very little or not outgassing up to 450°C.

Long time (8 years) *outdoors exposure* to weathering show these coatings very stable — as stable as glasses. They have excellent *stability in UV light* from 0.3 microns. This characteristic places these resins into a very special category among resins. In this unique UV behavior, they are really like inorganic glasses. Copper and aluminum panels were exposed for literally thousands of hours in the Atlas Twin Arc 655 *Weather-ometer* with little change in both the coating and the metal under the coating.

Metal-Metal seals are achieved by coating both substrate members and joining them mechanically by fixtures before the final cure.

Table 3 summarizes the essential properties of clear siloxane polymer coatings on aluminum and copper.

TABLE 3

Properties of Siloxane Polymer Coatings on Aluminum and Copper

	Aluminum	Copper
Thickness mils	0.5	0.5
Dielectric Strength Volts/mil	3,000	3,000-5,000
Heat Resistance (155°C in air 24 hours)	excellent	good
Flexibility	2%	1%
Hardness, Pencil	6H - 9H	6H - 9H
Chemical Resistance		
Acetone	excellent	excellent
5% NaOH	good	excellent
5% HCl	excellent	excellent
Adhesion	excellent	excellent

4.2 *Glass Sealing and Coating*

Glass sealing and coating applications are very similar to metal sealing. Most of the glass sealing and coating information is available from the development work done on glass cloth laminates. An experimental polysiloxane resin was specifically developed for high temperature applications that require considerable high flexural strength when the laminate is exposed to elevated temperatures for a prolonged period of time.

Table 4 lists the typical physical and electrical properties of 14 ply glass laminate with siloxane polymer, No. 908 (O-I). The glass was 181E glass composition. The resin was cured at 250°C (482°F).

TABLE 4

Physical Properties of Siloxane Resin +181E Glass Cloth Laminate

Physical Property	Value	Method ASTM No.
Tensile Strength, psi at R.T.	30,600	(D 638)
Compressive Strength, psi at R.T.	14,200	(D 695)
Bond Strength, psi at R.T.	1,150	(D 952)
Thermal Conductivity, BTU/Hr./Ft.^{2}/Inn/°F	3.5	(D 2214)
Heat Distortion, under 264 psi load	496°F	(D 648)

Electrical Properties of Siloxane Resin + 181E Glass Cloth Laminate

Electrical Property	Value	Value After 240 hrs. @ 300°C	Method ASTM No.
Arc Resistance, seconds	248	260	(D 495)
Dielectric Strength, volts/mil	260	288	(D 149)
Dielectric Constant, 10^6CPS	4.54	4.39	(D 150)
Surface Resistance, Ohms	1.7×10^{15}	5.5×10^{13}	(D 257)
Volume Resistivity, Ohms-CM	2×10^{16}	1.5×10^{15}	(D 257)

Table 4 indicates the outstanding retention characteristics of siloxane resins after long exposure of 240 hours. This characteristic was studied further in examining the flexural strength behavior under varying conditions. The testing was conducted in accordane with procedures described in ASTM D 790.

TABLE 5

Flexural Strength at Room Temperature after Exposure to Specified Elevated Temperatures for Specified Times

Exposure Time Hours	Exposure Temp. °C (°F)	Flexural Strength × 10^3 psi
None	None	41
200	300 (572)	31
500	300 (572)	25
500	350 (662)	16

TABLE 6

Flexural Strength at Specified Elevated Temperature, after Exposure to Specified Elevated Temperatures

Exposure Time Hours	Exposure Temp. °C (°F)	Testing Temp. °C (°F)	Flexural Strength × 10^3 psi
None	300 (572)	300 (572)	24
200	300 (572)	300 (572)	22
500	300 (572)	300 (572)	19
500	350 (662)	350 (662)	13

TABLE 7

Flexural Strength at Room Temperature after 24 Hours Water Soak and 2 Hours Water Boil

Conditions	Exposure Time Hours	Exposure Temp. °C (°F)	Flexural Strength × 10^3 psi
24 hr. soak	None	Room Temp.	38
24 hr. soak	240	300 (572)	21
2 hr. boil	None	Room Temp.	39
2 hr. boil	240	300 (572)	21

This table shows that 24 hours water soak and 2 hours water boil gave some flexural strength after 240 hours exposure to 300°C.

4. ELECTRONIC APPLICATIONS

Since the electrical and mechanical properties of siloxane polymers are truly outstanding, it is not surprising to notice that these resins found their first applications in the electronic industry. Among the many products which can use siloxane resins the following are benefiting by application of these resins:

- High Voltage Diodes
- Resistors
- Solder Stops
- Dielectrics
- Capacitors
- Insulators
- DIP Packages

The advantages offered by siloxane polymers for the electronic applications are:

1. Low temperature cure (80°C-250°C)
2. Nonflammable (vs. epoxies, silicones)
3. Good dielectric properties
4. Resistance to elevated temperatures (350°C)
5. Good strength at elevated temperatures
6. High purity
7. Low Outgassing
8. Stability under vacuum
9. Excellent moisture barrier
10. Good bond strength

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GLASS: FROM BOTTLES TO FIBERS

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INTRODUCTION

Glass can be fragile or tough; transparent or opaque; it can transmit heat or stop it; can conduct electricity or block it. Indeed the versatility of glass makes it one of the most valuable materials of modern industry. It is one of mankind's oldest manufactured products and yet today it is being produced, sold and used in greater quantity than ever before. Chief reasons are extended use in construction, increasing automobile requirements, greater demand for glass containers, continued growth in the electronics industry, constant expansion in the use of glass fibers and vast possibilities unfolding as we enter the Space Age.

Today in our concern for the conservation of energy, glass influences the economical use of heat and may be the vital factor for the control and utilization of solar energy. Other interesting factors relate to electronics, medical science, transportation and useful packaging. Glass starting as a curiosity has been indeed aided by glassblowing technology to the point where it is now a vital necessity. The industry has grown from crude developments in 4000 B.C. to a major industry of about 10 billion dollar annual business.

GLASS CONTAINERS

HAND BLOWING AND WORKING OF GLASS

Actually, the first important step in glassmaking technique was the use of the blowing iron or the glassworking blowpipe. It probably was first used as early as 200 B.C. in Egypt and Babylon. This invention was comparable to that of the potter's wheel of the clay industry and was of equal importance. The blowpipe consists of an iron tube 4 to 5 feet long with a knob at one end, a mouthpiece at the other, it enables the glassworker to shape, by blowing at one end, a blob of hot glass adhering to the other end.

It was only a short step from these early efforts until glassblowers were shaping glassware of circular cross-section by blowing in cast iron paste molds and cast iron molds that had been smoothly polished inside. The molds were usually made in halves, hinged together and held in place by an assistant who opened and closed them for the blowers. Shallow pieces or shapes easily removed from the mold were blown in a one piece mold. The molds were designed as to thickness of wall so as to remain hot while in use and thus insure continued operation of the process. Bottles were typical iron-molds ware. They were stuck-up or held in place by holders and necks reheated and finished by hand. Letters, numbers or trade marks were cut in the mold and appeared raised on the surface of the article.

The art of shaping glassware by pressing was an American Development and found considerable use. The process was simple and comparatively rapid. Hot glass was gathered on a gathering iron and transferred to the press mold and a plunger descended on the filled (hot glass) to cause the glass to take the shape of the mold. Simple pieces such as tumblers as well as various flat shapes were made by this procedure.

Tubing and rods were made by gathering a heavy gob of glass on a modified blowpipe after which an assistant by means of a hot iron disk is able to contact the gob of glass and pull the glass in the form of a tube as the blowers blow into the blowpipe and thus creates the tube which is kept round by intermittent blowing while the diameter and wall thickness are controlled by the speed of drawing.

In all hand blowing operations some sort of finishing operation is necessary because the joints between mold and ring and plunger leave sharp corners and small fins at the upper edge of the ware. To accomplish this finishing operation the ware is lightly "stuck up" reheated in a small furnace (glory-hole) and finished as in off-hand practice. Handles and lips may be added, or the entire shape of the piece may be changed by cupping, flaring, or other operations of the finishing tool. Some of the best examples of hand molded glass are tableware and various types of crystal ware and the beautiful modern Steuben ware of Corning Glass Works.

The various hand working operations briefly described above and others were the forerunner and precursor to the mechanical and machine operations that were to follow.

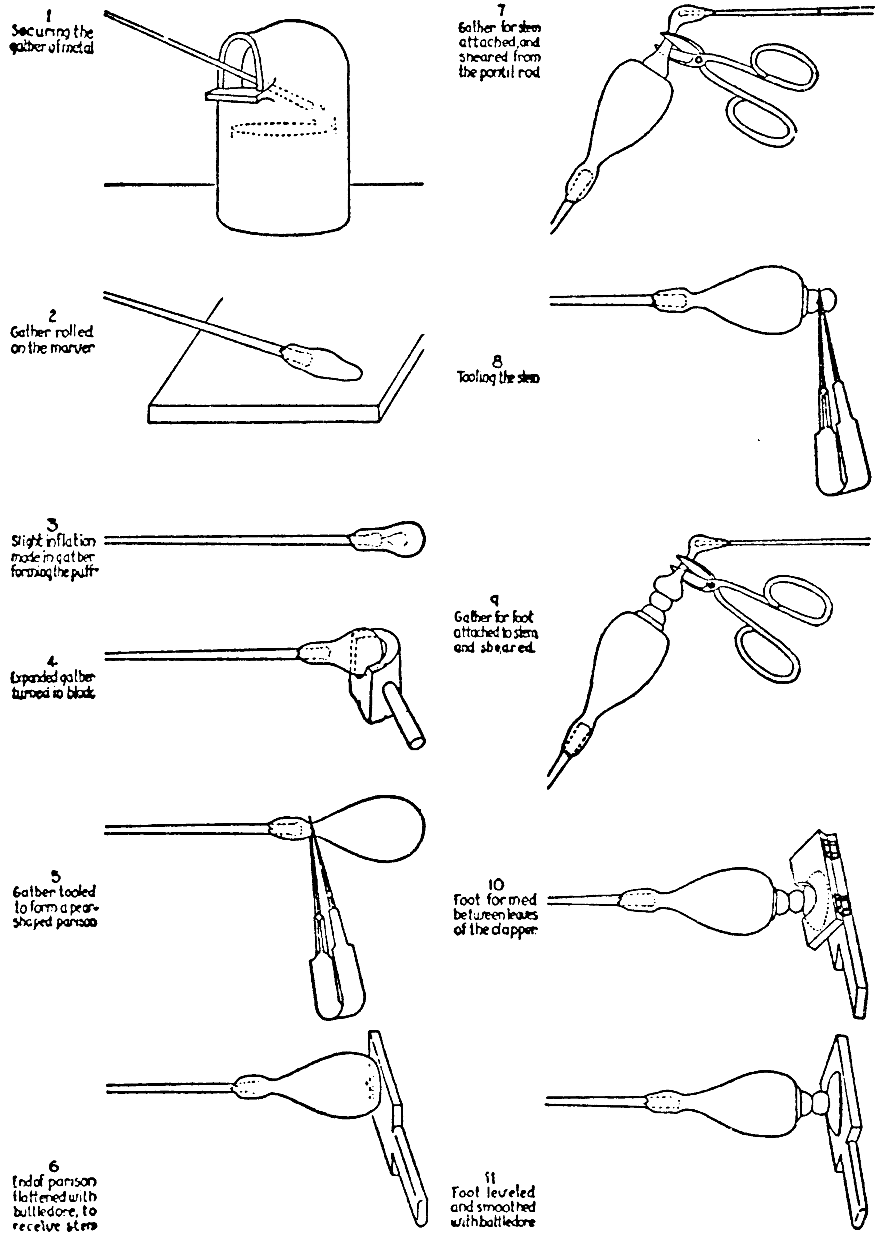
MECHANICAL GLASSWORKING

The mechanization of industry which began with the invention of the steam engine inevitably embraced glass manufacture. Machines have been devised for the production of nearly all the varieties of glassware that could be made by the older hand processes. The finish and luster of the ware and the artistry and individuality of design may be inferior in the mechanical product, it shows superiority, however, where accuracy of dimension is desirable and high production is needed.

It is interesting to note that most mechanical processes are for the most part attempts to duplicate in some way former hand processes, which emphasize the need for a knowledge of the properties and characteristics of hot glass.

The mechanical production of containers must begin with the making of a "parison", an imitation of the prepared gathering which the blower would form on his pipe before blowing a bottle in a mold. There are two distinct types of machines producing parisons in two different ways. One is the suction-fed or Owens Machine, the other is the gob-fed, represented by such machines as the Miller, the Hartford-Empire, the Lynch, and numerous others.

Molten glass for the automatic machines is produced in a continuous tank furnace. Actually there are three main mechanical methods of



Courtesy American Glass, *George & Helen McKearin, Crown Publishers, 1941*

Figure I
Parison Steps for Making a Stem Pitcher

CHARACTERISTICS

SUCTION PROCESS, BLOW AND BLOW.
ROTATES CONTINUOUSLY.
BOTH NARROW NECK AND WIDE MOUTH.
VERY WIDE FLEXIBILITY OF TIMING.
10 AND 15 HEADS.
SINGLE, DOUBLE, TRIPLE CAVITY.
VERY EFFICIENT FOR LONG RUNS.
MEDIUM OUTPUT RATE PER CAVITY.
HIGH OUTPUT RATE PER MACHINE.
2, 3, OR 4 MACHINES PER TANK.
PRODUCES UP TO CARBOY SIZE.
MOTOR DRIVEN AND CAM ACTUATED.

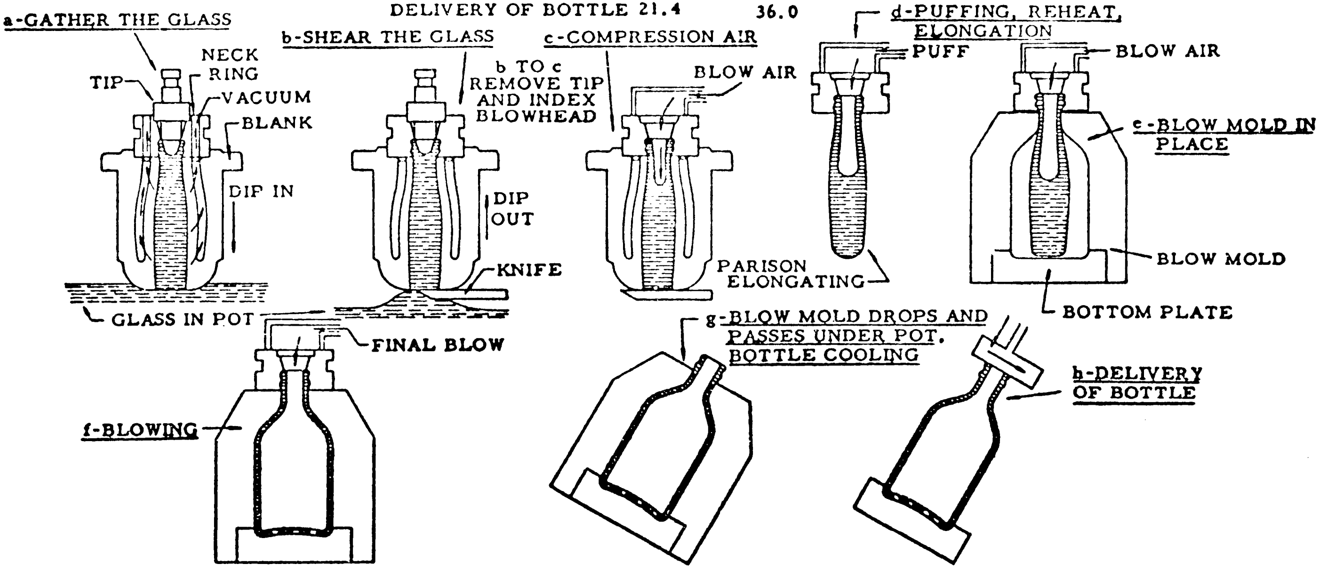
TIME CYCLE IN SECONDS	12 OZ. BEER	HALF GALLON
MACHINE TYPE	15 HEAD 2 CAVITY	10 HEAD 1 CAVITY
GLASS	AMBER	FLINT
DIP IN	-1.0	-2.0
DIP OUT	-0.5	-0.8
CUTOFF	0	0
KNIFE DROP	2.8	6.2
BLANK MOLD OPEN	4.0	7.0
BLOW MOLD CLOSE	8.0	14.0
BLOW AIR ON	9.0	16.0
BLOW AIR OFF	13.5	23.0
NECKRINGS OPEN	13.0	22.0
DELIVERY OF BOTTLE	21.4	36.0

TYPICAL SPEEDS PER MINUTE

OUNCES WEIGHT	PER CAVITY	PER MACHINE 10 HEADS	PER MACHINE 15 HEADS
O-1	7	140*	210**
4	4 3/4	95*	140**
8	3 1/2	70*	105**
12	3	60*	90**
20	2 2/5	24***	36****
40	2	20***	30****

ALL SPEEDS FROM PRESTONS PAPER.

* 20 CAVITIES ** 30 CAVITIES
*** 10 CAVITIES **** 15 CAVITIES



Courtesy - Owens-Illinois

Figure 2
Owens Machine Cycle

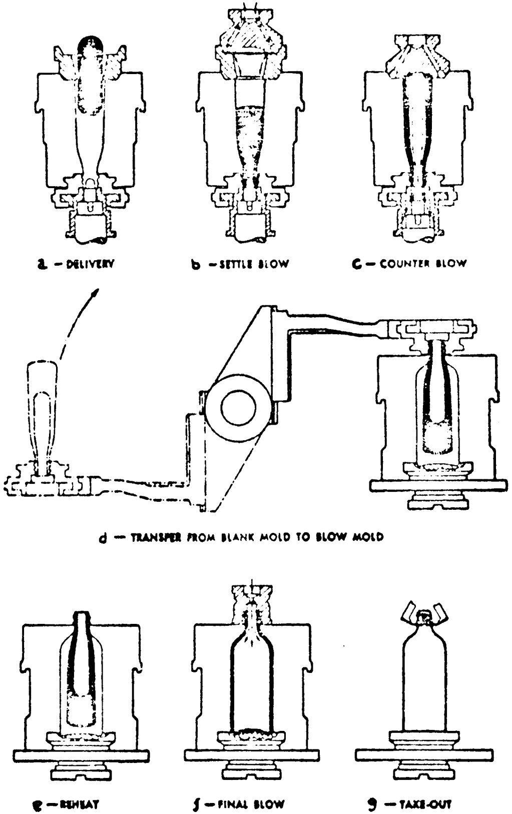


Figure 3
Narrow Neck Blow and Blow Process

CHARACTERISTICS

USES GOB FEEDER.
NON-ROTATING, SECTIONAL.
MOLDS ONLY OPEN AND CLOSE.
AIR ACTUATED.
INDEPENDENT TIMING, SECTIONS MAY BE IDLED INDEPENDENTLY.
VERY FLEXIBLE UNIT.
NARROW NECK ILLUSTRATED.
WIDE MOUTH P AND B ADAPTATION.
HIGH MOLD OVERLAP.
HIGH OUTPUT PER CAVITY.
INTERNAL COOLING BLOWHEADS.
FIRE FINISHING TAKEOUT.
DOUBLE CAVITY, SMALL JOBS.
1, 2, 4, 5, 8, 10 SECTIONS.
UP TO 1 GALLON SIZE.

TYPICAL SPEEDS PER MINUTE - 4 SECTIONS

OUNCES WEIGHT	PER CAVITY	PER MACHINE
O-1	16	63
5	10	41
10	9	36
20	7	29
30	5	19
40	3 1/2	14
50	2 3/4	11
DOUBLE GOB		
O-1	13	106
5	9	74
10	5	42

WARE SPECIFICATION LIMITS

	SINGLE	DOUBLE
MAX. HEIGHT UNDER FINISH	13 3/4"	8 7/8"
MAX. BODY DIAMETER	7"	3"
MAX. FINISH SIZE (MM)	89	53

Courtesy - Owens-Illinois

bottlemaking, which use two fundamentally different means of providing glass for the bottle: suction and overhead flow feeding.

Ribbon Machine

No comments about mechanical glassworking would be complete without mention of the Corning 399 bulb machine. In this device a stream of hot glass flowing without interruption from the orifice of a feeder passes between two rolls. One of these rolls carries a series of depressions and the other is smooth. The result is a ribbon of glass about three inches wide carrying a series of thin bosses closely spaced along its upper surface. The ribbon bends to the horizontal direction and is carried on a sort of caterpillar conveyor beneath a series of traveling blowheads, which register with the bosses on the ribbon. The glass is blown in a series of small hollow parisons down through the lower conveyor where presently a series of paste-lined molds close around the parisons, turning the while. A little further on these molds release the bulbs, which are still held to the traveling ribbon. Then a light blow with a hammer device and a puff of compressed air knocks the bulb off the ribbon, which passes on as cullet to be remelted. This machine operates at an unbelievably rapid rate. One thousand bulbs and more, depending on the size, can be blown per minute.

Most recently, modifications of this machine have been used to form a lightweight glass envelope shaped like a teardrop with a flat bottom that can and is modified into a container. This shape can obviously be made in various sizes.

Plasti-Shield

A new container, called Plasti-Shield has been developed by Owens-Illinois. The new container is a lightweight glass envelope shaped like a teardrop with a flat bottom, wrapped in a resilient sleeve of pre-decorated polystyrene that provides up to four times more space for decoration than present bottle labels. The lightweight glass portion of the new composite package is made on conventional bottle forming machines while the plastic shield is applied by equipment especially designed for the purpose. Efforts are also being directed to make the glass portion on modified ribbon machines at high rates of production.

The containers offer a number of benefits for the consumer: The insulating plastic jacket keeps the beverage cold longer. The foam jacket doesn't sweat, hence there is less chance to marring furniture. The jacket is pre-decorated so further labeling is not necessary. The container is easy to ship. The lighter weight makes significant savings of raw material resources. The container can be recycled in present glass furnaces. The bottle's shape gives it improved pressure retention.

Production Trends

The glass container industry has maintained a rather remarkable constant growth in recent years. The rate of increase has amounted to about 6 percent annually. There have been some years of variation. In 1974 production amounted to about 280.4 million gross (40.4 billion units). Domestic shipments were approximately 273.7 million gross (39.4 billion units) representing a value well in excess of $2 billion.

A profile of production and growth is shown in the following curves and data taken from the Glass Containers — 1972 Edition and "Current Industrial Reports" — U. S. Department of Commerce.

Flat and Structural Glass

The earliest method of making flat glass was by hand blowing. A sufficiently large gob of glass was taken up on the blowpipe and air was forced into it to form a roughly spherical globe of 50 or more centimeters in diameter. This globe was then reheated and attached to a pontil, removed from the blowing iron, and marvered so that it resembled a flat bottom flask not unlike a modern television picture tube. A circular hole a few centimeters wide was then cut from the flattened base and the glass thoroughly reheated and softened at the glory hole. It was then removed from the heat and the pontil twirled, causing the glass to spin rapidly. Suddenly, under the centrifugal stress created by rapid rotation, the open end of the globe would flare out and the soft glass took the form of a disk. Rotation had to be maintained until the cooling glass was sufficiently rigid to retain its form; but it was never really flat, and its two surfaces were usually far from parallel.

Plate Glass

The term "flat glass" includes sheets, plates and laminae of all sorts produced by a number of different methods. Flat glass formed by casting and rolling is known as plate glass, which may be rough, ribbed, figured, sand-blasted, etched, or otherwise decorated; or in its typical form, ground and polished on one or both sides. A second general division of flat glass called window glass, includes sheets formed by flattening blown cylinders, or by drawing continuous sheets from a bath of molten glass.

Rolled Sheets

The table-casting process of making rolled sheets is carried out by removing a pot from the furnace and pouring the glass upon an iron casting table. A heavy roller, moved by chains, passes over the glass and spreads it into a sheet whose thickness is gauged by guides at the ends of the roller.

In the Bicheroux process, the glass is poured from the pot upon a set of power-driven rolls which deliver a sheet of less thickness and of greater length.

The continuous-pour process, employed for quantity production of windows for automobiles, enjoys the economies of tank melting and continuous operation. The glass flows in a shallow stream several feet in width over a "lip" at the end of the tank, passing between two rolls which form the sheet. A continuous lehr several hundred feet in length anneals the plate.

The continuous pour process made a major part of the plate glass. The plate had a width up to ten feet and it moved as a continuous ribbon

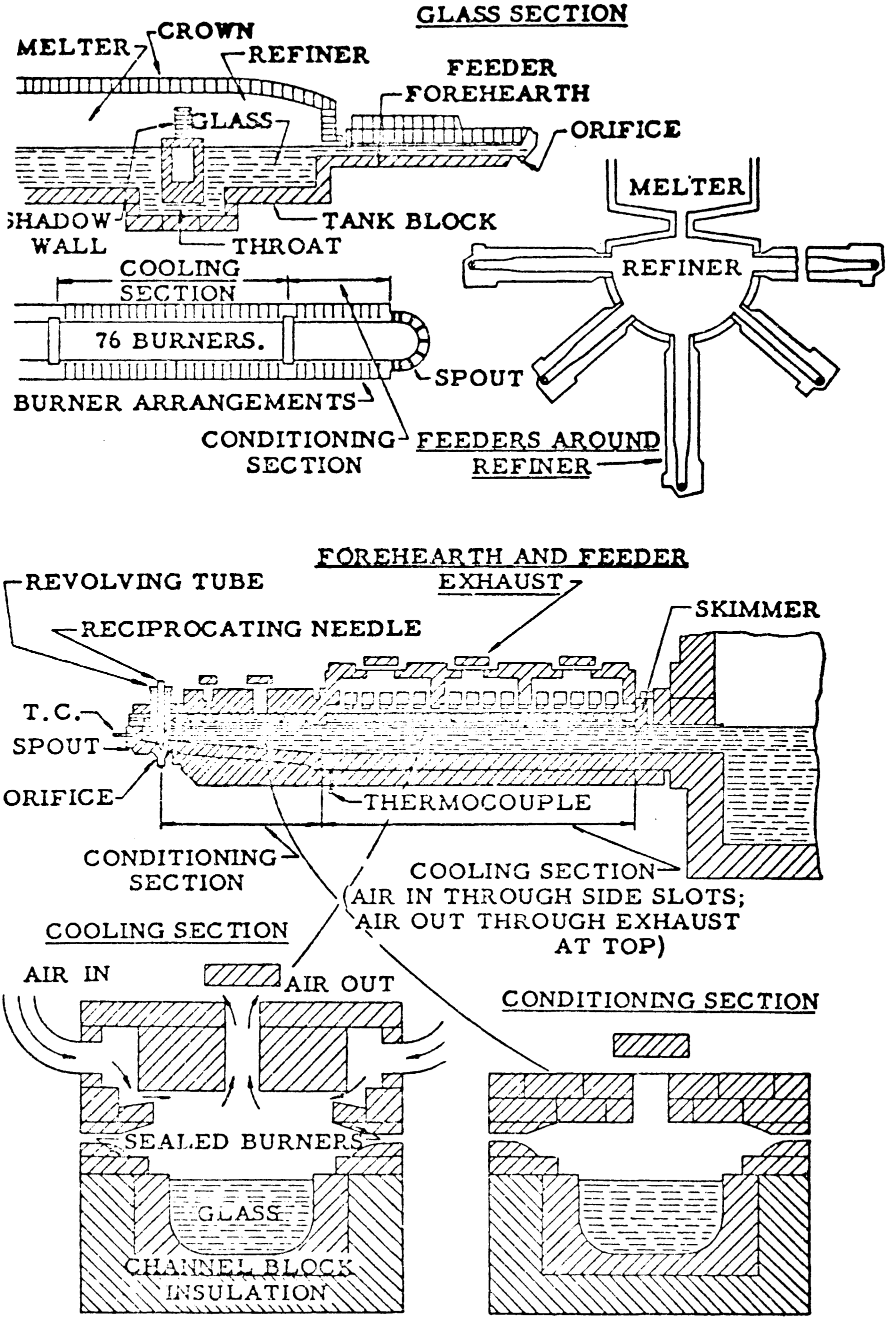
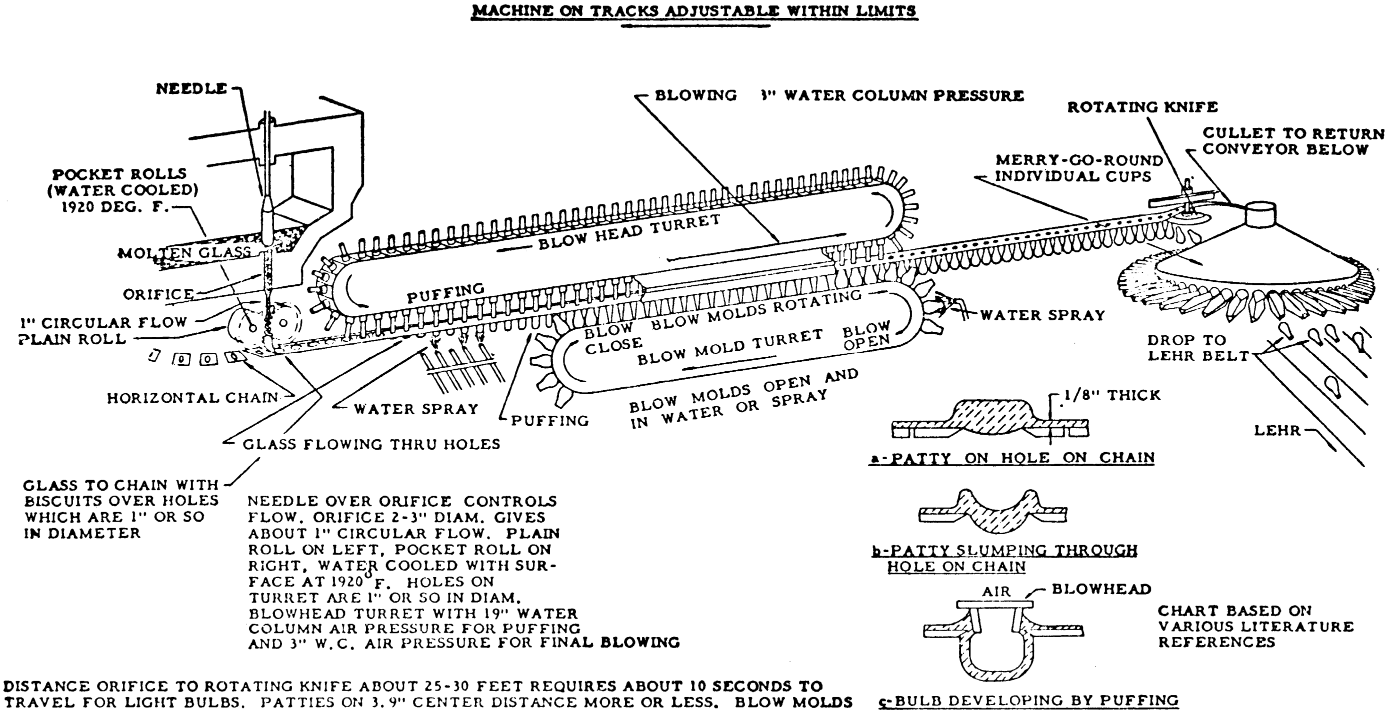


Figure 4
Flow or Gob Feeder Glass Conditioning

Courtesy - Owens-Illinois



NEEDLE OVER ORIFICE CONTROLS FLOW. ORIFICE 2-3" DIAM. GIVES ABOUT 1" CIRCULAR FLOW. PLAIN ROLL ON LEFT, POCKET ROLL ON RIGHT, WATER COOLED WITH SURFACE AT 1920° F. HOLES ON TURRET ARE 1" OR SO IN DIAM. BLOWHEAD TURRET WITH 19" WATER COLUMN AIR PRESSURE FOR PUFFING AND 3" W.C. AIR PRESSURE FOR FINAL **BLOWING**

CHART BASED ON VARIOUS LITERATURE REFERENCES

DISTANCE ORIFICE TO ROTATING KNIFE ABOUT 25-30 FEET REQUIRES ABOUT 10 SECONDS TO TRAVEL FOR LIGHT BULBS. PATTIES ON 3.9" CENTER DISTANCE MORE OR LESS. BLOW MOLDS ROTATE ABOUT 150-200 R.P.M. AND ARE PASTE LINED. SPEEDS OF 600-800 PER MINUTE FOR LIGHT BULBS, AND 160-190 PER MINUTE FOR TUMBLERS. THESE ESTIMATE AT 3-10 PER CAVITY PER MINUTE. PRODUCTIVITY 400,000 - 1,000,000 BULBS, AND 150,000 - 300,000 TUMBLERS PER DAY ARE INDICATED. ORIGINAL DESIGN ABOUT 1924-25. TWELVE MACHINES IN USE IN 1945 BY GENERAL ELECTRIC AND CORNING.

Courtesy - Corning Glass

Figure 5
Corning's Bulb Machine

through the annealing lehr at a speed of several feet per minute. Small quantities of plate glass, particularly colored plate and decorated plate are made by pot-melting and rolling on a table, or by the Bicheroux or by the ring-roll process.

GRINDING AND POLISHING

After being annealed, the rolled plates are inspected and the better pieces are set on tables in plaster of Paris that on setting holds the glass secure. A continuous process is devised in which the table handling the glass is mounted on a car and travels on rails passing under grinding heads and finally felt polishing heads to complete the polish operation. The grinding operation is aided by water and an abrasive as sand or garnet while the polishing operation is aided by iron oxide or rouge.

FLOAT GLASS PROCESS

The basic requirement for producing a flat, fire-finished distortion free glass is by some means to prevent contact between anything solid and the glass in the molten or plastic state, and at the same time avoiding mechanical stress on the glass. Pilkington were the first to apply the idea of floating the molten glass on the smooth surface of a molten metal at a strictly controlled temperature, and in an inert atmosphere to prevent oxidation of the metal. Tin was found to be the most suitable metal for the purpose and in 1959 the first glass was produced commercially by the process.

The molten glass leaves the furnace in a continuous strip and floats directly onto the surface of an enclosed bath of molten tin. During this crucial stage, the glass is enveloped in a controlled atmosphere and heated sufficiently so that irregularities on both sides of the glass ribbon become flat and parallel. As the continuous ribbon continues along the length of the bath it is gradually cooled sufficiently to avoid damage to the surface of the glass when it leaves the float bath chamber and moves into the annealing lehr.

The float process has grown to be the favored process of manufacture and now represents about 90 percent of flat glass production.

DRAWN GLASS

Drawn glass or window glass is flat glass formed into sheets with a natural fire-polish, without grinding and polishing. It is not made by rolling. It is generally thinner than plate glass but may be made quite thick. Optically, it is inferior to polished plate because its surfaces do not approach to a geometrically plane surface. Methods for making window glass started with various hand-blown operations and then progressed after much experimenting to machine drawn operations. There are several machine operations for drawing glass such as the Libbey-Owens-Colburn, Fourcault and Pittsburgh Process. I shall briefly describe the Pittsburgh Process.

The batch ingredients are fed into the melting furnace at one end at a rate controlled by withdrawal from the drawing kiln. The drawn sheet of glass is somewhat initially controlled by a submerged fireclay member tapered to a narrow edge near the surface of the glass. The position of the clay member produces a narrow zone of colder glass representing the highest viscosity and, therefore, highest tenacity of any of the surface glass and, therefore, it definitely controls the location of the sheet. There is also employed a pair of small wheels, called knurled rolls, at both edges of the sheet. The rolls exert a continuous pull outward and thus assist in maintaining the width of the sheet. In this way they help to overcome the greatest problem faced in the development of the method. One may clearly understand that the glass would normally have a tendency to pull itself together so that the sheet, after rising a short distance, would narrow to a thread.

TRENDS, PRODUCTION AND USE OF FLAT GLASS

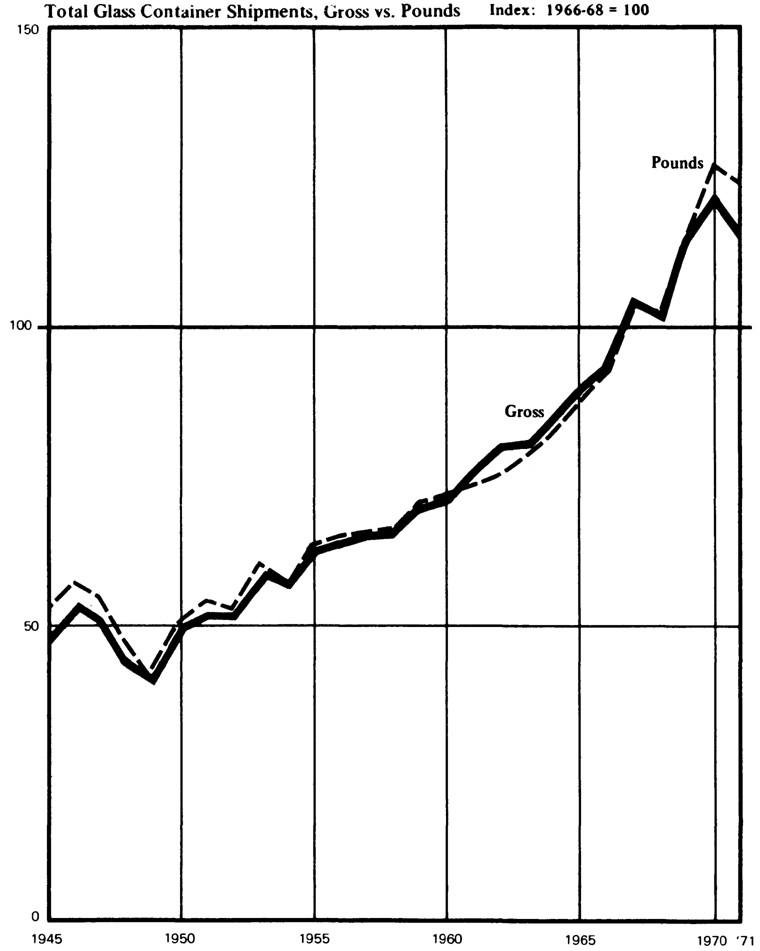
The versatility and useful application of glass has been enhanced by changes in compositions, small additions of colorants to control light and infrared transmission, use of coatings (metallic) double glazing, tempered glass to improve strength, laminated safety glass for safety and opaque glass cladding. Glass in almost every imaginable form — plate and sheet glass, wired, corrugated and patterned glass, tempered glass (thermally and chemically treated), translucent glass, blocks and various forms of decorative glass used with concrete and architectural metals have greatly enhanced architectural uses.

Modern buildings are practically entirely enclosed and encased in glass. By use of double glazing, heat absorbing, heat reflecting, glare reducing glass, the problem of climate control and personal comfort becomes pleasantly simple.

Moreover, increasing concern for safety in the home and other buildings has spurred market growth for safety glass. The stronger glass is used for such hazardous locations as storm doors, sliding patios as well as shower and tub enclosures.

The energy shortage has created a sharp rise in demand for insulation, double glazing, reflective glass and mirror-like transparent coatings to control the transmission of sun's heat and light. The practical use of solar energy to both heat and cool buildings demands the use of glass. Furthermore, the flat glass is becoming more involved with replacement work, renovations, repairs and restoring of the old to the new. These trends have brought a renewed interest in distribution and has encouraged tempering centers which provides a wider availability of the various products.

An analysis of production and shipment data taken from the U. S. Department of Commerce indicates the present trends of the industry.



	Thousands of Gross	Millions of Pounds		Thousands of Gross	Millions of Pounds		Thousands of Gross	Millions of Pounds
1971	254,975	22,208.3	1962	174,195	13,430.4	1953	127,516	10,558.3
1970	266,610	22,581.9	1961	165,656	13,252.5	1952	114,102	9,527.5
1969	253,193	20,787.1	1960	156,799	12,889.0	1951	114,738	9,626.5
1968	223,635	18,271.0	1959	153,102	12,707.5	1950	107,897	8,988.0
1967	231,046	18,506.8	1958	143,366	11,741.7	1949	89,827	7,581.4
1966	206,299	16,545.2	1957	143,467	11,678.2	1948	97,333	8,409.6
1965	198,131	15,751.4	1956	140,890	11,548.7	1947	110,170	9,816.1
1964	186,741	14,752.5	1955	137,278	11,256.8	1946	116,084	10,227.0
1963	177,886	13,928.5	1954	124,649	10,171.4	1945	106,318	9,483.6

Sources: U.S. Department of Commerce and GCMI

Figure 6
Total Glass Container Shipments, Gross vs. Pounds

PRODUCTION AND SHIPMENTS OF FLAT GLASS* — 1974

Sheet (Window) Glass (Not Rolled) Including Colored

	Production M Boxes	Shipments M Boxes	Value of Shipments ($1,000)
1st Quarter	6,705	5,573	40,524
2nd Quarter	5,460	4,981	35,806
3rd Quarter	4,420	4,297	31,842
4th Quarter	3,200	3,192	24,369
Total 1974	19,785	18,043	132,541

Plate Glass, Float Glass, Rolled and Wired Glass

	Production (M Sq. Ft.)	Shipments (M Sq. Ft.)	Value of Shipments ($1,000)
1st Quarter	453,053	333,908	88,250
2nd Quarter	428,099	381,747	103,513
3rd Quarter	513,358	359,825	113,648
4th Quarter	429,604	320,118	105,430
Total 1974	1,324,114	1,395,598	410,841

**Source: "Current Industrial Reports"—U. S. Dept. of Commerce*

The above tables show that in 1974 approximately 1.3 billion square feet of plate and float glass and 19.8 million boxes of window glass were produced. In comparison with 1973 (1.5 billion square feet), there has been a drop of about 13 percent in plate glass production, while window glass (1973—23.7 million boxes) dropped about 16.4 percent. The total value of shipments for the entire industry amounted to about $543 million. Shipments of sheet glass accounted for about 25 percent of the total and plate (largely float) accounted for about 75 percent.

The following companies (listed alphabetically) are major producers of flat glass and participate in varying degrees in the various achievements:

ASG Industries, Inc.
CE
Ford Motor Company
Guardian Industries, Inc.
LOF — Libbey-Owens Ford Company
PPG Industries, Inc.

Technical Glass

Technical glass over the years has had a profound influence over both science and industry. The wide range of products include laboratory ware, electronics, radio, television, communications, food preparation, illumination, industrial systems, information processing, health and medicine, science, transportation, optical glass as well as optical systems. Because less energy is required to produce a pound of glass than a pound of other basic materials, it would appear that glass is likely to continue as a material source for many products.

The industry has grown from a modest beginning to over a billion dollar annual sales.

Some very important developments, together with date of inception will show a history of development (courtesy of Corning Glass Works).

Heat Resistant Glass—Developed in 1912 the first glass ever made capable of withstanding sudden jolts of heat or cold. These borosilicate glasses (so-called because of the large amount of borax used in the formula) were used to make railway signal lanterns, later became well known to millions of housewives as Pyrex brand cooking and baking ware. Today borosilicate glass is used in a wide range of products, including laboratory glassware, signal lenses, boiler gauge glasses. The largest piece of glass in the world—a 200 inch, 20-ton disk produced by Corning as a mirror blank for the Hale telescope—is made of borosilicate glass.

Ribbon Machine—Introduced in 1926, the ribbon machine is one of the most fabulous glassmaking machines ever invented. It turns out incandescent lamp bulb blanks, radio tube blanks, and photoflash bulbs at speeds up to 2,000 per minute. It gets its name from a thin ribbon of glass, several inches wide, that runs continuously across a constantly revolving set of molds. Development of the ribbon machine helped reduce the cost of the household light bulb.

Silicones—These compounds, which might be called a cross between glass and plastics, were first developed in the Corning laboratories in the early 1930's. In 1943 Corning combined its technical know-how with the manufacturing facilities of the Dow Chemical Co. to form the Dow Corning Corp. to produce silicones. Silicones (which can be fluids, greases, putties, or rubbery materials) are used to improve a wide variety of products such as paints, gaskets, oils, waterproofing compounds, and many others.

Electrical Sealing—Prior to this development in 1938, most glass was sealed to other glass by heating the ends in a gas flame and then joining them—a method still used widely for a number of products. A new technique was developed using electricity. By striking an arc through both pieces of glass, current flows through the glass, thus raising its temperature and causing it to fuse. This new technique made possible a smoother, stronger, faster seal. Used in making television bulb blanks, the process gives perfect seals where other types of sealing had proved undependable. A technique to seal radar bulbs and glass piping was also used.

96% Silica Glass—In 1939 a method was found for removing practically all the constituents from a borosilicate glass—except the silica. This resulted in a glass that is nearly pure silica. The discovery was significant because it produced a glass so super heat-resistant that a piece can be heated to 900°C and then plunged into ice water without damage. Products were made from these glasses under the Vycor trademark. Because of their high heat resistance, 96% silica glasses are used for certain types of laboratory glassware, asbestos suit windows, welding torch tips, thermocouple protecting tips. Other properties make it ideal for sunlamps, aircraft camera windows, germicidal lamps, manned spacecraft viewports, and other products.

Optical Glassmaking—From 1790, when optical glass was developed, until the early 1940's, all optical glass was produced by the same wasteful and laborious method. A "pot" of glass was melted, cooled completely, then broken into chunks. The best pieces were then reheated to obtain the optical products. Thus much work produced little glass. In 1942 a completely new process was developed for making optical glass—the continuous melt method—making possible for the first time mass production of ultra high quality optical glass. At the heart of this development was a new kind of furnace which used electricity as well as gas, and new melting methods.

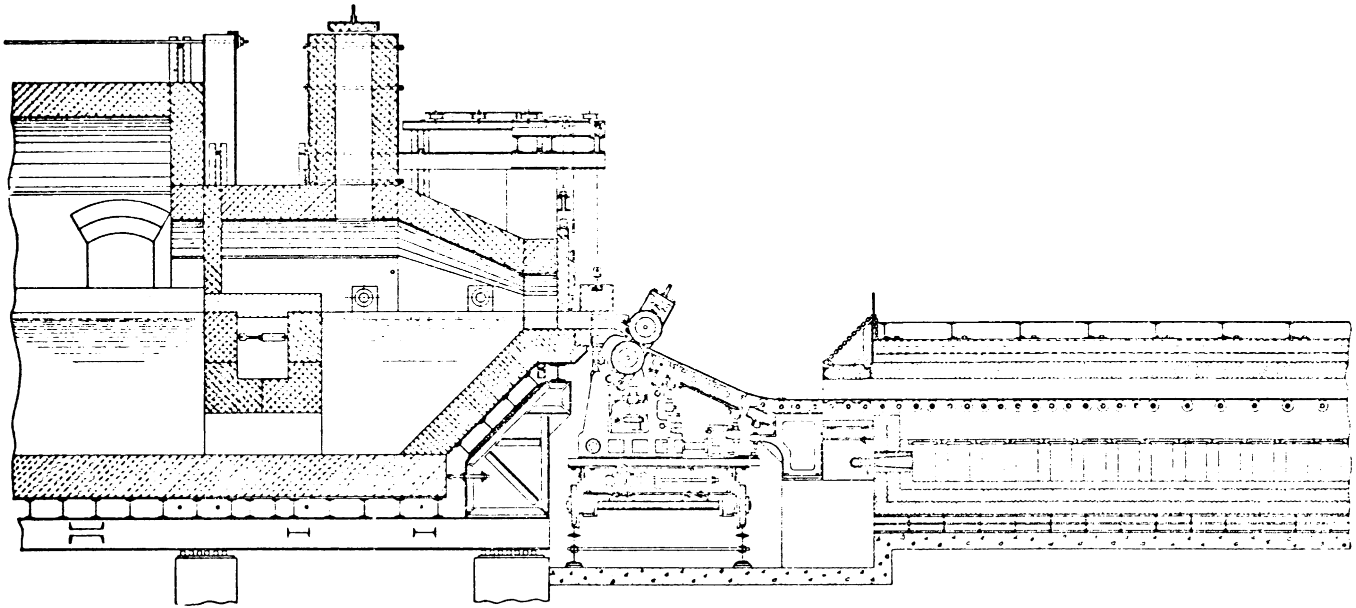
Thermometer Tubing—In the early 1940's a new method for continuously drawing thermometer tubing was developed. This speeded production and improved dimensional accuracy. Prior to this development, all thermometer tubing was produced in short runs by hand or semi-automatic methods.

Ribbon Glass—In 1943 a method was developed for drawing glass an inch wide and extremely thin—about as thin as newsprint. This process, together with special glass compositions, made the new "ribbon glass" ideal for use in electronic components such as capacitors. In this application the ribbon glass replaced mica, thus freeing the nation from its dependence on mica from India. Capacitors made with ribbon glass have set outstanding performance and reliability records and are widely used today in the nation's missile and space programs.

Centrifugal Casting—In the late 1940's a revolutionary new way to form glass—by centrifugal casting was used. In this method, a gob of molten glass is dropped into a mold, the mold spins, and the glass is thrown up the sides of the mold. This process is used to make the funnels of television bulb blanks.

Photosensitive Glasses—Developed in 1947, this family of glasses behaves much like photographic paper when exposed to ultraviolet light and developed by heat treatment. Thus, an image can be permanently formed through the entire thickness of the glass. The exposed areas will dissolve rapidly in acid, making it possible to produce highly intricate patterns in a piece of glass. Products made from photosensitive glass are trademarked Fotoform. Some of these glasses can be converted by further heat treatment into a crystalline ceramic material. In this state, the material is mechanically harder and stronger. Products made from this ceramic material are trademarked Fotoceram. Photosensitive materials have been used to make printed circuit boards, fine glass screens, dielectric spacers in electronic tubes, fluid amplifier components.

Electrically Conducting Coated Glass—By developing in 1950 a technique to coat glass with a metallic oxide film that conducts electricity, Corning reversed the classic role of glass as an insulator. The metallic film (dimensionally accurate to a few millionths of an inch), is fired into



Courtesy - Amsler-Morton

Figure 7
Continuous Rolled Glass Process

the glass, becoming an integral part of it. From this glass is made tin oxide resistors and heater panels for industrial drying, baking and curing.

Fused Silica—Although fused silica has been made for years, in 1952 a new manufacturing process was found which produces the purest fused silica ever made. Compared with other glasses, it has the highest softening point, the highest electrical resistivity, the highest resistance to thermal shock, and is the best transmitter of sound. Fused silica is used to make telescope mirror blanks, ultrasonic delay lines for radar ground systems, and other products.

Glass-Ceramics—Developed in 1957 and marketed under the trademark of Pyroceram, this family of materials results from a conversion process during manufacturing. The raw material is melted as a glass, then converted by special processing to a ceramic. The result is a super-strength glass-ceramic which has greater strength and hardness than the parent glass, greater abrasion resistance, improved electrical properties, and remarkable thermal characteristics. A line of cooking ware made of one Pyroceram brand glass-ceramic was introduced in 1958 under the Corning Ware trademark and has received outstanding acceptance from the American housewife. Other glass-ceramics are used to manufacture missile radomes for the government.

Cellular Ceramic Structures—Two thin-walled cellular ceramic structures are manufactured. A glass-ceramic material, trademarked Cercor, is used for gaseous heat exchangers in automotive gas turbine engines and industrial heat recovery systems. A cordierite ceramic material, trademarked Celcor, is used as the catalyst support in automotive catalytic converter systems designed to reduce harmful exhaust emissions. Both materials exhibit good resistance to high temperatures and thermal shock and excellent chemical durability.

Chemically Strengthened Glass—In 1962 Corning announced the development of methods (called the Chemcor processes) for strengthening glass chemically. By using these processes, glasses many times stronger than ever before possible were made—glasses so strong they could be bent or twisted. Centura tableware was the first commercial product to make use of these processes. Other chemically strengthened products include super-strong pipets and other laboratory ware, and aircraft windows of many kinds.

Photochromic Glass—In 1964 developments were announced of a series of special glass compositions which darken when exposed to light and clear when the light source is removed. Called photochromic glass, these light-sensitive materials are capable of cycling endlessly from clear to dark to clear again without wearing out. Their first commercial use was in lens blanks for prescription eyeglasses.

Machinable Glass-Ceramics—Announced in 1971, this is a new family of glas-ceramics that can be machined to precise tolerances with conventional metalworking tools. The materials have an insensitivity to surface damage and a high resistance to fracture, resulting from their unique microstructure. They also exhibit a broad range of outstanding dielectric and mechanical properties that lend themselves to potential applications in the electrical, electronics, metals-producing and aerospace industries.

Optical Waveguides—With today's urban telephone systems near the limit of their information-carrying capacity, scientists are working on new communications techniques that utilize beams of light transmitted through optical quality glass cables called waveguides. The technology offers the ability to compact the message-carrying capacity of 10,000 telephone wires into a single optical waveguide strand roughly one-fifth the diameter of a human hair. Researchers have long felt that a wave-guide material capable of keeping light signal losses below 20 decibels per kilometer would make establishment of such optical communications links feasible. Corning, recognized as a leader in optical waveguide technology, has fabricated long lengths of waveguide with measured losses of less than 4 dB/km at the light wavelengths suitable for communications use. Today, optical waveguide development is at the force of optical communications links.

Immobilized Enzymes—A range of processes were developed for permanently bonding active catalytic materials such as enzymes to inorganic substrates or carriers. The work was begun using Corning's controlled-pore glass (CPG) materials, but has since seen a range of inorganic materials such as ceramics, plastics and metals used as the carrier or substrate. The ability to permanently attach active catalytic materials to inorganic—and insoluble—carriers greatly extends the economies of such techniques for producing processes and for research by making the active materials reusable. In production situations, the immobilized enzymes can be packed in columns in continuous flow processes. Applications are foreseen in producing sweeteners, in biomedical research, in chemical processing and in pollution control.

FIBER GLASS

Glass fibers have been known for centuries as the Venetians used finely drawn glass in the 13th century for complicated decorations. It remained, however, for Owens-Corning Fiberglas starting in the early 1930's to bring the industry to its present position and important industrial status.

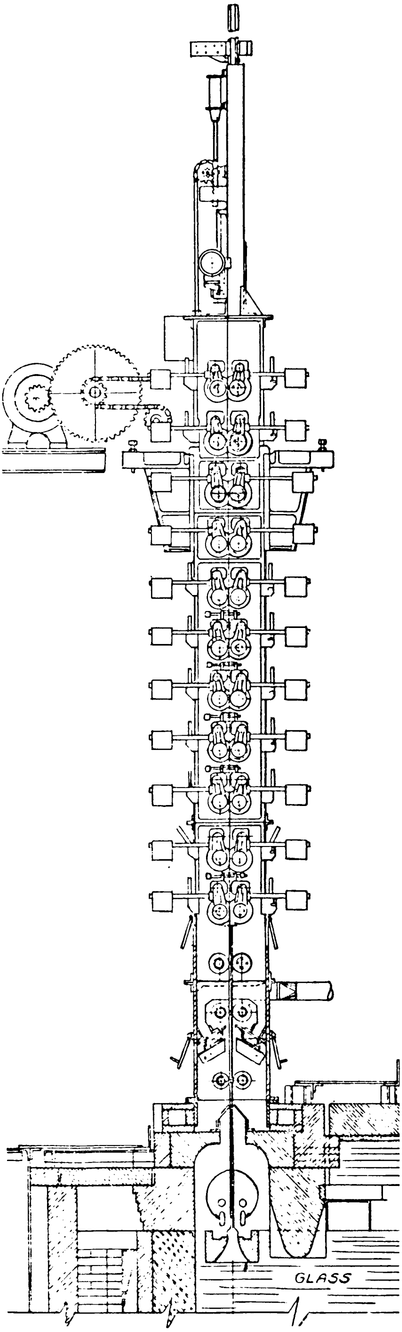
Modern glass fibers may be made by several different processes but two principal methods stand out as being the more important, namely, glass wool and continuous filament.

WOOL PROCESS

A process somewhat similar to that used in making mineral wool may be employed, wherein molten glass issuing from an orifice is blown by high pressure steam and attenuated into fibers and collected on a large belt as a blanket or mat.

CROWN PROCESS

A more modern process known as the crown process has been accepted. It was developed principally in France and the United States. A



Courtesy - Amsler-Morton

Figure 8
Machine Drawn Glass Operation

rather heavy stream of molten glass is permitted to fall from the forehearth of a fairly large glass furnace onto a rapidly rotating steel alloy dish that has many hundreds of tiny vents around its periphery. As the result of this centrifugal force glass is thrown out through these apertures forming fibers that are first controlled by blowing rings and, after spraying with a binder, are given further air treatment to insure random fiber distribution in the resulting mat. A conveyor belt then conveys the fibers to curing ovens and then to cutters and shapers that form he final product into either flexible mat or rigid board. Very fine textures are possible with individual fibers of a diameter of approximately 0.0007 cm. Lubricants are sprayed on the fibers to avoid breakdown or injury by friction. The final density of the mats may vary from 80 to 130 kg/m^3.

CONTINUOUS FILAMENT PROCESS

The objective of the continuous fiber process is to make a continuous strand made up of about 400 separate glass filaments. Actually two different methods are in use today. Continuous fibers may be drawn from orifices of refractory or platinum melting units and wound up on a drum. Molten glass is first prepared and carefully refined in a refractory tank after which the molten glass passes from the furnace through the forehearth to a series of platinum bushings containing numerous accurately dimensioned forming tips. Extremely fine filaments of glass are pulled down on rollers from the platinum orifices at a high speed, resulting in fibers of various diameters, some of which may be as fine as 1/300th thickness of the human hair. The filaments are then collected, sizing is applied to keep them together, and they are combined into a single strand on a high-speed winder.

A second and older method of drawing filaments directly is to provide already melted glass in the form of frit or preferably in the form of marbles into an electrically heated bushing. The filaments are then drawn similar to the process described above. The glass is fed into the bushing in the form of marbles largely for convenience and ease of controlling the weight of addition. This method of addition also minimizes the danger of uneven viscosity due to the introduction of cooler raw material to the melt.

In fiber form the many desirable properties of glass are retained. Fibers of glass are incombustible, moisture resistant, durable and dimensionally stable. In addition, glass in fiber form can be flexible, resilient, and have excellent thermal and acoustical properties.

The basic glass fibers are fabricated into various forms, depending on the properties desired in the end product. It is highly important to use glass composition of extremely high weather resistance since the great amount of surface exposed per unit weight renders the glass sensitive to the corrosive influence of even such moisture as present in air. The forming process is so precise that the raw materials must be extremely uniform and the use of cullet or premelted batch in the form of marbles is desirable. To accomplish satisfactory results most fiber glass compositions are of the borosilicate type which means rather high in Al_2O_3, CaO and B_2O_3.

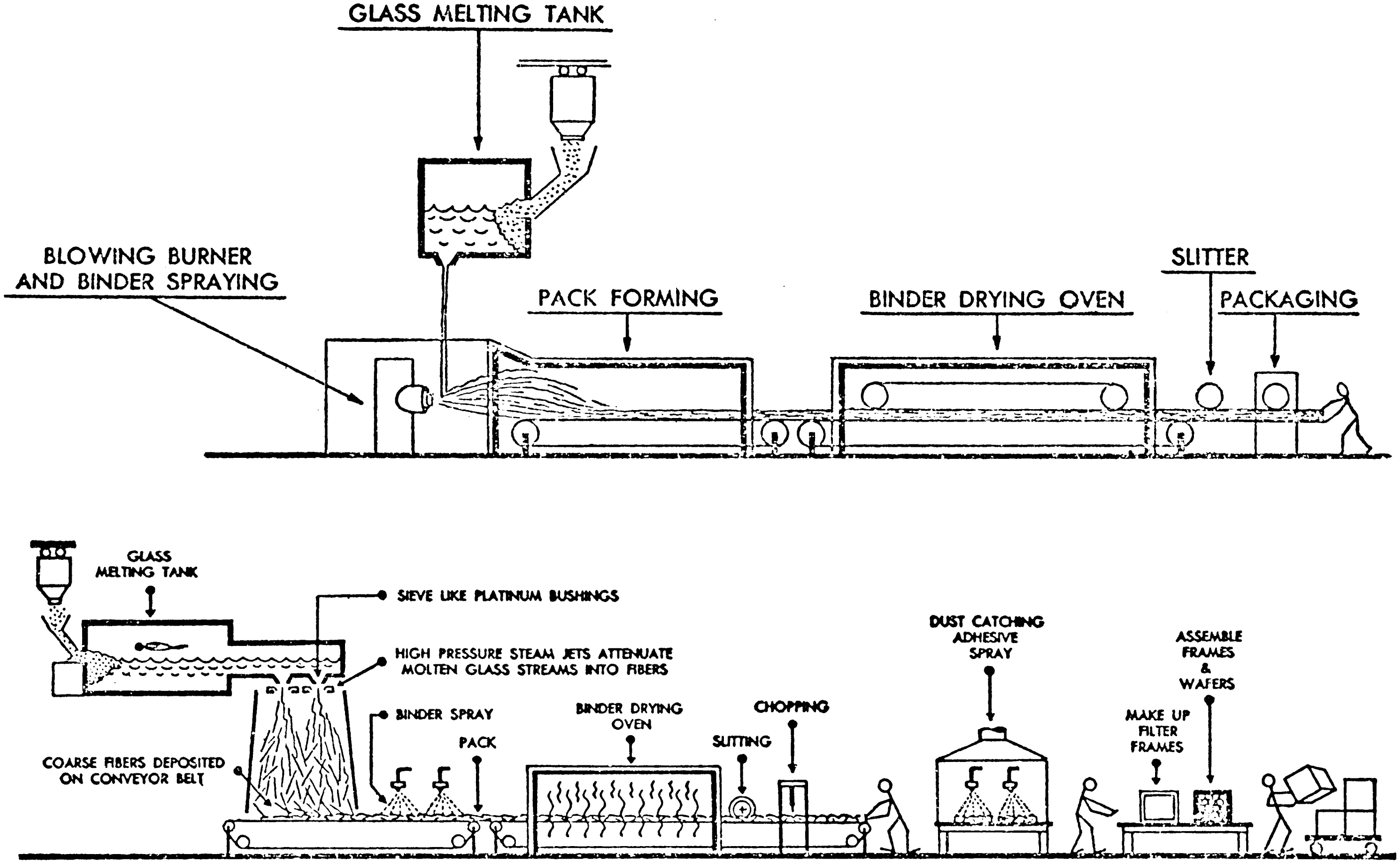


Figure 9
Process for Manufacturing Wool and Air Filters

Courtesy - Corning-Owens Fiberglas

In wool form, glass fibers are usually compressed and preformed to various densities producing products with outstanding thermal and acoustical insulating properties. Thin mats are used for air, gas, and liquid filtration. Considerable amounts of fibers are used for reinforcement in plastics, paper and rubber and more recently concrete where they supply unique and unusual properties to such products. Textile products form still another line of useful products where their high tensile strength and durability becomes a desirable factor.

Traditionally, glass fiber use falls into the following categories: construction, which is largely wool, fiber board, insulation and the like. Industrial, which encompasses reinforced products for industry, automobiles, boat hulls, pipes, tanks, etc. Textile, which involves woven cloth, fabrics, draperies and materials suitable for weaving. Special products, such as optical fibers and other specialized applications.

OPTICAL FIBERS (WAVE GUIDES)

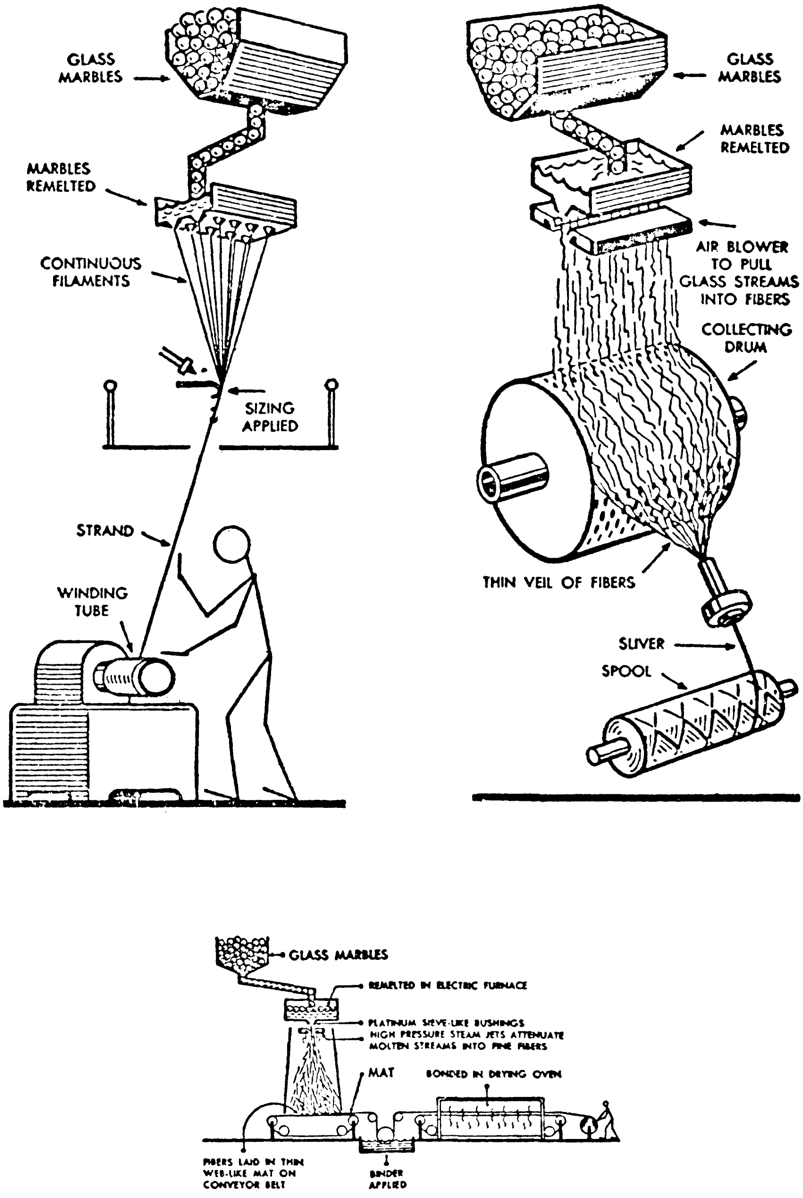
Recent work that has brought about greatly enhanced transparency of optical fibers has made their use in communications more encouraging. Glass optical wave guides with attenuations as low as 2dB/km have made possible a new approach to optical communications.

The quantity of information that can be carried by a transmission channel, for example, a pair of telephone wires or a radio beam, depends upon the frequency of the signal; the higher the frequency, the greater the amount of information. Since the frequency of light is roughly 1,000 times the frequency of the shortest radio waves, communication engineers have always dreamed of using light as a means of transmitting information. Hence, today the leading candidate for that function is the optical fiber; a hair-thin glassy filament of extremely high transparency.

Material dispersion does not present too many problems providing high purity is maintained. Impurity absorption arises predominantly from transition-metal ions such as iron, cobalt, chromium, nickel and the like. Still another important impurity is "water" which is present as OH^- ions. Scattering of light may also be caused by non-uniformity of the fiber, particularly in the interface between core and cladding and by particles or bubbles in the core material. One of the most transparent of all materials is fused silica and this material seems to be destined as the ideal material for wave guide use.

It should be pointed out that the feature which makes optical fibers most attractive for communications is their small size. One hundred individual, signal-carrying fibers bundled together might occupy a space that has the diameter of the lead in an ordinary wooden pencil. Each fiber might carry 1,000 telephone conversations, or several television programs at a time.

Optical fiber wave guide development has been a remarkable scientific break-through and appears to be on the threshold of fantastic applications. Bell Laboratories and Corning Glass have been responsible for the initial work to successively implement the program and their future joint efforts will no doubt carry it to still greater achievements.



Courtesy - Owens-Corning Fiberglas

Figure 10
Process for Manufacturing Fiber Yarns and Bonded Mat

CONTRIBUTION OF AMERICAN SCIENTIFIC GLASSBLOWERS AND FUTURE OUTLOOK

The American Scientific Glassblowers Society can make one unique and assuring claim and that is that they have been the motivating force and precursor to machines and mechanization that has now engulfed the entire industry. For example, glassblowing has aided the old Owens machine, the I-S machine, press and blow machine, ribbon machine and various operations formerly done first by hand to indicate and reveal the various operations that had to be perfected mechanically before precise machine operations were realized.

The same sequence as applied to the old cylinder process (made by hand) set up machine rolling of flat glass, which in turn later led to continuous rolling and drawing processes as well as float glass.

Glass for electronics, tubing, soldering, abrading, and intricate assemblage all became a foundation for logical progress of mechanization.

The inevitable improvement of mechanized effort lead to greater precision, excellent skill and vast increase in production capacity for manufacture.

The American man may not be an innovator, per se, but he can out-produce anyone.

It is strange to note that other facets of ceramics have followed a similar history but not quite to the same degree of perfection.

Medical science would be seriously handicapped without glass. There are hypodermic syringe parts of the glass "boot" that work miracles of healing in arterial disease cases; microscope lenses, germicidal lamps whose ultraviolet rays free the air of germs, and there is a tiny electric glass lamp, no larger than a grain of wheat which glows at the end of a bronchoscope enabling a doctor to see inside the bronchial tube.

Glass windows are constantly being used in greater areas for automobile glazing; glass fenestrations are playing an increasingly important role in the air-conditioned home and office building. The entire problem of heat control in building will demand more glass in the form of flat glass, glass blocks, and fiber glass as insulation.

Industrial television represents a tremendous potential for glass products. Because of its small size, relatively low cost, and extreme flexibility, closed circuit industrial television has limitless applications in industry, science, business, education, and numerous other fields.

Closely allied to television, but having application in all fields of communication, is microwave relay equipment, which, within the next five years, may represent one of the most comprehensive systems of communication in the nation. Because it is more efficient and less costly, the microwave system is rapidly replacing conventional open-wire telephone lines, lead-sheathed cables, and co-axial cables in television, telephone, telegraphy and other fields of communication.

Glass in electronics is closely allied to the demands of television.

Somewhere in the future, and perhaps not too far off, the glass industry will be furnishing vital parts for the manufacture of electronic air conditioning, electronic ignition systems, and other revolutionary devices.

Electronic sound-synthesizing devices have been developed which can, in theory, duplicate any sound, including musical notes and human voices. These devices can synthesize sounds never before heard on earth, literally bringing to man the music of the spheres.

There is an electronic cooling device in which six ounces of ice have been frozen. This is believed to be the first instance of attaining a freezing temperature in a sizeable area without mechanical refrigeration.

In other areas, the electronic light amplifier may lead to the possibility of making vision possible in darkness, adding greatly to the safety of all transportation.

It is important to imagine the future of electronic light. One certainty, though, like other major scientific innovations, is that it will cause the creation of entirely new instruments, appliances, services and industries.

Probably the most challenging of tomorrow's products is the variable transmission window whose opacity to light and radiant heat will be adjusted either manually or automatically.

Electroluminescent panels for lighting the interiors of homes and offices, are already being developed in the laboratory. These panels give off a low intensity of light when an electric field is applied to them, representing an extension of the mercury light concept. A mercury light has much lower intensity than the incandescent lamp, but the increased area results in a higher light value. The most practical base on which to apply this electroluminescent coating is glass.

The type of electroluminescent lighting is being used in the instrument panels of some new cars.

In the future, glass will serve not only as a decorative and durable surface for walls and ceilings, but it will also provide a base for coatings which will produce soft light for room illumination.

Glass covered with electrically conductive coatings has been used successfully as a source of heat. In the future, electrical energy will generally become lower in comparative cost, and this method for the controlled heating of buildings will be increasingly common. Coated glasses will be the most practical medium for supplying longlasting heating elements for completely enclosing and decorating rooms.

Scientists are returning to, and emphasizing, the sun as our principal source of available energy. In solar heat, glass will be used as a filter to allow the entrance of radiant heat enclosures, where it is absorbed; and also as a barrier to prevent the escape of heat by convection and conduction once it has been captured. Glass is being used today for this purpose.

There are certain applications where the temperature reached by direct sunlight is not sufficiently high. Under these circumstances, it is desirable to increase the temperature by collecting the sun's rays over a large area and concentrating the energy in a smaller area.

Reflecting mirrors for collecting the sun's rays will grow in importance, and most of them will be made from glass. In the not too distant future, such collectors will be able to furnish considerable quantities of power competitively in some parts of the world. In the more distant future following the inevitable deterioration of other power sources, enormous glass collectors will furnish very significant amounts of energy throughout the habitable world. Here could be one of the most extensive new mirror markets in many years.

Unbreakable glass will result from the extensive basic research being conducted today into the structure of glass, and the extension of its properties by new compositions. The products have long been talked about, but only recently have some of the puzzling mysteries surrounding the very old art of glassmaking being solved.

Such an unbreakable glass could compete directly with metals in many areas. Machinability is a characteristic not normally associated with today's glass, even though sand blasting and ultrasonic methods can now be used to fabricate glass at a cost that is satisfactory for very special purposes. Unbreakable glass, however, would probably be machined to an extent approaching that of metals.

As a packaging material, glass possesses certain qualities unmatched by any other material. It is chemically inert, impermeable, transparent, nonporous, sanitary, and odorless, and may be formed into an infinite variety of shapes and sizes in many colors.

Looking into the future, it is possible to see that glass, with its enormous annual investment in research and development, is on the threshold of a new era.

Since 1930 most glass containers have shed a third of their weight, and a further slimming of 20 percent has already been predicted for many styles of glass containers in the future, making lighter and even stronger glass containers no longer a matter of conjecture.

The modern glass container is capable of withstanding much more abuse than is generally believed. Proof of the strength and remarkable shipping qualities of modern, improved glass containers may be found in the Final Report of the Transportation and Packing Survey, sponsored jointly by the Railroads of U.S.A. and the Fibre Box Association.

Freight claims on foods and beverages in glass averaged $10.00 per car against $55.00 for juices in tin, and $44.00 for fruits and vegetables in tin.

The freight claim average of all commodities checked by the railroads is $29.00 per car, compared with $10.00 for glass.

Present-day glass production is expected to continue unabated. With the arrival of the Space Age and lunar probes, it is quite logical that the glass industry will direct its attention to studies in the satellite field. A broader use of glass is anticipated in this field, with the newer glasses having unusual and specific properties.

Glass-ceramic products will also multiply and electronic glasses will be more widely used.

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Corning Glass Works, Inc.
Glass in the Modern World

GLASS AS THE MATERIALS SCIENTIST SEES IT*

M. V. Nevitt
Argonne National Laboratory
Argonne, Illinois

It is a pleasure and a privilege to be with you today and to be afforded the opportunity to address your Society as the keynote speaker at your 21st Symposium and Exposition. I note that this Symposium Series is almost as old as the Society itself; the Society will celebrate its 25th anniversary of founding next year. I am pleased to take a small part in this kind of information interchange, which is so central to your Society's activity.

My background, as I think you know, is in materials science in general and metallurgy in particular. So I bring to this talk no real, direct expertise in glass and its forming, and I make no pretense that I do. If I have anything of use and interest to you it's along the line of some perspective on glass — indeed the limited perspective — that the material scientist now has regarding glass and some possible bases for a wider, deeper and more useful understanding in the future.

My thesis for this brief talk is simply stated: For many years the materials scientist has been *looking through* or around glass but not *looking at* it.

I mean by this that the materials scientist has used glass as an indispensable, transparent and durable material in his or her research tools, but has rarely made the glass the *subject* of research.

In the absence of the unique laboratory glassware that the members of this Society have provided, materials science research would have been seriously retarded and in some particular cases, blocked altogether. An equal, probably greater, roadblock to materials research would have existed if glass technology and, its partner, optical technology had not provided the lenses for microscopy and photomicroscopy. Microscopy has probably been the most essential tool of the metallurgist and materials scientist.

So glass in the form of these essential tools has been at the right hand of the materials scientist but he has not studied it extensively as a material.

Why has this been so? Mainly because the vitreous state — the state in which glass exists — has diverse properties, many of which have been unfamiliar and puzzling to the materials scientist. Not knowing how to describe glass, the materials scientist has relegated it to the back of the book, so to speak. In actual fact, most materials science textbooks have treated glass and the vitreous state briefly and perfunctorily, as kind of an after-thought. Glasses as a state of condensed matter have been thought to be too complex and too unyielding to rigorous analysis to be addressed as a scientific subject. It is pleasing to note, however, that glass,

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*Work performed under the auspices of the U. S. Energy Research and Development Administration.

which for centuries has been an artistic medium and more recently has been an industrial material, is now becoming a scientific subject. As we shall later discuss briefly, there are several incentives to develop a glass science. An evolution (not a revolution) is occurring in the understanding of the nature of glass.

Why the delay? Because the metallurgist/materials scientist has not been conditioned in a scientific sense to understand the vitreous state. The fledgling materials scientist has been given a "teething ring" that seems to have conditioned him against the glassy state.

You probably all know what a teething ring is. Just in case you don't, Figure 1 shows you one. I chose this example because the ring is hard, vitreous silica, and therefore appropriate to this Symposium. The silver bell, on its opposite side, carries the name of one of my children. As you know, the infant bites on the hard ring and thereby cuts his or her first teeth.

Figure 1

Now the materials scientist — because of his metallurgical ancestry — cuts his teeth, so to speak, on a rather softer material, the alloys of lead with tin or a comparably simple alloy system. His "teething ring" is the lead-tin equilibrium diagram, shown in Figure 2.(1)

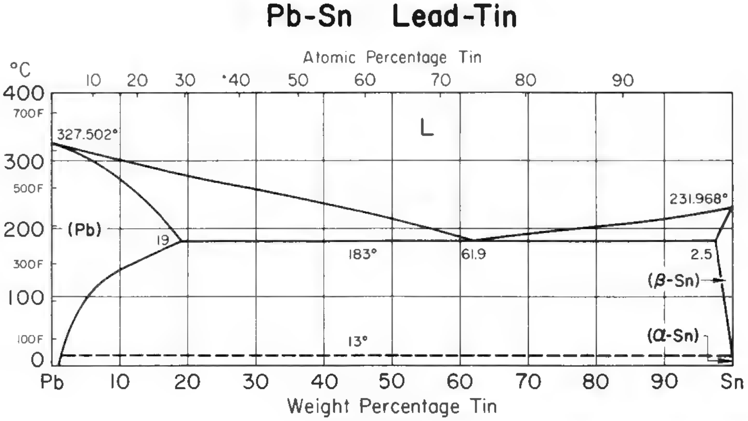

Figure 2*

This simple equilibrium diagram is basically a melting point diagram, with temperature as the ordinate and composition, from 100% Pb to 100% Sn, as the abscissa. Lead, which soldifies at 327.5°C, has its initiation of freezing lowered by the addition of Sn until at 62% Sn the freezing temperature has fallen to 183°C. Thereafter, with increasing Sn, the beginning of freezing is raised again until at pure Sn it is 232°C. Everything is simple: Temperatures are low. All melts are totally liquid above the liquidus line, and everything is a crystalline solid below the solidus line. The neophyte is shown photomicrographs like Figure 3(2) to convince him that only a crystalline solid exists below the solidus temperature.

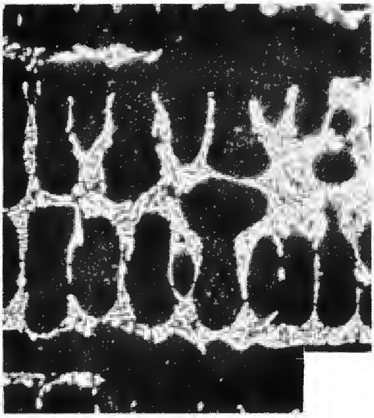

Figure 3**

Pb + 6% Sb **50X**

The young materials scientist sharpens his or her teeth on more complicated systems like Ni-Zr — Figure 4.(3) More complexity from a thermodynamic viewpoint, but everywhere there is order. Alloys are totally

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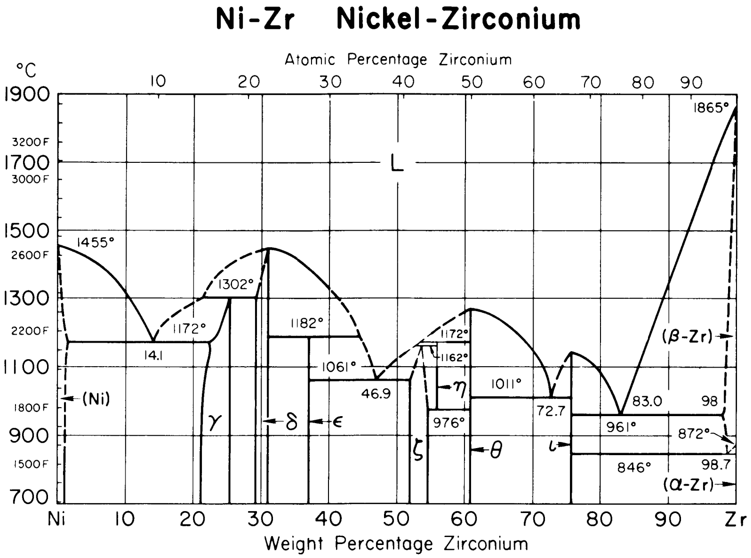

Figure 4*

liquid above the liquidus line and all are crystalline solids below the solidus line.

The student is sometimes shown — usually just in passing — what appears to be a deceptively similar phase diagram for two substances, such as CaO and Al_2O_3 (Figure 5).(4) He or she is told that the crystalline state is the state of lowest free energy, and thus the truly stable state, but that in certain composition regions a different, metastable form — the glassy state — prevails. Little discussion is customarily accorded this state of matter.

Why? Because this state of condensed matter is so poorly behaved. It is "scientifically uncivilized" from the viewpoint of materials science traditionalists.

Metals and certain alloys pass from the liquid state to the solid, crystalline state by tracing out the time-temperature relationship shown at left. At the freezing temperature, T_0, (Figure 6)(5) the heat of crystallization released during freezing just compensates for the heat lost by conduction, convection and radiation, so the temperature of the system remains constant as long as there is any liquid metal left. But the same kind of time-temperature plot for a glass former, such as GeO_2 (right) shows no comparable arrest at all at T_0, the temperature where the vitreous state occurs on cooling. The time-temperature plot shows that the glassy state is continuous with the liquid state. That is, crystallization has apparently not occurred and there has been no energy-liberating structural transformation that appears to accompany the onset of the vitreous state. Such behavior is alien to the thermodynamicist and to the materials scientist.

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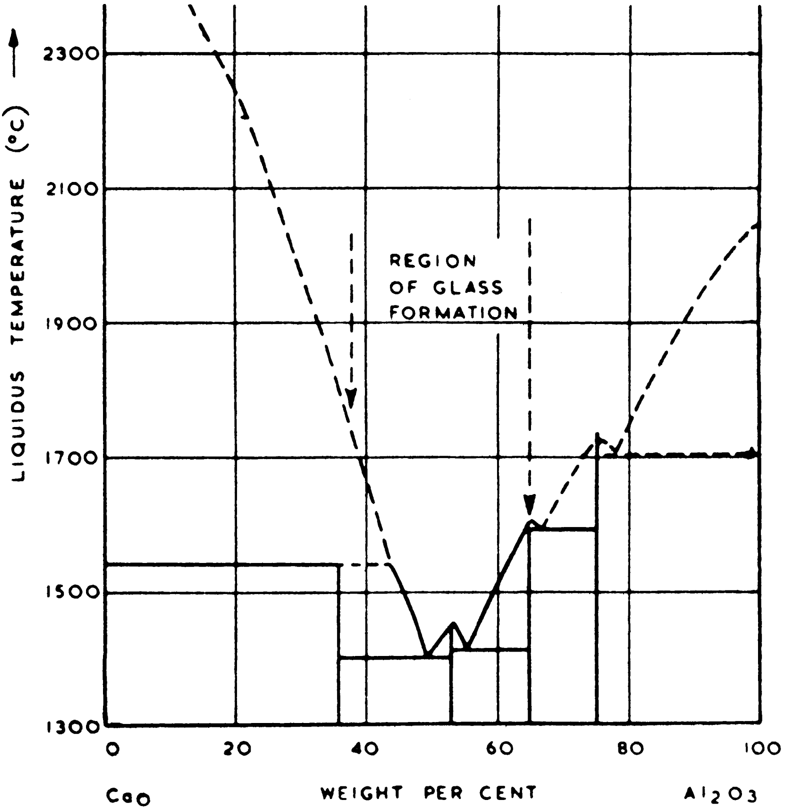

Figure 5
Region of glass formation in the system $CaO-Al_2O_3$.

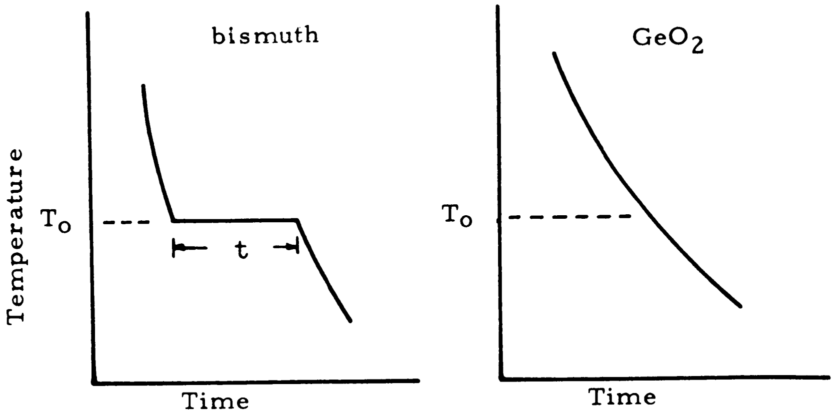

Figure 6
Typical cooling curves for a) bismuth, b) GeO_2. T_0 is different for each compound.

The materials scientist knows that when a metal or an alloy freezes the atoms arrange themselves in an orderly geometric array that has a repetitive, symmetrical configuration over many atomic spacings, as shown in the two-dimensional representation of Figure 7.(6)

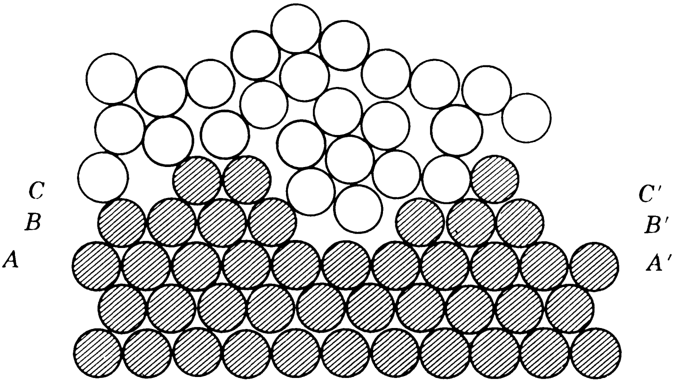

Figure 7
Interface between crystal and melt.

There is long range order, a profoundly important concept in the structure and behavior of crystalline solids. This long range order is not without some imperfections, but it is close to perfection. That is, the number of defects — or mistakes — is not large.

Figure 8(7) shows the perfect arrangement of atoms in a crystalline solid having the face-centered cubic crystal structure.

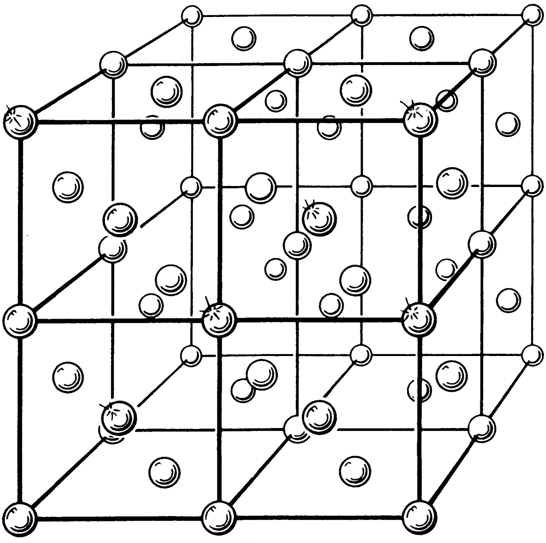

Figure 8
Positions of atoms in a face-centered cubic crystal.

Now, how about the comparable arrangement of atoms and/or molecules in a material having glass forming tendencies: Can the crystalline state occur? Yes, of course it can. Glasses do devitrify, i.e., partially crystallize, and more importantly from a scientific sense, large crystals can be grown under carefully controlled laboratory conditions.

When, for example, crystals of silica are grown and studied, one can determine a crystal structure such as that shown on the right in Figure 9.(8)

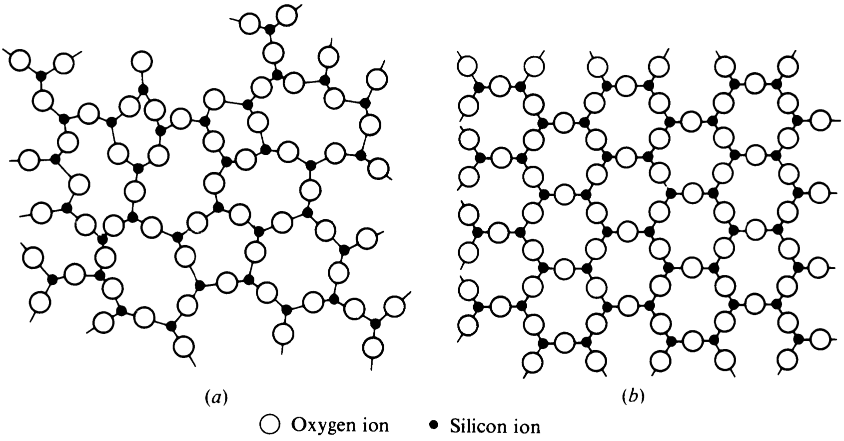

Figure 9
Silica as (a) a glass and (b) a crystalline solid. This two-dimensional representation shows only three of the normal four oxygen atoms around each silicon atom.

Silica as a crystalline solid has the same kind of long range order as the metallic crystal shown earlier. But there is a strongly competitive (from a thermodynamic viewpoint) metastable condensed state, the glassy state, which we have only recently begun to describe and understand.

The glassy state lacks long-range order, but it possesses short-range order. Short-range order is essentially the same in liquid or solid, crystal or non-crystal. There are the same bond lengths and bond angles. The presence or absence of long-range order is what makes the difference between the crystalline and vitreous states. As Figure 9 shows, the SiO_4 tetrahedron remains the basic structural unit in the vitreous state but with a high number of disordered sites. There is no long-range order. In the melt (liquid state) and in the vitreous state there is little ordering beyond the first coordination sphere, i.e., the first polyhedron.

Very unique mechanical properties are associated with materials bonded in this way. The modulus of elasticity (E) increases with stress for vitreous silica, which may be due to "straightening out" kinked Si-O-Si bonds at low strains, followed by distortions of the SiO_4 tetrahedra and stretching of the Si-O bond lengths at higher strains.

Figure 10[9] shows this polyhedron cluster. Glass is described as a "random network"; same immediate neighbors as in the crystalline state but there is randomness in the connections between the polyhedra.

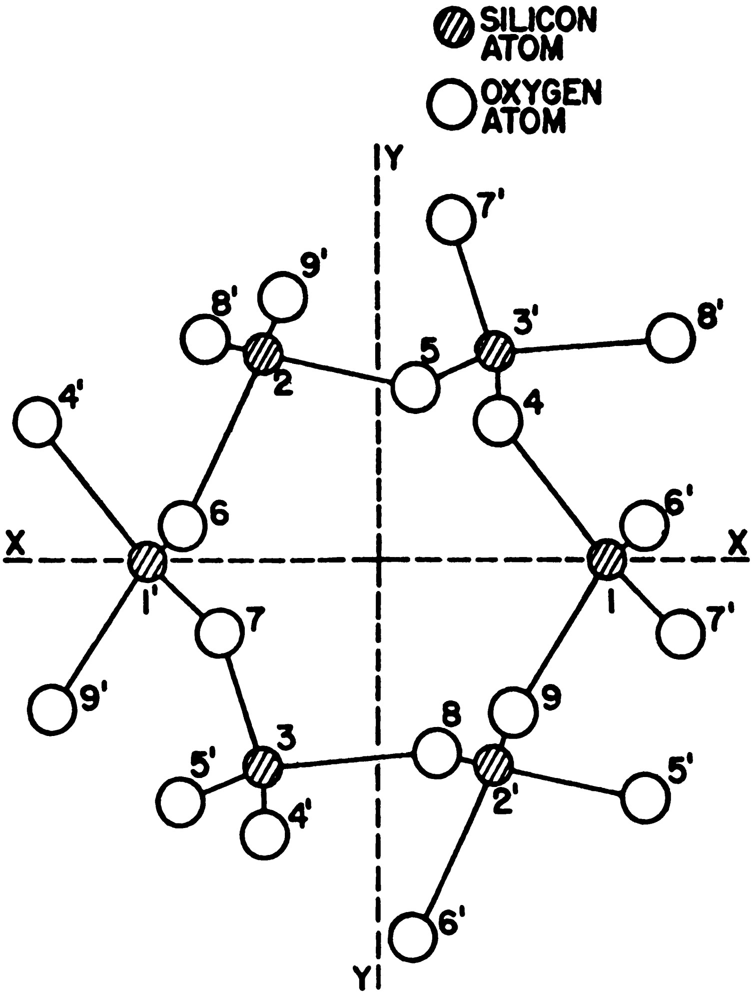

Figure 10
Structural unit for lattice model of vitreous silica.

It is important to note here that from an experimental point of view these first tentative descriptions of glass have been gleaned from very difficult experimental techniques. They are too complicated to be described in the short time provided me. Suffice it to say that the experimental techniques and the interpretations of the results are not as straightforward as those associated with studies of the crystalline state.

Why does this unique vitreous state occur in certain thermodynamic systems? The central feature of vitreous-inclined systems is the existence of strong, (particularly directional) bonds between the atoms. They are known as covalent bonds. Covalent bonds favor the vitreous state because they reduce the mobility of atoms. Crystallization is a dynamic process. It requires the forming, the breaking and the reforming of chemical bonds in order to change the material from a totally disordered liquid to a highly ordered crystal. So a high local bonding between atoms in a liquid reduces the bond-making/bond-breaking tendency and is an inhibitor or a retardant to crystallization and an encouragement to glass forming. An obvious clue to high interatomic bonding is a high viscosity.

Glass formation occurs during cooling through a temperature range where crystallization should occur, but crystallization is bypassed and the melt is cooled to a temperature low enough to act as a barrier to crystallization. This is a necessary condition for the formation of the vitreous state. Glass formation is favored by a high quenching rate and a low rate of crystallization.

Summing up, we find that melts with high viscosity show low rates of crystallization and thus a high tendency to glass formation because the individual bonds even in the liquid state are so strong that they aren't easily broken and this tight directional bonding inhibits the dynamic bond-breaking/bond-making activity that must occur to form crystals. Metals have non-directional bonding strength, i.e., they are not covalently bonded, and so they transform from liquid to crystalline solid much easier.

Why has the materials scientist recently become more interested in glasses? There are three reasons:

(1) It is now known that a wide range of substances can form glass. The vitreous state can occur in metallics, oxides, nitrides and in combinations of these. Every liquid can transform to glass under the right circumstance, without crystallization.

(2) Glasses, if they can exhibit their theoretical strength, are stronger than any known metals.

(3) Glasses are more energy-conservative than metals by many factors of ten.

Now let's briefly consider the first of these reasons. Table I(10) shows the wide range of materials that can form glasses.

TABLE I. *Representative Glass Forming Substances*

Elements	S, Se Bi, Ge (vapor deposited at low temperatures) Mixtures of As, Se, Te, S, In, Ge, etc.
Oxides	B_2O_3, GeO_2, As_2O_3, TeO_2 SiO_2, P_2O_5, V_2O_5 Mixtures of CaO-Al_2O_3, PbO-V_2O_5, TiO_2-K_2O, etc.

Salts	BeF_2, $ZnCl_2$ Mixtures of KNO_3-$CaNO_3$
Carbonates	$K_2Mg(CO_3)_2$ (at 1000 atmospheres)
Organic Compounds	Glycerol, Glucose Various high polymers.

Notice that even elemental metals and semi-metals can form glasses, although these are, in the main, elements whose bonding is in part covalent and thus they fit into the picture developed earlier that strong directional bonds facilitate the bypassing of the crystalline state and onset of the glassy state. Germanium is an interesting case here. It is interesting to note that from both theoretical and practical points of view the materials scientist and the solid state physicist had pretty well convinced themselves that the existence of the crystalline state was a necessary condition for the occurrence of the electronic properties of a semiconductor. All solid state devices have been crystalline. Now, however, in recent years it is becoming evident that this is not necessary and that Ge in the glassy state provides a concomitant set of electronic properties similar to those of the crystalline. Hence the strong current interest in the amorphous semiconductor, which you have no doubt heard about.

As I said at the start, my purpose has been to indicate the past and present perspective of the materials scientist regarding glass and to note that there is a gathering (and long overdue) interest in developing a better understanding of glass.

There now appears to be well-founded optimism that such a broadened understanding will lead to vastly improved glasses and to totally new applications of vitreous-state materials. The long history of metallurgy and the shorter but impressive history of materials science support this optimistic view. I am especially intrigued with the idea of improving the properties of glasses so that they can take their proper place as engineering as well as scientific materials and I am sorry that our brief time together this morning did not permit some of these ideas to be developed.

I am confident that the members of this Society will work with the materials scientist in gaining this understanding and as a result further developing the technology that is central to your profession.

Thank you.

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DESIGN PROBLEMS ASSOCIATED WITH THE USE OF EVACUATED GLASS RECEIVERS FOR SOLAR COLLECTORS

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ABSTRACT

This paper deals with the problems associated with the design and construction of practical, low-cost collectors for solar energy systems. The design of evacuated receivers being used with a compound parabolic concentrator (CPC) is discussed, and means of minimizing optical and thermal losses are described. Emphasis is placed on the identification of problems that need attention by the glass industry. An evacuated receiver designed for use with a lightweight, thermoformed plastic 3x CPC is used as an example.

INTRODUCTION

A major problem confronting designers of solar energy systems is that the solar energy reaching the earth's surface is very dilute (the peak power is ~1.0 kW/m^2 or 300 Btu/ft^2-hr.). Therefore, a practical low-cost system must not only provide efficient energy collection, but must minimize energy losses from the entire system. This is necessary to keep the overall size of the solar collector as small as possible and, therefore, more cost competitive. A description of a collector concept being developed at Argonne National Laboratory is presented. The overall system design is beyond the scope of this paper, and only the detailed design requirements of one component, the evacuated receiver (comprising the absorber and associated insulation), will be discussed.

The collector concept being developed at Argonne involves a compound parabolic concentrator (CPC). The CPC is one member of a family of nonimaging collectors currently under development at ANL[1,2,3]. The advantage of a nonimaging solar concentrator is that light may be accepted from a wide angle, and therefore the collector does not need to follow the daily motion of the sun as is required for an imaging (focussing) system. For example, a 3x CPC has an overall acceptance angle of 38° and would have to be tilt-adjusted only twice a year.

Figure I is an illustration of a lightweight collector panel based on this concept. The sets of compound parabolic reflectors concentrate the light energy onto an absorber within an evacuated glass tube. The reflecting surfaces may be thermoformed plastic substrates that have been metallized by vacuum evaporation of aluminum. The evacuated glass tube serves to thermally isolate the absorber from the reflectors allowing the use of inexpensive materials of construction. A 3-m^2 collector panel

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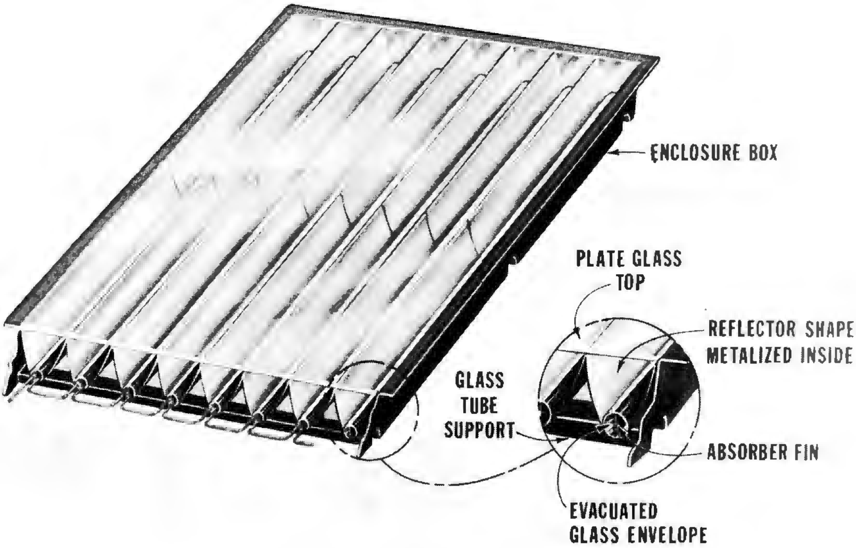

Figure I. Lightweight Collector Panel

using a 3x CPC would require 20 evacuated receivers 1.21 m x 3.8 cm (4 ft x 1½in.) which would be connected together with a heat-transfer-fluid manifold. The other major components of the collector are a cover glass and an overall enclosure box to protect the reflectors from the environment.

The collector is being designed to operate at temperatures of 120-230° C (250-450°F) with an efficiency of ~40% (at 350°F). This operating temperature range is between the range (50-90°C) for flat plate collectors and that (200-350°C) for line focus systems that must track the sun. This intermediate temperature range has many applications including air conditioning, process steam, and electrical power generation.

EVACUATED RECEIVER DESIGN

An evacuated receiver is composed of an absorber with a means of heat removal enclosed in an evacuated glass tube. The heat can be removed by use of tubing in contact with the absorber through which a gas or fluid flows, or a heat pipe. Figure II is a schematic of an evacuated receiver. This receiver is approximately 1.5 m (5 ft) long and has an absorber which is 1.21 m x 3.5 cm (4 ft x 1⅜ in.). The glass tubing is thin-walled, 38 mm (1½-in.) OD borosilicate. The tubing for the heat transfer fluid is bent into a U-shape and brazed onto the back of the absorber plate. The heat transfer fluid to be used is Dowtherm A. It will flow in one leg of the tubing and out the other, collecting heat from the absorber. One possible location for the getter required to maintain the vacuum is shown.

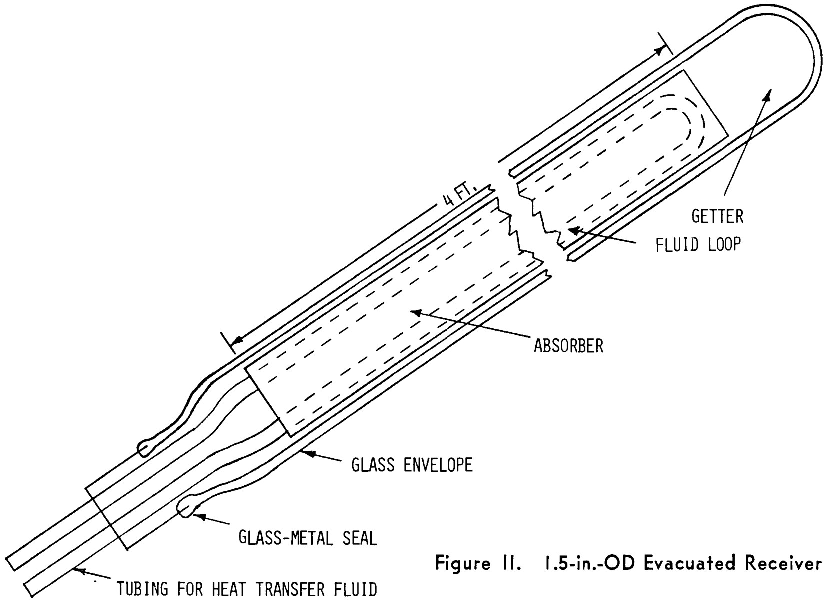

Figure II. 1.5-in.-OD Evacuated Receiver

The inlet and the outlet for the heat-transfer-fluid flow tubes are placed on the same end of the glass tube for two reasons. The first is that the thermal expansion of the absorber is larger than that of the glass because the temperature of the absorber is much higher. This expansion would put pressure on the glass and glass-metal seals which could result in breakage unless a bellows were used to accommodate the differential expansion. The second is that placement at opposite ends of the glass tube would require two glass-metal seals, which would increase the cost of the completed evacuated receiver.

The glass-metal seal is an area that needs more improvement. Currently borosilicate glass-metal seals are either the Housekeeper or the Kovar® type. The advantage of the Housekeeper is its lower-cost, the disadvantages are the low strength of the seal and temperature-cycling-induced fatigue.[8] The Kovar seal meets the strength and durability criteria, but is curently too expensive. Because the cost of an evacuated tube receiver should be less than $2.00 and the lifetime 10-20 years, it may be necessary to consider different types of materials, both glass and metal, for the seal. Soft glass tubing used by the fluorescent lamp industry is much less expensive than borosilicate glass. A glass-metal seal made with soft glass and an alloy such as Niron-52®, as illustrated in Figure III, might be amenable to production on an assembly line, using induction heating followed by annealing. This procedure has the potential for reducing the costs for both the glass and glass-metal seal.

Insulation of the manifold portion of the receiver which is not vacuum insulated will be critical. The metal of the glass-metal seal will be in

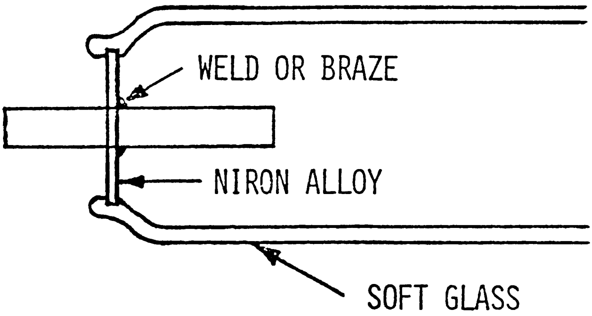

Figure III. Proposed Soft Glass-Metal Seal

direct contact with the heat transfer fluid tubing and therefore is at the same temperature. The glass-metal seal and manifold area must be carefully insulated to prevent heat losses. Also, since heat losses are a function of area, the area of the glass-metal seal and manifold should be minimized. Heat will also be conducted away from the glass-metal seal by the glass and lost from the glass surface. For that reason, approximately 10 cm (4 in.) of the glass tube will be insulated along with the heat transfer fluid manifold. The heat losses from conduction along the glass wall are minimized by reducing the cross-sectional area of the glass available for heat transfer, i.e., by using thin-walled glass tubing. While it is important to minimize heat losses at the manifold end of the evacuated receiver tube, the temperature of the glass tube at the CPC reflector surface must not be above the maximum temperature rating of the reflector base material. This maximum temperature rating will vary from 65-150°C (150-300°F) for plastics to much higher values for metals. With reflector materials having a relatively low maximum temperature rating, the receiver design will include an uninsulated section of glass tubing 5-7 cm long between the insulated manifold and the reflector to allow heat dissipation.

An absorber support positions the absorber in the glass tube. A good absorber support will minimize heat losses due to conduction as well as optical losses. Optical losses can be caused by frontal area blockage and by improper positioning of the absorber with respect to the mirror. Work with the Sterling Spring Company of Chicago, Illinois has led to the development of two supports. The first is a four-turn compression spring made from 0.787-mm (0.031-in.) OD stainless steel wire; the two end turns are small and bear against the absorber, while the two middle turns are larger and bear against the glass envelope. The disadvantages of this support are the optical losses resulting from the shadowing by the four turns of wire and the fact that eight notches have to be cut into the absorber to position the spring. Also, the absorber can move up and down from the proper position with respect to the mirror. The second design is a spring clip shown in Figure IV. The two ends of the spring point in opposite directions along the length of the glass tube, a design that gives

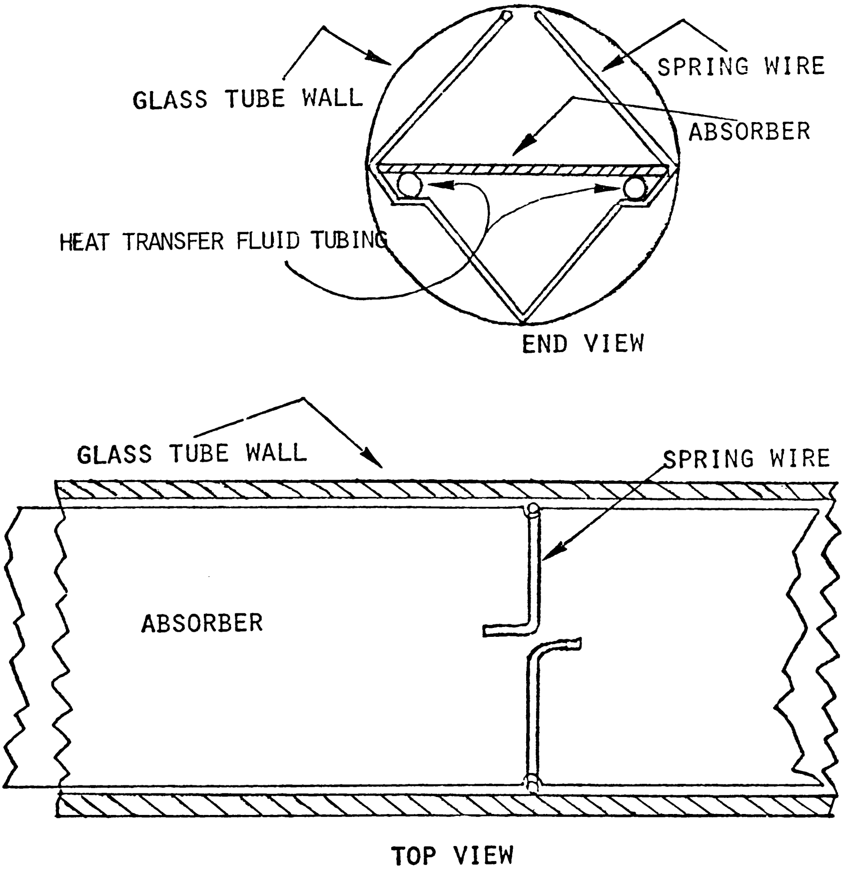

Figure IV. Spring Clip for Centering Absorber

the support stability from tipping which can occur during insertion of the absorber into the glass tube. The two horizontal areas above the bottom V-shaped section support the heat-transfer-fluid tubing. The advantages of this design are the reduced optical losses due to smaller frontal area blockage, accurate location of the absorber at the center of the glass tube, and the need for only two notches per support. This support, which can be made on a wire former with one manual operation, should cost about 1-2¢. Other types of supports should still be investigated.

Vacuum insulation helps in a number of ways. The vacuum reduces heat losses by suppressing both conduction and convection losses associated with the gas phase surrounding the absorber. Convection losses are suppressed with vacuums in the range of 12-25 torr, and are completely eliminated at 1 torr$^{4 \& 5}$. The conduction losses through the gas phase remain constant down to a pressure of about 10^{-1} torr. At 10^{-4} torr the conduction losses are negligible.

The use of getters to remove N_2, O_2 and H_2O will be necessary to maintain the vacuum at 10^{-4} torr. Before a getter can be used, the glass tube has to be evacuated to 10^{-5}-10^{-6} torr, baked out to about 400°C for 4 hours and then sealed. The common getter used in the T.V.-tube industry is the barium or evaporable getter, which can be expected to maintain a pressure of less than 10^{-5} torr.[6] The barium getter has the advantage of reasonable cost. The disadvantage is that it must be distributed over a large area to be most effective. However, with careful placement of the barium, it may be possible to utilize the hot areas of the glass tube near the glass-metal seal to help the total gas absorption. Another possibility is the use of the nonevaporable or bulk getter. Nonevaporable getters need high temperatures for activation, usually 300°C (575°F) for the diffusion of surface-absorbed gases into the getter. These temperatures can be achieved with evacuated receivers under conditions of no heat removal. The main advantages of nonevaporable getters are large capacity and the potential for long-term gettering. The disadvantage is the higher cost, but this could be reduced, especially through a larger demand. A combination of evaporable barium getters with nonevaporable getters could have the advantage of initial high-speed gettering by the barium and large total-volume gettering by the nonevaporable getter.[7]

In the design of solar collectors, it is important to minimize optical losses. One important optical loss in the application of the evacuated receiver with the CPC is caused by the gap between the absorber and the lower edges of the parabolic mirrored surfaces. The use of thin-walled glass tubing helps minimize this gap. Optical losses can also be reduced by a modification of the design of the CPC, in which the focal points of the parabolas are at the edges of the absorber rather than at the base of each opposing parabola.

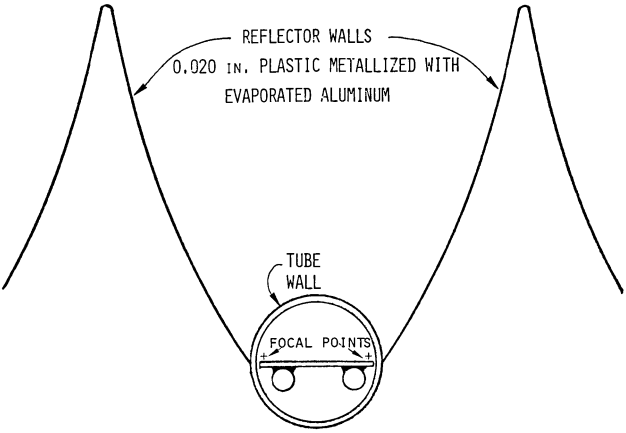

Figure V. Reflector and Receiver Cross Section

Selective coatings are available for the absorber surfaces for solar applications. These coatings have high absorptivities (α) for energy in the solar spectrum and low emissivities (ε) in the far infrared. A surface with a high absorptivity maximizes the amount of energy collected and with a low emissivity minimizes the energy emitted. The overall effect is achievement of higher temperatures. The coating currently being used in our prototype units is black chrome ($\alpha \geq 0.9$, $\varepsilon \leq 0.1$). The maximum temperature that black chrome can be heated to without undergoing irreversible degradation is about 400°C. This temperature is an important consideration in the glass sealing, annealing, and bake out operations. The vacuum environment can also help protect some selective surfaces which are not stable in air.

CONCLUDING REMARKS

The expected performance of the 3x CPC with an evacuated-tube receiver is shown in Figure VI. Also shown for comparison are the published performance curves for a commercially available (Owens-Illinois) nonconcentrating collector with an evacuated tube receiver and a previously tested, conventionally insulated 3x CPC built by Argonne National Laboratory.[9] This figure demonstrates the advantages of a design that combines an evacuated tube receiver with a concentrating collector. The CPC design has the additional advantage that fewer evacuated tubes are required to collect the equivalent energy when compared with nonconcentrating evacuated tube collectors. This reduction in the number of evacuated tubes used should lead to reduced cost.

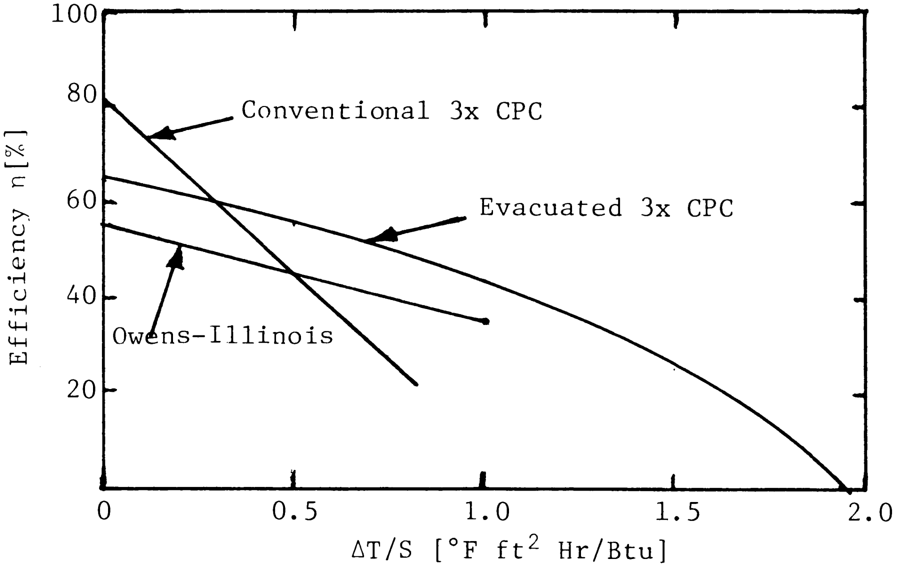

Figure VI. Expected Performance of a CPC (3x) with an Evacuated Receiver

One of the principal objectives of our program is early commercialization of the CPC solar energy system. This is being accomplished by information dissemination through publications and oral presentations. Also, industrial firms are being encouraged to send participants to work at Argonne National Laboratory, where they both contribute to the effort and learn about CPC development. Presently, an industrial participant from Chamberlain Corporation, Waterloo, Iowa is working at Argonne on the collector program. Contracts with industry for component development and other cooperative approaches are being used to encourage the development of the CPC.

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IN ATTENDANCE

The following are on record as having attended the Twenty-first symposium on the Art of Glassblowing held at the Sheraton-Chicago Hotel, Chicago, Illinois, June 7-11, 1976. As fully paid registered participants, these persons are entitled to a copy of the Proceedings.

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