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CONTENTS

DELAYED FAILURE OR SUBCRITICAL CRACK GROWTH IN GLASS

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Introduction

Delayed failure or subcritical crack growth in glass is a phenomenon that has been known by many different names. Some of them are static fatigue, dynamic fatigue, and stress corrosion. Each term has a different connotation about either the applied load or about the mechanism of the failure. Delayed failure implies the object broke after the load has been applied for some time. Static and dynamic fatigue refer to delayed failure when the loads are respectively unchanging or constantly increasing. Stress corrosion and subcritical crack growth refer to what is physically happening in the material rather than considering only the gross results of the load application. However, by whatever name it is called, this characteristic behavior of glasses (and ceramics) can cause serious problems in their effective use.

This report is going to review and discuss this behavior called fatigue by first considering some history, then some theories describing fatigue, and finish up with some examples of where delayed failure had to be evaluated for proper design.

History

Glass is a material that has been used for many centuries. The first glasses were natural such as obsidian or pumice[1]. Then came the man-made kind, first made in some unknown locale over six thousand years ago. However, it is only in the past century or so, that glass has been studied and analyzed for behavioral characteristics on a scientific basis. The following brief history focuses on people and papers that are important with respect to the development of the understanding of delayed failure aspects of glasses. Admittedly this section is incomplete as it is impossible to review fully the many recent publications and to acknowledge properly all of the steps taken to our current understanding of fatigue.

A.J. Holland[2] and S.M. Wiederhorn[3] have reported that the first published commentary on delayed failure was by L. Grenet in a French

1

publication in 1899[4]. The next really important event in the development of fatigue analysis was the publication of A.A. Griffith's paper on "The Phenomena of Rupture and Flow in Solids" in 1920[5]. This paper is the basis of the field of study known as "fracture mechanics", the study of sharp flaws in materials. Although this paper did not discuss fatigue directly, it did start the analysis of flaws necessary for fatigue characterization.

In 1940, Holland and Turner[2] reviewed some of the earlier work and presented data on what is known now as static fatigue. They did observe that the applied stress and failure time were linear on log-log axes; a consequence modern theories can explain. In 1946, the *Journal of Applied Physics* had a series of articles on experimental aspects of delayed failure by Baker and Preston, [6] [7] [8] and by Glathart and Preston[9]. Again static fatigue was being studied, this time over a range of time from 0.01 second to a day. At the end of this series, the data were being analyzed as linear in a 1/stress versus log time. In 1949 Gurney and Pearson[10] demonstrated that water in the atmosphere was an important factor in the delayed fracture of glass.

Activity in the study of this fatigue phenomenon increased during the fifties, sixties, and on up to the current time. R.J. Charles published two works in the late 50's that considered a power law type of theory (more about that in the next section) for analyzing dynamic and static fatigue cases.[11] [12] Starting in 1959 and continuing for the next two years, R.E. Mould, by himself and with R.D. Southwick, wrote a series of papers on the "Strength and Static Fatigue of Abraded Glass Under Controlled and Ambient Conditions:...".[13] [14] [15] [16] In 1964 Hillig and Charles presented their theory on the stress accelerated attack of water on glass[17] by saying that the critical dimension/factor is the sharpening of the crack/flaw tip radius, not just the length of the crack as was first considered. Then in 1974 Wiederhorn presented some data and theory on fatigue measurements and predictions based on an exponential approach.[18] Evans also expanded on theoretical analyses based on the "power law".[19]

In the seventies and eighties, more data were being generated, and more extrapolations were being made. Proper usage of these data was the theme of several papers including Pletka and Wiederhorn,[20] Ritter et al.,[21] and Jacobs et al.[22] This is a rather short paragraph to summarize the hundreds of recent papers on fatigue behavior; however, for the latest

developments read the *Journal* and the *Bulletin of the American Ceramic Society*, and the *Journal of Materials Science* to catch most of the published work. For more detailed and longer reviews than presented here, Adams and McMillan,[23] and Doremus[24] are suggested.

Theory

The question arises why is there so much more work being done on delayed failure behavior now than in the past? The main reason is the need for improved design on new and old materials. Glasses and ceramics are being used at higher temperatures, in harsher environments, for longer times, or conversely, for shorter times. Because of costs in money or weight, the designs tend to be closer to the ultimate capabilities of the materials, which implies the need to know the behavior closer than before. And, of course, some compositions have not been around long enough to have developed a good lifetime data base. All of this means that a good theory or model of this fatigue behavior is necessary for successful and safe design.

Past experiences have shown that glass will fatigue when the following three conditions are present:
- •surface flaws
- •moisture
- •tensile stress

The purpose of a theoretical model is to incorporate these "well-known" facts into a formula that can be used to predict future behavior from past behavior.

There are two basic approaches to theorizing about fatigue—a "macroscopic" or an "atomistic" approach. The former is more concerned with models that can be used directly and relatively easily for design purposes by making various assumptions about the overall behavior of the material, while the latter is trying to consider the "why" and "how" on a molecular scale without an immediate concern about engineering ease of use. The differences between these two approaches is getting smaller although much remains to be done.

The *macroscopic models* of fatigue behavior can be categorized into either a rule-of-thumb or a fracture mechanics relationship. The fracture mechanics is currently subdivided by two major assumptions. But first, the rule-of-thumb: the safe long term applied stress should be less than one third of the characteristic fast fracture or proof strength. This is a

3

conservative value that has been observed by glass manufacturers to provide a safe design strength for long life.[25]

The fracture mechanics or continuum approach to characterizing the delayed failure of glass provides a more rational basis for predicting short or long time spans. (The rule of thumb is much too conservative for short life spans!) The fracture mechanics approach (see [26] for a review) assumes that the material is a homogeneous, isotropic continuum with one, or more sharp flaws that could cause failure if they were to grow to some critical value. The criterion as to whether or not the flaw is at this critical value is determined by its stress intensity, K. Stress intensity has units of $Pa\sqrt{m}$ (psi$\sqrt{in}$) and is equal to $YS\sqrt{a}$ where S is the applied stress, "a" is the crack length, and Y is a geometric factor determined by the size, and shape of the stressed flaw with respect to the flawed object. Each material has a critical or limiting value of K which determines the relationship between the applied load and the extant flaws. From the above relationship, it can be seen that a material will appear weaker with the larger flaws. Fatigue is said to occur when subcritical flaws grow to become critical under a load.

One of the primary dividing opinions is in the governing "law" that describes a rate of- and the variables in-control of- the growth of this flaw or crack. The two main growth laws are a "power law" and an Arrhenius
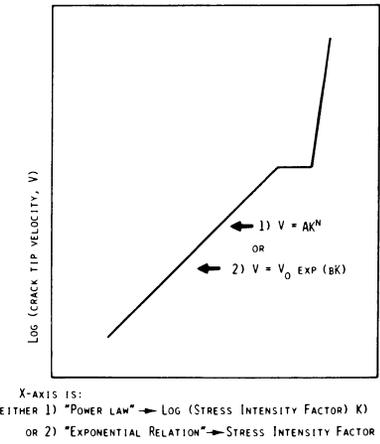
type "exponential relation". The major difference between these two can be best seen graphically as in Figure I where the log of the crack tip velocity, V is a direct function of the stress intensity factor, K for the exponential law, and is a direct function of the log of K for the power law. That is, the form of these subcritical crack growth models are:

- power law: $V = AK^n$
- exponential law: $V = Ae^{BK}$

The functional difference in two contending fatigue models is illustrated by the choices of plotting axes used to describe the crack tip velocity dependence on the stress intensity. The exponential relation uses K for the abcissa while the power law uses log K.

where V is the crack tip velocity, K is the stress intensity, and A, n, and B are constants for a given combination of environment and material. Both laws are semi-empirical in that the original basis was due to the fit of data although there are some theoretical bases for both of these descriptive laws.[27] [28]

After choosing the form of the crack growth to be used, the load history, or the load prediction is integrated into the form to obtain a relation which can characterize the lifetime. The power law is the more common form used—partially because it is easier to manipulate mathematically. It should be pointed out that both of these models of subcritical crack growth seem to fit current, short term data reasonably well. However, in long term prediction abilities, the answers are not in yet as no critical multi-year tests have been done. This is a serious difficulty as the predicted long term lives can be significantly different when based on either a power-law or an exponential-law growth model.

The *atomistic approach* tries to determine or explain on the atomic level why, and how, this fatigue behavior in glass occurs. While the above macro-models assume that the material is a homogeneous, isotropic continuum, atomistic models consider the material as a collection of individual atoms with an aim to understand the behavior of the flaw tip with respect to individual atoms. These theories are not developed enough yet to be able to make practical predictions of long term behavior, but can sometimes explain some behaviors. Most of these theories can be characterized as either a lattice/thermally activated process or as a molecular/chemical mechanism. The difference between these is not very clear as there is much hand waving in these concepts. Part of the difficulty in properly formulating on such a small scale is the lack of good experimental work on that scale to help lead theoretical development. Nevertheless, some experimentation has lead to proposed atomistic scale mechanisms for the crack enhancing effects of water and some other polar, i.e. electron donor, fluids.[29] [30] Some other recent work in chemical formulation has been done by Thomson,[31] and by Fuller and Thomson.[32]

All of the above has assumed that we have only one flaw that will grow and then fail. The problem is that most glass surfaces have more than one flaw, and they are of various sizes, shapes, and orientations. Since we usually cannot tell which flaw will be the culprit leading to failure, we have to use statistics to describe the critical flaw.

5

The most popular statistical description of flaws is the Weibull distribution.[33] This distribution is relatively easy to manipulate mathematically and fits most strength (flaw) data as well as, or better than any other distribution. The basic two parameter form is:

$$P = 1 - \exp(S/S_O)^m$$

where P is the cumulative failure probability, S is the applied stress, and S_O and m (the Weibull modulus) are the material behavior parameters. This form can be linearized to form straight lines on special plotting paper. Figure 2 is an illustration of some data on the strength of tensile tests on optical waveguides. The data seem to fall on a straight line so extrapolation to other probability levels is feasible.
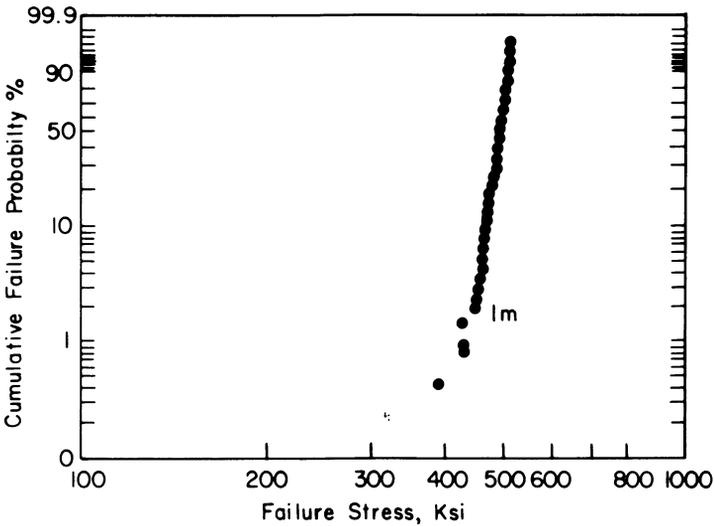
An example of strength distribution plotted on Weibull probability axes. Here, one-meter long optical waveguides with 125 um diameters were broken in tension.

Since the distribution of strengths or flaws can be described by an equation, the fatigue model can be algebraically adjusted to include such behavior.[34] Thus, we now have a mathematical model that allows us to provide numbers on when, and how likely it is a glass article will fail under a given loading condition. And, conversely, given a failure probability, one can indicate when the glass will fail at any particular load. Unfortunately, the calculated numbers may not be to the liking of the design engineer, especially since the probability values may be much too

6

high for useful commercial needs. That is, the size of the largest flaws may be too large for safety.

One way out the dilemma of there being a flaw weak enough to cause failure too early, is to process the material so that all flaws are smaller than the value required by the safe life/load combination. This is typically done by "proof testing" [35] [36] [37] the article to a load whereby all flaws are loaded enough to ensure breakage if the flaw is larger than desired. This procedure cuts off the low end of the flaw/strength distribution, leaving only the flaws that can cause breakage at loads higher than expected or at longer times than are expected for the article.

Examples

In order to understand that this study of glass fatigue is not just an academic exercise, a couple of examples will show when knowledge of the article's lifetime is important. The first example considers the windows of spacecraft. The second example brings out the many difficulties in using something more down to earth for long periods of time; the survivability of optical waveguide communication lines for decades of use.

Spacecraft windows are obviously a critical part of the vehicle. A break in one of them could lead to serious difficulties in surviving in a vacuum or in landing safely. However, one cannot just overpower the problem by using thicker glass to lower the stress to increase the life. Weight is very important—the less the vehicle weighs, the more payload one can put into orbit. Thus the need to design the windows to the exact needs, to insure safety at minimum weight.

Wiederhorn, Evans, and Roberts[38] did "A Fracture Mechanics Study of the Skylab Windows" in 1974 which presented some data and analysis for the proper design of these windows. They used an exponential form of a crack growth model combined with static loading to determine a load/failure time relation. By adding a proof stress to this relation, the authors developed a design diagram or guide for predicting load/life behavior. This process of determining design criterion and appropriate proof stresses for space craft windows was also presented in 1976 by Gulati and McCartney.[39] These writers were concerned with the space shuttle windows which were to be designed to survive for a minimum of 100 missions. Again with the vital need to be sure that the glass will not fracture early due to fatigue, each piece of glass was proof loaded with a special pressure fixture to demonstrate that any flaw still on

7

the surface was too small to cause failure before the designed lifetime.

The previous example was of the space shuttle window which was designed for a relatively short life, but with a significant constraint of minimizing weight. The next example is one of a need for a very long life—30 years—with a constraint of needing to use a particular product which cannot be physically changed. The product is an optical waveguide. These thin ($\sim$ 0.005 " dia.) glass fibers are being used for telephone lines. One of these fibers can replace a large coaxial cable many times its diameter. This saving in size is but one of the many features that has attracted the telephone companies. However, telephone companies have a long term outlook as they need to be sure that once the fiber is put in the ground, or hung on the poles, the fiber will have a useful life of 20 to 30 years. The great optical properties will mean nothing if the waveguide will fall apart too soon. And, for optical reasons, the general size and compositon cannot be significantly changed as a method to improve the mechanical characteristics for longer life at higher stresses. The way out of this dilemma is by making the cable enclosing the waveguide strong enough and stiff enough to absorb any loads over the waveguide's maximum allowable.

The maximum design load of the waveguide has to be determined by extrapolation from relatively short term tests. Short term tests are ones that require only a few weeks or months to perform. The tests done on these fibers include both dynamic and static fatigue.[40] The direct measurement of the subcritical crack growth velocity on 5 mil diameter fiber is currently impossible. As illustrated in Helfinstine,[41] the fatigue constants obtained by various researchers do differ. These differences are partially due to different glass compositions, to different coatings, and to different test conditions. These differences indicate the need to do extrapolations from test on similar fibers under environmental conditions as close to that seen in use as possible.

Concluding Comments

This article has attempted to discuss a characteristic behavior of glass known as delayed failure. This phenomenon of glass has been observed and much discussed since first published in 1899. During this period of time, this behavior has been called with many different names including fatigue and subcritical crack growth. While there are many details concerned with fatigue behavior, there are several general com-

ments one can make about the subject—

- •fatigue can be a serious design problem
- •several theories are available based on different assumptions
- •to minimize or decrease fatigue failures—remove or reduce
  - -flaws
  - -moisture
  - -stress

Although these are overly simplified comments, they do provide a good basis for the study and understanding of the delayed failure of glass.

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QUARTZ ROTARY-TABLE SUBSTRATE HOLDER
FOR EPITAXIAL GROWTH REACTOR

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Introduction

This paper concerns the design, fabrication and operation of a rotating quartz substrate holder which was developed for a vapor phase epitaxy (VPE) reactor used to grow the semiconductor InP. This substrate holder is appropriate for exploratory laboratory use and is not readily adaptable for manufacturing applications. This InP epitaxial reactor is arranged so that the vapor species flows parallel to the surface of the substrate during deposition. The crystalline quality of the epilayers grown in a reactor with this geometry has been found to be superior to epilayers grown in a vapor which flows perpendicular to the substrate surface. Under the parallel arrangement, however, distribution of both temperature and vapor species at the substrate can be nonuniform resulting in variations of thickness and composition, especially in large area epilayers. To enhance uniformity of conditions, the holder and therefore the substrate must be rotated during growth.

Figure 1 illustrates the arrangements for obtaining substrate rotation in both the perpendicular (Fig. 1a) and parallel (Fig. 1b) geometries. The origin of motion is the end of the tube exiting the reactor on the left side. It is apparent from Fig. 1a that rotation is easily obtained with the perpendicular geometry. However, rotation is difficult with the parallel geometry (Fig. 1b) where the axis of rotation is perpendicular to the support tube and the manipulation originates from a remote location. The rotator described here provides an effective means of achieving this motion in an exploratory growth system.

Design

Several designs were reviewed before deciding on the one used in this work. The chosen idea was thought to provide the mechanism with the

highest degree of reliable rotation at the possible expense of a more uniform rotation rate for the exploratory growth conditions to be used. In formulating the design, several fundamental factors were considered. First, VPE is generally carried out at temperatures in excess of 650°C, which requires that the reaction tube and substrate holder be fabricated entirely from quartz. Second, the mechanism must be reliable since viewing of the table is impossible when it is in the oven. Finally, a bevel-gear arrangement, if at all possible to produce, would be prohibitively expensive. Also, any gear mechanism would be likely to have jamming problems. This could be due either to vapor condensation or the 48-inch distance between the rotary table and source of motion (Fig. 2).

A device was designed which incorporated a manipulator which could slide and be turned within a fixed support tube. The rotating table is mounted on a spindle which is sealed to the end of the support tube. A labeled photograph of the mechanism is shown in Fig. 3 A series of slots was ground on the underside of the table. The inner manipulator, which extends through both ends of the support tube, can be translated and rotated within the support tube. An engagement rod is sealed to the end of the manipulator so that it may engage and disengage the slots in the table. A sequence of engagement, translation and disengagement serves to rotate the table through a given angle. Thus substrate rotation is achieved by means of a given series of such cycles.

Since these manipulations must be.executed from the end of the tube outside the reactor, and rotation cannot be viewed directly, a system of stops must be incorporated. The first stop is the engagement arm contacting the end of the support tube on the return stroke. (Refer to Fig. 3.) The other stops incorporate decal marker lines and serve to define the precise position of the engagement rod relative to the slots, enabling precise rotation cycles to occur. (See Fig. 4.)

Fabrication

The ideal size for the rotary substrate holder was determined to be a 25-mm-diameter bv 3-mm-thick quartz disc. This was adequate to hold the substrate and allowed space for turning the disc in the reactor tube. The first procedure was to concentrically drill the disc with an 1/8-inch diamond-core drill to provide clearance for the 3-mm spindle. The drilled hole also served to center the disc on the indexing table. A 0.090-in.-dia-

meter diamond-core drill was used to grind the slots. An angular spacing of 24⁰ between slots provided an optimum configuration for the chosen slot width (Fig. 5). The slots were then ground in depth increments of 0.010 in. due to the limit of strain on the core drill shank. Six passes were required to obtain the total depth of 0.060 in., which was half the disc thickness. The top of the disc was then counterbored to receive the retaining washer (Fig. 6). This procedure completed the disc. Next the spindle was sealed to the support tube (Fig. 7). The pre-drilled bearing plate was then placed over the spindle and also sealed to the support tube. The disc was then put on the spindle and the retaining washer was set into the counterbore. At this point, a 0.001-inch shim was inserted between the disc and the bearing plate, and the seal was made between the top of the spindle and the retaining washer. With the shim removed, sufficient clearance was provided for smooth and easy rotation.

The engagement rod was then fabricated. A 2-mm-diameter rod size was chosen as having sufficient strength while allowing a compatible fit with the slot size in the base of the disc. The rod was fabricated with the correct radial spacing to provide accurate engagement of slots. This distance between the rod and the center of the table was also critical so that sufficient rotary travel could be realized without exceeding the mechanical limits of the design. The decals were then positioned and baked on, giving orientation information about the engagement rod to the person using the manipulator. After fusing small hooks around the rim of the table and testing the table rotation for reliability, the device was ready to be used in the epitaxial reactor.

Operation

The sequence of operation of the rotary table is illustrated in Fig. 8. First the manipulator is rotated so as to insert the engagement rod into a slot in the bottom of the rotary table. The manipulator is then moved forward to the marker which rotates the table 1/5 turn. The manipulator is next counterrotated, which enables the engagement rod to clear the table as it is pulled back against the stop. This returns the manipulator to the starting position. A sequence every 3 min. results in a rotation rate of 4 revolutions per hour. A cycle can be started in any slot. Although only 5 slots are needed, the extra slots provide optional rotation cycles.

Results

The rotary table substrate holder was operated in an experimental reactor for the growth of InP epilayers where it was necessary to place the substrate parallel to the flow direction of the vapor species (Fig. 9). Substrates were typically 1 sq. in. in area. Prior to using the rotary table, epilayer thickness could vary as much as 25% over the area of a nominally 6-μm-thick layer. Electrical characteristics were found to be nonuniform. As a result of the incorporation of the rotary table, epilayer thicknesses varied less than 10%, and electrical uniformity characteristics were significantly improved.[1]

The design choice proved to be mechanically reliable. The cleaning procedure between runs originally included a lengthy and concentrated HF acid etch. This was detrimental to the spindle and retainer clearances, and the table loosened slightly. The etch time and acid concentration were reduced, and the table continued to work satisfactorily.

Conclusions

In this paper, the design, fabrication and operation of a quartz rotary-table substrate holder for exploratory experimental InP epitaxial growth was described. In addition to the successful operation of the table, a significant factor of this presentation is the demonstration that using readily available resources, somewhat unconventional but adequate methods, and the glassblower's creative potential, most technical challenges can be met.

Reference

[1] P. Vohl, F.J.Leonberger and F.J. O'Donnell, "Lateral Epitaxial Growth of InP over PSG Films for Oxide-confined Optical Waveguides," Electronic Materials Conference, Ft. Collins, CO, 1982.
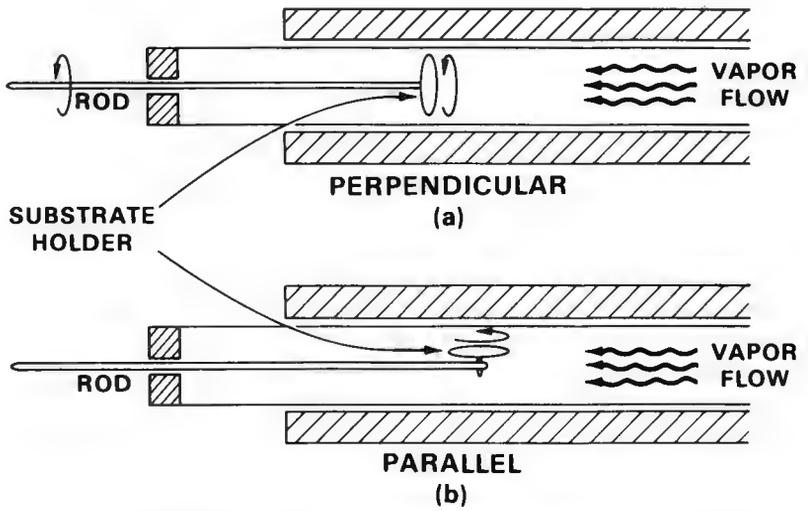
Fig. 1   Representation of Perpendicular and Parallel Vapor Flow in a
Vapor Phase Epitaxial Reactor

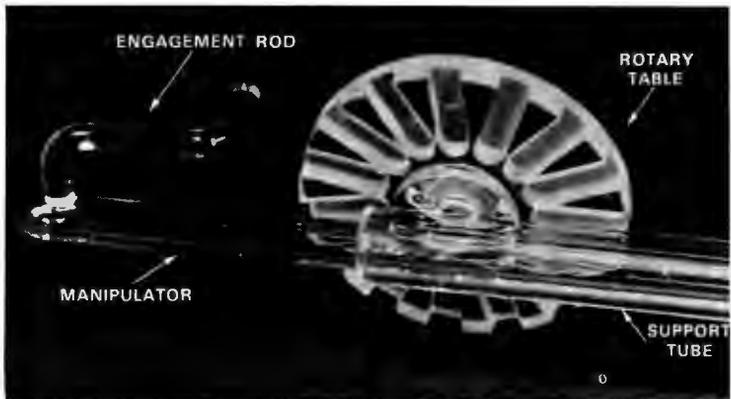Fig. 2   Entire Rotary-Table Mechanism Showing 48-inch Length

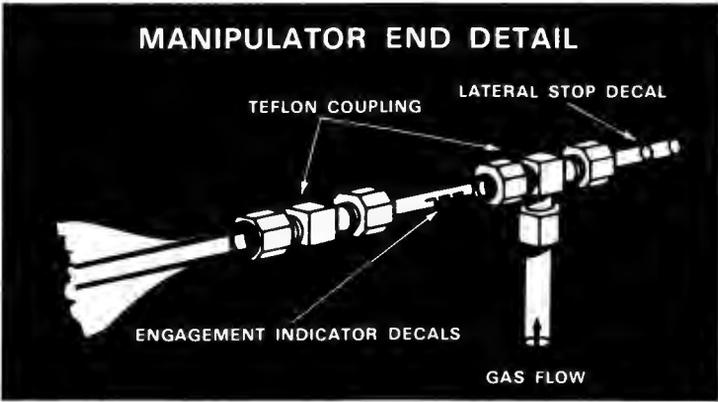Fig. 3   Quartz Rotary-Table with Major Parts Labeled

17

Fig. 4   Manipulator End of Support Tube Showing Decal Markers

Fig. 5   Quartz Disc Being Ground in Milling Machine

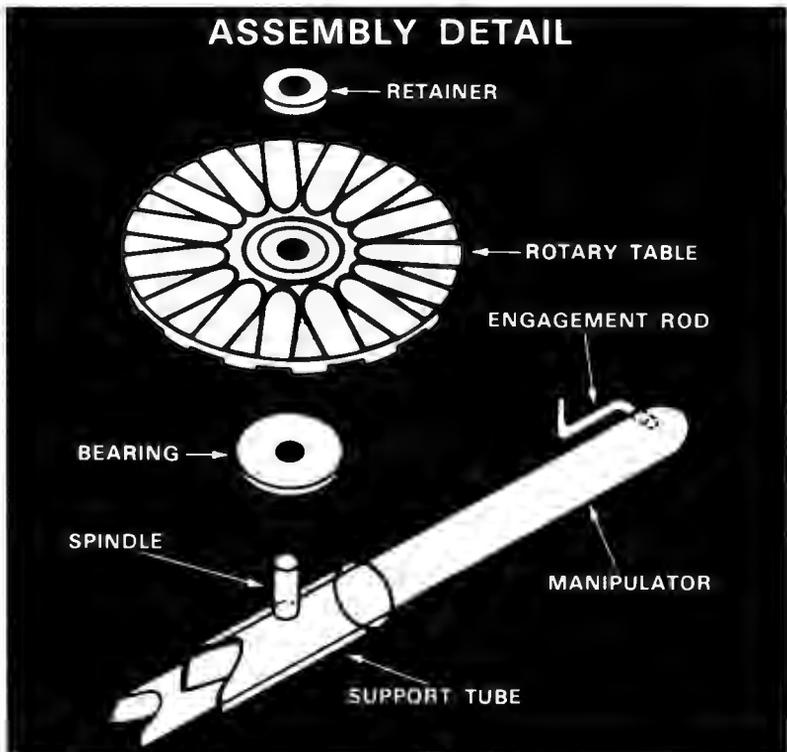*Fig. 6   Disc with Grinding Completed*

ASSEMBLY DETAIL

RETAINER

ROTARY TABLE

ENGAGEMENT ROD

BEARING

MANIPULATOR

SPINDLE

SUPPORT TUBE

Fig. 7   Blow-up of Table for Describing Fabrication Procedure
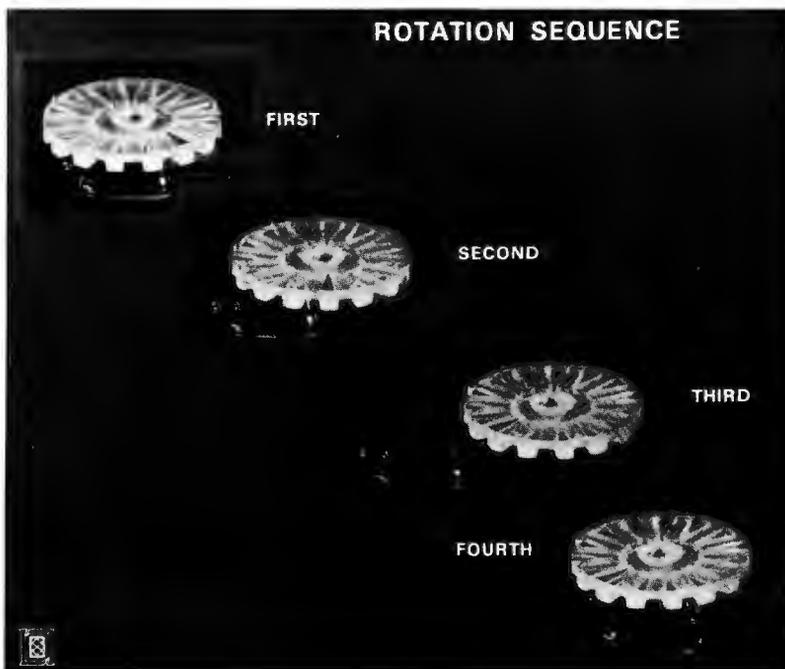
Fig. 8   4-Picture Composite Showing Rotation Sequence

Fig. 9   Rotary-Table Holding Substrate Inside Reaction Tube

20

SEALING GLASS TO 99.9% ALUMINA

Foster E. Tennant

Scientific Glass Blower
Organization 7472-3
505/844-7740
Sandia National Laboratories
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Abstract

Special applications require glass seals to furnace tubes and other apparatus that will operate at higher temperatures and vacuum than those assembled from fused silica and mullite. One example is a furnace tube that will withstand 1300°C under a vacuum of 10^{-7} torr with as little outgassing as possible. The material chosen to meet these requirements was alumina (99.9% Al_2O_3) because of its purity and ability to withstand high temperatures. Because of the contraction coefficient of this material it was difficult to find a suitable sealing glass. Glass blowers are prone to consider coefficient of expansion and forget the hysteresis during contraction of a given material when sealing glasses to them. Owens-Illinois glass N51-A was chosen as a sealing glass to alumina because of its similar contraction behavior.

A full radius was ground on the end of the alumina tube. Then the alumina tube and N51-A glass were placed in the lathe and set up to allow the glassblower to be able to blow when making the seal. A Litton annealing burner was placed under the alumina tube and a gas only flame turned on, allowing it to blacken the tube. After about 3 minutes, air was gradually added until the carbon was burned off. A National hand torch equipped with a size N-2 tip mounted in the back of the lathe was turned on with gas and enough air to produce a bushy flame. Air was gradually added to both burners until the alumina tube showed an orange color (750-800°C). The glass was then brought up to the flame so as to preheat it and allow it to be shaped so that the I.D. was about 4 mm larger than the O.D. of the alumina tube. Then the glass was brought over the alumina and, with a regular sealing flame on a 6-jet Litton lathe burner, the glass was heated and paddled down on the alumina. After the glass was sealed to the alumina, the lathe burner was moved out about 3/8"

and the glass was flame cut. The glass was paddled around the end of the tube which sealed it inside; the glass was then sealed to this end of the tube, fabricating a housekeeper seal. The N51-A glass seals very well to Corning's 7052 glass which in turn seals to Kovar and hence to a Varian flange that can be readily connected to a vacuum system.

It should be emphasized that extreme care must be taken that the sharp portion of the lathe burner flame or the paddle does not touch the alumina tube. Also, both the Litton annealing burner and the National hand torch are kept on during the sealing process.

COMBINING SCIENTIFIC METHODS AND TECHNIQUES TO FORM ARTISTIC DESIGNS

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The technique of lampworking was originally developed to make small beads and religious figures. One of the larger pieces of lampworking of the 19th century is a lace ship in the Albert and Victoria Museum in London. Up until quite recently, artistic lampworkers used soft glass and the techniques of weaving to achieve some degree of size to their pieces. The ability to utilize ring seals, massive solid shapes and implement such machines as the glass lathe were very useful for the artistic lampworker.

As a scientific and occasional ornamental glass blower, I have encountered many instances where a refined scientific glassblower insists that one must be an artist as well as a glassblower to create artistic pieces. This is not so. An artist is a person skilled or versed in one of the fine arts. Therefore, all glassblowers are artists whether it be scientific or ornamental. By applying these methods I am going to show, any glassblower will be able to create beautiful pieces of art work. Even those of you who have mastered both forms of glassblowing can benefit from these ideas.

There is a large variety of artistic glassblowing around us today. A lot of the artistic glass, with the exception of large solid glass figures, and woven glass, employs many similar techniques as in much of the scientific work we do today. Some of these pieces include a variety of lathe techniques as well.
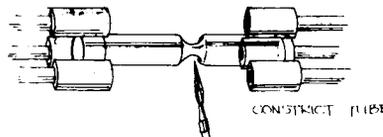
I have drawn some illustrations and put together a few slides which illustrate some of the methods I am talking about.
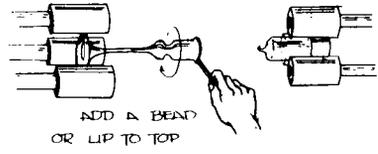
Project I. Candleholder

This particular candleholder employs quite a few scientific techniques. 1) First, a constriction is made for the top portion of the candleholder. 2) Then the tube is heated directly behind the constriction with a large bushy fire. Before the tube is molten hot it is stretched to its desirable length. 3) The bottom part of the holder is flared out such that it
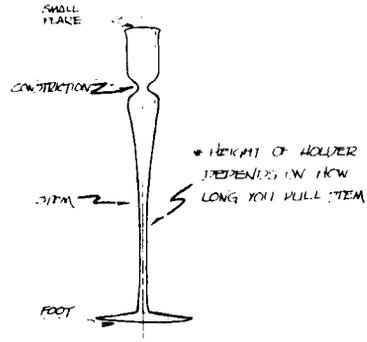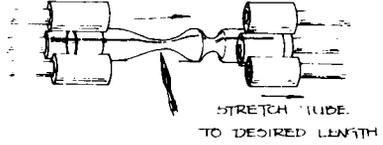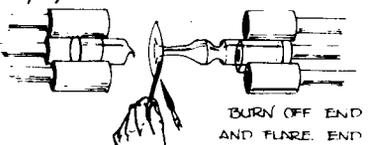
CANDLE HOLDER

1) CONSTRUCTION

CONSTRICT TUBE

2) STEM

STRETCH TUBE.
TO DESIRED LENGTH

3) FOOT

BURN OFF END
AND FLARE. END

4) SMALL FLARE

ADD A BEAD
OR LIP TO TOP

SMALL
FLARE

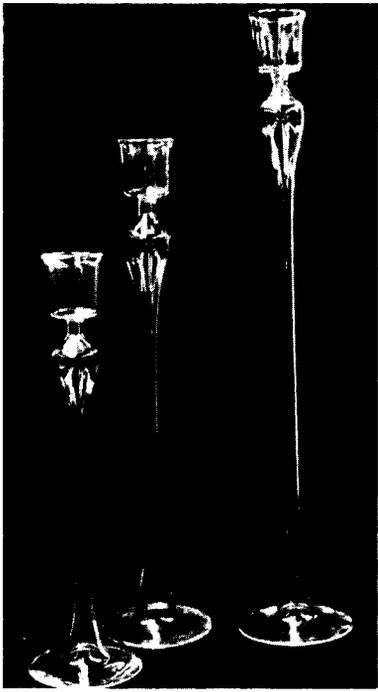CONSTRICTION

STEM

FOOT

* HEIGHT OF HOLDER
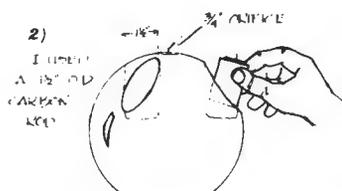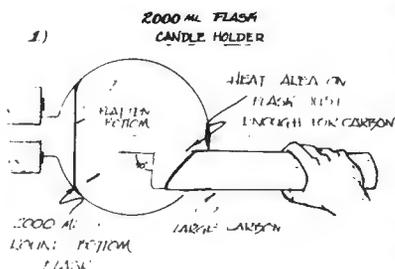DEPENDS ON HOW
LONG YOU PULL STEM

is proportional to the height of the candleholder. 4) It is up to the glass-blower to use his imagination to finish the top part of the holder. Frills, a bead, or even a cobalt ring can be added for finishing touches. White candles accent the glass very nicely.
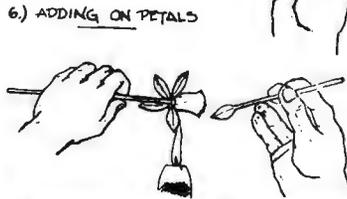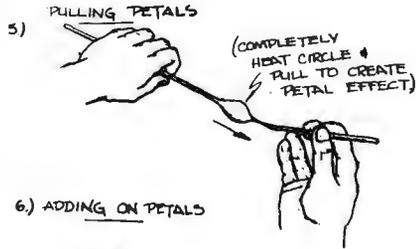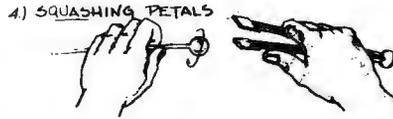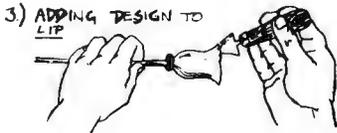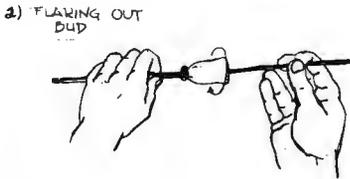
Project II. 2000ml Flask

Another unusual candleholder is this one derived from a 2000ml round bottom flask. The fabrication of this unusual candleholder is fairly simple. By using a 1½" carbon rod, indentions are made every 120° or 3 evenly spaced apart. Let me emphasize the importance of making the indentions deep enough the first time. It is extremely difficult to go back and make new indentions without making the glass paper thin in places. After the indentions are made, a ¾" hole is blown in the top center. The holder is then reversed, the neck pulled off, and the bottom of the flask flattened. The size of the carbon rod is not critical, but the 1½" indentions are perfect for accompanying a small white butter server candle. Again, the white candles enhance the glass but the color is not critical. Colored water or perhaps a flower in the top orifice can be added for variety.

25

DAFFADIL

1.) SEALING IN RINGSEAL

DOUBLE MARIA

28mm TUBING

2.) FLARING OUT BUD

3.) ADDING DESIGN TO LIP

4.) SQUASHING PETALS

5.) PULLING PETALS

(COMPLETELY HEAT CIRCLE & PULL TO CREATE PETAL EFFECT)

6.) ADDING ON PETALS
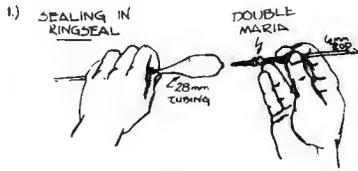
Project III. Daffodil

When fabricating a daffodil, we first start by using the standard ring seal method. This is used for attaching the stem to the bud of the flower. The stem inside of the glass represents the pistol or the inner filament of the bud. Next the tube is pulled off just above the tip of the inner filament, and flared out to resemble a bell. Frills are added to the top for design. This next step requires a pair of squashers for the petals. After the petals are squashed and pulled, they are sealed on to a second stem just below the bud of the daffodil.

Project IV. Birdcage

The final piece of work is a large glass birdcage. A bit more complex than the others, and employs a few lathe techniques.

We start by cutting several (20-24) 6mm rods to equal lengths proportional to the diameters. Also needed is another 2000ml flask. Next, a ring seal is made at the top center part of the flask. This ring seal has a hook on the inside of the flask to hang the bird by, and a loop on the outside to support the birdcage. After the ring seal is made, the flask is reversed, the neck pulled off, and the opening flared out to accommodate the 6mm rods being sealed on. These 6mm rods are held in place by using several different methods.

The simplest type of jig is to simply tape the rods around a large (6") tube. In order to prevent overlapping or congestion at the connecting point I used the old circumference equation, 2 r.

Another jig can be made by drilling ¼" or 6mm holes, in a 1-2 inch thick circular cut piece of wood.

The ultimate jig is one milled out of alluminum, with nylon set screws. The screws are nylon to prevent crushing the glass rod ends, and also hold the rods firmly in position so they will not loosen during fabrication.

The flask and 6mm rods are chucked into the lathe to be sealed together. Once they are sealed, the rods can be heated and spun out to add design to the birdcage. I used a large, bushy hydrogen oxygen flame to prevent sharp curves. Once the rods are sealed on to the flask, the jig is removed from the bottom portion of the rods. A tube is then sealed on to the rods and pulled off about 3-4" from the seal. A design can be added at the bottom to enhance the birdcage.

Now that the scientific techniques of the birdcage are completed, we are ready to start on the bird. Let your imagination go, preferably making a quick sketch of how you want it to look. I chose a long slender bird with a lengthy tail to be proportionate to the cage. I also sandblasted the bird to prevent the cage from dominating over the bird inside.

I have presented several ideas and techniques employing scientific approaches. By using your experience, ability and imagination, there is no limit to what you can design and create.

28
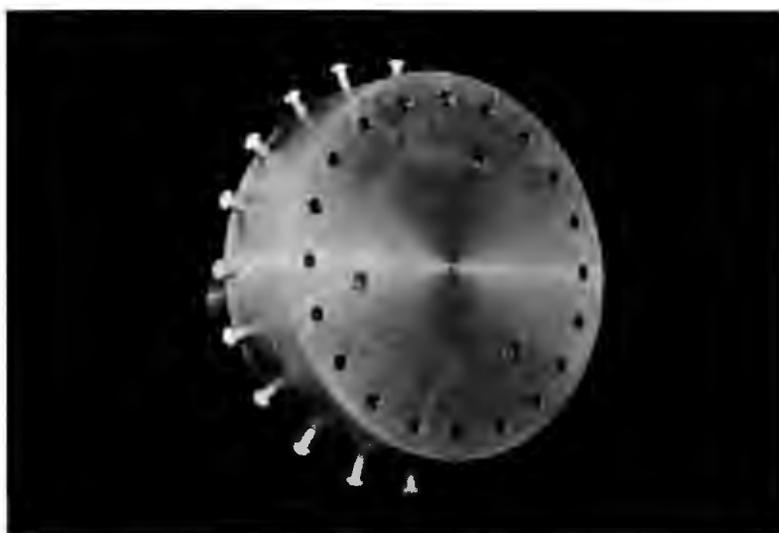
JIGS FOR BIRDCAGE
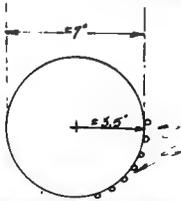
= 7" TUBE
= 2πr CIRCUMFERENCE
2(3.14)3.5 = 21.98
≈ 22" = 1" APART
USING 6mm RODS

EVEN A CIRCULAR
CUT PIECE OF
WOOD WITH 6mm
HOLES SPACED
EVENLY APART
MAKES A NICE
JIG.

30

ARMOLLEX

Joseph S. Gregar

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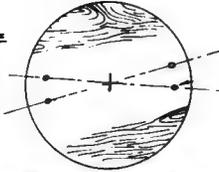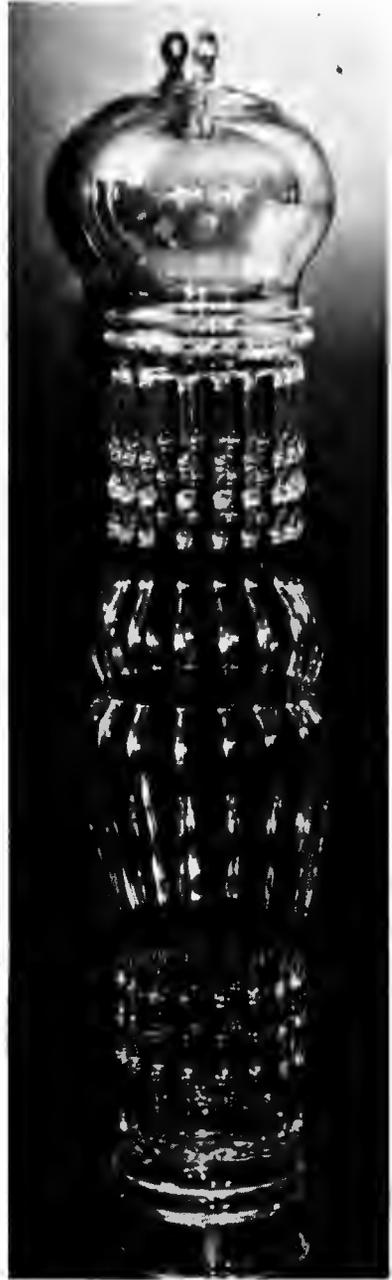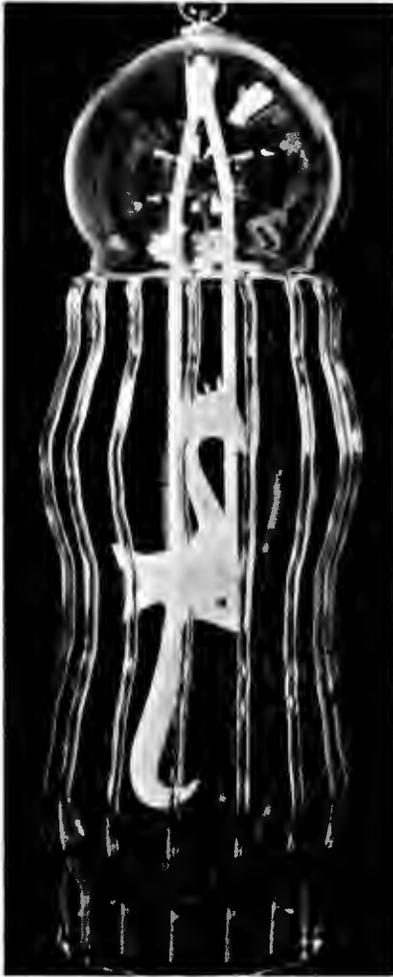*An apparatus for Solvent Extraction Kinetic Measurement.*

Armollex is a new forced-convection constant-interfacial-area stirred cell, suitable for studying kinetics of liquid-liquid extraction of metal cations. The cell allows the stirring of organic aqueous phase solutions in contact with each other at very high RPM without breakup. As the stir rate is increased the stagnant organic and aqueous layers at the interface diminish in thickness.

Constant interfacial area stirred cells, also called Lewis cells,[1] have been used to obtain solvent extraction kinetic data in experiments, however this kind of cell does not permit efficient stirring of the regions close to the liquid-liquid interface. In order to overcome this disadvantage, Nitsch and Hillekamp have developed an apparatus with cylindrical inserts containing screens at the ends closest to the interface and impellers with blades inclined 45° to the surface. This arrangement produced a very efficient forced convection and allowed one to stir the aqueous and organic phases at very high speeds without breaking the interface. The original apparatus was designed to handle rather large volumes (about 1 L). The experiments took long times and the concentrations of solute were determined by discontinuous withdrawals.

However, a continuous monitoring of the concentration variations and the use of small volumes are highly desirable to reduce the amount of experimental work required and to allow the use of organic reagents often available only in limited quantities.

This new cell retains the efficient hydrodynamic features described in reference 1 and permits the use of small volumes (100 ml) and a continuous monitoring of the concentration variations. The new forced convection, constant interfacial area stirred cell, where concentration variations are monitored by radiometric flow counting is called **ARMOLLEX**, an acronym for **Ar**gonne **Mo**dified **Le**wis cell for **L**iquid-liquid **Ex**traction kinetic measurements.[2]

32

The objective is to extract the metal cation from aqueous to organic phase and record the data. The reaction time should be kinetic limited. If you have a thick stagnant interfacial zone and not a thin film interface this will make the experiment diffusion limited rather than kinetic limited. The benefit of this cell is that it can produce a very thin interface layer between the two phases and enables high stir rates up to 800 RPM without breakup of the interface layer allowing accurate studies of kinetics of extraction.

Testing of the ARMOLLEX apparatus leads to the conclusion that in the apparatus a wide range of hydrodynamic conditions exist where the thickness of the diffusion films becomes small enough to make diffusional processes negligible with respect to the rate of the interfacial chemical reactions, or (alternatively) a constant thickness of the diffusion film, independent of the stirring speed, is reached at 350 RPM.

The construction of the ARMOLLEX cell is basically a temperature controlled chamber with cylindrical inserts. These inserts are 28mm OD supported by 4 mm rods, symmetrically inside of 45mm OD tubing. The chamber is then enclosed by a water jacket for temperature control. After annealing, the end seals of the jacket are ground flat for a grease seal between the chamber and the caps. To enhance the "Fluid Mechanics" of this cell, stainless steel screens were mounted to the inside ends of the 28mm cylindrical inserts. This insures that the pressure of the flow is evenly dispersed across the interfacial area. It also acts as a stabilizer for the interface. The screens were mounted with tiny glass rivets and must be parallel to each other. Slots were cut into the screens to allow passage of circulating and filling tubes. The caps were made from 45mm OD tubing and flanged. These flanges were ground to mate with the cell body. The top cap has an Ace stirring bearing and three luer joints for the filling and flow tubes. The bottom has a similar stirring bearing and a stopcock for draining the system. The propellers were hand formed and attached to Ace ground stirring shafts.

A total of five ARMOLLEX cells were built and used for this research experiment, all with great success. It was a great honor working with the scientists of Argonne National Laboratory, Chemistry Division, and very gratifying to present it to the American Scientific Glassblowers Society.

Acknowledgements

References

[1] W. Nitsch and K. Hillekamp, Chem. Zts. 96, 254 (1972).

[2] P.R. Danesi, C. Cianetti, E.P. Horwitz and H. Diamond.

ARMOLLEX: An apparatus for solvent Extraction Kinetic Measurement and Separation Science and Technology, 17(7) pp. 961-968, 1982
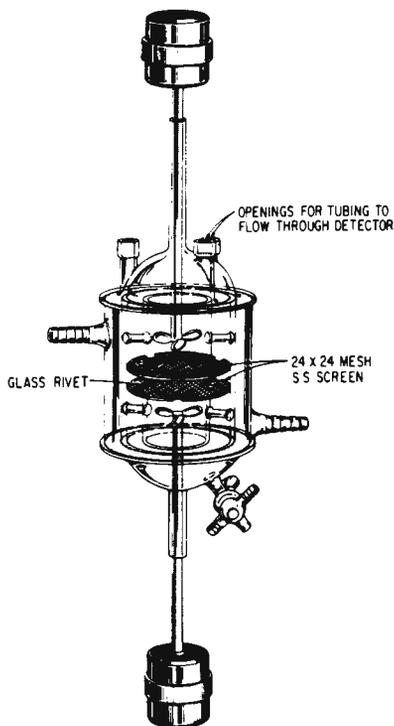
Figure 1.
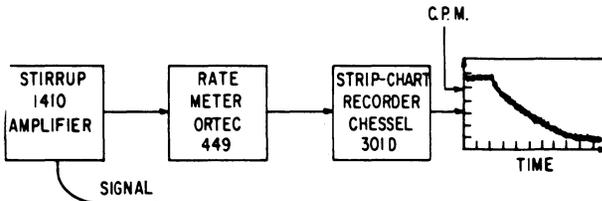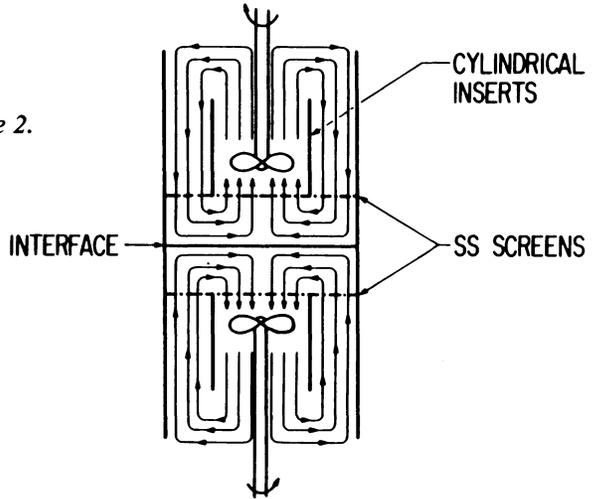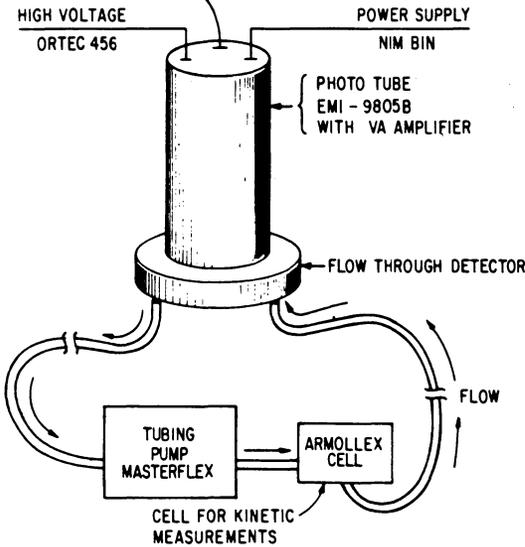
OPENINGS FOR TUBING TO FLOW THROUGH DETECTOR

24 x 24 MESH S S SCREEN

GLASS RIVET

34

Figure 2.

INTERFACE

CYLINDRICAL
INSERTS

SS SCREENS

| STIRRUP 1410 AMPLIFIER | RATE METER ORTEC 449 | STRIP-CHART RECORDER CHESSEL 301D |

C.P.M.

TIME

SIGNAL

HIGH VOLTAGE
ORTEC 456

POWER SUPPLY
NIM BIN

Figure 3.

PHOTO TUBE
EMI - 9805B
WITH VA AMPLIFIER

FLOW THROUGH DETECTOR

FLOW

TUBING PUMP MASTERFLEX

ARMOLLEX CELL

CELL FOR KINETIC
MEASUREMENTS

35

EFFECTIVENESS OF EYE GLASS TO REMOVE DAMAGING RADIATION FROM GAS FLAMES

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Introduction

The process of sight requires the interaction of light with the eye. The average person in his/her life is exposed to "normal" levels of visible light from the sun and artificial light sources. Both these sources can contain a small amount of ultraviolet (UV) light which can cause damage to the lens and cornea of the eye. For example, many scientists believe that senile cataracts are formed by the interaction of UV light with the lens.

A glassblower, as part of his/her everyday job, can be exposed to higher than normal intensities of visible and UV light from the flames with which he/she works. This paper will discuss the types of damaging radiation a glassblower can be exposed to in his/her everyday job, the types of damage this radiation can cause to the eye, and the effectiveness of presently available eye glass to screen damaging radiation.

Types of Photochemical Damage

UV irradiation below about 300 nm can cause molecules that absorb this light to ionize which leave very reactive free radicals. These free radicals cause damage to the DNA, RNA, protein and membrane of the cell. Some researchers believe that senile cataracts are caused by the chemical reaction of free radicals[1][2]. Since light of these wavelength con-

36

tain enough energy to cause ionization of some biomolecules, the production of free radicals requires only one photon of light.

In contrast to ionizing radiation, light with wavelengths greater than 300 nm do not usually contain enough energy to ionize most biomolecules. However, this light can be damaging at high intensity. The most damaging feature of high intensity radiation is due to thermal damage. Molecules that absorb light usually only stay excited for a short period of time (nanoseconds to milliseconds). Very often the excess energy is lost as heat. Under normal conditions this heat is dispersed rapidly but under conditions of high intensity, radiation heating is more rapid than dispersion, which results in burns.

Types of Radiations Emitted by Flames Used by Glassblowers

Most glassblowers use H_2 flames for quartz glass and methane, propane or acetylene flames for pyrex. These flames alone contain damaging radiation. H_2/air or H_2/O_2 flames spectrum contain a number of emission peaks in the UV due to the OH radical which is created in the flame. The major peaks are at 261, 268, 282, 288, 295, 302, 306, 312, 319, 326, 343 and 350 nm. The 302 nm peak is the most intense followed by the 288 and 295 nm peak[3].

Organic flames (methane, propane and acetylene) also contain emission peaks due to the OH radical. Although the peaks due to the OH radical in these flames are not as intense as in the H_2 flames, organic flames also contain emission peaks from the CH, CHO and C_2 radicals. The strongest emission peaks from the CH radical is at 431 nm with weaker peaks at 314 and 390 nm[4]. The major peaks from the C_2 radical are at 434, 517 and 564 nm[5]. The CHO radical emission peaks are relatively weak with their major peaks between 300 and 390 nms[6].

In addition to the radical emission peaks, glassblowers are exposed to light emitted from the glasses they use. The major emission peaks that come from Pyrex glass are due to Na. There are minor emission peaks from other elements in the glass such as Si. The Na-D-line at 589 nm is by far the most intense peak of this type. Na also has emission lines at 285 and 300 nm[7]. Si emission peaks are centered around 252 nm and are usually masked by the OH radical peaks of the flame.

Working with quartz glass adds an additional problem. Although quartz contains little or no Na the higher temperatures required to work with this type of glass causes an intense blackbody emission over most of

the visible spectrum. The overall intensity of this blackbody radiation is much higher than that of the Na-D-line in Pyrex glass.

Types of Damage that can Occur to the Eye

The three areas of the eye most susceptible to photochemical damage are the cornea, lens, and retina. It has been shown that near UV and low wavelength visible light can cause the cell membrane in the cornea to break down. The lower the wavelength the more damaging the radiation. For example, light of 320 nm is 13 times more damaging than the light of 390 nm[8]. The most damaging radiation to the cornea is that of less than 293 nm because it absorbs all the radiation of these wavelengths. Fortunately, the cornea has repair mechanisms that can fix this damage as long as it is not too extensive. Actually, the cornea protects the lens and retina from most of the ionizing radiation by absorbing all wavelengths less than 293 nm.

The lens of the eye is more susceptible to permanent damage than the cornea or retina because it is actually dead. Only the intersurface contains living cells, thus the repair mechanisms are not as effective as that of the cornea. Once the cells in the lens are formed they are never replaced. The center of the lens of an adult contain the same cells that made up his/her lens at birth[9].

It is thought that most of the photochemical damage that occurs to the lens is initiated by the absorption of light by the amino acid tryptophan[2]. Tryptophan absorbs light of 305 nm or less, thus it is protected from much of the damaging radiation by the cornea. However, light between 293 and 305 nm does pass through the cornea and into the lens causing a slow yellowing of the lens due to photo-oxidation of tryptophan. These photoproducts of tryptophan absorb light between 300 and 360 nm making the lens more susceptible to photochemical damage[9]. Thus, the most damaging radiation to the lens are those between 293 and 360 nm.

The retina is protected from most of the ionizing radiation because it is absorbed by the cornea and the lens. The most damaging radiation to the retina is not from flames but the high intensity visible radiation of the Na-D-Line or from quartz glass. This radiation can burn the retina and lead to permanent damage. Although the shorter the wavelength of the light the greater the damage, all wavelengths of visible light are close to equal in their efficiency to cause thermal damage to the retina.

Effectiveness of Commercially Available Glasses to Remove Damaging Radiation

As can be seen from figure 1 and 2 both didymium and Bausch and Lomb (B + L) G-33 glasses have little or no transmittance below 320 nm. This removes the ionizing radiation and protects the lens and cornea from the wavelength of light most damaging to them. Almost any type of normal eye glass will remove these radiations, even plastic lab glasses. The didymium and B + L G-33 glasses also have very low transmittance between 570-590 nm ($\sim$ .2%) removing most of the Na-D-line that can burn the retina. There is also relatively low transmittance at around 440, 480, and 540 nm removing much of the emission from the C_2 and CH radicals. In general, the B + L G-33 glasses have lower transmittance than the didymium lens, thus offering more protection.

The glasses used for work with quartz glass are the same as those used by welders. As can be seen from figure 3 these glasses have very low transmittance in all parts of the visible and UV spectrum. There is a slightly higher transmittance between 500 and 600 nm. This is mainly to allow the wearer to see what he/she is doing. The eye is the most sensitive to light in this range so that this low transmittance allows enough light through for reasonable visibility.

Conclusion

There are a number of sources of damaging radiation that come from flames and glasses that glassblowers use. This radiation can cause permanent damage to the cornea, lens, and retina of the eye. Commercially available eye glasses are effective in removing this radiation and protecting the eye.

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Figure 1. Percent transmittance of eye/glass containing didymium lens. Lines at top denote position of major emmission lines due to OH, C_2, CH and CHO radicals in gas flames. Line at 589 is due to Na emission from Pyrex glass used in flame and is at least 10 times more intense than show.

Figure 2. Percent transmittance of eye/glass containing Bausch and Lomb G-33 lens. Lines at top denote position of major emission lines due to OH, C_2, CH and CHO radicals in gas flames. Line 589 is due to Na emissions from Pyrex glass used in flame and is at least 10 times more intense than show.

42
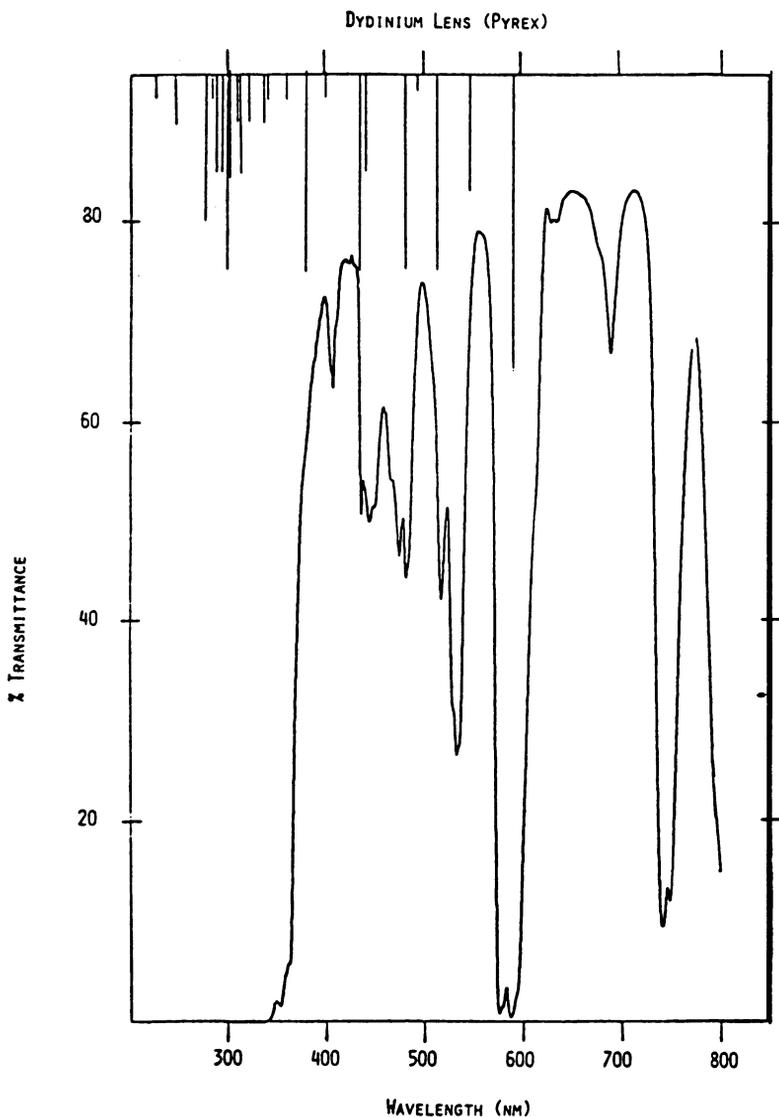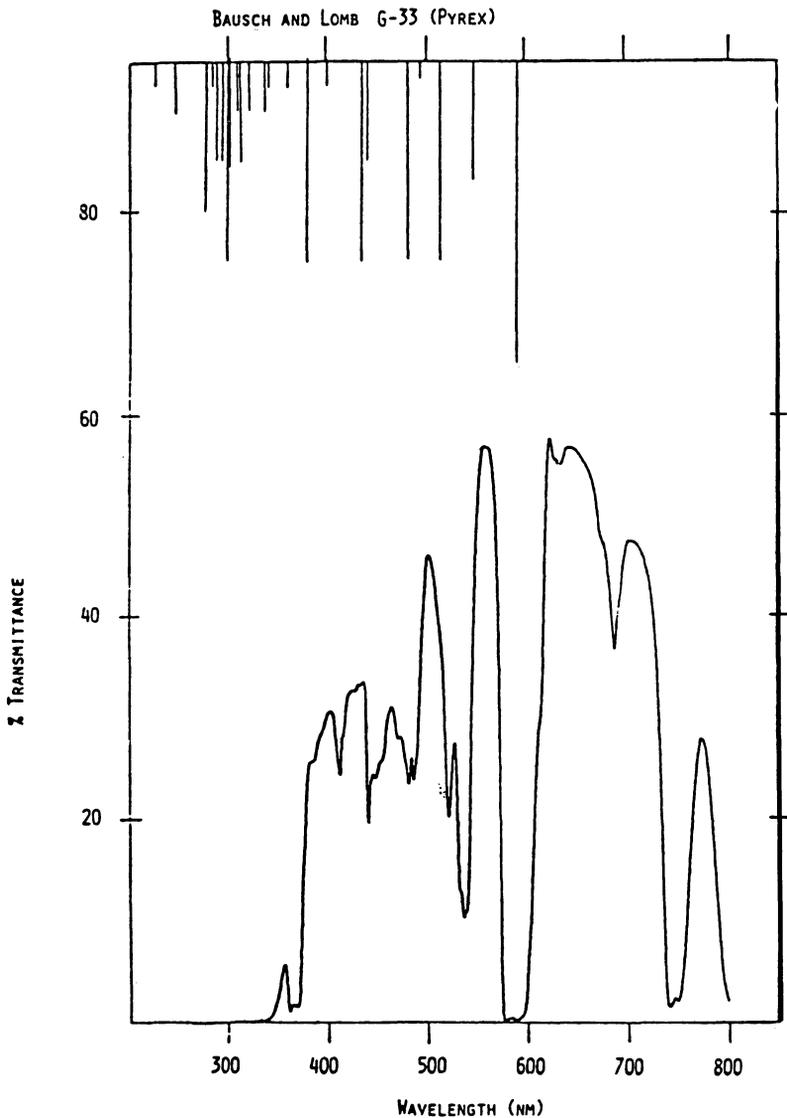
WELDER'S GLASSES (QUARTZ)
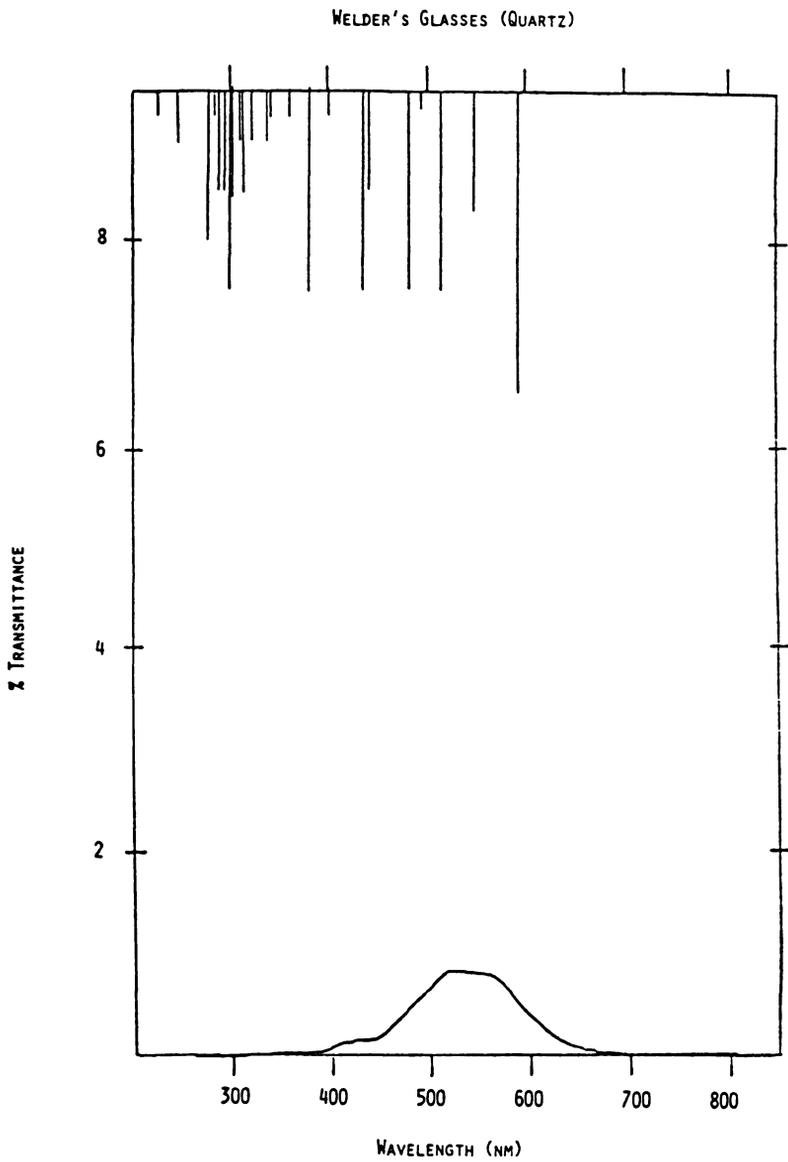
% TRANSMITTANCE

WAVELENGTH (NM)

Figure 3.- Percent transmittance of eye/glass used for quartz work. Lines at top denote position of major emission lines due to OH, C2, CH and CHO radicals in gas flames.

43

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57

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