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Stressed Glass Technology for Actuators and Removable Barrier Applications

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ABSTRACT

There are commercial and military applications in which a material needs to serve as a barrier that must subsequently be removed. In many cases it is desirable that once the barrier has served its function that it then be reduced to small pieces. For example, in pipelines and in downhole drilling applications, valves are needed to function as barriers that can sustain high pressures. Later the valves must be removed and essentially disappear or be rendered to such a small size that they do not interfere with the functioning of other equipment. Military applications include covers on missile silos or launch vehicles. Other applications might require that a component be used once as an actuator or for passive energy storage, and then be irreversibly removed, again so as not to interfere with the function or motion of other parts of the device. Brittle materials, especially those that are very strong, or are pre-stressed, are ideal candidates for these applications.

Stressed glass can be produced in different sizes and shapes and the level of strength and pre-stress, both of which control the fragmentation, can be manipulated by varying the processing. Stressed glass can be engineered to fracture predictably at a specific stress level. Controlling the central tension allows the fragment size to be specified. The energy that is stored in the residual stress profile that results from ion exchange or thermal tempering processes can be harnessed to drive fragmentation of the component

once it has been deliberately fractured. Energy can also be stored in the glass by mechanical loading. Energy from both of these sources can be released either to perform useful work or to initiate another reaction. Once the stressed glass has been used as a barrier or actuator it can never be “used” again because it fragments into many small unrecognizable pieces during the actuation. Under some circumstances it will interfere with the motion or functioning of other parts of a device.

Our approach was to use stressed glass to develop capabilities for making components that can be used as barriers, as actuating devices that passively store energy, or as a mechanical weaklink that is destroyed by some critical shock or crush load. The objective of this project was to develop one or more prototype devices using stressed glass technology and demonstrate their potential for applications of interest. This work is intended to provide critical information and technologies for Sandia's NP&A and MT&A customers, and is relevant to commercial applications for these same materials. Most of the studies in this project were conducted using the Corning 0317 sodium aluminosilicate glass composition.

Stressed Glass Technology for Actuators and Removable Barrier Applications

Introduction

Stressed glass is favored for architectural and automobile glazing applications because of its high strength and fracture properties. Stressed glass has strengths that are typically three to ten times higher than the strength of annealed soda lime silicate glass. As a result stressed glass can sustain much higher loadings (wind, impact, flexural) than annealed glass and it has improved contact damage resistance. Another advantage of stressed glass is that when it fractures the fragments are small, regular shape and size fragments that are much less lethal with respect to personnel safety.

The following section provides information about the processing and mechanical properties of stressed glass, its energy storage capacity, and other technological information relevant to a variety of applications including high consequence hardware. Section 2 describes the project objectives of this program and applications and requirements. Section 3 describes approaches for enhancing the fracture of stressed glass because its high strength makes it too difficult to break for some applications that require fracture or require the release of its stored energy. Section 4 describes preliminary experiments conducted to evaluate ways for making geometries more complex than the simple plates that the glass is generally available as.

1. Stressed Glass Technology

1.1 Stressed Glass Processing and Mechanical Properties

Stressed glass is made using two different processes. The first, which is used for the majority of the glass for architectural and automobile applications, is a thermal tempering process in which the glass is rapidly cooled using air or liquid jets.¹ Because of the difference in cooling rates between the surface and interior of the glass, the surface is under compression and the interior is in tension when the glass reaches room temperature. The magnitude of the surface compression is twice the magnitude of the interior tension. The compressive stresses achieved in thermal tempering are usually in the range of 60-200 MPa (29-44 ksi), which approximately doubles the strength of the base glass. The level of internal tension is in the range of 30-100 MPa. At these moderate values of internal tension, the fragments from broken tempered glass tend to range from 3-10 mm in size, depending on the original thickness of the glass plate.²

The second process for making stressed glass uses an exchange of large ions for small ions at the glass surface.^{3,4} This exchange is a diffusion-controlled process that occurs faster at elevated temperatures. The presence of the larger ions in spaces in the glass structure previously occupied by small ions produces residual surface compression. This is balanced by residual internal tension. The exchange depth and the number of ions that can be replaced by larger ions determine the compressive layer depth and the magnitudes of the compressive and tensile stresses (Fig. 1). The depth of the exchanged layer can be controlled by the time

and temperature of the exchange process. Both the strength and the fragmentation can be controlled by controlling the time and temperature of the ion exchange process. For the same ion-exchange treatment a thinner piece of glass will develop a much higher internal tension than a thicker piece, with correspondingly smaller fragments when it fails. Using chemical ion-exchange processes it is possible to achieve very high values of both the surface compression and internal tension. For Corning 0317 glass (a sodium aluminosilicate composition) surface compressions that approach 1000 MPa and internal tensions of >200 MPa have been achieved for a K^+ for Na^+ exchange.⁵ For these very high values of central tension, the fragments are sub-millimeter in size as shown in Fig. 2a.

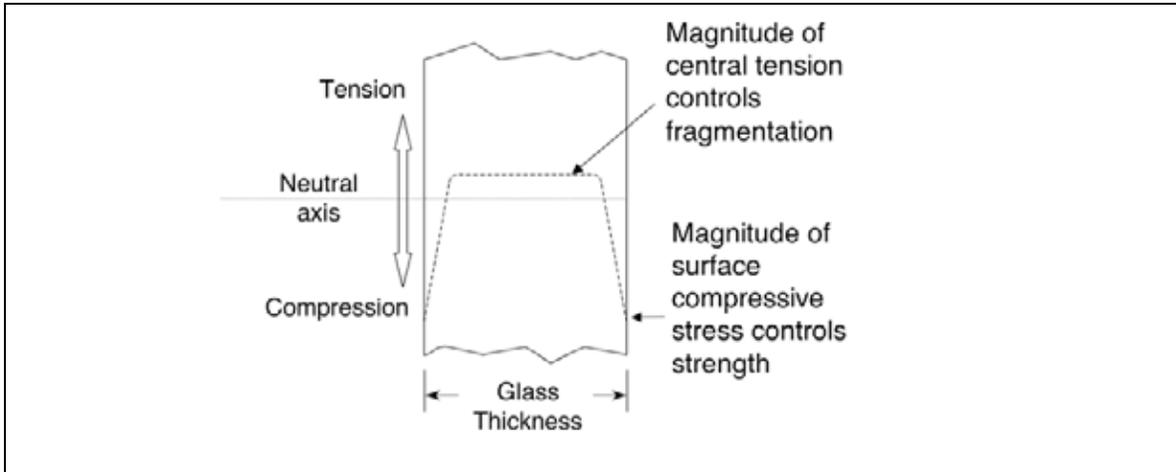


Fig. 1 Schematic of residual stress profile through the thickness of ion-exchanged glass.

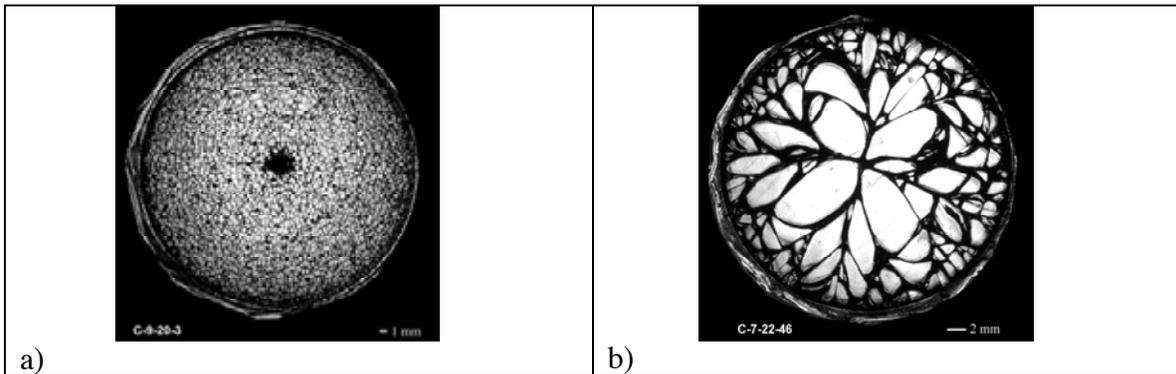


Fig. 2 Optical photographs of fragmented ion-exchanged Corning 0317 stressed glass disks. Fracture and fragmentation of the 1" diameter disks were initiated at the center of the disk face with a sharp-tipped diamond indenter. The disks were taped to hold the fragments together. a) glass with high residual tension b) glass with moderate level of residual tension

The glass used in most of our studies has been Corning 0317, a sodium aluminosilicate composition optimized for ion exchange. It is available as 1.8 or 2.2 mm thick plates. The disks' surface was left in the as received condition; no additional polishing or grinding was used unless thinner glass samples were used. Samples were exchanged in laboratory grade KNO_3 at temperatures from 400-550°C.

Regular ion-exchanged glass and thermally tempered stress glass have the compressive stress maximum right at the surface. The Engineered Stress Profile (ESP) process for making stressed glass uses a two-step ion exchange.⁶ During the second step some of the large ions introduced in the first exchange step are removed, partially relieving the surface stress and producing a compressive stress maximum below the surface. In ion-exchanged glass with this type of stress profile the strength distribution is very narrow compared to that of regular glasses and ceramics. A typical glass has a low Weibull modulus, e.g., $m=5-10$. ESP glass has Weibull modulus values as high as 60, which means the distribution is very narrow. A designer's confidence in the glass's ability to survive or fail at a given stress is therefore increased significantly. In an application where the glass must sustain 80% of the average failure stress, ESP glass with $m=60$ has an only a two in one million chance of failing. In contrast, regular annealed glass has a 30% failure probability. A two-step or multi-step ion exchange process provides flexibility in terms of engineering the stress profile to optimize, strength, reliability, and fragmentation behavior for different glass compositions and applications.

Stressed glass is being used and considered for applications in which the strength, fragmentation, and reliability are all critical. For example, rupture disks made out of stressed glass were designed and made for Halliburton Energy Services for oil well casings.^{7,8,9} In this application the glass rupture disks needed to be able to sustain a specific pressure and then rupture at a narrowly defined higher pressure. Both the reliability of survival and failure of the disks were critical. Normally glasses do not have a failure stress distribution that is narrow enough to meet this reliability requirement. Through a collaboration with Penn State University and the University of Trento in Italy,^{10,11} Sandia developed and implemented the process for making Engineered Stress Profile (ESP) glass that would have the desired reliability. This process can be used for specialty compositions such as Corning 0317 glass and for the soda lime silicate (SLS) glass compositions that are used for automotive and architectural applications.¹²

1.2 Energy Storage Capacity of Stressed Glass

Energy is stored in both the interior tensile layer and in the surface compressive layer of thermally tempered or ion-exchanged glass; however, it is only the energy that is stored in the interior that is available to create fracture surfaces once the outer layer has been penetrated by a crack. The more energy that is available in the interior tensile layer, the more fracture surface that can be created, and the smaller the fragments will be. Once the crack pattern is established in the interior of the glass (all within a period of less than a millisecond) then the energy that was stored in the surface compressive layers is available to drive the fragments apart and disperse them. Calculations and measurements of the energy

that is stored in the glass due to the residual stress profile indicate that it ranges from 0.6×10^5 to $10 \times 10^5 \text{ J/m}^3$.^{13,14,15}

Another mechanism for storing energy in stressed glass takes advantage of its high strength. When a brittle material is loaded (in bending or tension), elastic strain energy is stored in the body. With glasses and other brittle materials, the amount of energy stored in the loaded sample when it fails is proportional to the square of the fracture stress, σ_f and inversely proportional to the elastic modulus (stiffness) of the material. As an example, the energy density that is available in the stressed glass, due to loading only, if we assume biaxial flexure loading (e.g., a ball pushing on a disk that is supported on a ring), ranges between 4×10^3 and $1 \times 10^5 \text{ J/m}^3$ for glasses that have strengths of 100 and 500 MPa, respectively. The benefit of using stressed glass over annealed glass for energy storage is that because of its much higher strength, the amount of energy that can be stored in it due to loading is much higher. Details about the stored energy in stressed glass due to both of these mechanisms are available.¹⁶

The total energy storage capacity in mechanically loaded stressed glass is less than the typical energy densities of $1 \times 10^7 \text{ J/m}^3$ for shape memory alloys, $1 \times 10^9 \text{ J/m}^3$ for batteries, and 10^{10} J/m^3 for lead azide at 50% of the theoretical density. Although the amount of energy is modest, it is very stable and could be used on demand to do work or to help initiate it. The stressed glass would act as an energy source that would not lose energy over long periods of time. Some fraction of the energy can be released either by unloading or breaking the glass. Configurations that could take advantage of this energy storage capability of stressed glass include a curved disk that is loaded to flatten it, a flat disk that is bent significantly and bonded to another material, or a cantilever that is deflected significantly and clamped in the deflected position. In each of these configurations the material or device that is holding the glass in its deflected position could be released to let the glass return to its unloaded position. The glass would essentially act as a loaded spring. In addition, some of the energy in the glass due to the residual stress profile (due to thermal tempering or ion exchange) could also be released under conditions in which the glass was designed to break.

1.3 Other Information Relevant to the Application of Stressed Glass

1. To allow electrical connections with other components, stressed glass can be coated with conductive materials and some of these coatings can be soldered to. Conductive coatings can also be used on stressed glass for applications that require the disruption of a critical electrical circuit. By fracturing the coated stressed glass, the resulting fragmentation and separation of the fragments disrupts the continuity of the electrical path.¹⁷

2. Stressed glass can be made with different form factors including disks, plates, and rods. Some form factors, including small diameter round cross section rods (and fibers) do not fragment completely, i.e., fragmentation of the part is not complete as shown in Fig. 3. Low aspect ratio shapes such as strips of glass will fragment. More details about this can be found.¹⁸

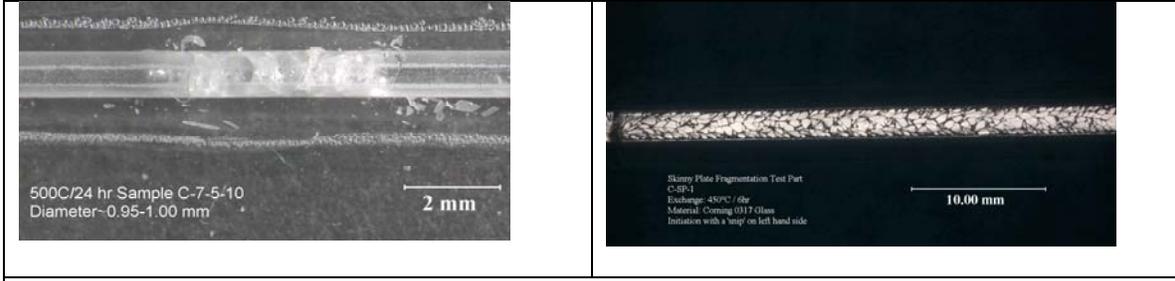


Fig. 3 a) Incomplete fragmentation of a round cross-section rod of ion-exchanged Corning 0317 glass. b) Complete fragmentation of a long thin strip of ion-exchanged Corning 0317 glass.

3. Stressed glass can be patterned using various masks (photoresist, amorphous silicon with photoresist, and metal coatings) and etches (plasma, ion mill, and wet). Fig. 4 shows one such pattern on the surface of stressed glass. The patterning can be done either before or after ion exchange, while maintaining the desired mechanical properties of the glass.

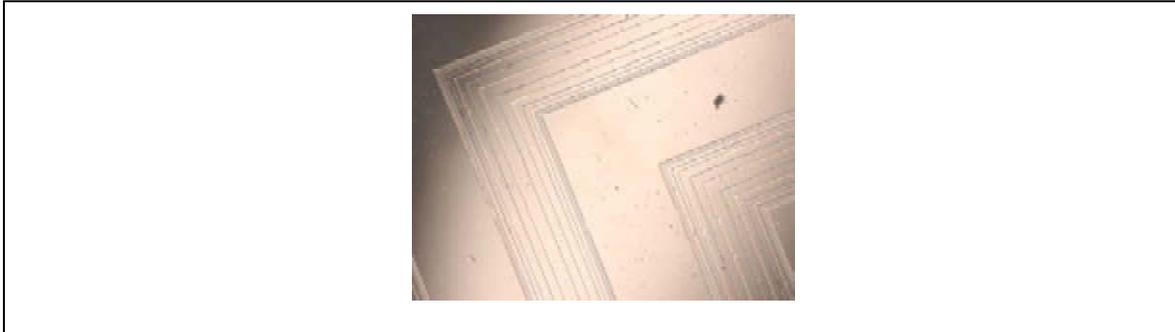


Fig. 4 Surface of stressed glass following a masking and etching process that produces a shallow series of grooves.

2. Project Objectives and Applications of Stressed Glass Technology

The unique and tailorable properties of stressed glass provide opportunities for a variety of applications. The first goal of the program was to meet with potential customers and to identify applications of interest from their own technology needs, and from their knowledge of the needs of their customers. We also conducted a literature review to determine what had been done previously for several potential applications; however, we were unable to locate any information.

The second goal of the program was to prioritize the identified applications based on needs of the customers and the feasibility of using stressed glass for the applications of interest. The feasibility of making applications out of stressed glass relates to the form and sizes of glass that are available, processing (times and temperatures), and the performance requirements. The form of the glass that is normally available from vendors is flat plates with thicknesses of several to tens of mm. Thicknesses less than the available thicknesses need to be prepared by grinding and polishing. More complex shapes require more extensive

machining, bonding individual glass pieces, or molding or shaping the glass. Because the residual stress profile in the glass is produced by an ion-exchange treatment that is a diffusion-controlled process, more complex shapes can also lead to different stress profiles in different regions of the glass.

Applications and general requirements noted by were:

- a) Hardware or barriers that are out of the way they function;
- b) Hardware that is in the way after it functions (or provides some kind of disruption to other device functions);
- c) Devices that store usable energy to do useful work or initiate another event.
- d) Materials that can sense and detect changes in the environment, including those materials that experience a change in a property of interest

A feasibility study of two prototype applications for **a)** was made for this program. For the first prototype application, simple hardware with the desired form factor was fabricated and demonstrated to function as needed. Obstacles to the use of glass for this application included the difficulty to machine the glass to the required level of detail, the sensitivity of the glass to fracture during subsequent processing and use, and problems with the corners and edges during use.

The second prototype for **a)** demonstrated the use of the stressed glass as a removable barrier between two reactive chemicals in a closed container. A clear two-piece box was designed and constructed with a microscope slide-size piece of stressed glass as a barrier between two chemicals introduced in the cavity of the box. Fracture of the stressed glass allowed mixing and reaction of the two chemicals. A photograph of this prototype is shown in Fig. 5.

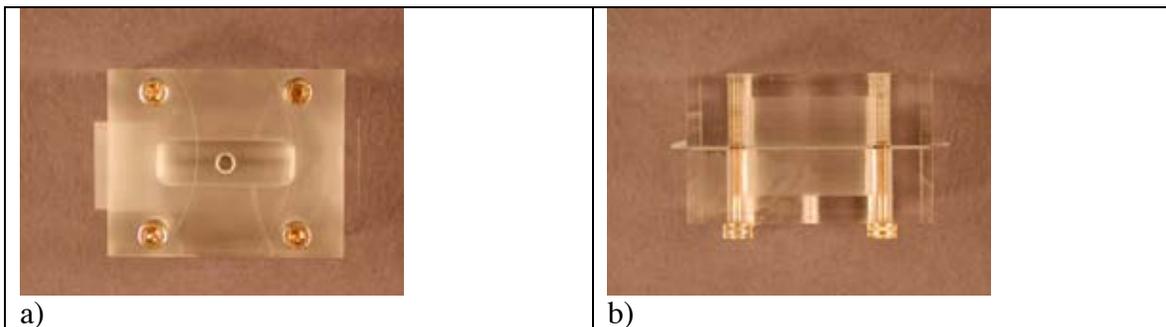


Fig. 5 a) Top and b) side views of the prototype chemical mixing box. A stressed glass plate separates the upper and lower sections of the cavity in the polycarbonate box. A hole (easily visible in top view) allows the introduction of the second chemical once the two parts of the box and the glass plate have been assembled. The hole also allows venting of any gaseous reaction products once the glass plate is broken and the chemicals mix.

There was also considerable interest in stressed glass as d) a material that can sense and indicate the occurrence of different events or exposures, or change its properties in response to one of these. An example of this is a mechanical weaklink, a material that changes irreversibly in response to a mechanical insult. The application of stressed glass as a mechanical weaklink was evaluated and reported in detail for another project that was taking place in parallel with this project.¹⁹ In this project a stressed glass, mechanical *shock* weaklink was demonstrated in a component that included a critical electrical circuit on the stressed glass. A stressed glass *crush* weaklink was demonstrated in a rolled Mylar capacitor. Following fracture of a stressed glass strip in the Mylar windings, the capacitor was no longer able to hold off charge. This application of stressed glass for nuclear weapons safety applications might also be of interest for other customers' applications.

The final objective was to expand our general understanding of the possibilities and limitations of stressed glass for various applications. This included studies to identify ways to make it easier to fracture stressed glass and evaluation of approaches for making glass parts with different form factors including enclosed volumes. More details about the last two objectives are described in the following sections.

3. Enhancement of Fracture and Fragmentation Initiation in Stressed Glass

One of the constraints on the use of stressed glass for applications in which it needs to be broken is that it is so strong after ion-exchange strengthening that it is very difficult to initiate the fracture and fragmentation process. For this project and another, several methods of changing the shape and processing of the glass were tested and demonstrated as possible approaches for making it easier to fracture.

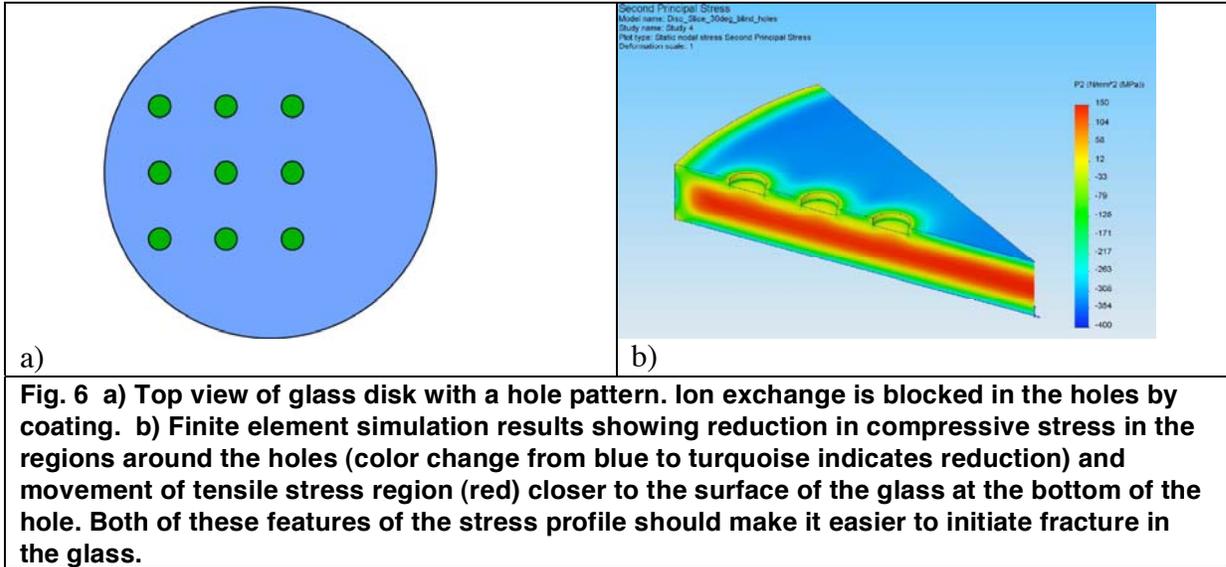
3.1 Ion-Exchange Blocking

One approach is to locally weaken the glass by using coatings on the glass surface to block exchange. Several thin films were tested that partially blocked the ion-exchange process and changed the stress profile in the glass. Further work is being done in an ongoing project to identify better coatings and procedures. Additionally finite element analyses were used to model how preventing ion exchange in localized regions changes the residual stress profile in a favorable way. This was done for the case in which a hole or a series of holes was machined into the surface of a glass surface.

During the ion exchange process all the glass inside the hole would either be:

- a) blocked from exchange
- or
- b) allowed to be exchanged, while the glass on the top surface around the hole would be blocked from exchange

Both of these configurations produce a situation in which there is a localized region in or near the hole where the compressive stress is significantly reduced from what it would be without blocking the exchange. Fig. 6 shows the situation in which the holes are blocked from being ion exchanged and how this affects the residual stress profile.



3.2 Geometrical Approach

Another approach to making it easier to break stressed glass is to reduce the amount of force required to produce a specified stress level in a localized region. This can be done by fabricating glass samples with regions with small cross-sections or regions that have high stress risers due to the geometry and applied loading. One approach that showed some success was to bond the end of a thin glass rod to a glass disk using glass blowing techniques. The thin glass rod was then heated and pulled like a fiber, which produced a geometry that was thickest near its attachment with the glass, and increasingly thin as the distance from the disk increased as shown in Fig. 7. These structures are similar to Prince Rupert's drops.^{20,21} The assemblage was then ion exchanged to produce the appropriate residual stress profile in the disk and the fiber. Under the right conditions it was possible to initiate fracture of the fiber with a relatively small bending load, and the fracture propagated down the fiber into the disk, causing the disk to fragment. More study of this approach is needed to ensure that fragmentation of the fiber, followed by the disk, occurs reliably.

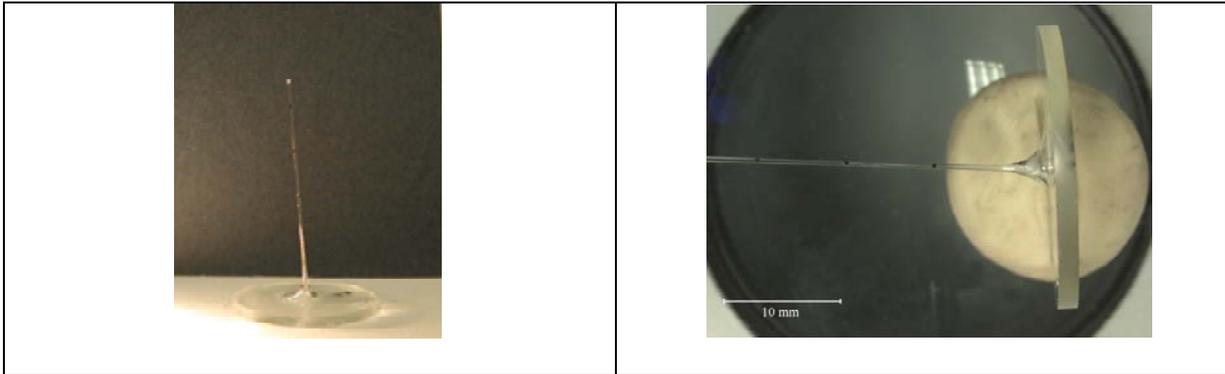


Fig. 7 Glass and disk bonded using glass blowing techniques. After a thin glass rod was attached to the disk, it was heated and pulled, producing a fiber with a thicker cross-section near the disk.

3.3 Directed Laser Energy Approach

Another approach for fracturing and initiating fragmentation of ion-exchanged glass was to use directed laser energy to create flaws in the tensile region of the glass. The first set of experiments used a femtosecond laser²² to write lines of damage of different thickness and width inside glass bars of annealed Corning 0317 glass. The visible lines are regions in which the glass has undergone a structural change. Fig. 8a shows the direction and position of the laser, and the focus spot in the glass. Fig. 8b shows an optical photo of the damage lines that were created as the glass was moved transversely with respect to the laser position for three power levels. The width of the damage line increases with laser power.

A second set of femtosecond laser experiments was conducted to investigate whether damage lines could be created inside the tensile region of stressed glass samples, that would lead to their fracture and fragmentation. The 2.2 mm thick Corning 0317 glass disks were ion exchanged in KNO_3 for 96 hr at 500°C . These exchange conditions give a central tension value of approximately 110 MPa. The laser parameters (power, pulse repetition rate, and pulse width) and damage line length were used to control fracture initiation. The pulse repetition rate and pulse width were kept constant at 1 kHz and 120 fs respectively. The sample was translated horizontally relative to the beam at a rate of $\sim 17 \mu\text{m}/\text{sec}$. At power levels of approximately 720 mW, with the laser focused at the center of the glass thickness (in the zone of residual tension), the glass fractured as soon as the laser was turned on. At lower power levels (~ 100 -500 mW) to the glass could be translated a considerable distance to create a long damage line before it fractured. For samples that broke power levels of 500-600 mW the damage line length at fracture was just under one mm. The critical level of damage appears to be a combination of the laser power (which determines the width of the damage line) and the damage line length.

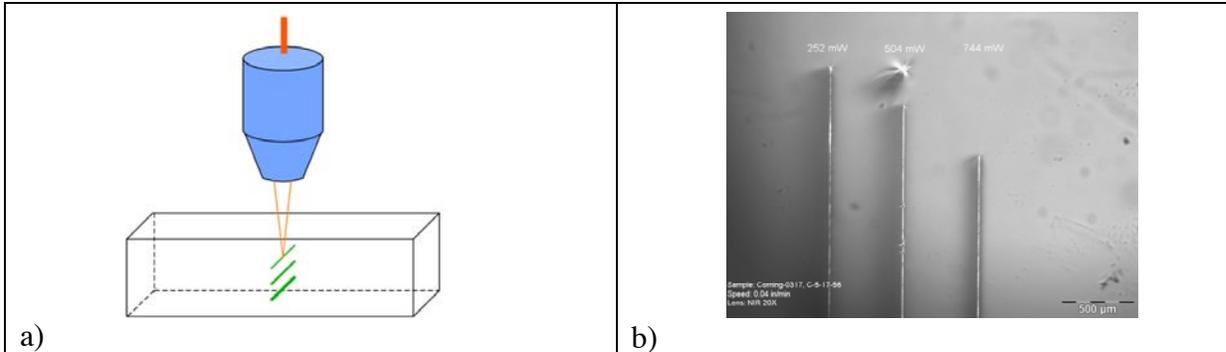


Fig. 8 a) Schematic showing laser and focus position in glass used to create b) Lines of damage. Laser power indicated above each damage line.

3.4 Energetic Materials Approach

We investigated explosive shock wave propagation methods for releasing the energy that is stored in the tensile region of stressed glass. The shock waves were created using small amounts of explosive in contact with the surface of a glass disk. Tests were done with the explosive directly on the surface or in a blind hole in an ion-exchanged glass disk. With the explosive in contact with the surface not even the unstrengthened glass disk could be fractured. When the explosive material was used in a small hole machined in the glass surface it was possible to break the stressed glass. The containment of the explosive material appears to increase the amount transmitted to the glass to break it.

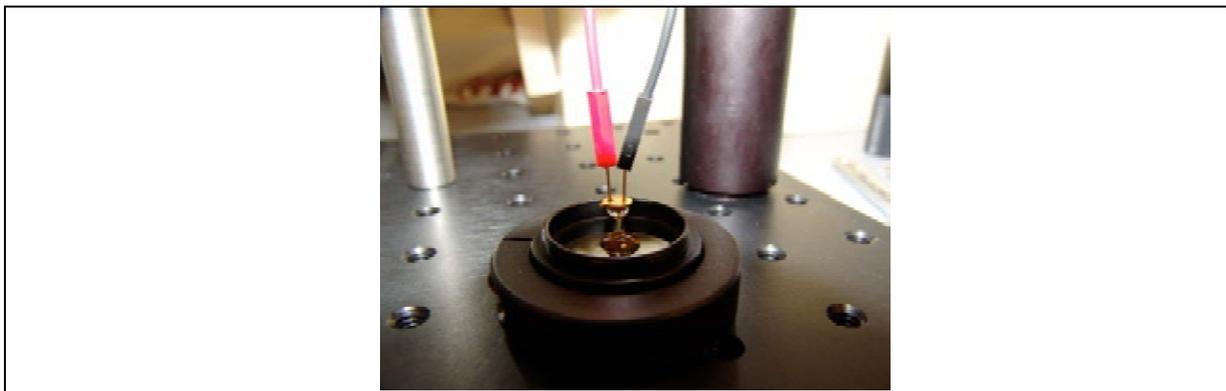


Fig. 9 Fixture used to test use of energetic materials to initiate fracture in glass. A one inch diameter glass disk is sitting in the cup and has explosive material and igniter bonded to it.

4. Investigation of Methods for Making Parts with More Complex Shapes

The ion-exchange process has been used primarily for strengthening glass parts with a plate or disk geometry. Technologies for making complex shapes such as containers, were investigated, including machining cavities in glass, and using hot forming approaches including glass blowing and high temperature molding of the glass. Because the Corning 0317 glass has a high melting temperature, it was very difficult to melt or mold the glass starting with plate stock. The use of a frit (glass powder) may be a better approach for the mold approach. Traditional glass blowing was unsuccessful because the high softening temperature of Corning 0317 glass makes it hard to work; it also appears to offgas under certain conditions. Machining glass plate into more complex parts is feasible, but great care needs to be taken to avoid creating mechanical damage and other stress risers that weaken the glass, possibly causing it to fail during ion exchange or in its intended applications. The thickness of the available plate stock limits the size and shape of the container that can be fabricated. An example of a cavity machined into a plate is shown in Fig. 10.

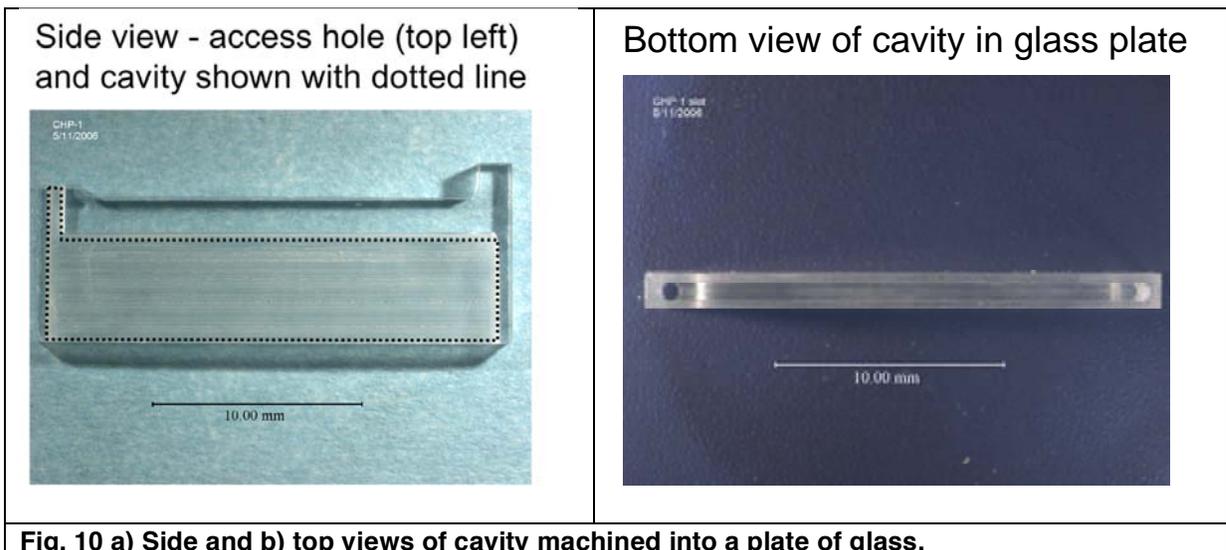


Fig. 10 a) Side and b) top views of cavity machined into a plate of glass.

Complex shapes can also be made by bonding different pieces of glass together, either before or after the exchange process. Previous work has shown that it is possible to bond two pieces of stressed glass together with a low temperature frit bonding process after ion exchange. Fracture can be transferred across the bond interface from one piece of stressed glass to another; however, the glass bonding material is weak and is not transparent. As such it does not provide an invisible or low visibility joint.

5. Conclusions

The information gained in this program has further defined the processing and property space for stressed glass, allowing us to extend the range of applications for which it can be utilized. Several prototype applications were developed and fabricated. This work has also helped demonstrate that stressed glass can be used as part of a stress/shock sensor with directional discrimination for components such as mechanical weaklinks.

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