

519-36

162613

p-10

1993-3009  
49151

N93-22198

## HIGH PERFORMANCE SAPPHIRE WINDOWS

Stephen C. Bates  
Advanced Fuel Research  
87 Church Street  
East Hartford, CT 06108

Larry Liou  
NASA Lewis Research Center  
Cleveland, Ohio 44135-3191

### ABSTRACT

High-quality, wide-aperture optical access is usually required for the advanced laser diagnostics that can now make a wide variety of non-intrusive measurements of combustion processes. Specially processed and mounted sapphire windows are proposed to provide this optical access to extreme environments. Through surface treatments and proper thermal stress design, single crystal sapphire can be a mechanically equivalent replacement for high strength steel. A prototype sapphire window and mounting system have been developed in a successful NASA SBIR Phase I project. A large and reliable increase in sapphire design strength (as much as 10x) has been achieved, and the initial specifications necessary for these gains have been defined. Failure testing of small windows has conclusively demonstrated the increased sapphire strength, indicating that a nearly flawless surface polish is the primary cause of strengthening, while an unusual mounting arrangement also significantly contributes to a larger effective strength. Phase II work will complete specification and demonstration of these windows, and will fabricate a set for use at NASA. The enhanced capabilities of these high performance sapphire windows will lead to many diagnostic capabilities not previously possible, as well as new applications for sapphire.

### INTRODUCTION

The study of the physics and chemistry of combustion and other high temperature and pressure processes is increasingly being performed by advanced laser diagnostics that have extensive and steadily broadening capabilities. These diagnostics often require high-quality wide-aperture optical access that is not currently possible using conventional materials. The combined pressure and temperature constraints of the contained environment are beyond current window materials technology. Glass has too low a mechanical strength, sapphire is thought to be too sensitive to thermal stress and shock, zirconia cannot be manufactured in large pieces, and diamond is eroded too rapidly by an oxidizing atmosphere and is too expensive in large sizes.

### SAPPHIRE

Of all of the commonly available materials sapphire is the hardest and has the highest melting point. The weakness of sapphire as a material and a major cause of its limited application has always been its poor thermal stress behavior. Because sapphire is such a strong crystalline solid it is difficult to shape and polish. Its strength, enhanced in single crystals, is far superior to that of glass. The potential of strong sapphire, however, is even greater if ways can be found to extend to large scales the strength that has been demonstrated for small single crystals. It has long been known that the relatively low practical strength of sapphire is a direct result of surface flaws that are generated during the fabrication of sapphire in a particular shape; the bulk crystal itself can be made essentially perfect by using modern crystal growing techniques.

Stoichiometrically there is only one oxide of aluminum: alumina,  $Al_2O_3$ . However, alumina can form various polymorphs, hydrated species, etc., depending on the conditions of its preparation. There are two forms of anhydrous  $Al_2O_3$ :  $\alpha-Al_2O_3$  and  $\gamma-Al_2O_3$ . In  $\alpha-Al_2O_3$  (sapphire) the oxide ions form a hexagonal close-packed array where the aluminum ions are distributed symmetrically among the octahedral interstices. The  $\alpha-Al_2O_3$  material is stable at high temperatures and is also indefinitely metastable at low temperatures. It occurs naturally as the mineral corundum. The  $Al_2O_3$  that is formed on the surface of aluminum metal has still another structure: a defect rock-salt structure with an arrangement of Al and O ions in a rock-salt ordering with every

third Al ion missing. Sapphire is a unique material in a number of ways. Its extreme hardness and chemical inertness is complemented by its very broad bandwidth for optical transmission (0.16 - 5.5  $\mu\text{m}$ ). Sapphire has special optical properties in that it has a large surface reflection and is optically active as well as birefringent. The forming and polishing of single crystal sapphire is difficult and time consuming. Common properties are given by manufacturers [1], while the primary reference for sapphire is a Russian book by Belyaev [2].

### Mechanical Characteristics of Sapphire

The strength of brittle materials, which at normal temperatures includes single crystal sapphire, depends on a number of factors: rate of testing, temperature, quality of the surface of the specimen, ambient conditions, size of specimen, etc. Sapphire is a single crystal; its properties are orientation dependent and determined by the properties of the crystal itself. Its macroscopic behavior depends strongly on the perfection of the crystal, both internally and at its surface. Stress failure occurs as a result of the formation and propagation of cracks in the crystal. Near 900 °C sapphire becomes plastic, as the thermal energy in the crystal becomes large enough to enable the weakest slip planes in the crystal to move. Of all of the properties of sapphire, those related to its strength are the least well defined because the failure of sapphire is statistical; design values must be based on the minimum possible strength. The mechanical properties of sapphire are given as:

Tensile strength:	at 25 °C: 410 MPa (60 kpsi) (design criterion)		
	at 500 °C: 280 MPa (design criterion), at 1000 °C: 350 MPa (design criterion)		
Bulk modulus:	2.4 x 10 <sup>11</sup> Pa,	Young's modulus (60° to c-axis):	3.45 x 10 <sup>11</sup>
Modulus of rigidity:	1.5 x 10 <sup>11</sup> Pa,	Modulus of rupture:	450 - 700 MPa, Poisson's ratio: 0.25

There are four primary characteristics of a sapphire window that together determine the design strength of the sapphire. These are 1) Bulk crystal quality, 2) Crystal orientation relative to the window planes, 3) Surface preparation, and 4) Avoidance of mechanical design features that cause stress concentration. Defining the current standard practices with respect to these factors determines the baseline case for any strengthening comparison and leads to an understanding of how the fabrication and use of sapphire can be optimized.

Mechanically, sapphire is currently characterized by the optical quality of the bulk single crystal. There are no strict standards for describing the crystal, only approximate grades. The primary reason for this accepted imprecision is the lack of correlation of identifiable defects with the macroscopic behavior of a crystal - except for optical clarity. Optical grading is done both because large sapphire is usually used as an optical material, and because optical testing is the simplest and easiest technique that is used for identifying crystal defects. A microscopic property that does translate to macroscopic behavior is the crystal orientation, where bond strength translates to directional crystal strength.

For the purpose of determining the global mechanical strength of a high quality single crystal of sapphire the condition of the surface is the determining factor. The characterization of the surface finish must be supplemented by a knowledge of the subsurface damage layer that has been created by the mechanical deformation of the surface during the shaping or polishing of the piece. Often sapphire pieces are rapidly diamond polished to high quality without removing the associated damage layer in the crystal. This damage layer often cannot be easily detected, except by a separate chemical surface etching process.

Stress concentration is usually caused by holes or sharp edges. Window design excludes holes, but sharp edges are a common feature of windows simply because edges are rarely specified by the design engineer. Sharp edges produce chips easily, and these chips can scratch the window surface and significantly reduce strength. Sharp corners also occur when a window is made in one piece with two diameters; the large diameter is used for mounting and sealing purposes. The corner between the two diameters is not only a location of stress concentration but a location where there is likely to be surface damage from machining and no polish at all. This edge is a primary cause for the failure of windows using this design, and should be radiussed and polished.

### Surface Strengthening

The process of strengthening sapphire by modifying its surface has long been known and practiced in the form

of fire polishing. More recently research using glazing to strengthen glasses has been extended to the strengthening of sapphire by a similar glazing process [4,5]. The precise mechanism for the strengthening in either case is unknown, except that it is known that surface flaws are either eliminated (fire polishing) or their effects on overall mechanical strength are somehow diminished. One of the purposes of the research described here has been to identify the important mechanism involved in the strengthening of sapphire, so that a practical process can be developed to achieve a reliable increase in the strength of any appropriately processed piece. Fire polishing itself cannot be used, since the thermal stress inherent in this process breaks large pieces. Unfortunately for the purpose of identifying important strengthening mechanisms, practical experimental strengthening techniques almost always improve the surface through a number of mechanisms simultaneously.

Since the condition of the surface determines the overall mechanical strength of the piece, techniques have long been sought that can reliably improve the condition of this surface. The following mechanisms have been demonstrated to be associated with improvements in the strength of sapphire: 1) Polishing, 2) Healing of surface flaws (chemically or through high temperatures), 3) Protecting the surface 4) Sealing surface flaws (solid solution layers), 5) Compressive surface layers, and 6) Crack propagation prevention (dislocation pinning). The most used of these techniques has been compressive surface layers. Such layers improve the strength of brittle materials by preventing surface flaws from acting to cause failure; compressive stresses are created in the surface that cause much larger tensile stresses to be required for a crack to grow and propagate.

In the work of Kirchner [4], a series of methods were used to obtain compressive surface layers on a variety of ceramic materials. Emphasis was placed on strengthening by quenching, and by glazing and quenching, because these experimental methods yielded the highest strengths. Strengthening sapphire was emphasized as a result of the inherent strength and ready availability of sapphire. Substantial improvements in flexural strengths were observed. In some cases improved tensile strength, thermal shock resistance and delayed fracture properties also were demonstrated. Treatment of sapphire single crystals resulted in three-fold improvements in strength. A glazed and quenched alumina rod 3.2 mm in diameter with a glaze layer 0.04 mm thick may have a measured flexural strength of 820 MPa. At failure, the tensile stress in the outermost portion of the alumina under the glaze on the tension side of the rod was at least 807 MPa. Much of the improvement in strength was retained when the samples were abraded. The stresses in the glaze are much lower because of the initial compressive stress and the low elastic modulus. It is apparent that the bulk of the material is inherently strong, but that the untreated material fails at low stress levels because of processes originating at the surface. More recently, Dr. Bates used this glass glazing process to strengthen a large (100 mm diam., 5 mm thick, and 150 mm long) sapphire cylinder for use as the transparent cylinder shell in a research internal combustion engine [5,6].

An important issue associated with surface strengthening is surface protection. There are two important contributors of the environment to the strength of sapphire. One is environmental chemistry, and the other is handling or mounting damage. The effect of environment on sapphire surfaces is usually that of moisture in combination with stress. Other effects arise from corrosive atmospheres (they must be very corrosive to affect sapphire), very high temperatures, or a combination of these factors. Handling protection prevents hard particles from coming into contact with the surface and causing damage that would weaken the piece. Although sapphire is very hard and very scratch resistant, it is easy to microscopically scratch an unprotected surface. The reason for this is the omnipresence of hard particles in the form of alumina on "sand" paper, and chips from the sapphire piece itself. Simple aluminum cannot scratch sapphire; although there is an aluminum oxide film on the surface, this film is in a different chemical form than sapphire, and is much softer. Protective layers can consist of any coating ranging from a glass glaze applied to strengthen the piece, to an antireflective coating applied for optical reasons. Even plastic films can be used. The coating need not be very thick to protect the surface; coatings that have poor transmission in wavelength regions where the transparency of sapphire is required can still be used because the coating is too thin to have significant absorption.

For surface strengthening to be effective the condition of the surface must be the determining factor for the macroscopic strength of the single crystal, rather than any bulk flaws. The bulk material of a window by definition has only a few flaws, since it is good optically. Such a window must be of even better quality as an imaging or laser diagnostic window. Current commercial window sapphire is thus always of a bulk quality such that the surface finish probably determines overall material strength. The surface quality is most important at locations where there are large local tensile or shear stresses present. The surface condition at the exact

position of maximum stress may not be relevant, however, because some other location on the surface may have half the stress but be unpolished (typically the side of the window). Stress distribution is highly three dimensional and determined by the combination of mechanical and thermal loading with residual stresses.

Almost all of the applications relevant to the present work are those where the surface which controls the strength of the piece is in a benign environment. These windows face a hostile environment - an environment that almost always includes high temperatures. Thus the side toward the hostile environment is hot, and the face away from it is cooler; the hot face is in compression and the cooler face is in tension as a result of thermal expansion differences. The face in tension is the face whose surface condition determines strength, and it is on the benign side. For a window into a combustion chamber, the benign side is the outside. For a window on a supersonic plane the benign side is the inside. In both cases the surface condition of the important side can be easily and passively maintained with the proper care.

## SAPPHIRE WINDOW FAILURE TESTING AND ANALYSIS

The Phase I program [7] had a goal of providing an experimental demonstration of the strengthening of sapphire as an isolated material for the specific application of a window that provides optical access to a high pressure, high temperature environment. The mechanical strength of a sapphire window was to be significantly increased by appropriate processing of the surface that is in tension during use. In another sense the project goal was to greatly increase the design strength of sapphire with respect to current design practice and accepted engineering criteria. As will be shown, large additional gains in effective design strength can also be achieved by using improved mounting design techniques that are appropriate only to the material properties of sapphire.

### Sapphire Surface Processing

The goal of the surface processing effort was twofold. The original goal of Phase I was to demonstrate sapphire strengthening through surface processing, with a secondary goal of identifying the mechanism of strengthening. A critical part of the work was the examination of the window surfaces by a scanning electron microscope (SEM), which led to a correlation of window failure strength with surface finish. Seven different types of surface processing were performed: 1) Standard 80/50 polishing, 2) "Epi" polishing, 3) Antireflection coating, 4) Molecular beam implantation, 5) Glass glazing, 6) Surface annealing, and 7) CO<sub>2</sub> laser melting. Different strengthening mechanisms were evaluated by using a surface processing technique that isolated a particular mechanism. Of the surface processing techniques epi polishing was found to be most effective for strengthening sapphire; the present discussion will center on polish strengthening experiments. The other techniques were found to be less effective, but were used to confirm the importance of the polishing as the most important effect among the many that could contribute to strengthening.

Small sapphire windows were used in an inexpensive hydraulic failure pressure testing facility. These windows were 2.5 cm diameter and 1 mm thick, sized such that the pressure required to break them is within the accepted rating for a commercial medium-pressure hydraulic assembly (up to 140 MPa). A set of 30 standard grade, random orientation windows with an 80/50 scratch/dig optical polish was obtained from Meller Optics (Providence, RI). This polish is standard when no specific finish is requested except that it be an optical finish. Final polishing is performed using a fine grit diamond compound. SEM images of this polish indicate that the polished surface is a mass of randomly oriented scratches on the order of 1  $\mu$ m wide. Another set of 32 best quality, 0° orientation windows with an epi polish was obtained from Crystal Systems (Salem, MA). An epi finish is the best available polish for sapphire, where final polishing is performed by chemical removal of the sapphire surface using a colloidal silica solution at elevated temperature. SEM images of these windows reveal a variety of types and concentrations of defects. Characteristic of the colloidal silica polish is pits of varying size randomly distributed over the surface. The pits can occur in isolation or in clusters of varying size. A high quality epi polished surface has few defects widely spaced, while a low quality epi finish not only leaves remnants of scratches in place, but introduces quite a few streaks of deeper erosion. It should be noted that features visible in the SEM may not be actual surface features, but rather subsurface scattering sites, since they are actually identified only by changes in the deflection of electrons from the SEM beam. No polishing was required or performed on the edges of these windows, since the edges were not stressed at all, and chipping could not damage the window surface before testing.

### Sapphire Window Failure Testing

A hydraulic facility as shown in Fig. 1 was constructed to failure test the processed sapphire windows. The configuration consisted of a hydraulic hand pump, medium pressure hydraulic tubing, a pressure gauge, and a window mounting fixture surrounded by a metal safety shield. The primary pressure containment was done by a pair of standard "Conflat" vacuum flanges that seals using copper gaskets. One flange was modified to simulate a clear-aperture fixture for optical access, and the other was drilled and tapped to accept a fitting from the hydraulic system. The sapphire windows were not clamped, but held against the O-ring seal by the internal pressure of the system. This prevented any possible complications from clamping, and turned out to be instrumental in the development of the high-pressure window mounting technique. The fixture included two safety shields; a case around the entire window mount fixture to contain pieces and a thin plate on the top of the aperture flange itself. Any significant air volume at high pressures would otherwise result in an extreme safety hazard when a windows failed.

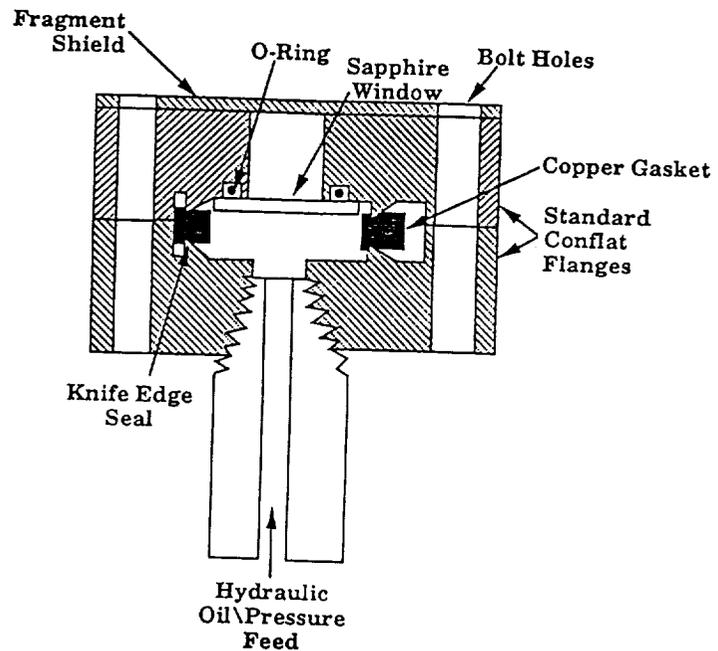


Figure 1. Sapphire window hydraulic failure testing facility; detailed drawing of window mount fixture.

Failure testing of 31 windows was done, sampled from a set of 62 windows that had undergone combinations of 6 different surface processing techniques (laser melting was an isolated test). The pressure behind each window was increased until the window broke, and the peak pressure was recorded. The results of failure testing that demonstrate the feasibility of strengthening of sapphire by surface processing are shown in Fig. 2, where the strength of the 80/50 polish windows is compared with that of epi polished windows. Also indicated is the predicted window failure strength based on the design failure strength of sapphire as specified by the manufacturer. It is crucial to note that epi polish windows were selected on the basis of minimal flaws under SEM inspection, and then failure tested. Every window chosen for minimal flaws had a large failure pressure.

After demonstrating a correlation between minimal surface flaws and strengthening, two epi polished windows were identified as having major flaws, and these were tested, expecting them to have significantly less strength. One of these two windows did have a strength comparable to the 80/50 polish windows, demonstrating that the polish was responsible for the increased strength. The other one was shown by the SEM not to have been polished with colloidal silica at all. Apparently the rough diamond machining of this window did not reduce its strength, an intriguing fact. The correlation between minimal flaws and increased strength was 100%, while the correlation between flaws and reduced strength was not perfect because there was not a large enough flaw density to guarantee that a failure causing flaw would occur in the highly stress center of a window. This contrasts with the 80/50 polish, which was continuously flawed.

The absolute magnitude of the failure stress for these 2.5 cm diameter windows is very large: 25-27.5 MPa (almost 4,000 psi), although the windows are only 1 mm thick. The high contained pressures before failure demonstrates the potential of sapphire windows. The clear viewing aperture was 1.9 cm. As will be shown, a significant part of the load capacity of this window arises from the mounting system.

Strengthening ratios vary from 4.7 (maximum strength of strong samples/minimum strength of weak samples) to 1.87 (minimum strength of strong samples/maximum strength of weak samples). The most appropriate factor is that of a design strength, which can be taken as the minimum strength of the stronger samples relative to the predicted failure strength based on manufacturer specifications. As expected, this predicted failure strength is

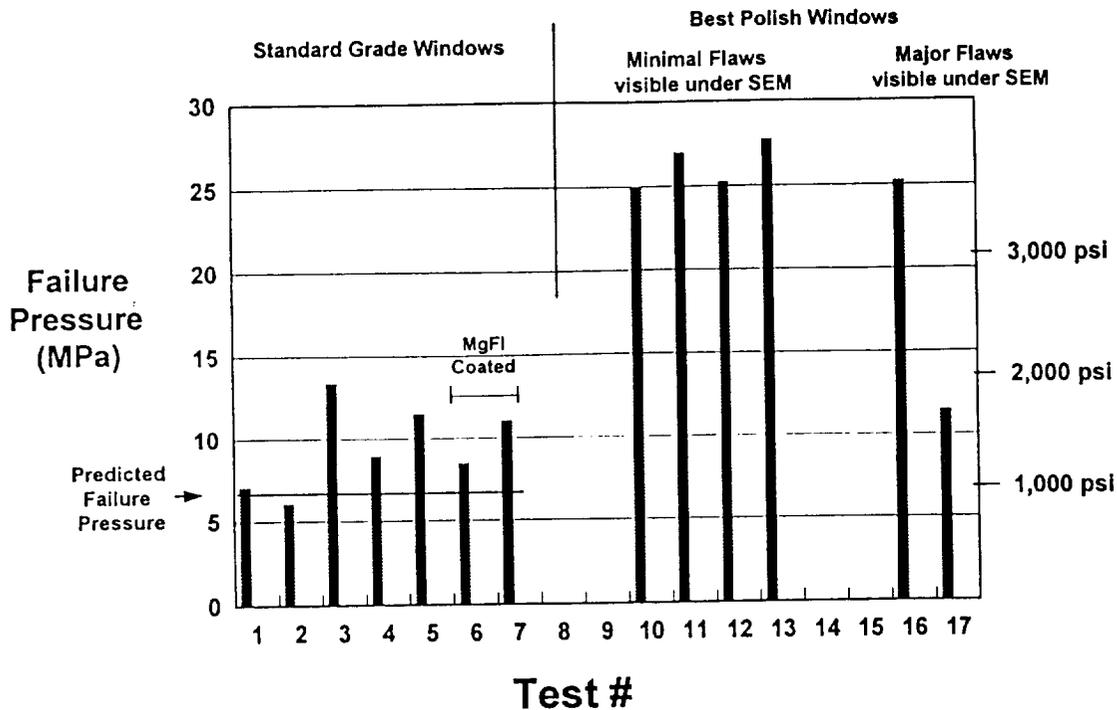


Figure 2. Sapphire window strengthening feasibility demonstration pressure testing results.

close to the minimum tested strength, because the design strength must be based on the worst case analysis. Thus, a design strengthening factor of 3.4 has been achieved through surface processing. This design factor increase implies that a new design strength of 1.4 GPa (203,000 psi) has been achieved for sapphire.

Other failure tests were performed to try to isolate the important strengthening processes. A MgFI antireflection coating isolated the surface from any environmental chemistry that might affect window strength, but did not cause strengthening. Both argon ion implantation and glass glazing resulted in strengthening of the 80/50 polished windows but to a lesser degree than epi polishing. Furthermore these processes did not increase the strength of epi polished windows. Testing of all of these techniques on both the 80/50 and epi polish windows separately also confirmed the role of the epi polish in providing the major strengthening effects measured.

There are two other possibilities for the difference in strength of the windows. The first is that the 80/50 polish windows are of lower bulk crystal quality. The second is the probable difference in crystal orientation of the two different types of polished windows. That these are not the cause of the strength difference can be shown from the data. Considering the quality of the bulk crystal first, if that were controlling the strength, the perfect crystals would always be stronger than the standard grade crystals. That is not the case is shown by case # 17 in Fig. 2. In this case the strength of the perfect and standard crystals are identical - only the surface processing has changed. That the orientation is almost certainly not the cause is demonstrated by the same case, where the degradation of the strength of the C-axis ( $0^\circ$ ) windows to that of the low strength 80/50 polish, and probably 90 degree orientation windows. Other data support this argument in a similar manner. The strengthening effect must be caused by surface finish.

CO<sub>2</sub> laser melting was attempted on a test window to determine if this could be a practical means of more reliably achieving a high quality surface. A 25W CW CO<sub>2</sub> laser emitting radiation absorbed by sapphire (10.6  $\mu\text{m}$  wavelength) was focussed to a spot on a 80/50 polish window. Melt polishing was clearly visible in SEM images, smoothing out the scratches from the 80/50 polish, but fracture lines were always present. These fractures were caused by the thermal stress associated with the large local thermal gradients of the heating. This polishing process may be successful if it is performed while the sapphire piece is at a high enough temperature to allow the plasticity of the material to absorb the thermal deflections and prevent fracture.

## Window Mechanical Strength and Mounting Analysis

Although the standard technique for strength testing discs is the ASME four-point bending technique, it was decided to use an equivalent real window mounting system to perform the failure tests. The window can be modeled as a thin, uniform-thickness disk surrounded by a pressurized fluid up to the O-ring seal. Without pressure, the window rests flat on the metal support structure between the inner edge of the clear aperture and the outer diameter of the window. As the pressure is increased, the center of the window is pushed into the aperture and the outer diameter lifts off the metal. This is possible because the O-ring is pressurized and deforms to fill the space between the metal O-ring groove and the window, even if this space grows slightly. The load modeling is as shown in Fig. 3, together with the window deflection. For a uniformly loaded thin disc the maximum stress is related to pressure by:

$$S_m = k (wr^2/\delta^2) \quad (1)$$

where  $S_m$  is the maximum stress,  $k$  is a constant equal to 1.27 for a thin circular plate,  $w$  is the pressure,  $r$  is the disc radius, and  $\delta$  is the disc thickness. For a 410 MPa tensile strength of sapphire at room temperature, a 11.7 mm O-ring radius, and a 1 mm thickness, the predicted failure pressure is 2.55 MPa; a pressure far below even the worst test results.

However, as discussed above, the actual case of the loading on the window is a disc where the deflection is constrained by the inner edge of the clear aperture. As long as the maximum stress at (a) is significantly larger than at (b) the piece will fail at the center and surface processing will result in strengthening. This applies only to sapphire, since it is so hard that the inner metal edge does not cause the unusually high line stress and piece failure that occurs using glass. The loading outside the fulcrum diameter balances some of the load inside of the fulcrum diameter and causes a reduction in the effective load at the center. Using the simple area balance indicated in Fig. 3, a new effective loading area can be calculated, giving an effective radius for failure loading of 6.85 mm. This compares with the initially assumed radius of 11.7 mm. It also leads to an effective reduction in loaded area by a factor of 2.9 and an increase in predicted failure pressure by the same factor to 7.4 MPa. This prediction is in excellent agreement with the window testing data, as indicated in Fig. 2.

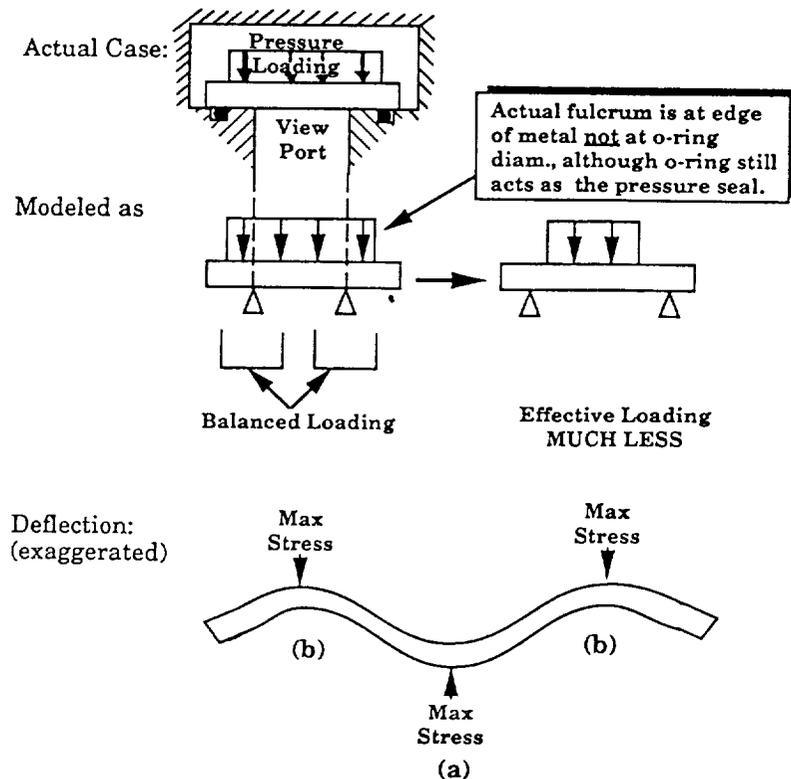


Figure 3. Window failure stress analysis.

Furthermore, the new effective load area is almost a factor of 2 less than the clear aperture, illustrating the advantage of such a mounting scheme for increasing the design aperture of a given window.

### Sapphire Thermal Analysis

The methods for increasing the strength of sapphire windows are predicted to be independent of temperature, but thermal stress effects are known to be an important contributor to the overall stress developed in a typical high pressure, high temperature window application. Thermal stress analysis tends to be complex, but it can be monitored experimentally. Basic thermal stress analyses have been performed, and these, together with previous research for the sapphire cylinder engine project [5], confirm the validity of the feasibility demonstration for harsh thermal environments but indicate the necessity of detailed work in Phase II. Experiments have been designed and prepared for thermal testing of strengthened sapphire.

### Sapphire Window Strengthening Assessment

An assessment of the Phase I program to develop high performance sapphire windows must begin with the fact that current sapphire window design practice is very poor in general. This poor practice begins with a lack of adequate specification of the bulk material and the surface quality of sapphire windows, and a lack of design work to eliminate geometries that lead to stress concentration. Furthermore, the elimination of sharp edges is rarely specified. Poor practice continues with a total lack of the use of thermal isolation to minimize thermal stresses, and no consideration of modifying experimental operating procedure to lessen the severity of thermal transients. Mounting procedures that are used are those appropriate to glass but not to sapphire, and these procedures usually degrade the thermal as well as the mechanical performance of any type of window.

Another factor in the misuse of sapphire (and glass) windows is the lack of an understanding of statistical failure of brittle materials. Commercial design strengths must be based on the minimum strength of a material. For the case of statistical failure, such as is the case for sapphire, the majority of pieces are stronger than the quoted value. This means first that proof testing can easily be done to increase the safety factor of expendable pieces. It also means that the strength of a replacement will not be the same as the original - one window may survive for extended periods, whereas the next may fail immediately. Also ignored is the effect of environment on strength. It has been mentioned previously that it is relatively easy to scratch unprotected sapphire, and that scratches can lead to the failure of the entire piece.

The advances made in this program must be considered relative to these facts. Strengthened sapphire windows with improved mounting can only result in superior performance in the context of a thorough understanding of their proper use. The full benefits of the excellent properties of sapphire can only be obtained with proper manufacture, design, construction, and operating procedures.

### Large Sapphire Window Design

A preliminary design of a large-aperture (15 cm) sapphire window on a combustion chamber was performed based on Phase I advances. Although such a window was previously thought not to be practical, the new design implies a window that is only moderately thick. A large aperture window to high temperature and pressure environments is a major goal for this program and for NASA; it would allow much more extensive use of current advanced laser diagnostics, and new information on difficult and important problems.

## APPLICATIONS

Sapphire is widely used because of its attractive characteristics: excellent strength, hardness, chemical inertness, temperature resistance, and broadband transmissivity. It is also known for its high cost and its reputation for poor response to thermal stress and shock. Therefore, sapphire must be used with planning, both in its specification and design details, in order to utilize its attractive characteristics while avoiding the thermal stress and shock problems. A description is given below of the applications of sapphire as well as the potential of the improved sapphire. The listed applications provide the context for understanding the potential of improved sapphire.

## General Applications

The following is a comprehensive list of the uses of sapphire as supplied by Meller Optics, Inc. Many applications are of specialty nature. Among the listed uses, note that sapphire fiber optics applications are expanding rapidly to provide diagnostic access to high temperature environments. Also, in addition to the single crystal sapphire on which this work is based, polycrystalline sapphire is used extensively in aerospace structures and for armor.

### **MECHANICAL**

1. Fluid and gas nozzles
2. Hole and cap jewels for instruments
3. Wire and thread guides for electro erosion and printing machines
4. Blades for equalizing magnetic sound recorders
5. Various orifices for air, gas, and liquid meters
6. Capillaries for the semi-conductor industry
7. Chromatography pistons, pumps and valves for laboratory use
8. Probes for measuring instruments
9. Magnetic tape cleaners
10. Insulators
11. Pivots and machining stops
12. Balls-bearings, flow meters, check valves
13. Washers, valve seals (for particular flows)
14. Tubes
15. Narrow tolerance pieces
16. Point of sale windows for cash registers
17. Micrometer rotors
18. Microtome blades and knives
19. Tape guides

### **ELECTRONIC**

1. Wafer carriers thin film deposition:
  - a. Silicon on Sapphire
  - b. Gallium Arsenide for field effect transistors
  - c. Mercury Cadmium Telluride for detector arrays
2. Base substrates for thick film deposition of various metals for a wide range of circuitry
3. Acoustic delay lines

### **OPTICAL (Windows, lenses and prisms)**

1. CO<sub>2</sub> and O<sub>2</sub> blood gas analysis
2. Environmental smokestack and auto emissions
3. Other toxic gas and fluid analysis
4. Industrial oven and furnace windows
5. Cryogenic analysis
6. Ultraviolet industrial lamps
7. Hermetic vacuum ports and seals
8. Thickness guides for paper machines
9. Fire and smoke detection instruments
10. Optical wavelength detectors and filters
11. Infrared missile seeker and camera covers
12. Centrifuge cell windows
13. Laser optics, etalons and reflectors; high power
14. Sight glasses
15. High pressure windows
16. Polarization optics
17. Engine turbine pyrometry
18. Refractometry
19. Sight Reticles
20. Telescope Optics
21. Fiber Optics
22. Lenses and prisms
23. Solar cell cover plates
24. Radiation damage environmental optics

### **CHEMICAL**

1. Reactor components
2. Corrosion resistant cells, crucibles and tubes

### **MISCELLANEOUS**

1. Transparent armor
2. Hollow wave guide for laser systems
3. Fiber optic tips for surgical lasers
4. Watch crystals and jewelry
5. Charges for vacuum coating

## Windows Applications

Sapphire windows are used in a wide variety of research applications to provide optical diagnostic access to pressurized apparatus. They are also used in deep submersion vehicles. Commercial window applications include: 1) High pressure optical cells, 2) High temperature optical cells, 3) High pressure and high temperature optical cells, 4) Diagnostic cylinder wall for Internal Combustion engines.

NASA has many uses for sapphire windows, including a wide variety of diagnostic windows which provide the access to fluid and combustion studies. Future applications include structural windows in hypersonic vehicles.

One major application in defense is for missile and aircraft radomes. A radome consists of a hemispherical sapphire crystal mounted to permit tracking and guidance. The domes must be transparent to the proper wavelengths (infrared) and robust enough to survive the impacts and aerodynamic heating encountered in this application. Sapphire is the most competitive material for this application.

## Potential

The potential is great for the strengthened sapphire; any improvement of a factor of 10 in strength will lead not only to improvements in present applications, but will also open the door to many new ones. For example, experiments in the more extreme environments such as those in high speed aerospace research can now be studied for longer duration, and new applications in structural windows, transparent armor, and visible high temperature furnaces, can now be considered.

## CONCLUSIONS

Phase I research has successfully demonstrated the feasibility of greatly strengthened sapphire windows. Through surface processing, improved mounting designs, and minimization of thermal stresses, single crystal sapphire can be a mechanically equivalent replacement for high-strength steel. A factor of 10 increase in reliable design strength has been demonstrated for a strengthened, properly mounted sapphire window. The highest quality surface polishing resulted in experimental strengthening of sapphire windows by a factor of 3.4 relative to the strength of windows with a standard optical finish, which failed at the manufacturer's design strength. A new window mounting scheme provided a further increase of a factor of 2.9 in effective strength. The clear aperture that can safely be designed for a prespecified window thickness can be improved by this factor using the new mounting design. Guidelines have also been developed for specifying strengthened sapphire and minimizing thermal stress.

## REFERENCES

1. Crystal Systems Technical Brochure, Crystal Systems Inc., Shetland Industrial Park, 35 Congress St., Salem, MA, 01970.
2. L.M. Belyaev (Editor), **Ruby and Sapphire**, Nauka Publishers, Moscow, Available from National Technical Info. Service, Springfield, VA, 22161, (1974).
3. H.P. Kirchner, R.M. Gruver, R.E. Walker, "Strengthening Sapphire by Compressive Surface Layers," J. of Appl. Phys., Vol. 40, No. 9, 3445-3452, (1969).
4. R.L. Gentilman, E.A. Maguire, H.S. Starrett, T.M. Harnett, and H.P. Kirchner, "Strength and Transmittance of Sapphire and Strengthened Sapphire," Comm. of the Am. Chem. Soc., Sept., C-116,117, (1981).
5. S.C. Bates, "A Transparent Engine for Flow and Combustion Visualization Studies," SAE Transactions, Vol. 97, Section 6, 892-908, (1988).
6. S.C. Bates, "Insights into Spark-Ignition Four-Stroke Combustion Using Direct Flame Imaging," Combustion and Flame, Vol. 85, Nos. 3 & 4, 331-352 (1991).
7. S.C. Bates, "High Performance Sapphire Windows," SBIR Phase I Final Report, NASA Contract # NAS3-26330, August, (1992).