Sapphire Optical Fibers: Splicing and Sensing Applications

by

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Fiber optic sensors fabricated from standard silica fibers have many advantages over conventional sensors like small size, portability, durability and immunity to electromagnetic fields. Unfortunately, these sensors are not suitable for use in harsh environments where the temperatures are greater than 700°C and large working stresses are involved. Sapphire fiber-based sensors present an attractive alternative for use in such environments. The material properties of sapphire like high melting point, extreme hardness and relative imperviousness to chemical reactions, coupled with the advantages of optical fiber sensing, enhance the performance of these sensors for rugged use. Unfortunately, commercial sapphire fiber that is currently available has higher optical attenuation than silica fiber and is costlier. So, it is prudent to use a small length of sapphire fiber as a sensor head, which is then spliced to a standard singlemode silica fiber which acts a lead-in/lead-out fiber to the sapphire sensor head. This thesis investigates possible splicing techniques to fabricate such a sensor set-up. Comparative results from experiments performed on splices that have been obtained by each of these techniques, are presented. Furthermore, two different sensor configurations using a sapphire fiber, spliced to a silica fiber, are developed, and the results of preliminary tests are presented.
ACKNOWLEDGEMENTS

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INTRODUCTION

Optical fiber sensors have been researched for the nondestructive evaluation of materials since they were proposed for that application by Heyman in 1978 and demonstrated by Claus and Cantrell in 1980 [1]. These sensors can be attached to a surface to measure surface strains, or they can be embedded in a material to measure internal strains [2]. Alternatively, the sensors can be configured to yield the temperature of the environment. These sensors use the effect of perturbations on the light carrying capability of optical fibers to sense physical observables such as temperature, pressure, displacement and strain. Intensity, phase and polarization are among the properties altered by the perturbations of geometry and index. Optical fiber sensors can be divided into two basic classes, intrinsic and extrinsic. An intrinsic sensor uses changes internal to the optical fiber as the mechanism to alter the output characteristics of the light propagating through the fiber. Examples are modal domain sensing and microbend sensing. Extrinsic sensors utilize alterations to optical energy which occur external to the optical fiber. The light may or may not be coupled into another optical fiber after alteration. Examples of extrinsic sensors are intensity-based sensors such as air gap sensors. Many of the advantages of fiber optic sensing over conventional sensing is based on the small size, low weight, high strength and immunity to electromagnetic fields of optical fibers.

Recently, interest in the use of optical fiber sensor techniques to instrument advanced aerospace materials in high temperature environments has stimulated research in the use of sapphire fiber optical sensors [3]. Methods such as interferometry, polarimetry, and intensity-based optical techniques have been demonstrated for the measurement of strain and temperature on carbon/carbon and metal matrix composite materials. Silica optical
fibers can be used for these measurements at temperatures up to about 1000 °C, but at temperatures exceeding that, diffusion of dopants in the fiber defeats the waveguiding properties of the fiber. Also, since the vitreous form of a glass fiber is metastable, prolonged exposure of an optical (vitreous) silica fiber to harsh environments may promote devitrification of the glass, leading to increased attenuation in the fiber from scattering. For these reasons, single crystal sapphire fibers are being researched for instrumentation applications at temperatures up to 2000 °C.

**Sapphire Optical Fibers**

With the development of modern aerospace and high energy applications, new sensing requirements have emerged for sensing technology. Due to the extremely harsh combined environments of the high temperature, high pressure and chemically corrosive atmospheres, sensing in these environments requires fibers with these rigorous attributes. Single crystal sapphire fibers are a very good candidate for these type of applications.

Sapphire is the common name given to a single crystal α-alumina, or corundum, although it can refer to the crystalline material with various impurities. Sapphire is an attractive optical material due to its hardness, high melting point, and wide spectral window of transmission (145nm to 5500nm). It is chemically inert, and insoluble in any solvent except at high temperatures. The single crystal form is optically anisotropic, but the resulting birefringence may be used advantageously to realize polarimetric sensors [4]. Unlike glass fibers, sapphire fibers must be fabricated by crystal growth techniques. Recent advances such as edge-defined film-fed growth (EFG) techniques permit the

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simultaneous growth of many sapphire fibers in parallel. The resulting single-crystal fibers are used primarily for structural reinforcement in advanced composite materials, but have been investigated for use as waveguides. The fibers are not clad with a waveguiding layer, and the diameter fluctuation that results from the growth process introduces high levels of optical losses. The optical attenuation of these structural grade fibers has been reduced slightly by flame polishing in our laboratory with an oxy-propane torch.

Reference 5 illustrates the different growth techniques used to grow single crystal sapphire fibers. Two methods that are commonly used currently are the Laser Pedestal Growth (LPG) and Edge Defined Growth (EDG). In the LPG growth technique [6], the tip of a rod of source material is heated using two to four focussed lasers forming a molten droplet. The crystal seed is dipped into the molten area and slowly pulled out. The rate of the source material in and the amount of fiber pulled out can be carefully controlled. Outer shape of the fiber is determined by the surface tension. One of the advantages of this method is that the fiber grown will be as pure as the source material since no contact is made in the melt zone, i.e, with crucibles or dies as illustrated in Fig. 1.

In the Edge-defined, Film-fed, Growth (EFG), as illustrated in Fig. 2, sapphire fibers of reasonably controlled geometry are grown from the melt. The edge of the fiber is defined by a capillary tube (typically fabricated from molybdenum) which is attached to the bottom of a crucible (also from molybdenum) in such a way that liquid is able to enter the bottom of the capillary. Because the contact angle of the molten alumina is less than 90°, liquid rises in the capillary an amount, h cm, given by [7]

**Introduction**
\[ h = \frac{2 \gamma \cos \theta}{\rho r g}, \]

where \( \gamma \) is the surface tension (dyne/cm), \( \rho \) is the density of the melt (gm/cm\(^3\)), \( r \) is the tube radius (cm) and \( g \) is the acceleration due to gravity (981 dyne/cm). The use of a capillary arrangement provides a technique for maintaining a constant liquid level for filament growth as the melt is consumed. This introduces a considerable amount of thermal and mechanical stability. The temperature at the top of the capillary is arranged to be close to the melting point and an \( \alpha \)-alumina seed crystal of the desired orientation is dipped into the melt at the top of the capillary. As the seed crystal is pulled, the curvature at the solid-liquid interface is increased, with a consequent reduction in pressure within the liquid. If the

**Introduction**
temperature conditions are adjusted appropriately, the filament will solidify at the rate at which it is being pulled, maintaining an essentially constant Δp to continue the supply of liquid. Figure 3 illustrates the growth process. It should be noted that the EFG growth process is adopted by Saphikon to grow the single-crystal sapphire fiber which has been predominantly used throughout this research effort.

Fig. 2 Edge-Defined Film-fed Growth (EDFG) apparatus
Sapphire optical fiber sensors

The Fiber and Electro-Optics Research Center at Virginia Tech has pioneered the use of sapphire fiber sensors for the measurement of strain and temperature at high temperatures. The use of such high-temperature sensors represents new capabilities for the nondestructive evaluation of aerospace materials in trans-atmospheric aircraft such as the National AeroSpace Plane, or for measurements of parts in jet engines.

Several optical techniques have been demonstrated for measurement of strain and/or temperature at Virginia Tech. Both intrinsic and extrinsic Fabry-Perot configurations were demonstrated with sapphire rods. In the intrinsic approach, coherent light from a laser was guided by a single-mode fiber to a gradient index (GRIN) lens, which collimated the light and launched it into the sapphire rod. Interference between the reflection from the far end of the rod and the Fresnel reflection from the sapphire rod/GRIN lens interface was responsible for the sensor output. In the extrinsic approach, an air gap between two
sapphire rods held by a tube forms the cavity. The interfering reflections are generated by the two air-to-sapphire interfaces that form the cavity boundaries.

**About this thesis**

This thesis is concerned with development of a splicing technique to splice a sapphire fiber to a silica fiber and the subsequent use of this splice in the development of sapphire fiber-based extrinsic and intrinsic Fabry-Perot sensors. Sapphire is stronger (9 moh), more resistant to chemical abrasions and thermal shock, and has a higher melting/softening point than conventional silica fibers. Therefore, single-crystal sapphire fibers can be used in extremely harsh environments where conventional silica fibers would fail.

Four different approaches to the problem of fabricating a mechanically-strong and optically transparent splice between a sapphire and a silica fiber are evaluated in this thesis. The first approach is based on material diffusion properties of silica and sapphire fibers. The second approach focuses on the splice obtained by the controlled melting of the endfaces of both the fibers at the silica-sapphire interface, using an arc-fusion splicer. The third and fourth techniques employ alumino-silicate glass (ALG) as an intermediate to act as a binder and a filler, respectively, between the two fibers. The importance of these splices can be envisioned from the fact that present day growth techniques limit the transmission capabilities of the sapphire fiber to very lossy optical waveguides. Besides, the extremely prohibitive cost of fabricating these sapphire fibers also limit the use of sapphire fibers to very short length. On the other hand, all the advantages listed above would make sapphire fibers very good candidates for sensing in very harsh environments. Thus, as a trade-off,
a long length of silica fiber is spliced to a short length of sapphire fiber with the sapphire fiber functioning as a probe and the silica fiber as a lead-in/lead-out fiber.

Sapphire fiber-based interferometric sensors—both intrinsic and extrinsic—are detailed in this thesis. The intrinsic Fabry-Perot interferometric sensor consists of a small length of sapphire fiber, which works as a Fabry-Perot cavity, spliced to a silica fiber using one of the techniques described in chapter 2. The two reflections of the input light—one at the silica/sapphire interface and the other at the sapphire/air interface—interfere to produce a differential phase modulated interferometric output. Phase modulation can be achieved by changing the path length of the input light in the sapphire fiber. This is done by the variation of parameters such as temperature, strain and displacement.

A sapphire fiber-based extrinsic Fabry-Perot interferometric sensor has also been developed. The working principle of EFPI sensor is similar to that of an intrinsic sensor, in that differential phase between two reflections of the input interfere to produce an interferometric output. The Fabry-Perot cavity, in this case, is an air-gap between the input and the reflector fiber. Again, a small length of sapphire fiber is spliced to a lead-in silica fiber and another small length of sapphire fiber is placed in line with the input sapphire fiber, to act as a reflector. The air-gap between the two sapphire fibers is then enclosed in a sapphire tube, to give the air-gap sensor mechanical and chemical protection. The problem of the unwanted reflection at the silica/sapphire interface is effectively dealt with by using a laser diode with sufficiently large spectral bandwidth. A detailed description of the fabrication, principle of working and testing of a sapphire fiber-based interferometric sensor is presented.

Introduction
2.0 SILICA - SAPPHIRE FIBER SPLICING TECHNIQUES

A method for connection of sapphire optical fiber with silica optical fiber was investigated as a part of this thesis effort. The basic objective is the fabrication of a system for the low loss and low cost bonding of sapphire fibers to silica lead-in fibers for subsequent high temperature instrumentation.

During the early stage of the project, a careful study of all the possible techniques was performed, before reaching a decision about the technique that is to be applied finally. The results of this study are enumerated in the following sections.

2.1 Diffusion Splicing Approach

As a first step, a careful study was made of the diffusion process between silica and sapphire. After a thorough literature survey, several phase diagrams for the silica-sapphire system were obtained and a careful study of these was consequently made to better understand the various phases of the silica-sapphire system. The possibility of using mullite formation to enhance the joint between the silica and sapphire fibers has also been studied. A detailed study of the properties of mullite was undertaken.

To begin with, a fused silica rod (~96% pure, 2 mm diameter) and a sapphire rod (single crystal, 1.8 mm diameter) were put in contact and the interface was gradually heated using an oxygen-propane torch (maximum achievable temp. = 2000°C). The temperature of the interface was carefully monitored using a TYPE B thermocouple with a digital read-out.
Several attempts at promoting diffusion across the interface were tried. A typical heating profile consisted of heating the interface to a temperature of $\sim 1400^\circ$C and maintaining that temperature for $\sim 20$ minutes, followed by heating at $\sim 1700^\circ$C and for $\sim 10$ minutes, and then heating the interface to a little over $1770^\circ$C for half a minute. This is followed by cooling the interface to $\sim 1230^\circ$C and keeping the temperature constant for $\sim 10$ minutes. This profile is illustrated in Figure 4.

![Graph showing temperature vs. time](image)

**Fig. 4.** Heating profile for fabricating a diffusion splice

During the diffusion experiments, a small but observable bond between the silica rod and the sapphire rod was apparent. In the first few experiments, this bond appeared to break as the rods were cooled. To minimize the impact of thermal contraction during cooling, in subsequent experiments the clamps holding the rods were loosened before the cooling phase of the experiment was started. In these trials, the bond did not break during cooling, but was sufficiently strong to permit careful handling.
Careful examination of the fractured interface under an optical microscope indicated a definite diffusion across the interface, though it appeared to be predominantly the diffusion of silica into alumina. This can be explained on the basis of the structures of silica and sapphire molecules along with their relative mobilities. The silica rod has begun to melt at \( \sim 1770^\circ C \) which is consistent with the phase diagram of the system. The physical bonding between the rods is found to be very weak and is easily given to fracture at the interface. This may be due to the difference in the thermal coefficients of expansion of silica and sapphire which would affect the interface during the cooling process. The difference in the thermal coefficients of expansion is a source of stress that might break the weak physical bonding between the two rods.

To counter this problem, the rod system was cooled gradually. This was achieved by enclosing a sapphire rod and a silica rod in close proximity, in a ‘box’, built with mullite ceramic, and the interface was heated using an oxy-propane flame. The temperatures attained during a typical experiment were greater than 1200\(^o\)C, the softening point of silica, but were less than 1700\(^o\)C. This is approximately the temperature at which an introduction of liquid phase at the interface would be expected for most initial compositions of the silica and alumina. This meant that any reaction across the interface would be predominantly due to diffusion. The heating was performed twice, each time using two different orientations: one with vertical endfaces and the other with the endfaces slanting at \( \sim 45^\circ \). The reason for slanting them was that it was felt that the bond at the interface would be better if the liquid silica that melted off the silica endface would run down the sapphire endface and secondly, this resulted in an increase in the interface area for diffusion. The heating resulted in a weak bond at the interface, the bond being appreciably stronger in the second (i.e., the

Silica-sapphire fiber splicing techniques
slanting) case. It should be mentioned that the splice strength was not quantitatively tested, and this conclusion is based on qualitative and subjective evaluation. Even gradual cooling of this interface did not appreciably strengthen the bond and it was observed that this bond was not permanent and would break after some time (usually, several days).

Photomicrographs of the sapphire rod endface (after the heating) were obtained using Scanning Electron Microscopy (SEM) and are shown in Figures 5 and 6. It can be seen that there is a definite interaction across the interface. A highly magnified micrograph shows in detail a intricate network of short, pin shaped structures. The chemical composition at a point on the interface was also obtained using EDX, and are enclosed (Figure 7). Details of these results strongly suggest that there is a high probability of mullite formation at the interface. The enclosed photomicrographs show a distinct crystalline phase on the silica endface, different from that of sapphire. The structure suggests that this phase could be that of mullite, according to reference [8]. A study of these photomicrographs and comparing them with the photomicrographs of the reference mullite plate suggests that mullite formation does occur at the interface.

2.2 Arc-Fusion Splicing technique of silica/sapphire fiber splices

As an extension of the experiments being carried out with rods, the splicing of sapphire and silica (without coatings) by an arc-fusion splicer was attempted. The splicer used was manufactured by Power Technologies, Inc. The endfaces of the sapphire fiber were polished using metallurgical polishes prior to the splicing (as described in section 2.6), as it was impractical to obtain perfectly cleaved ends, owing to the crystalline structure of Silica-sapphire fiber splicing techniques.
sapphire. The sapphire fiber had a 125 μm outside diameter (Saphikon, inc.) without a jacket or a cladding. It was coated with a water soluble methyl cellulose, which was removed before splicing. The silica fiber was 125 μm outer diameter and 50μm in core diameter (Corning, inc.). The jacket was a CPC acrylate polymer. It should be noted that the fusion splicer does not include an enclosure or an arrangement to enable specific control of the heating profile. Moreover, the maximum temperature attained during the fusion process is not known.

Each of the splices thus obtained were considerably stronger mechanically, and were 'permanent' unlike the bonding obtained by diffusion. Some anomalies were observed. In some cases, the sapphire and the silica fiber melted at the same current settings. However, a numerical value for the temperature at which this occurred has not been determined. This was not the case with all types of silica fiber though. This was likely due to difference in chemical doping of the telecommunication optical fiber used. It was speculated that the cause of this apparently low melting temperature was the presence of impurities in the sapphire fiber. An elemental analysis of the flame-polished (described in 2.6.2) as well as unpolished sapphire fiber was made using the Scanning Electron Microscope and Energy Dispersive X-ray (EDX) spectroscopy. The EDX results show no detectable amounts of impurities in the commercially obtained sapphire fiber. Thus, the reason for this behavior is still unknown.

From the SEM photomicrograph of the splices that have been obtained (Figure 8), it can be noted that a compound, with a structure very similar to that of mullite, is formed. This suggests that mullite may indeed form at the interface that helps in the bonding. In one of...
the splices, the intermediate compound appears to be highly intricate and porous. This results in some loss of transparency at the interface. In fact, reference [9] states that mullite is a poor glass former, due to the crystalline phase.

The EDX results of this splice (Figure 9) as well as the EDX results of a splice obtained from diffusion-enhanced splice and the EDX results of the 'reference' mullite sample are listed below. In this table, 'coatless' refers to a splice between uncoated silica and sapphire fibers.

<table>
<thead>
<tr>
<th>Element</th>
<th>Mullite Reference Atom %</th>
<th>'Diffusion' splice Wt %</th>
<th>'Coatless' splice Wt %</th>
</tr>
</thead>
<tbody>
<tr>
<td>ALUMINUM</td>
<td>28.67</td>
<td>27.72</td>
<td>31.08</td>
</tr>
<tr>
<td></td>
<td>34.08</td>
<td>34.68</td>
<td>38.52</td>
</tr>
<tr>
<td>SILICON</td>
<td>19.63</td>
<td>21.08</td>
<td>19.71</td>
</tr>
<tr>
<td></td>
<td>24.19</td>
<td>27.35</td>
<td>25.33</td>
</tr>
<tr>
<td>OXYGEN</td>
<td>48.60</td>
<td>51.20</td>
<td>49.22</td>
</tr>
<tr>
<td></td>
<td>34.23</td>
<td>37.97</td>
<td>36.15</td>
</tr>
</tbody>
</table>

The above results indicate that the compositions of the three splices, obtained using two different techniques, closely agree with that of the reference mullite sample. The small variations could be due to various reasons, including the different splicing techniques that were used, and the variation of chemical composition over the minute scan regions chosen as the electron gun target in the SEM to perform the EDX analysis on these splices. The observations tabulated above support the conclusion that mullite formation does take place.
in the two splicing techniques examined so far.

The spectral attenuation of the splice was obtained by using a Photon Kinetics FOA 2000 Fiber Optic Analyzer, using the ‘cut-back’ approach. Figure 10 details the variation of the attenuation with the input wavelength. It is obvious that the attenuation is very high. It is important to note that this measured attenuation is probably due to many factors not only including splice loss, but also the geometrical structure of the splice, mismatch of the numerical apertures of the fibers at the splice region, etc.

![Attenuation vs Wavelength](image)

**Figure 10.** Attenuation in an arc-fusion splice

The best of these results, with total losses varying from 13 dB for long wavelengths to 17 dB for short wavelengths, show a definite improvement in the splice loss when compared to the results obtained previously on a similar splice. The 5 cm length of the sapphire fibers account for approximately 7.5 dB, yielding splice losses of 5.5 to 9.5 dB for the Silica-sapphire fiber splicing techniques
best splice. One more very important factor to note is that the numerical aperture of the sapphire fiber is very high, approximately in the order of 0.7, mainly due to the large core size (140 µm). The FOA-2000 characterizing system's accuracy in measuring the output power of a test fiber is limited by a numerical aperture of ~ 0.4. Thus, the value of the output power, as indicated by the FOA-2000, is less than the actual output. So, the value for the attenuation in the splice is probably more than the actual losses.

A large percentage of these splices (greater than half) broke while removing the spliced fibers from the fusion splicer. In order to protect the splices, finger nail polish was applied to some of the splices before removing them from the fusion splicer. Splices protected with nail polish were sufficiently strengthened to permit their removal from the fusion splicer without breaking the splice. The mechanism by which the nail polish strengthens the splice is not yet fully understood. It may involve redistribution of the stress around the splice, or retardation of water vapor induced stress corrosion weakening of the bare silica fiber.

In order to investigate the effect of the nail polish on the splice loss, a splice without polish and a splice with polish were analyzed on the Photon Kinetics FOA 2000 Optical Fiber Characterization System. There were only small differences in the attenuation at the splice in both the cases, indicating that the nail polish does not incur additional optical losses. This implies that the arc-fusion splice can be strengthened, mechanically, by applying nail polish to the splice. It should be noted that the maximum temperature that the nail polish
can withstand is approximately 70°C. Consequently, the maximum operable splice temperature is limited by this low melting temperature of the nail polish. Alternative coating materials, with higher melting temperatures, could be investigated in further work. The load frame was used to qualitatively test the splice strength of the splices (as described in 2.4.1), obtained by arc-fusion. The best of these splices broke at a load of 5.9 N, which corresponds to a tensile stress of 69.7 kpsi.

2.3 Splicing of silica-coated sapphire fiber with silica fiber

Sapphire fiber samples of ~2 inches in length were deposited with 2 μm thick silica, using Chemical Vapor Deposition (CVD) by Dr. Desu’s group in the Materials Engineering department. The coated sapphire fiber samples were then spliced with silica fiber using the same fusion splicer that was used before. The modification in the splicing technique was that the sapphire fiber was not clamped to the fiber holding platform and the fibers were moved closer to each other while the arc-current was present (as illustrated in figure 12). Figure 13 illustrates the variation of the splice loss with wavelength. The splice loss varied from 14 to 17 dB. It should be noted that the sapphire fiber that was spliced to the silica fiber was approximately 4 cm long. Therefore the attenuation in this piece of fiber was approximately 6 dB. This indicates that the splice loss was about 8 to 11 dB. Some of this loss may be accounted for by imperfect polishing of the sapphire endface.

Unfortunately, the mechanical strength of the resultant splice could not be analyzed quantitatively. The grippers on the I.J. Lloyd T-20,000 load frame that was used to test the splice strength did not accommodate the small lengths of the silica-coated sapphire fiber (~
4 cm). The reason for this small length of the sapphire fiber used for this particular splice

![Diagram]

Figure 12. Splicing between a silica-coated sapphire and a silica fiber

![Graph]

Figure 13. Attenuation in a splice between a silica-coated sapphire and a silica fiber

Silica-sapphire fiber splicing techniques
experiment is that the CVD furnace that was used for the silica deposition could only accommodate a maximum of 2 inches of fiber. It may be noted that subjective evaluation of the splice strength (by manually exerting tension on the splice) seemed to suggest that splices prepared by this technique were stronger than those prepared, as described in the previous sections.

2.4 Aluminosilicate glass intermediate splice

2.4.1 Splicing with aluminosilicate glass as a binder

A large effort was directed toward the possibility of using aluminosilicate glass materials as an intermediate material between the sapphire and silica fibers. Aluminosilicate glass ‘melts’ at about 950°C to form mullite [10]. This phenomenon could be used to enhance the splice strength by using the alumino-silicate glass as a filler material. The refractive index is greater than that of silica (1.458) and is less than that of sapphire (1.768). This could result in minimizing the optical losses due to the refractive index variation at the silica-sapphire interface. Some of the important specifications of this glass are:

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Thermal expansion</td>
<td>$4.6 \times 10^{-6}$ cm/cm/°C</td>
</tr>
<tr>
<td>Strain point</td>
<td>665 °C</td>
</tr>
<tr>
<td>Annealing point</td>
<td>710 °C</td>
</tr>
<tr>
<td>Softening point</td>
<td>908 °C</td>
</tr>
<tr>
<td>Working point</td>
<td>1168 °C</td>
</tr>
<tr>
<td>Density</td>
<td>2.64 gm/cm$^3$</td>
</tr>
<tr>
<td>Young’s Modulus</td>
<td>$8.8 \times 10^3$ Kg/mm$^2$</td>
</tr>
</tbody>
</table>
Refractive index : 1.547

Upper working temperature (mech.) : 650 °C

An aluminosilicate glass made by Corning Glass Works (Corning glass code #: 1723) was obtained. A platinum crucible was used to heat the aluminosilicate glass up to its working point (~ 1160 °C). The heating was done gradually over a period of about 10 minutes. As the temperature increased to approximately 1160 °C, the glass began to ‘flow’. The silica and sapphire fibers were then dipped into the molten glass. The ends of the fibers were cleaved (or polished in the case of sapphire fibers) before being dipped into the molten glass. These ‘coated’ fibers were then spliced using the arc-fusion splicer. Three different approaches were attempted:

1) a coated sapphire fiber was spliced with an uncoated silica fiber.
2) a silica fiber, coated with the glass, was spliced with a coated sapphire fiber.
3) a coated silica fiber was spliced with an uncoated sapphire fiber.

Some experimentation was required to determine the appropriate current and feed rate settings on the arc-fusion splicer. Subsequently, it was observed that in the first of the three approaches, the ALG glass softened and pulled on the uncoated silica fiber, resulting in a strong bond between the ALG glass and the silica fiber. Cooling did not seem to adversely effect the splice. In the other two approaches, the ALG glass on the coated silica fiber did soften to form a bond with the coated and uncoated sapphire fiber, respectively. But, upon cooling, the ALG glass on the silica fiber cracked and separated from the silica fiber. This was observed regardless of the duration of the arc time. This is probably due.
to the large disparity that exists between the coefficients of thermal expansion of the aluminosilicate glass (~ $4.6 \times 10^{-6}$ / °C), and the silica fiber (~ $0.75 \times 10^{-6}$ / °C), which will result in thermal stresses on cooling. The splice obtained in the first approach was strong because the CTE of sapphire fiber (~ $5.3 \times 10^{-6}$ / °C, along the c-axis, and ~ $4.5 \times 10^{-6}$ / °C, perpendicular to c-axis) is in close agreement with that of the aluminosilicate glass. So a number of splices were attempted with an uncoated silica fiber and a coated sapphire fiber, each time varying the settings on the arc-fusion splicer to obtain the optimal values as reflected in the quality of the splice. The coated sapphire fiber was polished so that the polished endface of the sapphire fiber was exposed, i.e., there was no ALG covering the sapphire endface.

![Splice loss vs Wavelength graph](image)

**Figure 14.** Attenuation in the ALG ‘binder’ splice

Silica-sapphire fiber splicing techniques
The optical losses of three splices, thus obtained, were measured using the FOA 2000 fiber characterization system. The results of the best splice are illustrated in Figure 14. The attenuation in the sapphire varied from 1.4 dB to -0.2 dB. This represents the best optical result of the splices that have been made with the techniques discussed in this thesis report. This technique is the ‘binder’ splice.

The breaking strength of the splices obtained by this technique were quantitatively evaluated using a J.J. Lloyd T-20,000 load frame. A 2 KN load cell was used to measure the applied load. An illustration of one of the grippers used is shown in Figure 15. The fiber was wrapped three turns around one of the one inch diameter mandrels and then secured under the clamp. With the fibers firmly wound around the mandrel, the two grippers are slowly pulled apart, thus increasing the tension on the splice. The grippers are pulled apart until the splice breaks. This procedure was attempted on a number of splices.
In a majority of the tests, the splice or the sapphire fibers broke while loading the fibers onto the grippers, due to the stiff, brittle nature of the sapphire. For the three splices successfully tested so far, the breaking loads were 4.7 N in one case, 5.8 N in another case, and greater than 6.3 N in a third case. In the last case, the silica fiber broke with the splice intact, so that the strength of the splice exceeded 6.3N. Converting this value into the units of stress would result in a breaking stress of greater than 74.5 kpsi for the best splice. It should be noted that the proof test limit on the Corning silica fiber is 50 kpsi. It is also imperative to note that these measurements of breaking stress of splices were done with splices with minimum alignment between the sapphire and silica fibers. So, these splices were strong mechanically, but were not as good optically.

2.4.2 ‘Modified’ binder splice approach

The ‘modified’ binder splice approach is essentially similar to the splicing technique described in 2.4.1. Previous work involving aluminosilicate glass, reported in the last section, employed the glass as a binder between the sapphire and silica fibers. But, the procedure of polishing the coated sapphire fiber, with no ALG left to cover the endface meant very little ALG was present on the sapphire fiber to enhance the splice strength. Thus, the splices obtained with this technique were very weak. A revised technique was attempted in which the coated sapphire fiber was polished with a very thin layer (<100μm) of ALG covering the polished endface of the sapphire fiber. Splicing of silica fiber to the coated sapphire fiber was then done using the fusion splicer. This difference in the techniques is illustrated in figure 16.
The resultant splice appeared to be mechanically strong (based on subjective evaluation), but has not yet been quantitatively tested for strength. The splice loss in this improved splice was obtained on the FOA-2000 fiber characterization system. The combined loss of the splice and the sapphire fiber at 820 nm was 8.5 dB. Subtracting the 7.1 dB loss due to the sapphire, the splice loss was found to be 1.4 dB. The splice losses for varying wavelengths, calculated in a similar fashion, are plotted in Figure 17.

The splice was analyzed for its structure and composition using a scanning electron microscope. Figure 18 shows a side view of the splice, and Figure 19 is a view of the fractured end of the splice after load-to-failure tests. Fig. 20 tabulates the surface elemental composition determined by EDX spectroscopy. In the photomicrographs, there is no

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(i)  

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(ii)  

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Figure 16. Modification of the ‘binder’ splice approach

trace of the needle-shaped structures that are characteristic shape of mullite crystals. To Silica-sapphire fiber splicing techniques
confirm the absence of mullite formation, EDX analysis was performed on the interface obtained by cleaving the spliced sapphire fiber, right on the splice. The results of EDX analysis indicate that the percentage by weight of silicon in the composition is 50%, while that of aluminum is 15%. This does not correlate with the corresponding compositions for a reference mullite sample (as reported in the previous report), nor with those obtained with other splicing techniques. The absence of mullite is likely responsible for the low splice losses that have been observed with this splicing technique, because mullite is generally not totally transparent. Presently, it appears that the ALG forms an intermediate compound with silica that enables a good mechanical as well as optical bond.

![Graph showing attenuation in a 'modified' binder splice](image)

*Figure 17. Attenuation in a 'modified' binder splice*

**Silica-sapphire fiber splicing techniques**
2.4.3 Splicing with intermediate aluminosilicate glass fillers

Another splicing technique was attempted and evaluated, in which a short, fiber-like piece of aluminosilicate glass was used as an intermediate filler between the silica and the sapphire fibers. In the first step, a piece of aluminosilicate (ALG) glass was heated until fluid in a platinum crucible, and a fiber of aluminosilicate glass was ‘drawn’ from the molten flux of aluminosilicate glass. A piece of sapphire fiber was used as a ‘seed’ in this drawing process, which resulted in the glass fiber having approximately the same diameter as the sapphire fiber. This ALG ‘fiber’ was then cleaved using the same procedure that is used to cleave silica fibers. This resulted in a clean, flat endface on the ALG fiber. In the next step, the cleaved end of the ALG fiber was spliced to a sapphire fiber on an arc-fusion splicer. One difficulty in splicing the ALG fiber to the sapphire was that the ALG fiber

![ALG 'fiber' splice diagram]

Fig. 21 ALG ‘filler’ splice

Silica-sapphire fiber splicing techniques
melts at a lower temperature than the sapphire, leading to non-uniform melting of the ALG glass on the sapphire fiber endface. This was solved by adjusting the rate at which the ALG glass was fed into the splice on the arc-fusion splicer. In the final step, the spliced ALG fiber was then cleaved again, and spliced to a silica fiber.

The resulting splice was measured for optical losses by using the Photon Kinetics FOA 2000 optical fiber characterization system. The results are given in Fig. 22. The average optical loss in the splice and sapphire fiber was approximately 13 dB at 850 nm. Given that the length of the sapphire fiber used was 3.7 cm, the loss in the sapphire fiber was about 5.5 dB, assuming an attenuation of 1.5 dB/cm in the structural grade sapphire fiber. This suggests that the splice loss was 6.5 dB. This is comparable to the attenuation of some of the splices obtained with other techniques.

![Attenuation in the filler splice](image)

**Figure 22.** Attenuation in the filler splice

Silica-sapphire fiber splicing techniques
2.4.4 Aluminosilicate glass intermediate splice enclosed in a silica tube

A splice technique was developed in which a silica capillary tube was used to align the fibers and contain the aluminosilicate glass fiber used as a jumper. The capillary tube allows better control over the positioning of the fibers, as well as the flow of the molten aluminosilicate glass. In the splices using aluminosilicate glass, discussed in 2.4.1, the aluminosilicate glass would contract into a ball upon melting, and accumulate on the sapphire fiber, causing a weak bond between the silica fiber and the aluminosilicate glass. This aluminosilicate glass sphere also made alignment of the silica and sapphire fibers difficult. During the actual process of the splicing, the molten aluminosilicate glass sphere would pull on the fiber in a non-uniform manner.

In this technique (as illustrated in Fig. 23), a small piece of the aluminosilicate glass 'fiber' of about one millimeter length was inserted into a silica capillary tube. Subsequently, a sapphire fiber (polished at both the endfaces) and a silica fiber (with cleaved endfaces) were inserted into the capillary tube from each end. The capillary tube was then positioned on the arc-fusion splicer, such that the aluminosilicate glass fiber piece was between the electrodes. The silica fiber and the sapphire fiber were then clamped to the fiber platforms, allowing the fibers to be moved in and out of the capillary tube. The capillary tube is free to move axially, and is not clamped along its length. The inner diameter of the capillary tube was approximately 190 μm, while the diameters of the sapphire and silica fibers were 145 μm and 125 μm, respectively. The structural grade sapphire fibers used in the experiment were prepared by flame polishing their surfaces.

Silica-sapphire fiber splicing techniques
The arc-current setting on the arc-fusion splicer was adjusted so that the aluminosilicate glass fiber piece inside the hollow core melted without any effect on the structure of the hollow core fiber. The clamped sapphire and silica fibers were then 'pushed' together in the molten aluminosilicate glass, while the arc was maintained. In this way, the longitudinal offset between the silica and sapphire fibers was reduced, with the excess aluminosilicate glass melt being squeezed into the gap along the sides of the fibers. This was intended to produce a uniform layer of the aluminosilicate glass on the fibers, and reduce the lateral offset of the fibers.

![Diagram](image)

**Figure 23. Schematic of the splicing technique**

The optical attenuation of one splice, obtained using this technique, was measured by the cut-back technique using a FOA-2000 optical fiber characterization system. The results are illustrated in Figure 24. The loss of the splice was found to be about 3 dB. The actual **Silica-sapphire fiber splicing techniques**
splice losses may be lower for this splice, since the sapphire fiber endfaces were not polished to an optical grade and may be responsible for some of the losses.

Attempts to measure the mechanical strength or the breaking stress point of the splice using a J.J. Lloyd T-2000 Load frame were unsuccessful because the splice was very weak and could not be loaded onto the load frame. The low strength of the splice could be due to stresses induced in the capillary tube by the aluminosilicate glass, which expands radially faster than the silica tube when heated. In some unsuccessful attempts to fabricate a splice with this technique, cracks were evident in the silica tube after splicing, when examined under a microscope. The use of sapphire tubes rather than silica tubes will eliminate this problem, since the coefficient of thermal expansion of the sapphire is close to that of the silica-sapphire fiber splicing techniques.

Figure 24. Attenuation in a silica tube splice

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aluminosilicate glass.

Since the silica tube has a relatively low softening point, close to that of the aluminosilicate glass (908°C), it is difficult to obtain an optimal temperature to melt the aluminosilicate glass without effecting the capillary tube. Also, the coefficient of thermal expansion (CTE) of silica (~0.53 x 10⁻⁶) is approximately 10 times lower than that of the aluminosilicate glass (4.6 x 10⁻⁶). This mismatch in the CTE’s of the two materials causes thermal stresses in the splice region, on cooling, tending to weaken the splice. To counter both of these problems, it was proposed to use sapphire capillary tubes. The large disparity in the softening points of sapphire and aluminosilicate glass would result in a greater range to optimize the splicing temperature. Secondly, the similarities in the CTE’s of the two materials would result in reducing the thermal stresses in the splice region, thus improving the strength of the splice.

A modification to the above technique of splicing a sapphire fiber to a silica fiber with an aluminosilicate glass fiber as an intermediate material was attempted, in that the oxy-propane flame was used instead of the splicer. In this approach, a sapphire tube was used as an ‘enclosure’ or splice tube.

In the technique using the fusion splicer, the heating zone on the arc-fusion splicer is very small, defined primarily by the size of the tip of the electrodes. This means that a very little portion of the aluminosilicate glass will soften, constraining it to a very narrow space at the interface. So, to provide heat spread uniformly over a larger area, an oxy-propane torch was used instead of the arc-fusion splicer. Three splices were fabricated with silica
capillary tubes and aluminosilicate intermediates, using the oxy-propane torch to heat the assembly. In each of the three attempts, the silica tube bulged and fractured before the aluminosilicate glass softened enough to flow. This result indicated that the size of the heating zone was not the cause of the problem, but that the CTE mismatch was more likely the cause.

The use of a sapphire capillary tube appears to present the most reasonable prospects for success. The CTE of sapphire is very close to that of the aluminosilicate glass used, so that little or no compressive stresses should be generated when the assembly is heated.

In the modification employing the sapphire tube, a small piece of the aluminosilicate glass ‘fiber’ (approximately 1 mm long by 125 μm diameter) was introduced into the sapphire tube. Subsequently, a sapphire fiber (polished at both the endfaces) and a silica fiber (with cleaved endfaces) were inserted into the tube, from the two ends. The inner diameter of the tube that was used was ~ 250 μm, while the diameters of the flame-polished sapphire and silica fibers were ~ 140 μm and ~ 125 μm, respectively. The sapphire fiber and the silica fiber were then fixed to a stationary and a translational stage, respectively. Both fibers were then moved into the tube until they touched the aluminosilicate glass endface. The silica fiber was inserted into the tube an additional distance approximately equal to the length of the aluminosilicate glass piece in the tube, so that the fiber was bent, indicating an axial compression of the silica fiber. The flame from a oxy-propane torch was introduced gradually and the tube was heated to about 950°C, so that the aluminosilicate glass softened and allowed the silica fiber to force itself into the molten aluminosilicate glass. The heating was maintained until the silica fiber straightened out. The technique is illustrated in Silica-sapphire fiber splicing techniques.
Figure 25. It should be noted that the sapphire tube was free and was not clamped along its length.

The flame of the oxy-propane torch was adjusted so that the aluminosilicate glass fiber piece inside the sapphire tube melted without any effect on the structure of the tube. This required some trial and error methodology, before the correct flame size could be determined. The distance through which the silica fiber moves into the melt was approximately equal to the initial length of the aluminosilicate glass. This means that the longitudinal offset between the silica and sapphire fibers was minimized as much as possible, with the excess aluminosilicate glass melt being squeezed out onto the sides of the fibers. This, in turn, should mean that there is a uniform layer of the aluminosilicate glass on the side of the fibers, and the lateral offset of the fibers should be minimized. To

![Diagram of splicing technique]

Fig. 25 Schematic of the splicing technique

Silica-sapphire fiber splicing techniques
minimize the thermal induced stresses during the cooling process, at the splice region, the
flame was gradually removed from the sapphire tube. Secondly, both the fibers were
gently pushed toward each other during the cooling phase of the splice region.

The optical attenuation of the splice, thus obtained, was measured on the FOA-2000 and
Fig. 26 illustrates the losses.

![Graph showing attenuation in a sapphire tube splice](image)

**Figure 26.** Attenuation in a sapphire tube splice

### 2.4.5 Splices using high temperature epoxies / adhesives

Since the proposed maximum operating temperature for the silica/sapphire splice is only
500°F, the possibility of using optically transparent epoxies or adhesives to act as a
mechanical binder between the two fibers was investigated. The chosen compound must be

**Silica-sapphire fiber splicing techniques**
able to withstand at least 500 °F, on a continuous basis. It should also be rigid enough to
give mechanical support to the splice and to prevent any misalignment between the fibers,
after the splice has been fabricated.

As a step in this direction, a survey of vendors that manufacture high temperature epoxies
or adhesives was undertaken and a candidate adhesive was identified. The specifications
for this material, a silicone adhesive, are given below:

* Fixmaster High Temperature Red Silicone:

FEATURES: High temperature resistance, permanent flexibility, excellent resis-
tance to aging, vibration and shock resistant, bonds to most clean surfaces.

PHYSICAL PROPERTIES: Tensile strength - 630 psi / 4.34 MPa

% Elongation - 200%

Tear strength - 12.3 lb./in.

Cure time - 24 Hours

Max. Temperature 600°F / 315°C.

A splice was fabricated using this epoxy, and characterized for its optical as well as
mechanical properties.

Another approach that was investigated involved using the silicone adhesive to cast a
‘mold’ of a splice tube, similar to the commercially-available elastomeric splice tubes. This
approach closely follows the design disclosed by Virginia Tech researchers K.A. Murphy
Silica-sapphire fiber splicing techniques”
Making Same,” awarded on 10 April 1990. In that design, a room-temperature-vulcanizing (RTV) elastomer is cast in a glass cylinder mold, with an optical fiber held in the center of the mold by two glass funnels. The funnels can be made from common laboratory pipettes. When the elastomer has cured, the funnels and the fiber are withdrawn, leaving a clear bore into which two optical fibers may be inserted from opposite ends. Insertion of the fibers is facilitated by the impression left by the funnels in the ends of the elastomer. If the optical fiber used in the fabrication of the mold is chosen to have a diameter slightly less than that of the fibers to be connected in service, then the elastomer will exert a radial compressive force on the spliced fibers, centering them and holding them in place.

![Diagram of the fabrication process](image)

Figure 27. Cross section showing fabrication of Murphy/Zimmerman optical fiber splice.

In the modification of this concept for the silica-to-sapphire splice, the Fixmaster high temperature silicone adhesive was used in place of the RTV elastomer. The glass tube used had an inner diameter of 5 mm. After letting the silicone cure for several days at room
temperature, it was observed that the inner layers of the silicone were not cured. The experiment was repeated with the silicone cured at 100 °C for eight hours. With this cure schedule, the silicone cured throughout the volume of the specimen. However, in this case, the silicone adhered so well to the fiber and funnels that they could not be withdrawn from the mold. For the next step in this approach, the fiber and the funnels could be coated with a mold release agent before the silicone is cast in the mold.

2.5 Measurement of attenuation in the sapphire fiber

In order to more accurately gauge the optical losses occurring at the splice between the silica and sapphire fiber, the attenuation in the sapphire fiber was measured. The particular fiber investigated was a 125 μm diameter structural-grade uniaxial crystalline sapphire fiber made by the EFG technique by Saphikon, Inc. The measurement was accomplished by using a Photon Kinetics FOA 2000 optical fiber characterization system. The measurement technique is illustrated in Figure 28. A sapphire fiber approximately 6 cm long and polished on both the ends was fusion spliced to a silica optical fiber using alumino-silicate glass, as described in the later part (Section 2.2) of this report. Light from a white lamp filtered by a monochromator was coupled into the silica fiber and the output power was measured at the sapphire fiber endface for varying input wavelengths (Figure 28i). Then, without moving the input end of the silica fiber, the sapphire fiber was cut into two parts, and the spliced part of the sapphire fiber was polished (Figure 28ii). Subsequently, the output power out of the re-polished sapphire output endface was measured to determine the reference power (the power input into the sapphire fiber). The attenuation in the sapphire fiber is given by

Silica-sapphire fiber splicing techniques
attenuation (dB/cm) = \frac{10}{\text{length (cm)}} \log_{10} \frac{P_{\text{out}}}{P_{\text{in}}}

(i) measurement of power $P_{\text{out}}$ out of sapphire fiber

(ii) cut-back and polishing of sapphire fiber

(iii) measurement of reference power $P_{\text{ref}}$

Figure 28. Technique for measuring attenuation of structural sapphire fiber.

Silica-sapphire fiber splicing techniques
The attenuation in the sapphire fiber was measured to be about 1.8dB/1.3 cm, or 1.5dB/cm.

One reason for this high attenuation is the surface roughness of the fiber, which results from the edge defined fiber growth (EDFG) fabrication technique. It was reasoned that by smoothing out the surface roughness, the optical losses can be diminished by reducing the surface scattering effects. The use of an oxy-propane torch to “flame polish” a sapphire fiber in order to reduce the surface scattering, and consequently the optical transmission losses of the fiber, was explored.

In this technique, a piece of sapphire fiber is held by a fixture. A moving torch is brought into close proximity to heat the sapphire fiber. The flame is moved along the length of the sapphire fiber, repeatedly, so that the outer periphery of the sapphire fiber melts slightly and then quickly recools. The achievement of the proper temperature, where it is only enough to melt the superficial layers and not melt the whole fiber, was found to be critical to the success of the technique. The attenuation in a fiber thus obtained was measured using the FOA-2000 characterization system. The results are detailed in Fig. 30. The attenuation at 850 nm was 4.3 dB for a length of 3.6 cm. Thus, the attenuation normalized for length was 1.1 dB/cm. It should be noted that the procedure used to measure the attenuation in the flame-polished fiber was same as that used to measure the attenuation in an unpolished fiber.

The attenuation of the flame-polished sapphire fiber is likely to be less than that reported due to difficulties encountered in polishing the sapphire end after the sapphire was cut back for the reference (input) power measurement. Rather than risk breaking the fiber or splice...
during additional polishing, it was thought prudent to finish the measurement and accept a conservative measure of the attenuation. Since the attenuation in the sample is given by equation (1), scattering from an improperly prepared endface will reduce the measured reference power $P_{in}$, increasing the calculated attenuation. Due to the promise of this approach for lowering the sapphire fiber loss, work was focused in the development of the technique and the measurements were repeated.

2.6 Polishing of sapphire fibers

Since single or polycrystalline sapphire has basic crystalline structure, it is almost impossible to achieve a perfectly flat surface on cleaving the fiber using a diamond-tipped scribe. The cleaved endface has many facets. To overcome this problem, the sapphire silica-sapphire fiber splicing techniques
fiber has to be polished on it's endface, everytime a sapphire fiber with a flat or perpendicular endface is required.

The procedure that has been adopted to polish the structural grade fiber primarily involves the use of a metallurgical polishing wheel. The sapphire fiber is mounted into a groove on a polishing block. Care should be taken to let a small length of the fiber to overhang the polishing edge of the block. This is illustrated in Fig. 31. The ‘overhang’ of the sapphire

![Schematic of sapphire fiber polishing technique](image)

Figure 31. Schematic of sapphire fiber polishing technique

fiber is then completely covered with phenyl salicylate, which is a solid at room temperature. This covered end of the sapphire fiber is then polished using polishing pads of increasing smoothness. To begin with, a 600 grid polishing pad is used, followed by a 3 \( \mu \)m grid, and then followed by a 0.5 \( \mu \)m grid polishing pad. During the polishing, extreme care should be taken to ensure that the sapphire fiber is as perpendicular to the

Silica-sapphire fiber splicing techniques
polishing pad as possible. Besides, pressing the polishing block against the rotating pad should be avoided, as this would cause destructive abrasion.

Conclusions

A conclusion that was made during the course of this thesis effort is that using mullite - either as a coating on the fibers or as an intermediary compound in the silica-alumina system- appears to be impractical and is not recommendable based on our current understanding. A primary reason is that obtaining transparent mullite commercially has proven to be very difficult. Coating the fibers with transparent mullite, synthesized in-house, is the other alternative. The process is highly involved, including the grinding the alumina and silica source into very finely ground powders. This would result in smaller grain size of the resultant mullite, resulting in smaller pores and hence transparency. The next step would be to make a very accurate stochiometric measurement of these powders to make up a composition (~33% silica) that would induce the formation of mullite on gradual heating and subsequent cooling of the mixture. Further, the coating of the transparent mullite, thus obtained, would be problematic, as the melting point of mullite is ~1840°C, much higher than that of silica. This would mean that the coating can be done only on the sapphire fiber. Even after obtaining fibers coated with transparent mullite, heating them to melt the mullite, with subsequent cooling, would probably lead to the recrystallization of the mullite that could result in loss of transparency in the mullite coating.

The experiments that have been carried out thus far, to study the possibility of diffusion across the silica-sapphire interface have been relatively discouraging. The resulting bonds
produced to date have been neither strong nor permanent. These results suggest that diffusion-induced splicing of silica and sapphire fibers may not produce an optimal splice from a mechanical and optical viewpoint.

A careful study of the results of various experiments performed on the arc-fusion splices (like attenuation and breaking stress) suggests that the arc-fusion approach produces splices with relatively low optical losses (9.5 - 5.5 dB), but, the mechanical strength of the splices is very questionable. One of the reasons for this is the large difference in the CTEs of sapphire and silica fibers, that results in great thermal-induced stresses everytime the splice experiences variations in the surrounding temperature. As was described in the appropriate section, the splice was required to be ‘reinforced’ with finger nail polish or similar material to enable the splice to withstand stresses applied during normal usage. This, in turn, means that the maximum usage temperature of this splice is limited by the temperature that this ‘reinforcement’ material can withstand. An advantage of this splicing technique is the excellent control on the alignment of sapphire and silica fibers that can be obtained. Thus, in environments where the temperature is not greater than, say, 70 °C, this splice can be used satisfactorily.

The splice obtained by fusing a silica coated sapphire fiber to a bare silica fiber exhibited relatively low optical losses while the mechanical strength was greater than that of the splices obtained by the diffusion and arc-fusion approach. Unfortunately, the problem of large disparity in the CTEs of sapphire and silica still existed which resulted in large thermal stresses at the splice region for large temperature fluctuations. The problem was not as aggravated as in the other two cases because the major interface was between two similar Silica-sapphire fiber splicing techniques -Page43-
materials, i.e., silica. Another disadvantage with this splicing technique is the necessity of the relatively expensive and time consuming process of coating silica on the sapphire fiber using Chemical Vapor Deposition (CVD). Besides, the length of the sapphire fiber that can be coated with silica is limited by the size of the substrate holder in the CVD chamber. Once again, a very good control over the alignment of the fibers to be spliced is possible. Hence, sensing environments with relatively high temperatures (less than 650°C) and low working stresses are conducive for a splice fabricated using this technique.

The splices using aluminosilicate glass hold out promise of providing an ‘ideal’ splice. The reason is that using aluminosilicate glass enhances the possibility of formation of intermediate compounds between silica and sapphire thus improving the quality of the splice. The ‘binder’ splice using the aluminosilicate glass as a coating material on the sapphire fiber endface results in a splice with very low optical losses at the splice. In fact, a splice with losses less than 1 dB (the best during the course of this research effort) was obtained by using the ‘binder’ splice technique. One reason is that this technique makes it possible to have remarkable control in the accurate alignment of the core regions of the singlemode silica and multimode sapphire fiber, which is imperative to obtain a splice with low optical losses. Secondly, the silica and sapphire fibers can be positioned in immediate contact with each other before melting the aluminosilicate glass onto the sides of the silica fiber. Thus, there is aluminosilicate glass in between the silica and sapphire fibers. Dispersion losses (as in the non-uniform melt of aluminosilicate glass) are reduced with no glass in between the two fibers. Unfortunately, the mechanical strength of the splice, thus obtained, is not as good as its optical performance because the splice is, in actual terms, being held by the little melt of aluminosilicate glass that is present on the sides of the sapphire fiber. In other

Silica-sapphire fiber splicing techniques
words, there is a limit on the amount of glass that can be melted on the endface of the sapphire fiber, prior to the splicing. This is because the larger the melt is, the harder it gets to melt the entire glass onto the silica fiber, using the arc current between the electrodes on the fusion splicer. This limited quantity of the glass prohibits the fabrication of a mechanically strong splice. It should be noted that in this splice, neither the silica fiber nor the sapphire fiber is melted. It is only the aluminosilicate glass that is melted over the interface. Thus, in applications which demand a very low optical loss splice, with relatively low working stresses, the ‘binder’ splice is very well suited.

The ‘modified’ binder splice is an improved version of the binder splice. Unlike the previous technique, in the ‘modified’ approach, there is some aluminosilicate glass in between the silica and sapphire fibers. This results in a stronger splice but, with slightly higher optical losses for reasons explained in the above paragraph. Thus, the modified binder splice technique provides a ‘trade-off’ mechanism between the mechanical strength and the optical losses of the splice. The degree of this trade-off can be controlled by the thickness of the aluminosilicate glass layer that is left to remain on the sapphire fiber endface, before splicing it to a silica fiber.

The ‘tube’ splice approach should result in the best splice form the mechanical strength viewpoint because the heating and cooling of the splice region is performed gradually, thus preventing thermally-induced stress generation. Moreover, with the aluminosilicate glass uniformly spread out on the sides of the fiber and enclosed by the tube, the splice is strengthened considerably. The tube also provides an adequate means to align the fibers and the ALG ‘fiber’ accurately before splicing. As was discussed, splices with optical

Silica-sapphire fiber splicing techniques
losses of less than 2dB were fabricated. Unfortunately, sapphire tubes are currently available only in 250μm and 125μm diameter sizes. The former is too large when compared to the singlemode clad diameter and the core diameter of the sapphire fiber and the later is too small. Due to this large disparity in the diameters, perfect control over the alignment of the silica and sapphire fibers, inside the sapphire tube, has not yet been achieved. With the availability of sapphire tubes of the suitable diameter (~ 145 μm inner diameter), this problem can be solved and splices with losses of less than 1dB can be made possible. Besides, another advantage of this splicing technique is the relative strength. The splices fabricated with this technique offer the greatest strength and flexibility as they are enclosed in a sapphire tube. This tube provides excellent protection to the splice region from environmental degradation.

Using high temperature epoxies to mechanically hold the sapphire and silica fiber together has not shown much of a promise, so far. One reason is the limited number of high temperature epoxies (greater than 500°F) that are commercially available. Secondly, the requirement that they have high post-cure rigidity for providing mechanical support to the splice, further narrows down the available choice. The limit on the cure time (less than 24 hours) has also caused problems in fabricating a usable splice, using this technique.
Figure 5. Sapphire rod end enlargement (SEM)

Figure 6. Silica rod enlargement (SEM)

Silica-sapphire fiber splicing techniques
EMI-QUANTITATIVE ANALYSIS: SIO2 FIBERS
NORM. K-RATIO

$-K \ 0.25554 \quad +\pm \ 0.00286$
$-K \ 0.48535 \quad +\pm \ 0.00225$
$I-K \ 0.25912 \quad +\pm \ 0.00159$

AF CORRECTION 20.00 KV 34.00 Degr

no. of iterations 0

---  K [Z] [A] [F] [ZAF] ATOM.% WT.%
C-K 0.256 0.966 2.763 0.999 2.669 51.20 37.97 *
Al-K 0.485 1.038 1.247 0.991 1.284 27.72 34.68
Zn-K 0.259 1.005 1.087 1.000 1.897 21.08 27.35 *
* - High Absorbance

Figure 7. EDX Analysis of the diffusion splice

Silica-sapphire fiber splicing techniques
Figure 8. Photomicrograph of an arc-fusion splice

Silica-sapphire fiber splicing techniques
Figure 9. EDX analysis of the arc-fusion splice
Figure 18. Side view of the modified binder splice (SEM)

Figure 19. Photomicrograph of the fractured end of a modified splice after load-to-failure tests

Silica-sapphire fiber splicing techniques
**SEMIS-QUANTITATIVE ANALYSIS: ALG 'BINDER' SPLICE**

**EL**  NORM. K-RATIO

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**ZAF CORRECTION**  20.00 KV  26.92 Degr

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* - High Absorbance

**SEMIS-QUANTITATIVE ANALYSIS: ALG 'BINDER' SPLICE**

**EL**  NORM. K-RATIO

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**ZAF CORRECTION**  20.00 KV  26.92 Degr

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* - High Absorbance

Figure 20. EDX analysis of the modified binder splice

Silica-sapphire fiber splicing techniques
3.0 SAPPHIRE FIBER-BASED INTRINSIC FABRY-PEROT INTERFEROMETRIC SENSOR

3.1 Introduction

Fiber-optic Fabry-Perot (FP) sensors have been shown to be highly sensitive to strain, temperature, pressure and acoustic waves [11]. Since such sensors offer the advantages of simple configuration, single-ended operation, high resolution and potential low cost, different methods for the construction of FP cavity and output signal demodulation have been developed over the past few years [12, 13, 14]. Fiber FP sensors can be categorized into two classes, namely intrinsic and extrinsic FP interferometric sensors (IFPI and EFPI). A balanced fiber IFPI sensor for temperature measurement has been demonstrated by Lee et al [15]. Recently, Wang et al. demonstrated a lensed fiber EFPI sensor by splicing a length of multimode fiber, functionally equivalent to a quarter- pitch GRIN lens, to a single mode fiber [16]. In this way, the dynamic measurement range of the sensor has been significantly increased.

Since silica fiber becomes soft when its temperature of operation exceeds about 1000°C, silica fiber-based FP sensors are strictly limited to low temperature applications. Over the past fifteen years, several fiber-optic high temperature sensors have been demonstrated by different researchers. A sapphire rod-based high-temperature sensor was demonstrated by Dils et al [17]. In this sensor, platinum with a high melting point was deposited onto the fiber tip to serve as a blackbody radiator. The measured temperature information was extracted by taking the ratio of two specifically selected wavelengths emitted by the Sapphire IFPI sensor.
blackbody. Later, Murphy et al. proposed and demonstrated single crystal sapphire-rod based fiber EFPI sensor for measurement of temperature and acoustic waves [18]. This chapter of the thesis describes a sapphire fiber-based IFPI sensor in which the FP cavity is based on a length of multimode sapphire fiber. An external perturbation of the sapphire fiber (such as a temperature change) would result in a change in the length of the propagation path. The sapphire fiber is operated as a low-finesse Fabry-Perot cavity.

3.2 Experiment

The schematic of the sapphire fiber interferometer is shown in Figure 32. The interferometer is comprised of a sapphire fiber as the probe which can withstand harsh environments and high temperatures. A length of multimode sapphire fiber is connected by fusion splicing to a single mode silica fiber to form the Fabry-Perot (FP) cavity. A laser beam is launched into the single mode silica fiber and propagates to the sapphire fiber. Due to the difference in the refractive index between the silica (1.46) and sapphire fibers (1.73), a fraction (about 0.4%) of the incident laser power is reflected. The light transmitted into the sapphire fiber excites propagation modes. Since the numerical aperture of the single mode fiber is very small, primarily low order modes in the sapphire fiber are excited. The propagating light in the sapphire fiber is partially reflected at the free endface in air. Without reflection coatings, this endface provides about 7% reflectance caused by Fresnel reflection. At the silica-sapphire fiber splice, a fraction of this reflected light from the free endface of the sapphire fiber is recoupled into the silica single mode fiber. The interference of the two reflections - from the silica-sapphire fiber splice and the free endface of the sapphire fiber, respectively - gives rise to the interference fringe output. Since the optical Sapphire IFPI sensor
phase of the light traveling in this sapphire fiber is highly sensitive to temperature and longitudinal strain, this sensor is useful for the measurement of these parameters.

The experimental set-up is illustrated in Fig.33. A laser beam from a 1300nm pig-tailed semiconductor laser injects optical power into one leg of a 2x2 fused biconical optical fiber coupler. Approximately, 50% of the this power is divided into each of the output legs of the coupler. The leg which is not attached to the single mode fiber is placed in index-matching gel, to prevent any noisy back-reflections. The other leg is attached to a single mode silica fiber with a dimension of 9/125 μm and a numerical aperture of 0.13, using a GTE splice tube.

A 31 mm length of uncoated ‘structural-grade’ sapphire fiber with a diameter of 140 μm was spliced to this single mode silica fiber using the ‘modified’ ALG binder splice technique, as described in the previous chapter. The small length of sapphire fiber thus forms a low-finesse Fabry-Perot (FP) cavity. The sapphire fiber, which has a melting point of 2053°C, was fabricated by Saphikon, inc. of Milford, NH, by an edge defined fiber growth (EFG) technique. The light traveling back from the sapphire fiber cavity is divided by the biconical coupler and a part of this output is detected by a photodetector and the related electronics transform the detected output into an interference fringe pattern on the oscilloscope. The problem of laser drift, caused by the back reflections from the light of the other leg of the coupler can be avoided by optically isolating the laser. An obvious advantage of this configuration is that the alignment problem that is encountered in a typical air-gap sensor is no longer a problem. this eliminates one potential source of signal degradation. A disadvantage is that the entire length of the sapphire fiber is used as the Sapphire IFPI sensor
sensing region, i.e., the gage length of the sensor is long and so the dynamic range as well as the stability of the sensor is limited. This is aggravated if the sensed parameter, like temperature or pressure, is not uniformly distributed along the length of the sensor.

In the experiment, it was found that the fringe contrast of the sensor output was dependent on the quality of the silica fiber to the sapphire fiber splice. Specifically the fringe contrast is a function of the relative position between the silica and sapphire fibers at the splice. To optimize the sensor performance, it is necessary to study this effect. To precisely change the relative position between the fibers, both the silica and sapphire fibers were mounted onto two micro-positioners. To eliminate the Fabry-Perot cavity formed by the air-glass interfaces at the fiber splice, an index matching liquid with a refractive index of 1.548 (refractive index of ALG) was used to fill the gap between the two fibers.

This set-up is similar to that of the sensor that was used for high temperature measurements. In that sensor, a thin layer of ALG is sandwiched between the fiber endfaces at the fiber splice and the refractive index of the ALG glass is 1.547. To determine the dependence of the fringe contrast of the sensor output on the relative position between the silica and sapphire fibers at the fiber splice, the temperature of the sapphire fiber was changed slightly to obtain a several fringe shift in the sensor output for each relative position. Figures 34 and 35 show the normalized fringe contrast as functions of the lateral offset and the longitudinal gap between the endfaces of the two fibers at the fiber splice. It can be noted that the lateral offset and the longitudinal gap should be controlled within ~10 µm and ~25 µm, respectively, for the fringe contrast to be within acceptable limits. In the experiment, the splice was achieved using alumino-silicate glass deposited on Sapphire IFPI sensor.
the sapphire fiber end and a cleaved single mode silica fiber was then spliced to the ALG-covered end of the sapphire fiber. To achieve more reflection power at the free endface of the sapphire fiber coupled into the single mode fiber, it is imperative that the single mode fiber be positioned as close to the center of the sapphire fiber endface as possible. Besides, it is also imperative that the endface of the sapphire fiber must be polished as close to perpendicular to the c-axis of the sapphire fiber, as possible. The lateral offset was controlled to within 10 μm at the fiber splice. Also, the thickness of the glass layer between the sapphire and single-mode fiber endfaces was controlled to within about 20 μm. Since the softening point of the alumino-silicate glass is 908°C, the silica-to-sapphire fiber splice may be operated only at temperatures up to approximately 800°C. The free endface of the sapphire fiber was polished to be perpendicular to the axis of the fiber.

This intrinsic interferometric fiber sensor was demonstrated for high temperature measurement. To obtain a high temperature up to 1500°C, an oxy-propane torch was used to directly heat the sapphire fiber. About 5 mm of the sapphire fiber was heated, and a conventional high temperature thermocouple (type B) was located very close to the heated sapphire fiber region to monitor the temperature simultaneously. The experimental results are presented in Figure 36.

3.3 Principle of Operation

The sapphire fiber used in the experiment had a large diameter (much larger than the diameter of the single mode fiber core), with no cladding. Due to this, the propagation constant of the fundamental mode in the sapphire fiber should be very close to the France
propagation constant in bulk sapphire. Thus, the sensor interference fringe output can be approximately written as

\[ \phi = 2nL \]  

(1)

where \( n \) is the refractive index of sapphire, and \( L \) is the length of the sapphire fiber. Then we obtain an expression for the phase difference responsible for the interference fringe variations of the sensor output as

\[ \Delta\phi = \frac{4\pi}{\lambda_0} [n \Delta L + L \Delta n], \]  

(2)

where \( \lambda_0 \) is the source wavelength in vacuum.

Figure 3 shows the oscilloscope trace of a typical interference fringe output of the sensor. The fringe contrast was found to be 0.18. This IFPI sensor was also demonstrated for the measurement of high temperature using a 1 cm\(^3\) chamber constructed of mullite ceramic walls heated by a oxy-propane gas torch. About 5 mm of the sapphire fiber was placed in this chamber, and a high temperature thermocouple (type B) was located close to the sensor to monitor the temperature simultaneously.

The number of fringes of the output intensity of the sensor, \( N \), is given by

\[ N = \frac{2\alpha \Delta T L}{\lambda}, \]  

(3)

where \( \alpha \) is the coefficient of thermal expansion (CTE) of the sapphire fiber, \( \Delta T \) is the change in temperature of the sapphire fiber due to heating, \( L \) is the length of the sapphire fiber and \( \lambda \) is the wavelength of the input light source. In this case, \( L \) is 31 mm, the CTE is \( 5.3 \times 10^{-5}/^\circ\text{C} \), \( \lambda \) is 1300 nm, and therefore, \( N = 0.2528 \times \Delta T \).
The minimum detectable temperature change, i.e., the sensitivity of the sensor (expressed in rad/°C) can be determined by,

$$\frac{\Delta T_{\text{min}}}{\text{SNR}}$$

(4)

$\Delta T_{\text{min}}$ can be found using Equation (3).

The temperature co-efficient of the sensor output is the slope of the curve that defines the relationship between the temperature and the number of fringes (like Fig.4). It can be calculated as,

$$C = \frac{\Delta \phi}{L \Delta T}$$

(5)

where, $\Delta \phi = N \times 2\pi$. It is expressed in rad/mm.°C.

The resolution of the sensor can be calculated from the temperature co-efficient, thus obtained, as well as the signal-to-noise ratio (SNR).

The SNR (in dB) is given by,

$$\text{SNR} = 20\log_{10} \frac{\Delta \phi}{\Delta \beta}$$

(6)

where $\Delta \beta$ is given by,

$$\Delta \beta = \frac{v_{\text{noise}}}{v_{\text{signal}}}$$

(7)
3.4 Results and Discussion

The experimental results are presented in Figure 36. A linear output of phase change as a function of temperature was obtained for the measurement range of $310^\circ C - 975^\circ C$. The temperature coefficient of the sensor output was measured to be $6.66 \text{ rad.} \cdot ^\circ C^{-1} \text{ mm}^{-1}$.

The attenuation of the sapphire fiber used, was measured to be $1.77 \text{ dB/cm}$ at $1300 \text{ nm}$. The fiber surface roughness, caused by the EFG fabrication process, should be responsible for this large attenuation.

As Fig.36 illustrates, a linear output of phase change as a function of temperature was obtained for the measurement range of $256^\circ C - 1510^\circ C$. The noise level in the sensor output was measured to be $23 \text{ mV}$ and the amplitude of the sensor output fringe was $72 \text{ mV}$. Based on the sensor temperature coefficient of $6.66 \text{ rad.} / ^\circ C$, as shown in Figure 5, at quadrature point operation the resolution of the sensor was determined to be $\approx 0.1 ^\circ C$ for signal to noise ratio of $3 \text{ dB}$. It is believed that the noise in the sensor output was mainly caused by random laser phase changes, induced by the backward reflection. This phase noise may be reduced by inserting an optical isolator. Due to the optimal fabrication of the sensor, an undistorted interference fringe output of the sensor was observed without any coatings at the free endface of the sapphire fiber. The fringe contrast was measured to be 0.41. Since no film was deposited onto the free endface of the sapphire fiber, the problem of the oxidation of the film at high temperature has been solved and the sensor may thus be capable of being operated at temperatures up to $1800-1900^\circ C$.
In summary, recent advances in the development of a practical sapphire fiber-based interferometric sensor have been presented. A 31 mm length of bare structural grade sapphire fiber was used as a Fabry-Perot cavity and spliced to a silica single mode fiber. The splice was achieved by making use of the 'modified binder' technique with alumino-silicate glass deposited on the sapphire fiber end, and a cleaved silica single mode fiber then spliced to the glass-covered end of the sapphire fiber. The dependence of the fringe contrast of the sensor output on the lateral offset and the longitudinal gap separation at the silica to sapphire fiber splice has also been determined. Fringe contrast was measured to be 0.41. A temperature resolution of 0.1°C has been obtained over a measurement range of 256°C - 1510°C in the laboratory.
Figure 32. Sapphire fiber intrinsic Fabry-Perot cavity

Sapphire IFPI sensor
Figure 33. (a) Schematic of sensor set-up for lateral and longitudinal offset measurements. (b) Method for measuring the dependence of lateral offset on the fringe contrast. (c) Method for measuring the dependence of longitudinal offset on the fringe contrast.

Sapphire IFPI sensor
Figure 34. Normalized fringe contrast as a function of the lateral offset

Figure 35. Normalized fringe contrast as a function of longitudinal offset

Sapphire IFPI sensor
Figure 36. Fringe number vs temperature curve

Sapphire IFPI sensor
4.0 SAPIRE FIBER-BASED EXTRINSIC FABRY-PEROT
INTERFEROMETRIC (EFPI) SENSOR

4.1 Introduction

Fiber optic Extrinsic Fabry-Perot Interferometric (EFPI) sensors have been the focus of
intense research during the last ten years. A number of sensor configurations, highly
sensitive to temperature, mechanical vibration, acoustic waves, and magnetic fields have
been reported in the literature [11]. Kersey et al. were the among the first to demonstrate
such a sensor where the FP cavity was created by the use of air-glass interfaces at the fiber
ends as the reflectors [19]. Murphy et al. demonstrated a quadrature phase-shifted sensor
for the detection of the amplitude and the relative polarity of dynamically varying strain [12].

In most of these sensor configurations, the maximum temperature of application is limited
by the temperature at which the silica fiber begins to soften (∼ 1000 °C). This prohibits the
use of these sensors in harsh environments involving high temperatures and large working
stresses. For reasons discussed in chapter 1, sapphire fiber is an ideal candidate for use in
these harsh environments. Chapter 3 details the principle and fabrication of a sapphire
fiber-based Intrinsıc Fabry-Perot sensor configuration. One advantage of this
configuration is the ease of fabrication which essentially involves splicing a small length of
sapphire fiber to a singlemode silica fiber. The disadvantage is that the gage length of the
sensor is defined by the length of the sapphire fiber, which has a finite minimum value to
protect the splice region from the high temperatures at the tip of the sapphire fiber. This
length can be detrimental to accurate measurements for high-temperature strain sensing.
applications. Moreover, the use of the sapphire fiber as a Fabry-Perot cavity results in the performance of the sensor being dependent on the material properties of the sapphire fiber. This, in turn, results in inaccurate measurements as well as renders the performance analysis of the sensor very complex. This is aggravated if the sensed parameter, like temperature or pressure, is not uniformly distributed along the length of the sensor.

As an alternative, an Extrinsic Fabry-Perot Interferometric (EFPI) sensor configuration was demonstrated to sense strain at high temperatures. The advantage of the EFPI sensor is that the gage length is defined by the air-gap between the two sapphire fibers. Thus, the performance of the sensor can be optimised by choosing a suitable distance for the initial air-gap separation and the sensor characteristics are not as closely dependent on the material properties of the sapphire fiber as the IFPI sensor. The disadvantage is the relative difficulty in fabricating this sensor as well as dealing with an unwanted reflection at the sapphire/silica splice region.

4.2 Experiment

The schematic of the sapphire fiber interferometer is shown in Figure 37. A single mode fiber ($\lambda_0 = 1300$ nm), used as the input/output fiber is spliced to a small length of sapphire fiber which acts as a reference fiber. The reflector is obtained by using a polished sapphire fiber of approximately the same diameter as the former to form an air gap that acts as a low-finesse FP cavity. The far end of the ‘reflector’ sapphire fiber is shattered so the reflections from the far end do not add to the detector noise. The Fresnel reflection from the glass/air interface at the front of the air gap (reference reflection) and the reflection from Sapphire EFPI sensor
the air/glass interface at the far end of the air gap (sensing reflection) interfere in the input/output fiber. Although multiple reflections occur within the air gap, the effect of reflections subsequent to the ones mentioned above can be shown to be negligible. The two fibers are allowed to move in the sapphire tube and changes in the air gap length cause changes in the phase difference between the reference reflection and the sensing reflection. This changes the intensity of the light monitored at the output arm of a fused biconical tapered coupler.

The splicing of the multimode sapphire fiber to the input/output singlemode silica fiber is done by using the ‘modified’ binder approach, as described in chapter 2. The lateral and longitudinal offset between the singlemode silica and sapphire fiber is optimised to obtain maximum coupling between the two fibers. A laser beam launched into the single mode silica fiber propagates to the sapphire fiber. Due to the difference in the refractive index between the silica (1.48) and sapphire fibers (1.73), a fraction (about 0.4%) of the incident laser power is reflected at the silica/sapphire interface. The light transmitted into the sapphire fiber excites low order propagation modes as the numerical aperture of the single mode fiber is very small. The propagating light in the sapphire fiber is partially reflected at the first sapphire/air interface (reference interface). Without reflection coatings, this endface provides about 7% reflectance caused by Fresnel reflection and 93% transmittance. At the second air/sapphire interface (target interface), a fraction of the transmitted light (∼7%) is reflected. At the reference interference, a fraction of this reflected light is coupled back into the reference sapphire fiber. The interference of the two reflections - from the reference and target interfaces, respectively - gives rise to the interference fringe output. Since the optical phase of the light traveling in this air-gap Fabry-Perot cavity is highly

Sapphire EFPI sensor
sensitive to temperature and longitudinal strain, this sensor is useful for the measurement of these parameters.

The experimental set-up is illustrated in Fig.39. A laser beam from a 1300nm pig-tailed semiconductor laser injects optical power into one leg of a 2x2 fused biconical optical fiber coupler. Approximately, 50% of the this power is divided into each of the output legs of the coupler. The leg which is not attached to the single mode fiber is placed in index-matching gel, to prevent any noisy back-reflections. The other leg is attached to a single mode silica fiber with a dimension of 9/125 μm and a numerical aperture of 0.13, using a GTE splice tube.

A 25 mm length of uncoated sapphire fiber with a diameter of 190 μm was spliced to this single mode silica fiber using the ‘modified’ ALG binder splice technique. The sapphire fiber, which has a melting point of 2053° C, was fabricated by the Optical Materials Research Group at MIT, Cambridge, MA. The light traveling back from the air-gap cavity is divided by the biconical coupler and a part of this output is detected by a photodetector and the related electronics transform the detected output into an interference fringe pattern on the oscilloscope. The problem of laser drift, caused by the back reflections from the light of the other leg of the coupler was avoided by optically isolating the laser. The disadvantage of this configuration is the alignment problem between the reference and reflector sapphire fiber to enable the maximum coupling between the reference and the sensing reflections.

To achieve the maximum coupling between the two reflections, it is imperative that the Sapphire EFPI sensor
single mode fiber be positioned as close to the center of the sapphire fiber endface as possible. Besides, it is also imperative that the endface of both the sapphire fibers must be polished as perpendicular to the c-axis of the fibers, as possible. The thickness of the glass layer between the reference sapphire and single-mode fiber endfaces was controlled to within 5 μm. This is very important to achieve because of the large disparity of the numerical aperture of the silica (0.13) and the sapphire fiber (~0.7). Since the softening point of the alumino-silicate glass is 908°C, the silica-to-sapphire fiber splice may be operated only at temperatures up to approximately 800°C.

This extrinsic interferometric fiber sensor was demonstrated for high temperature measurement. The reference, spliced to a silica fiber, and the target sapphire fibers were first enclosed in a sapphire tube with an inner diameter of ~250μm and were subsequently aligned to optimise the coupling between the two fibers. The alignment was performed by mounting the target sapphire fiber on a five-axis positioner (X-Y-Z and two angles) and aligning the fiber with respect to a firmly fixed reference sapphire fiber to obtain the maximum fringe contrast in the output fringe pattern. The purpose of using the sapphire tube enclosure is three-pronged. First, restrict the movement of the sapphire fibers within the tube, thus preventing gross misalignment. Second, protect the exposed polished endfaces of the sapphire fibers from dirt and other environmental degradations. Third, prevent uneven heat flows during the heating and cooling of the air-gap, which is the case with an unenclosed air-gap. It should be noted that the sapphire fibers were not attached to the tube and the fibers were free to move in the tube. The enclosed sapphire fibers were then carefully placed on a titanium plate (3”x 0.5”x 3.2mm; 99.7% pure). Both the fibers were then attached to the plate using a ceramic adhesive (Cotronics Corp.- 906 magnesium

Sapphire EFPI sensor  

-Page70-
Care was taken to position the contact points as close to the sapphire tube as possible so as to minimise the gage length, which is defined by the distance between the two contact points.

After curing the adhesive for the appropriate period, the titanium plate, along with the sensor, was clamped between two heavy metal blocks. Thin ceramic (carbon-carbon) plates were introduced between the titanium plate and the metal clamps, on either side of the plate, to thermally isolate the titanium plate from the blocks. So, the metal clamp blocks did not act as a heat sink for the plate, enabling high plate temperatures to be achieved. The metal clamp blocks were firmly fixed to a vibration-free table. To obtain a high temperature up to 1300° C, an oxy-propane torch was used to directly heat the titanium plate. A conventional high temperature thermocouple (type K) was located in close contact with the titanium plate to monitor the temperature simultaneously. By knowing the initial and final temperatures of the titanium plate, the change in the air-gap separation is calculated, using the co-efficient of thermal expansion of the titanium plate. To achieve controllable strain on the sensor, the titanium plate was bent using a metal block which was mounted on a X-Y micropositioner. Thus, by noting the initial and final reading of the micrometer, the distance through which the tip of the titanium plate was deflected is known, which in turn was used to calculate change in the gap-separation of the sensor, due to the strain on the plate. The top-view schematic of the whole set-up is illustrated in Figure 38. The experimental results are presented in Figure 41.
4.3 Principle of Operation

The theoretical analysis of the performance of this sensor can be attempted on the lines of a conventional singlemode silica fiber sensor. This is because, although the sapphire fiber is a multimode waveguide, the small numerical aperture (NA) of the single mode silica fiber allows only the lower order modes in the sapphire fiber to be excited. Besides, it can be shown that only the fundamental mode contributes appreciably to the interference ring pattern at the output.

The electric field at any point outside the reference sapphire fiber is given by the Kirchhoff diffraction formula [20]

$$E(P) = \frac{1}{4\pi} \int \int \left\{ E_{01} \frac{\partial}{\partial z} \left( \frac{\exp(jkS)}{S} \right) - \frac{\exp(jkS)}{S} \frac{\partial E_{01}}{\partial z} \right\} ds$$  \hspace{1cm} (8)

where $k$ is the free space propagation constant, given by $k = 2\pi/\lambda$, and the integral is evaluated over the core region of the fiber endface. The factor $S$ is the distance between a point $Q$ at the reference fiber endface and the point $P$ in the air-gap. Since most of the propagating light field is strongly restricted within the fiber core, taking the integral over the core region will yield a good approximation. Because only the electric field distribution within the core region of the reference/sensing fiber is of interest and the air-gap separation is restricted to a few tens of microns, the endface of the target fiber can be approximated by an infinite reflector. The electric field contributed by the sensing fiber at the reference fiber endface can be given by

$$E_t = C(P) + j \ D(P)$$  \hspace{1cm} (9)

Sapphire EFPI sensor
where \( C(P) \) and \( D(P) \) are two expressions obtained simultaneously solving the Kirchhoff diffraction equation and the equation for the scalar field of the fundamental mode, \( LP_{01} \) in the core region of the reference sapphire fiber. The amplitude of the electric field re-entering the reference fiber due to the reflection at the sapphire/air interface of that fiber is given by

\[
E_i = C_1 \psi_{01} \exp(-j\omega t) \quad (10)
\]

for the case where \( z = 0 \) at the reference fiber endface. \( C_1 \) is given by \( (n_2 - n_1)/(n_2 + n_1) \), where \( n_1 \) and \( n_2 \) are the refractive indices of the core and the cladding, respectively, of the sapphire fiber. Hence, the total output optical power returning through the reference fiber is given by

\[
P_{\text{total}} = \int \int |E_t + E_i|^2 ds \quad (11)
\]

The integral is taken over the endface surface of the core region of the reference sapphire fiber.

The number of fringes of the output intensity of the sensor, \( N \), is given by

\[
N = \frac{2(\alpha_{\text{cti}} - \alpha_{\text{sap}})\Delta T L}{\lambda} \quad (12)
\]

where \( \alpha_{\text{cti}} \) and \( \alpha_{\text{sap}} \) are the coefficients of thermal expansion (CTE) of the titanium plate and the sapphire fiber, respectively. \( \Delta T \) is the change in temperature of the titanium plate due to heating, \( L \) is the gage length and is given by the distance between the two contact points. \( \lambda \) is the wavelength of the input light source. In this case, \( L \) is 13 mm, the CTE of Sapphire EFPI sensor
titanium is $8.6 \times 10^{-6} \degree C$ while that of the sapphire fiber is $5.3 \times 10^{-6}$. $\lambda$ is 1300 nm, and therefore, $N = 0.062 \times \Delta T$.

The signal to noise ratio (SNR) of the sensor and the minimum detectable temperature change, i.e., the sensitivity of the sensor (expressed in rad/\degree C) can be determined by, equation (6) and (4). The temperature co-efficient of the sensor output is the slope of the curve that defines the relationship between the temperature and the number of fringes. It can be calculated as by equation (5).

![Diagram of bending strain in a clamped plate](image)

**Figure 40.** Schematic of bending strain in a clamped plate

The measurement of strain in the titanium plate with the EFPI sensor can be explained with reference to Fig.40. The free (unclamped) length of the plate is denoted by $L$ and the deflection, due to the force $P$ is given by 'dY'. 'z' is the distance of the surface of the plate, which is in contact with the force $P$, and the neutral axis of the plate. $X_1$ and $X_2$ are the two contact points between the sensor and the plate and $(X_1 - X_2)$ is the sensor gage length.

**Sapphire EFPI sensor**
The incremental increase in the length of the plate, along the x-axis, is given by

$$\delta x = \int_0^L \epsilon_x \, dx$$  \hspace{1cm} \text{(13)}$$

where $\epsilon_x$ is the strain function of the plate, at a point on the plate and is given by,

$$\epsilon_x = \frac{P(L-X)}{EI} \frac{z}{z}$$  \hspace{1cm} \text{(14)}$$

where $X$ is the distance of that point from the clamped end, $E$ is the Young’s modulus of the plate and $I$ is moment of inertia.

Substituting Eqn. (13) in (14), we have,

$$\delta x = \int_0^L \frac{P(L-X)z}{EI} \, dx$$  \hspace{1cm} \text{(15)}$$

But, we know that,

$$\delta EI = \frac{PL^3}{3\delta Y}$$  \hspace{1cm} \text{(16)}$$

Substituting eqn. (16) in (15), we have

$$\delta x = \frac{3\delta Yz}{L^3} \int_0^L \frac{(L-X)dx}{x}$$  \hspace{1cm} \text{(17)}$$

Integrating Eqn.(17), within the limits of $X_1$ and $X_2$, gives the incremental change in the air-gap separation of the sensor. Thus, the change in length is given by

$$\delta x = \frac{3\delta Yz}{L^3} \left( LX - \frac{X_2^2 X}{2} \right)$$  \hspace{1cm} \text{(18)}$$

In the present case, $X_1$ is 0 mm and $X_2$ is 13.04 mm. The free length of the titanium plate was measured to be 95.45 mm. ‘z’ was measured to be 1.5875 mm. Given these values, the incremental change in length can be calculated for different values of deflection. The **Sapphire EFPI sensor**
averaged strain in the homogenous plate can be calculated as,

$$\varepsilon = \frac{\delta x}{\text{gage length}}$$  \hspace{1cm} (19)

The strain as sensed by the optical sensor can be obtained by the equation

$$\varepsilon_{\text{optic}} = \frac{n \lambda}{2 \text{ (gage length)}}$$  \hspace{1cm} (20)

The strains calculated using eqn. (19) and (20) for varying amounts of bending deflections are plotted as illustrated in Fig.41.

4.4 Results and Discussion

Figure 41 illustrates the strain produced in plate as a function of amount in deflection at the free end of the plate. A comparision between the two curves for the values of strain as measured by the EFPI sensor and theoritical calculations, shows that they are consistent with each other at lower values of strain but the margin of error between the two curves increases with the deflection or the force applied to bend the plate. This could be due to an error in the determination of the gage length of the sensor as well as 'z', the distance of the sensor from the neutral axis of the titanium plate. Figure 42 illustrates a typical fringe pattern obtained by straining the sensor. From this plot, the Signal-to-Noise Ratio of the EFPI sensor is calculated by using eqn. (6) to be 66.84 dB

The sensor was heated from room temperature to 650°C and the fringe number was counted when the plate was cooled from 650°C to 150°C. The fringe number was counted to be 30. This agrees very well with the theoritical value calculated for the given range of Sapphire EFPI sensor

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temperature change given by equation (12). This gives us a value of 32 for the fringe number. Higher temperatures could not be obtained in the vicinity of the sensor because the plate thickness (3.175mm) prevented from efficient heat transfer from the bottom surface (which was directly heated with the torch) to the top surface (where the sensor was attached). Besides, the large volume of the plate enabled the plate itself to act as a heat sink. Another problem was the mismatch of the coefficients of thermal expansions (CTEs) of the titanium plate and the ceramic adhesive. This resulted in the debonding of the adhesive after a few thermal cycles.

The single-mode semiconductor laser diode that was used for this particular experiment, had a very narrow spectral bandwidth thereby increasing the coherent length of the source by a large amount. This, in turn, resulted in unwanted interference between the light reflected at the silica/sapphire interface in the splice region, and the light reflected at the sapphire/air interface of the reference sapphire fiber ($R_1$ and $R_2$, as illustrated in figure 43). This is because the length of the reference sapphire fiber was less than the coherent length of the laser diode.

![Figure 43. Schematic illustrating the unwanted interference](image)

**Sapphire EFPI sensor**
\[ \Delta l = c \Delta t \approx \frac{c}{\Delta v} \ll \frac{\lambda_0^2}{\Delta \lambda_0} \]

So, the coherent length of the source is inversely proportional to the spectral bandwidth of the source. Thus, the output fringe pattern was a superposition of the fringe pattern of the intrinsic effect and those of the extrinsic effect. This meant that obtaining an accurate measure of the number of fringes was very difficult. To study the effect a little further, a multimode laser diode, with a finite spectral bandwidth (8nm) was used. This relatively broad bandwidth would result in a decrease in the coherent length of the source and if the length of the reference fiber is suitably chosen to be more than this length, the unwanted interference, as discussed above, can be avoided. This was experimentally verified as illustrated in Fig.44-46.

Fig.44 shows the relatively broad spectrum of the laser diode. Fig.45 shows the output fringe pattern of an EFPI sensor fabricated by using a 40mm long sapphire fiber as the reference fiber. These fringes were obtained by heating only the reference fiber and not the air-gap, thus, making the sensor to function in the intrinsic mode. As can be seen, the fringe contrast of the fringes is negligible and would definitely not affect the performance of the sensor. On the other hand, Figure 46 illustrates the fringe output pattern obtained by moving the target (sensing) sapphire fiber away from the reference fiber. The fringe contrast is much higher than that of figure 44. This demonstrates the fact that by decreasing the coherent length of the source (by using a broadband source), the unwanted interference between the reflection of the silica/sapphire and sapphire/air interfaces can reduced to a point where it would not adversely affect the performance of the sensor and

Sapphire EFPI sensor
fringes from just the extrinsic effect can be obtained. Figure 47 illustrates the dependence of the fringe contrast of the EFPI sensor on the gap separation between the reference and the target fiber. The fringe contrast increases as the gap is increased from its initial position (which is very close to zero). After the optimum gap separation is exceeded, the fringe contrast begins to drop and becomes negligible after ~ 100 μm. Thus, by making sure that the initial gap separation is close to the optimum value, the performance of sensor can be optimised to obtain the maximum fringe contrast, which improves the accuracy of the sensor.

In summary, a sapphire fiber-based extrinsic Fabry-Perot interferometric sensor has been developed and tested. A 25 mm length of bare structural grade sapphire fiber was spliced to a silica single mode fiber. A polished sapphire fiber (without any coatings on the endface) was aligned with the reference fiber and both the fibers were enclosed in a sapphire tube, to improve the performance of the sensor. The air-gap between the target and the reference fibers was used as Fabry-Perot cavity. The splice was achieved by making use of the ‘modified binder’ technique with alumino-silicate glass deposited on the sapphire fiber end, and a cleaved silica single mode fiber then spliced to the glass-covered end of the sapphire fiber. The dependence of the fringe contrast of the sensor output on the longitudinal gap separation between the reference and target fibers has also been determined. The sensor was demonstrated to measure strain with considerable accuracy and resolution. The sensor was also demonstrated to measure temperature (from room temperature to 650°C) and the results were found to agree closely with the theoretical values. Thus, sensor is effective for use in high temperatures to measure strain as well as temperature. To optimise the performance of the sensor, a broadband source is required.

Sapphire EFPI sensor
Figure 37. Sapphire EFPI sensor
Figure 38. Sensor arrangement for strain and temperature measurement (top view)
Figure 39. EFPI sensor set-up

Figure 41. Measured strain as a function of the deflection of the plate

Sapphire EFPI sensor
Figure 42. Typical fringe pattern obtained by straining the sensor

Figure 44. Spectral bandwidth of the laser diode

Sapphire EFPI sensor
Figure 45. Output pattern of the sensor when operated as an intrinsic sensor

Figure 46. Output pattern of the sensor when operated as an extrinsic sensor

Sapphire EFPI sensor
Figure 47. Fringe contrast of the EFPI sensor as a function of the gap separation
5.0 CONCLUSIONS

The preceding three chapters discussed, in detail, the various techniques of splicing sapphire fibers to silica fibers and their subsequent use to develop interferometric sensors to sense strain and temperature. Extrinsic and intrinsic Fabry-Perot interferometric sensors have been successfully demonstrated for use in harsh environments.

Chapter 2 gave a detailed description of all the techniques of splicing silica fibers to sapphire fibers, that have been attempted during the course of this research effort. Splicing involved uncoated, silica-coated and aluminosilicate glass-coated sapphire fibers. Direct arc-fusion splicing using the fusion splicer can be used to splice the fibers for use in environments without large working stresses. Using silica as a binding agent to enhance the splice strength has the disadvantage of being more time-consuming and the length of the sapphire is limited by the size of the substrate holder in the CVD chamber. Splices involving the use of aluminosilicate glass as a binding agent, showed great improvement in the quality of the splice. Splices with relatively low optical losses and high mechanical strength were fabricated. This technique essentially consisted of melting the ALG onto both the silica and sapphire fibers and holding them together, on cooling of the ALG. Adequate control over the alignment of the cores of the silica and sapphire fibers, before and during the splicing, was provided by this technique. This minimised the lateral and longitudinal offsets between the fibers. Besides, the softening point of the aluminosilicate glass (~908°C) is less than the softening point for silica (~1100°C) and the melting point of sapphire (2054°C). This meant that the heating of the ALG to melt, did not adversely effect the physical geometry of either the silica or sapphire fibers. This was not the case in the Conclusions

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arc-fusion splicing technique and the splicing of a silica-coated sapphire to silica fiber. The large mismatch of the CTEs of the ALG and silica is minimised by heating and cooling the splice region at a gradual and steady rate. Due to intermediate compound formation, ALG bonds very well with both silica and sapphire. This, in turn, means the splice obtained by this technique is very strong. Enclosing the splice in a sapphire tube enhances the strength even more and the splice is conducive to rugged use.

To summarize, the splice loss and the breaking stress (if available) for the best splice obtained by each of the technique is tabulated.

<table>
<thead>
<tr>
<th>SPLICE</th>
<th>SPLICE LOSS</th>
<th>BREAKING STRESS</th>
</tr>
</thead>
<tbody>
<tr>
<td>Diffusion splice</td>
<td>Not available</td>
<td>Not available</td>
</tr>
<tr>
<td>Arc-fusion splice</td>
<td>9.5 - 5.5 dB</td>
<td>69.7 kpsi</td>
</tr>
<tr>
<td>Silica-coated splice</td>
<td>10.7 - 8.5 dB</td>
<td>Not available</td>
</tr>
<tr>
<td>ALG Binder splice</td>
<td>1.4 - 0.2 dB</td>
<td>74.5 kpsi</td>
</tr>
<tr>
<td>Modified</td>
<td>2.0 - 1.25 dB</td>
<td>Not available</td>
</tr>
<tr>
<td>ALG tube splice</td>
<td>2.1 - 1.1 dB</td>
<td>Not available</td>
</tr>
</tbody>
</table>

The tube splice approach provides a good possibility for the whole process of splicing to be automated. The polished sapphire fibers and the cleaved silica fiber are firmly fixed on motorised platfroms and inserted into a sapphire tube containing a small length of ALG ‘fiber’. The flame is also mounted on a motorised platform. The fibers are moved into the tube through a pre-determined distance and the flame is introduced to heat the tube. After a fixed time interval, the flame can be withdrawn at a steady and gradual rate. To enhance

Conclusions
the alignment of the sensors, the silica-sapphire fibers can be connected to source-detector pair. Dynamic detection of the power out of the sapphire fiber and the feedback control to the silica fiber results in optimising the alignment between the fibers.

Chapters 3 and 4 describe the theory and development of sapphire fiber sensors. Besides being able to use these sensors in harsh environments, the sapphire fiber-based sensors are also very attractive for embedding in materials like composites. This is not true with the sapphire rod-based sensors that have been developed so far. Besides, the fiber-based sensors offer smaller size and greater flexibility when compared with the rod sensors. Moreover, the problem of the maximum usable temperature of the rod sensor being limited by the maximum temperature that the GRIN lens can withstand, is avoided in the fiber-based sensor as no GRIN lens is used.

The Intrinsic Fabry-Perot interferometric sensor has been demonstrated to measure temperatures exceeding 1000°C, with a resolution of 0.1°C. The fringe contrast was adequate, but can definitely be improved by polishing the endface of the sapphire fiber as close to the perpendicular of the longitudinal axis of the fiber, as possible. Secondly, improving the alignment of the fiber cores at the splice region enhances the coupling of light across the splice region and, thus, improves the fringe contrast. The sensor can be demonstrated to sense temperature higher than 1500°C if proper heating apparatus like a bigger torch and an insulated enclosure are used while heating. A disadvantage with this configuration is that the gage length of the sensor is dictated by the length of the sapphire fiber used. Thus, accurate measurements of strain (by bending) was not possible, as the bending of the fiber resulted in adversely effecting the coupling at the splice.

Conclusions
The extrinsic configuration was demonstrated to measure strain of greater than a 1000 μstrain, with considerable consistency. It was used to measure temperature up to 650°C, with satisfactory results. Again, the maximum temperature was limited by the capability of the torch as well as the heat sinking properties of the titanium plate. With the proper apparatus, the sensor can be tested at higher temperatures. It was conclusively shown that a broadband source would minimise the unwanted interference between the reflections from the silica/sapphire and sapphire/air interfaces. The extrinsic configuration works on the interference between the reflections at the endfaces of the reference and target fiber. This means that it is absolutely imperative that the splice between the silica and the reference sapphire fiber should be perfect with the minimum amount of losses possible. This also means absolute control over the alignment of the fibers must be achieved. The polishing of the endfaces of the sapphire fibers also is very critical to the performance of the sensor. It should be noted that with the availability of sapphire fibers with much lower losses (like the fiber grown at USF), the fringe contrast, and hence, the performance of the extrinsic as well as intrinsic Fabry-Perot sensors can be improved considerably. To summarize, a proof of concept was made to demonstrate the use of sapphire fiber-based sensors to measure parameters like strain, temperature and acoustic waves in harsh environments. They can also function effectively in embedded applications.

Conclusions
References


References


VITA

The author was born on November 8, 1967 in a sleepy little town called Visakhapatnam, located on the eastern sea-board in India. He spent all but two years of his life in this town. He graduated from High School in May 1985. In the fall of 1986, he started professional college as an undergraduate in the College of Engineering, Andhra University, Visakhapatnam. In July 1990, he graduated with a Bachelors Degree in Electrical Engineering. He has been pursuing graduate studies at Virginia Polytechnic Institute and State University since fall 1990, working in the Fiber and Electro-Optics Research Center in the department of Electrical Engineering.