

## ***Chapter 1: Introduction***

It is the goal of this work to develop a simply constructed and implemented, absolute fiber optic displacement measurement sensor capable of operation from ambient temperatures up to 1500 degrees Celsius. While there has been a wealth of development and research in the field of low temperature fiber optic sensors, the area of high temperature fiber optic sensors remains comparatively immature. This thesis both presents details of the sensor design and addresses some important issues, ranging from materials concerns to implementation details, raised during the development process.

Chapter 1 briefly introduces the concepts of optical fiber sensors and presents the factors that limit the use of glass-based optical fiber sensors to environments having temperatures not exceeding 700 degrees Celsius. Sapphire fiber is then presented as an alternative to glass fiber for high-temperature sensing applications, and the sapphire-based sensor research that has been reported in the literature to date is summarized.

### **1.1 The Role of Sapphire Fiber in Fiber Optic Sensors**

The practical applicability of fiber optic sensors was widely recognized over 20 years ago. Since then, the further development and implementation of these sensors has been driven by the versatility and properties inherent to optical fiber, as well as by the maturation of the fiber and device technologies associated with the telecommunications industry.[1] Attractive qualities of optical fiber include a dielectric composition which results in immunity to electromagnetic interference and ground loop networking problems,[2] the ability to support the high bandwidth signals required for multiplexing numerous high-performance sensors, and the possession of a small size and a sturdy

composition which allows them to be integrated directly into materials.[3] The flexibility and sensitivity of sensors fabricated from optical fiber permits the measurement of a variety of phenomena, including temperature, pressure, strain, degree of cure, chemical content, viscosity, acoustic waves, magnetic fields, and degree of rotation.[2, 3]

Fiber optic sensors act as transducers that encode information, which describes a non-optical external perturbation, onto an optical carrier.[4] In intrinsic sensors, such as microbend sensors, the perturbation acts directly on the fiber to modulate the optical carrier. In extrinsic sensors, such as those based on the Fabry-Perot etalon, the perturbation affects the optical carrier external to the optical fibers; the optical fibers act only to convey the optical signal to and from the sensor.[5] The design of the sensor system determines whether the external perturbation modulates the amplitude, phase, differential phase, or spectral distribution, of the optical carrier. Amplitude modulation directly affects the signal intensity, phase modulation is converted to intensity modulation through interferometry, differential phase modulation is converted to intensity through the determination of polarization, and modification of spectral distribution may be determined through spectroscopic analysis.[5]

Fiber optic sensors are commonly constructed from silica-based glass optical fibers.[3] When standard telecommunications fiber is used, sensor systems benefit from low optical signal transmission loss (0.16 decibels/kilometer at 1550 nanometers) of the optical fiber, an optical signal well confined to the waveguide, and the low cost and wide availability of the optical fibers.[6] Optical fibers frequently have a protective coating of acrylate, but they may also be encased in polyimide, metal, carbon, or some other protective jacket. This outer coating preserves the physical strength of the optical fiber by providing protection from the environment. Without the coating, the glass waveguides are exposed to moisture which causes significant weakening of the fiber: water penetrates microcracks on the surface of the fiber, resulting in the propagation of the fissures.[3]

Silica-based optical fiber sensors are generally restricted for use in environments below 700 degrees Celsius, because the integrity of the glass fiber is adversely affected at higher temperatures. Specifically, at 1000 degrees Celsius the migration of the dopants from the fiber core becomes significant.[3, 7] In addition, at temperatures exceeding 900 degrees Celsius the combination of strain and elevated temperatures will also induce creep and plastic deformation in the silica optical fiber.[7] Most silica-based sensor systems are specified for use under 700 degrees Celsius, to provide an adequate margin of safety.[3] The durability of fiber coatings at elevated temperatures must also be considered. Acrylate coatings degrade at temperatures above 150 degrees Celsius, while polyimide coatings deteriorate between 400 and 500 degrees Celsius. Metal coatings are capable of surviving temperatures at which the silica softens.[7]

There is interest in the development of sensors that can operate in high temperature and chemically harsh environments for which most sensors are not suited. Such high-temperature sensors would find use in the control of high-temperature combustion and industrial processes and in the development of advanced high-temperature materials. Platinum gauges are used to perform some measurements, but their applicability is limited.[7] Sensors constructed from sapphire fiber ( $\text{Al}_2\text{O}_3$ ), which has an index of refraction of 1.78 and a melting point of 2050 degrees Celsius are reasonable candidates for an alternate sensing technology in the high-temperature regime.[8] In addition, sapphire is noncorrosive, and it is insoluble in water, organic solvents and acids.[9]

Optical sensors made of sapphire are able to withstand environments where few other sensors can function, but the growth of optical-grade fibers is still in the research phase.[8] Growth methods do not permit sapphire fiber to be drawn as a singlemode waveguide or with a cladding. Cladding on optical fibers serves a variety of purposes: it effectively confines the propagating optical signal, limits attenuation, and discourages cross coupling with adjacent waveguides. It also provides support and strength for the core of the fiber which, for singlemode operation, is usually a few wavelengths, or less than 10 micrometers, in diameter. Without a cladding, the entire sapphire fiber may be

considered to be the core, with the surrounding lower index air the fiber cladding. In this case, the large diameter sapphire core supports the propagation of thousands of different modes. Sapphire fiber also lacks a coating, which acts to protect a fiber and discourages interactions with chemical contaminants.[10]

## **1.2 Sapphire Fiber and Rod Based Optical Sensors**

The ability of sapphire optical fiber to withstand chemically harsh and high-temperature environments, unsuitable for silica fiber, has generated interest in both the research and commercial communities. Sapphire is thermally, chemically, and mechanically stable, and it will potentially find innumerable uses in the development of high-performance structures and materials. The sapphire-based sensors generating the widest interest are those used as temperature sensors and those used for spectroscopic analysis of liquids and gases. Researchers have also constructed interferometric and polarimetric sensors from sapphire fiber, although these areas of investigation are of lesser popularity. The problematic characteristics of sapphire fiber, which limit the uses and performance of sapphire-fiber based sensors, will be addressed in detail in Chapter 2.

The first proposed design for a sapphire-based temperature sensor relies on the principles of blackbody radiation, and it is the basis of both commercial products and the majority of next-generation sapphire-based temperature sensors currently under development.[11] In this first sapphire-based sensor design, shown in Figure 1.2.1, a blackbody cavity is sputtered onto the end of a sapphire rod 0.25 to 1.25 millimeters in diameter and 0.05 to 0.30 meters in length. The non-metalized end of the sapphire fiber is butt-coupled to a standard glass optical fiber, and the output of the glass fiber is collected by a detector. The radiance emitted from the blackbody cavity is used to determine the temperature of the environment of the sensor; as the temperature of the environment increases, the spectrum emitted by the blackbody predictably shifts to shorter wavelengths according to the Planck radiation law. Although the sapphire rod employed in this sensor is flame polished to smooth the surface, the majority of scattering losses arise from the remaining

surface imperfections. At temperatures exceeding 1100 degrees Celsius, scattering, absorption, and reemission at internal defects and surface imperfections are also noted. The author of the paper observes that, because these sources of loss will likely challenge the accuracy of the sensor, only high quality crystals should be used. Commercial temperature sensors based on this design claim a temperature range between 300 and 2000 degrees Celsius, an accuracy of 0.10% at 1000 degrees Celsius, and a resolution of 0.000020 degrees Celsius.[12]

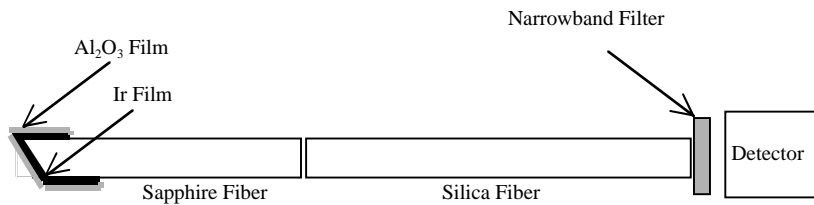


Figure 1.2.1: Schematic of a Blackbody Cavity High Temperature Sensor [11]

In addition to being commercially produced, [12, 13] and finding use in a variety of environments, variations on this temperature sensor, intended to improve the range and accuracy of the original, continue to be reported. The sensor has been used to monitor the temperature of internal combustion engines,[14] of aircraft turbine engines, and in high velocity combustion flows.[13] While platinum and iridium are commonly sputtered on the end of the sapphire fiber sensor to create a blackbody, these films can deteriorate at temperatures exceeding 1600 degrees Celsius. Success in doping the sapphire fiber end with Cr<sub>2</sub>O<sub>3</sub>, as an alternative to coating the end of the fiber in a comparatively fragile metallic film, has been reported.[15] The low-temperature measurement range of this sensor is limited to above 300 degrees Celsius because the low attenuation window of sapphire lies between 0.25 microns and 6 microns,[16] a waveguide transmitting longer wavelength light is needed for lower temperature measurement. Use of a hollow sapphire tube, which has a window of low attenuation between 9.6 and 17.2 microns, has been demonstrated to enable temperature

measurement between 45 and 900 degrees Celsius.[17] Other variations on the design of the sensor aim to improve the accuracy of the measurement: system errors coupled with changing transmission and emission losses can result in inaccurate temperature measurements. One solution is to base the temperature measurement on the ratio of the optical powers detected in two wavelength bands.[18] At low temperatures, the power in the two wavelength bands are nearly equal, and their division produces a number too small to result in accurate measurements. This problem has been addressed by comparing the value of the detected blackbody radiation in one wavelength band to that of a guided reference signal.[19]

Another popular use of sapphire fiber is in fiber optic attenuated total reflectance (FO/ATR) spectroscopy. The analysis of the transmitted infrared spectra of a material can be used to determine its chemical makeup. When a substance absorbs the infrared too strongly to enable this measurement, FO/ATR can be an alternative. In FO/ATR, as illustrated in Figure 1.2.2, a section of unclad and uncoated fiber is submerged in a sample of the material of interest. In order for total internal reflection to confine the source light to the fiber, the fiber must be of a higher index than the sample. When this is the case, an evanescent field extends into the sample and is partially absorbed by it. Spectral analysis of the light exiting the sensor determines the chemical composition of the sample.

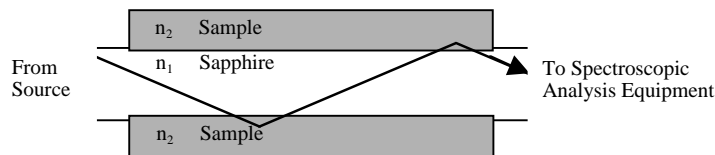


Figure 1.2.2: Illustration of the Principle of a FO/ATR Spectroscopic Sensor [20]

Several types of optical fibers have been employed in FO/ATR spectroscopy, including chalcogenide, silver halide, and heavy metal glass fibers, but only sapphire fiber possesses the combination of mechanical strength, chemical resistance, and high-temperature survivability required for a number of applications.[20] Sapphire-based FO/ATR spectroscopy systems have been used to determine the C<sub>2</sub> content of ethylene/propylene copolymers,[9] to monitor the thermal stability of jet fuel,[21] to monitor the coal liquefaction process,[20] for the on-line analysis of chlorinated hydrocarbons,[16] for the measurement of gaseous hydrocarbons at elevated temperatures,[22] and as a cure state monitor.[23]

Work has also been performed towards developing sapphire-based strain, displacement, acoustic wave detection, and related sensors. The first such reported design describes an extrinsic intensity-based strain sensor, depicted in Figure 1.2.3.[24] The intensity of light captured and guided by the second fiber decreases with the length of the gap; the gap length can be determined by optical power incident on the detector. The resulting curve of detected power versus gap length is highly nonlinear, and is thus not easily interpreted. The merits and problems associated with various sensor configurations is addressed in Chapter 3.

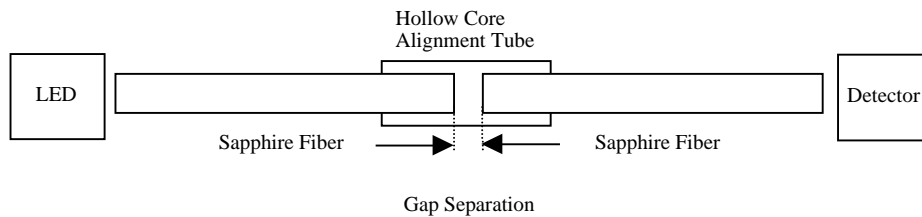


Figure 1.2.3: Intensity-Based Sensor Schematic [24]

While intensity sensors are attractively simple, more complex interferometers permit greater measurement accuracy. Common high-sensitivity sensor configurations include

the Mach-Zehnder interferometer, Michaelson interferometer, Sagnac interfrrometer, Fabry-Perot etalon, fiber grating, dual mode fiber sensor, and polarimetric fiber sensor.[3] The report of the first sapphire-based extrinsic interferometric sensor was made by Murphy *et al.*[7] The sensor, illustrated in Figure 1.2.4, uses a sensing head configured as a low-finesse Fabry-Perot cavity, and it is designed for use in strain and acoustic wave measurements. Radiation is directed into the sensing head and reflects from both the end of the 5 millimeter sapphire rod and the indicated reflector. The graded index (GRIN) lens acts to couple the radiation into and out of the center of the sapphire rod. The technique of injecting the light into the center of the sapphire waveguide greatly reduces the interaction of the beam with the surface of the waveguide, and it obviates the need to flame polish the surface of the rod to reduce surface scattering losses. The two reflected waves are incident on the detector where, because they have traveled different optical path lengths, their phases are shifted relative to one another.

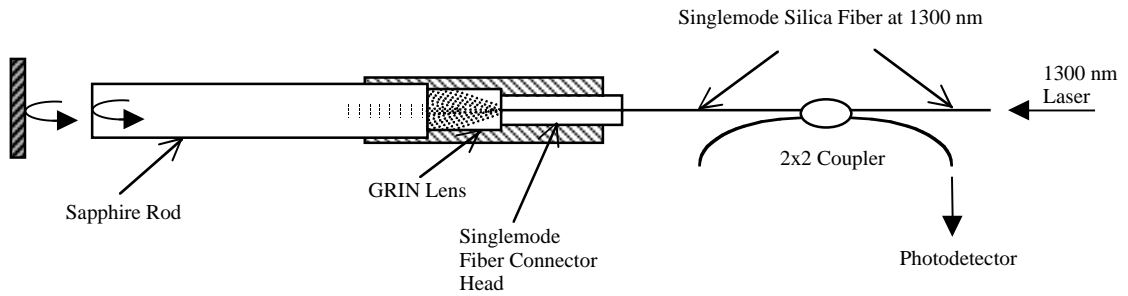


Figure 1.2.4: Sapphire Rod Displacement Sensor [7]

The phase of a wave is dependent on the optical path length it has traversed. The optical path length of a medium is the value obtained through the multiplication of the length and the refractive index of the medium. At the detector, information encoded in the phases of the optical signals are converted to intensity according to the equation

$$I = A^2(1 + m^2) + 2mA^2 \cos(\phi_1 - \phi_2) \quad 1.2.1$$

where  $I$  is the detected intensity,  $A_1 = A$  is the normalized complex intensity of wave one,  $A_2 = mA$  is the normalized complex amplitude of wave two,  $m$  is a scalar multiplier, and  $\phi_1$  and  $\phi_2$  are the phases of waves one and two. As the reflector moves relative to the end of the sapphire rod, the difference in the phases of the two waves changes. This change is manifested by the detected intensity assuming the form of a sinusoid with respect to gap length. One complete cycle is referred to as a fringe .[25] This subject is addressed in greater detail in Chapter 3. A variation of this sensor head configuration,[7] is shown in Figure 1.2.5. Two rods are inserted end-to-end into a

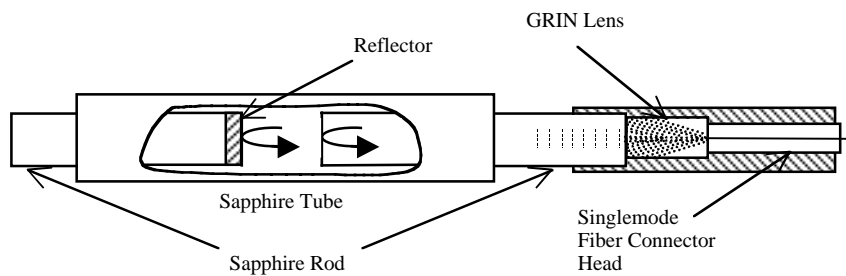


Figure 1.2.5: Sapphire Extrinsic Fabry-Perot Interferometer [7]

sapphire tube, and the second rod, possessing a metalized endface, acts as the external reflector. The tube protects the air gap and endfaces from undesired particulate accumulation.

While the extrinsic fiber optic Fabry-Perot interferometer, as described in the previous paragraph, is more accurate than the similarly constructed intensity based sensor, it is not an absolute sensor. The fringes reveal only relative changes in the gap length, and the sensor cannot distinguish between perturbations that widen and lessen the gap length. If the direction of the perturbation should change at a fringe maxima or minima, the change would be undetectable. One proposed solution to this problem is to use a broadband, instead of a single wavelength, source and to view the sensor output with a spectrum analyzer as shown in Figure 1.2.6.[26] The Fabry-Perot cavity permits the transmission

of only selected wavelengths. For two wavelengths that are  $2\pi$  out of phase,  $\lambda_1$  and  $\lambda_2$ , the gap length,  $d$ , is

$$d = \frac{\lambda_1 \lambda_2}{2(\lambda_2 - \lambda_1)} . \quad (1.2.2)$$

This proposed sensor design was implemented by other researchers, who, due to the degradation of fringe visibility with gap length, were limited to measuring gap lengths of under 30 microns.[27]

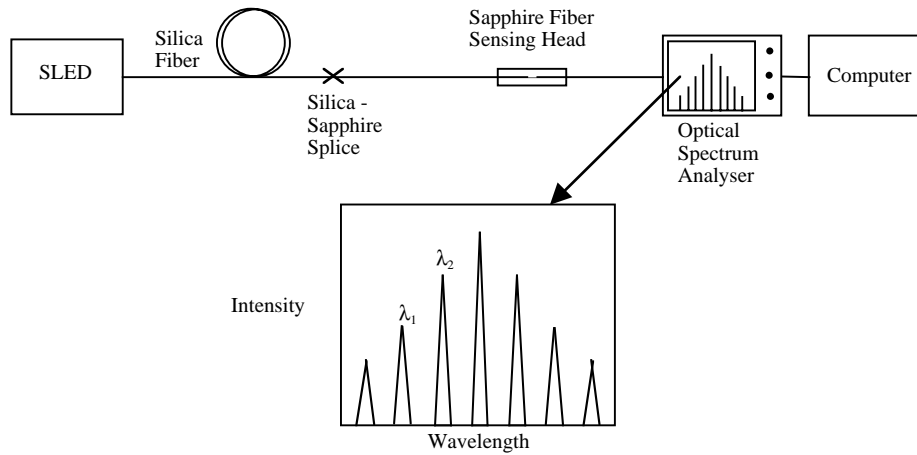


Figure 1.2.6: Absolute Strain Sensor and Illustrative Output [26]

Fringe visibility, also known as fringe contrast, is a measurement used to express the degree of fading experienced by the fringes:

$$V = \frac{I_{\max} - I_{\min}}{I_{\max} + I_{\min}} \quad (1.2.3)$$

Values for  $V$  range from 0 to 1, with 1 indicating the output of a perfect system, illustrated by the solid line in Figure 1.2.7. Fading results when only portions of the two reflected waves are capable of interfering at the detector and producing fringes. The remainder of the light, detrimentally altered by the system and incapable of interfering, contributes a uniform background intensity. As depicted by the dashed line in Figure 1.2.7, these fringes, compared with those of the perfect case, are smaller in amplitude and neither achieve the maximum or the minimum intensity values of the perfect system. This is described in greater detail in Chapter 3.[25]

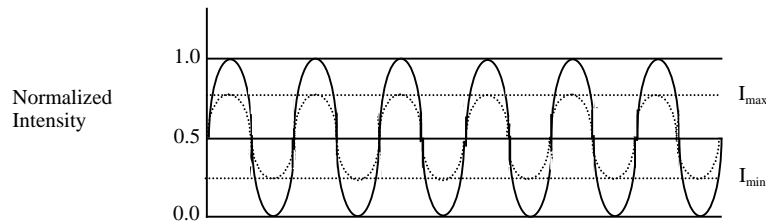


Figure 1.27: Determination of Fringe Visibility [25]

A related sensor, but one configured as an intrinsic Fabry-Perot cavity, utilizes the fact that both the length and the refractive index of the sapphire fiber change with temperature.[28] The operation of the sensor, depicted in Figure 1.2.8, relies on the interference of two reflected waves, the first reflected from the free sapphire fiber end and the second from the silica-sapphire splice, at the detector. As the temperature of the sapphire fiber changes, the optical path length of the sapphire fiber changes, altering the phase of the second reflected wave. The detected intensity changes according to equation 1.2.1, and results in a sinusoidal characteristic when the detected power is plotted against either temperature or against optical path length. It is reported that the quality of the silica-sapphire splice directly influences the fringe contrast achieved by the system.[29]

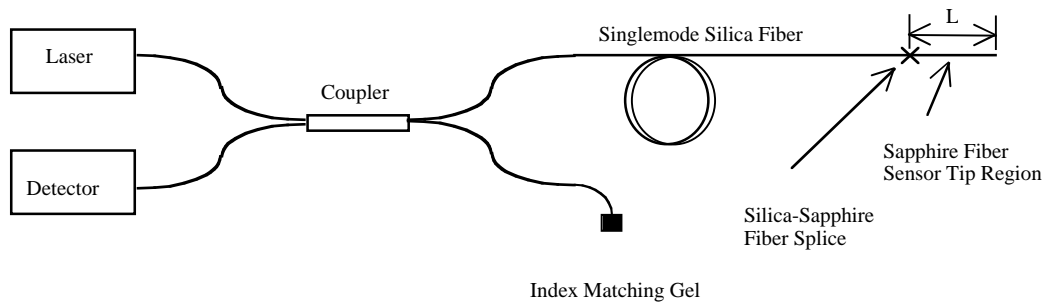


Figure 1.2.8: Sapphire-Based Intrinsic Fabry-Perot Interferometer [28]

Polarimetric sensors are those that utilize data obtained through the exploitation of such phenomena as the Faraday, Pockels, Kerr, and photoelastic effects. The Faraday effect occurs when certain materials act as polarization rotators under the influence of a static magnetic field, the Pockels effect describes the change of the refractive index of a material in proportion to the change of the applied electric field, the Kerr effect details the change of the refractive index of a material with the square of the applied electric field, and the photoelastic effect specifies that the refractive index of a material under strain will change in proportion to the value of the strain.[30] Many of the elements used in traditional polarimetric sensors cannot withstand high temperature environments. Sapphire fiber, whose birefringence and length change with temperature, has been used to realize a high temperature polarimetric temperature sensor.

The design of the sapphire fiber-based polarimetric temperature sensor is highly dependent on the characteristics of both the sapphire fiber and the intensity spectrum of the source.[31] In general the sensor would be configured as shown in Figure 1.2.9 (a) with the sensing head shown in Figure 1.2.9 (b): one polarizer functions as both the input and the output polarizer, and the slow axis of the fiber is rotated 45 degrees with respect to the axis of the polarizers. The light source is a light emitting diode with a Gaussian spectrum and a full width half maximum of 46 nanometers. Mathematical analysis, performed by the authors of the paper, shows that the fringe contrast of such a

combination of sensor and source is an exponentially decreasing function of the birefringence and the fiber length, and that for high temperature measurements the length of the fiber should not exceed 0.125 millimeters. This limitation is circumvented by constructing the sensing head from two sapphire fibers, which are oriented such that the fast axis of one coincides with the slow axis of the other. This is depicted in Figure 1.2.9 (c). By balancing the birefringence in this manner, the length of the sensing head may be made arbitrarily long. As a demonstration, 10 millimeters of the second sapphire fiber was heated from room temperature to 1500 degrees Celsius. A plot of the relative intensity of the output signal with respect to the temperature is nonlinear and shows a sensitivity of 5 degrees Celsius.

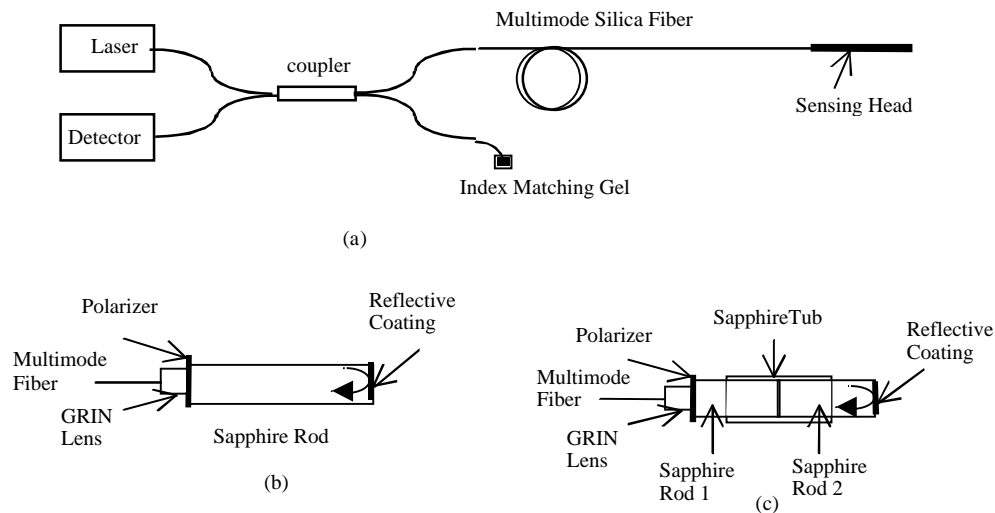


Figure 1.2.9: (a) General Design of a Polarimetric Sensor System, (b) Non-Birefringence Balanced Sensor Head Design, (c) Birefringence Balanced Sensor Head [31]

Work has also been performed to develop a sensor that operates on the principles of spatial modulation.[32] The work has been performed by a research group at Drexel University, which has developed a cladding and an overcoating for sapphire fiber. The authors of the paper fabricated a sapphire fiber cladding of polycrystalline alumina and an overcoating of silicon carbide, and they intend to eventually use the product in a sensor designed to test ceramic composites. In this sensor, shown in Figure 1.2.10, a

light source is coupled to a clad and overcoated sapphire fiber, which is embedded in a ceramic composite. The output of the fiber is directed onto a CCD camera. Because the fiber is heavily multimoded, microbends in the fiber result in the modification of the modal power distribution of the fiber. In preparing to test the sensor, the authors deliberately endeavor to excite only higher order modes, because they are most sensitive to the effects of the external perturbations. As the fiber is subjected to 5-point bending, the modal power is redistributed into the lower order modes of the fiber. The authors of the paper suggest that this behavior may be characterized and used to fabricate a sensitive displacement sensor.

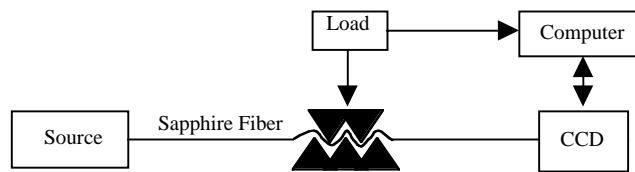


Figure 1.2.10: System Used to Test Viability of a Sensor Based on the Principle of Spatial Modulation [32]

### 1.3 Preview of Thesis

The objective of this work is to design and construct a fiber optic strain sensor to be used in the testing of advanced materials for use at temperatures up to 1500 degrees Celsius, as specified by a contract between the Fiber & Electro-Optics Research Center at the Virginia Polytechnic Institute and State University and the Wright Laboratories. The sensor must be capable of absolutely measuring crack displacements in a test material of up to 6.35 millimeters in width and possess a resolution of less than 10 microns. It is required to be robust enough to survive in a portable form outside of the laboratory. As an additional constraint, the operation of the sensor should be straightforward and easily mastered by people not accustomed to working with fiber optics. While the investigation

of the operation and implementation of the sensors described in the previous section is instructive, none adequately meet the specified requirements.

Chapter 2 presents information about sapphire optical fiber relevant to this work. The different manufacturing techniques and their impact on the optical quality of the sapphire fiber are detailed. The optical qualities of current state-of-the-art fibers are described, including the absorption spectrum, loss mechanisms, and the inherently multimoded behavior. The chapter concludes with a discussion of different attempts to clad and overcoat the sapphire fibers, and the reasons that such a goal is difficult to achieve.

Chapter 3 describes the basic operation of the Michelson interferometer and its adaptation to the sensor design used in this work. The definition and importance of white light interferometry to this sensor design is detailed. The expected output of the resulting sensor is then presented and determined to be capable of meeting specifications. As a confirmation of this optimism, a silica-based model is constructed and tested.

Chapter 4 details the implementation of the sensor design developed in Chapter 3 using sapphire fiber. Techniques used to overcome the problems associated with the highly multimoded and lossy sapphire fiber are described. A discussion of the restrictions placed on the construction of the sensor by polarization mode fading is included. The performance of the sensor is evaluated.

Concluding remarks are made in the final chapter, as well as a summary of significant results. Suggestions for future work are also included.