"MATERIAL PROCESS MONITORING WITH OPTICAL FIBER SENSORS"

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ABSTRACT

Our motivation for this work is based on the need to monitor the cure and in-service health of composite materials. We describe the continuation of an effort to design a multi-functional fiber optic sensor which can be embedded in polymeric composite laminates for monitoring the degree of cure during its fabrication, as well as internal composite strains occurring post-cure. In short, this dual-purpose sensor combines the characteristics of a Fresnel reflectometer with those of the extrinsic Fabry-Perot interferometer. For monitoring cure, a broadband source is used so the output intensity of the sensor is amplitude-modulated as the refractive index of the composite is increased during the polymerization process. Post-cure, a coherent light source is implemented so a sinusoidal variation of the output signal occurs when strains within the composite cause the sensor output to be phase-modulated. We demonstrate the measurement of refractive index with the Fresnel reflectometer/EFPI, and test it as an embedded refractive index monitor. Our experimental results demonstrate that the refractive index of 5-minute epoxy increases by approximately 2% during the cure process. In addition, the sensor can be used as an interferometer to measure internal composite strains, where the phase difference between consecutive fringe peaks is one-half the wavelength of the source.
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To my father, Ray Burford
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1 INTRODUCTION

1.1 MOTIVATION

The motivation for this work is based on the need to monitor the cure, as well as the in-service health, of composite material, which is defined as "a combination of two or more materials (reinforcing elements, fillers, and composite matrix binder), differing in form or composition on a macroscale."\(^1\) Even though the various constituents of a composite structure react together as one part, they retain their individual identities, and usually a physical interface exists between them.\(^1\) To cure a composite laminate is "to irreversibly change the properties of a thermosetting resin by chemical reaction, that is condensation, ring closure, or addition. Cure may be accomplished by addition of curing (cross-linking) agents, with or without heat and pressure."\(^1\)

The use of composite materials as primary structural members in critical applications such as aircraft and spacecraft has been proposed since they provide efficient structural integrity at lower weights than metallic structures.\(^1\) The rapid growth of these advanced materials is due to the substantial improvements that they offer in performance over conventional materials such as high strength and stiffness-to-weight ratios, good fatigue resistance, low radar cross-section, good corrosion resistance, and the flexibility to tailor the mechanical properties and shape.\(^3\) Such advanced composites are quickly becoming the primary material for use in aircraft structures, and are expected to completely reform the airframe industry in the next 5 to 10 years.

However, the deployment of composite structural members has lagged due to
high manufacturing costs. For example, it has been reported that about 80% of the total cost of advanced composite parts is due to the complexity of the fabrication process, which has been clearly targeted for cost reduction.\textsuperscript{2} Also, the performance properties of composites are strongly dependent upon the internal events, chemical and rheological, that occur during the cure process.\textsuperscript{2} In fact, partially cured composites will likely fail prematurely, and composites cured at excessive temperatures typically become brittle and weak.

Variations in cure are triggered for several reasons. First, the chemical reaction during cure is very dependent on the thermal properties existing within and around the laminate. Such properties are changed whenever the size and shape of the part design are altered, and can even be dependent on the thermal properties of the specific autoclaves and pressing tools used. In addition, the exothermic nature of the cure reaction leads to nonuniform heating within the composite which can result in residual stress build-up and uneven cure. Variations in chemistry between batches and changes within batches during storage can also alter cure time requirements.\textsuperscript{2}

Traditional cure cycles are inflexible and waste time and heat. Sensors are therefore needed to develop intelligent composite processing controllers that reduce uncertainties of the material cure state\textsuperscript{3} and yet minimize cure time. The parameters of interest include degree of cure, resin flow, refractive index, viscosity, temperature, pressure, voidage, and the formation of residual stress.\textsuperscript{2} The small size of optical fibers renders them particularly effective as embedded point sensors for measuring cure-related parameters in-situ, in real-time, and without affecting the structural integrity of the composite material. Furthermore, the same sensors could also be used post-cure to monitor parameters related to the composite structural integrity throughout its lifetime.
The focus of this research is on the cure monitoring of polymeric composite materials. The following sections briefly introduce the subjects of epoxy cure, some of the most relevant and useful sensor developments in cure process monitoring, the relationship between the refractive index of the curing material and the percentage of cure, and the objective and final optical fiber sensor design of this thesis work.

1.2 EPOXY CURE

Epoxy resins are low molecular weight organic liquids containing reactive epoxide groups that consist of an oxygen atom joined to each of two carbon atoms as shown in Figure 1.1.\textsuperscript{4} The cure process often involves the application of heat and pressure to a mixture of epoxy resin and an amine- or hydroxyl-based curing agent for predetermined time periods. Such a process transforms the liquid resin that surrounds the layers of reinforcing fibers of a laminate into a solid polymeric network, and thus forms a hardened composite material.\textsuperscript{1} The reaction is exothermic, i.e. it produces heat, and is accompanied by a small amount of shrinkage.\textsuperscript{5}

\begin{center}
\includegraphics[width=0.2\textwidth]{figure1.1.png}
\end{center}

\textit{Figure 1.1} Epoxide group.\textsuperscript{3}

Cure refers to an irreversible chemical reaction during which an epoxy branches, gels and cross-links,\textsuperscript{2} as shown in Figure 1.2.\textsuperscript{3} In recent years, cure processes have been primarily based on changes in viscosity.\textsuperscript{1} At room temperature, the viscosity of the resin and curing agent mixture in the prepreg is very high. As the resin is heated during the first stages of the cure cycle, its
viscosity drops significantly enabling the epoxy to thoroughly penetrate and wet the reinforcing fibers. Subsequently, the epoxide groups become more reactive and the oxygen atoms bond with donor hydrogen atoms from the curing agent. Neighboring polymers are then joined together to form branches which continue to grow until the reaction reaches gelation, at which point very
long networks have formed and the part has become a rigid semisolid. Pressure is often applied before the low-viscosity stage ends.\footnote{1}

After gelation, cross-linking between the long branches occurs which causes the viscosity to increase sharply and the epoxy to quickly become a hard solid. Cross-linking refers to the "the setting up of chemical links between molecular chains" such that "one infusible supermolecule of all the chains" is formed.\footnote{1} Figure 1.3 shows a typical change in viscosity for a 3501-6 resin as it is heat-cycled.\footnote{1}

![Figure 1.3 Viscosity of a 3501-6 resin as applied heat is increased at 0.56 °C/min.\footnote{1}](image)

1.3 DETERMINATION OF CURE EXTENT

There are several models that have been developed to describe the degree of cure of epoxy matrix composites. One of the most famous models came from research conducted by Loos and Springer in 1983 [27], which characterizes many cure parameters in great theoretical detail. Other models are empirical in nature, such as the non-linear differential equation curve fit, which requires a
few experimentally determined rate constants for the determination of cure percentage versus time.

One model of particular interest to this work is one that is based on the Lorentz-Lorenz equation, which is given by\textsuperscript{6,10}

\[ R = \left( \frac{n^2 - 1}{n^2 + 2} \right) \left( \frac{M}{\rho} \right). \]  

Here, \( R \) is known as the molar refraction, \( n \) is the refractive index, \( M \) is the molecular weight, and \( \rho \) is the density. Since this equation relates the chemical reactions which take place within an epoxy solution to its refractive index, it could possibly be used to find the correspondence between index of refraction and extent of cure.

The density and chemistry of an epoxy change as functions of cure percentage and temperature; and therefore, so does the refractive index.\textsuperscript{6} So, for isothermal cure cycles, \( n \) is a function of cure extent; however, for isothermal cures at different temperatures or for cure processes that require temperature cycling, the task of relating refractive index to cure percentage becomes more complicated. For example, when cured at different temperatures, an epoxy will gel at different cure extents. Also, if the cure temperature is not high enough, the epoxy will not fully cure. Thus, we must obtain more detailed measurements of the refractive index of curing epoxies as a function of cure percentage and temperature in order to verify that the Lorentz-Lorenz equation provides an appropriate model for relating the refractive index to the extent of cure of typical epoxies.
1.4 DEVELOPMENTS IN CURE MONITORING SENSORS

This section reviews four research efforts to develop fiber-based embedded sensors that are capable of measuring cure-related parameters in-situ, in real-time, at specific locations within, and without affecting the structural integrity of the composite material.

In 1986, Harrold and Sanjana embedded acoustic waveguides composed of both polyester fiber glass\(^6\) and nichrome wire\(^7\) in epoxy resin and graphite/epoxy composites, respectively.\(^2\) They measured the acoustic wave velocity in hopes that they could detect changes in the speed due to the small changes in acoustic impedance and viscosity that occur during the cure of the host material. They found that as the matrix approached gelation, the transit time of the acoustic wave quickly increased, corresponding to a decrease in wave velocity. Subsequently, they were able to pin-point gelation accurately. Once embedded, this sensor has the added advantage that it can be used throughout the lifetime of the material to sense impact events, stress/strain, etc. However, the sensors used for these experiments were approximately 1.5 mm in diameter. By designing sensors out of optical fiber, the sensor size can still be reduced by an order of magnitude.

Afromowitz also developed a polymer cure monitor in 1988.\(^8\) The sensor was designed to be sensitive to the difference between the refractive index of the curing polymer and that of a fully cured reference fiber made of the same thermoset material. First, a short length of fiber composed of fully cured resin was joined at both ends to multimode optical fiber and embedded inside a composite panel, as shown in Figure 1.4. Light from a broadband source was then injected into one of the fibers, through the reference fiber (which is the sensing region), and detected at the other end. The composite was heated and
as the uncured resin surrounding the reference fiber began to react, its refractive index increased until it matched that of the reference fiber, which led to a change in the guidance properties of the reference fiber such that the amount of light confined within and transmitted through the guide decreased to a very small value. Figure 1.5 shows results of one of Afromowitz' experiments, in which the relative light intensity transmitted through the sensing region versus time was detected for a sensor made from EPON® 828 Epoxy cured with 14 phr m-phenylenediamine.

![Schematic of sensor design](image)

**Figure 1.4** Schematic of the sensor design used by Afromowitz to monitor the cure of polymers.

Theoretically, the optical power detected at the output of this sensor is dependent upon the number of guided modes $M$, which is defined as:

$$M = \frac{V^2}{2},$$

1.2

when many modes exist. Such is the case if multimode optical fiber is used. Here, $V$ is the normalized frequency given by

$$V = \frac{2\pi a}{\lambda \left(n_1^2 - n_2^2\right)^{1/2}},$$

1.3

where $a$ is the radius of the cured polymeric optical fiber, $\lambda$ is the wavelength of the incident light, and $n_{1,2}$ are the refractive indices of the glass fiber and the
Figure 1.5 The relative light intensity transmitted through the sensing region versus time for a sensor made from EPON® 828 Epoxy cured with 14 phr m-phenylenediamine.

surrounding medium (curing resin), respectively.

From Equation 1.3, it can be seen that the transmitted intensity will be related to the difference between the squares of the two refractive indices. When the normalized frequency is less than or equal to 2.405, the cured polymeric fiber guide becomes single-mode. A further decrease in V leads to a less strongly bound modal intensity distribution within the sensor head so the light penetrates further and further into the surrounding medium where it is lost. When the refractive index of the curing resin is perfectly matched to that of the sensor, which occurs when polymerization is complete, the interface effectively ceases to exist and the light is no longer guided by the sensor. This effect occurs no matter what the temperature of the system.

There may still be some power transmitted through the sensor, depending upon the length of the cured resin fiber. Some of the light emitted from the transmitting multimode fiber is not spread (due to its numerical aperture)
beyond the core region of the receiving multimode fiber. This light does not need to be guided by the sensor anyway. The concept of numerical aperture is discussed in Chapter 3. Note that the numerical aperture of the input multimode fiber is dependent on the refractive index of the fully cured resin of the sensing region, which changes with temperature. This dependence causes the amount of light that is accepted by the output multimode fiber to change with respect to temperature. Thus, the length of the cured resin fiber should be long enough to minimize the amount of light that can be transmitted through the sensing region without guidance, and the system should also be isothermal at the end of the cure in order for this sensor to retain its self referential feature as an endpoint cure-detector.

According to Afromowitz, this sensor may also be implemented to monitor an epoxy throughout the cure process as soon as more information is gathered concerning the relationship between refractive index and extent of cure at various temperatures. This knowledge would then permit a calibration of the fiber response using Equation 1.3 from which one may determine the extent of cure at any point in an isothermal cure process.

Afromowitz also conducted some experiments to determine the refractive index as a function of cure extent at several isothermal cure temperatures. For this study, he used an optical fiber as a simple Fresnel reflectometer. He also used a differential scanning calorimeter (DSC) to analyze the exotherm of an epoxy cured with the same hardener and under identical isothermal conditions. Figures 1.6a-c show the refractive index and cure extent versus the cure time of EPON® 828 Epoxy cured with 14 phr m-phenylenediamine at 60, 90, and 130 °C. Figure 1.6d shows the index of refraction versus cure percentage for the same experimental tests. This data shows that it may not be assumed that the
Figure 1.6 The refractive index and cure extent of EPON® 828 Epoxy cured with 14 phr m-phenylenediamine at a) 60, b) 90, and c) 130 °C. The refractive index of the same epoxy mixture versus cure percentage at 60, 90, and 130 °C.

refractive index varies linearly with cure for all temperature cure cycles.

An effort to develop a sensor that could be used to monitor the cure of a polymeric composite during its fabrication, as well as the structural integrity of the composite throughout its lifetime, has also been conducted by Sanderson et al. at the Fiber & Electro-Optics Research Center at Virginia Tech. They researched the possibility of adapting the conventional extrinsic Fabry-Perot interferometer (which is described in detail in Chapter 2) so it could be used to
monitor the degree of cure of a polymeric matrix composite, as well as the presence of static or dynamic strain, or stress waves originating from acoustic sources such as acoustic emission, ballistic impacts, or ultrasonic nondestructive evaluation (NDE) sources in the material post-cure.

Although the details of optical fiber sensing are explained in the subsequent chapters of this thesis report, a brief summary of the experimental setup and sensor design that Sanderson et al. proposed is included here. Figure 1.7 depicts the basic experimental setup and sensor design that they proposed to use for strain measurement and cure monitoring. A light emitting diode (LED) or a laser diode, depending on whether a noncoherent or coherent source is needed, was to be used to inject light into a singlemode fiber coupler, where 50% of the optical power would be directed to the sensor head. Upon reflection from the sensor, the light would propagate back through the splitter and to a photodetector where its intensity would be monitored. The sensor itself was to be constructed by inserting the cleaved fiber endfaces of the output coupler leg and a homogeneous glass fiber into a hollow core fiber. Also, the far endface of the homogeneous fiber was to be coated with a metallic film.

For the measurement of strain, a coherent light source was used so the detected signal could be phase-modulated when the air gap length changes. The number of periodic variations, or partial variations for that matter, of the resultant sinusoidal signal output could then be related to the air gap length displacement, and thus to the strain experienced by a certain length of the sensor head. For cure monitoring, a broadband source was to be implemented so, in the absence of any air gap displacement, the output intensity of the sensor would decrease to zero as the refractive index of a curing epoxy resin that surrounds the homogeneous glass fiber increased to a value equal to that of the fiber.
Figure 1.7 The proposed design by Sanderson et al. for the measurement of the refractive index of a curing polymer and strain throughout the material lifetime.

Although they were successful in demonstrating that the sensor could be used to measure strain, several problems prevented them from implementing the final sensor design to quantitatively measure the refractive index of a curing polymer. One area of difficulty was that mode mixing occurred as the light propagated along the length of the homogeneous fiber, causing the light power to be distributed more evenly across the cross sectional area of the fiber. This lack of confinement resulted in much loss of power since most of the light could not be accepted back into the small core of the singlemode fiber. Another problem source was that much power loss is incurred due to the spread of light as it propagates through the air gap, especially in the direction toward the
singlemode fiber.

1.5 OBJECTIVE

We describe the continuation of the effort to design a multi-functional fiber optic sensor which can be embedded in polymeric composite laminates for monitoring the degree of cure during its fabrication, as well as internal composite strains occurring post-cure. In short, this dual-purpose sensor combines the characteristics of a Fresnel reflectometer with those of the extrinsic Fabry-Perot interferometer developed by Murphy et al. at the Fiber & Electro-Optics Research Center at Virginia Tech. For monitoring cure, a broadband source is used so the output intensity of the sensor is amplitude-modulated as the refractive index of the composite is increased during the polymerization process. Post-cure, a coherent light source is implemented so a sinusoidal variation of the output signal occurs when strains within the composite cause the sensor output to be phase-modulated.

This thesis is composed of five chapters. The following chapter provides an overview of the concepts and techniques involved in optical fiber sensing. Chapter 3 describes a preliminary experiment involving the Fresnel reflectometer and discusses the theoretical development of the dual-use sensor. Chapter 4 includes a detailed account of all of the experiments conducted with this sensor, and finally, Chapter 5 summarizes this research and provides suggestions for future work.
2 INTRODUCTION TO FIBER OPTIC SENSING

Optical fiber sensors have many advantages over conventional electronic sensors. These advantages include light weight and small size, large bandwidth, high resolution, immunity to electromagnetic interference, sensitivity to a wide range of measurands, ability to perform in high temperature environments (since the softening point of silica is about 1000 °C), and multiplexing capabilities.\textsuperscript{11}

Optical fiber sensors may be classified in several ways. As sensors, they are often grouped according to the perturbation which they measure, such as strain, temperature, pressure, or vibration. As optical fiber sensors, they are often grouped as either extrinsic or intrinsic depending upon whether or not the propagating light exits the fiber. Optical fiber sensors can also be considered as either amplitude- or phase-modulated, which is the classification choice for the descriptions and detailed examples provided in this chapter.

2.1 AMPLITUDE-MODULATED SENSORS

Amplitude-modulated, or intensity-based, sensors measure changes in received optical power. They are simple in operation and require only modest signal processing. Hence, they are inexpensive. The main disadvantage of intensity-based sensors is that they are sensitive to source fluctuations, and optical fiber macro- and microbends in any fiber leg in the system. Two examples of such sensors are described in this section.
2.1.1 The Fresnel Reflectometer

The optical fiber reflectometer is a simple sensor design that utilizes the relationship between the reflection intensity at a boundary between two media, and the refractive index of the two media. The sensor design is based on the following theory.

First, let an optical wave be propagating at normal incidence to the boundary between two lossless media, and let it be represented as a perpendicularly polarized plane wave with electric field, $\vec{E}_i$, and magnetic field, $\vec{H}_i$, of the form\textsuperscript{12,13}

$$\vec{E}_i = \hat{x}E_{i0}\exp\{j\phi\}, \quad 2.1$$

and

$$\vec{H}_i = \hat{y}H_{i0}\exp\{j\phi\}, \quad 2.2$$

where the optical phase, $\phi$, is

$$\phi = \omega t - \beta z + \varphi. \quad 2.3$$

$E_{i0}$ and $H_{i0}$ are the field amplitudes, $\hat{x}$ and $\hat{y}$ represent the field directions, $\omega$ is the frequency, $t$ stands for time, $\beta$ is the propagation constant, $z$ represents the propagation direction, and $\varphi$ is the phase constant. Figure 2.1 shows this interface, the incident fields, and the reflected and transmitted fields which have forms similar to Equations 2.1 and 2.2 except that these fields are denoted with $r$ and $t$ subscripts.

Boundary conditions derived from the integral form of Maxwell's equations state that the tangential components of the electric field across an interface between two media are continuous, so\textsuperscript{13}
Figure 2.1. The incident, reflected, and transmitted electromagnetic fields at the boundary between two media.

\[ \vec{E}_i + \vec{E}_r = \vec{E}_t. \]  

Likewise, the tangential components of the magnetic field across an interface are continuous, or\textsuperscript{13}

\[ \frac{\vec{E}_i}{\eta_1} - \frac{\vec{E}_r}{\eta_2} = \frac{\vec{E}_t}{\eta_2}, \]

where the subscripts 1 and 2 denote the first and second media that create the interface and \( \eta \) is the intrinsic impedance of the medium as given by\textsuperscript{13}

\[ \eta = \sqrt{\frac{\mu}{\varepsilon}}. \]

Given that \( \mu_1 = \mu_2 = \mu_0 \) for dielectrics, and since the index of refraction, \( n \), is equal to the square root of the relative permittivity, or \( \varepsilon \), the Fresnel reflection coefficient, \( \Gamma \), can be found from the ratio of the reflected field to that of the incident field, as in\textsuperscript{14}
\[
\Gamma = \frac{\bar{E}_t}{E_i} = \frac{n_2 - n_1}{n_2 + n_1} = \frac{n_1 - n_2}{n_1 + n_2}.
\]

The ratio of the reflected to the incident intensity is called the reflectance, \(|\Gamma|^2\). It is a parabolic function with respect to the refractive indices of the two media, or

\[
|\Gamma|^2 = \left(\frac{n_1 - n_2}{n_1 + n_2}\right)^2.
\]

When observing reflections from a flat optical fiber endface submerged in a curing material, \(n_1\) is the modal refractive index of the fiber (for singlemode fiber) and \(n_2\) is that of the material being processed.

The theoretical reflectance (described by Equation 2.8) from an optical fiber (\(n_1 \equiv 1.46\)) as a function of the refractive index of the material in which the fiber is immersed is shown in Figure 2.2. The intensity drops to zero as the refractive index of the processing material becomes more closely matched to that of the fiber. As \(n_2\) becomes greater than \(n_1\), a phase shift of 180° occurs for perpendicularly polarized light.\textsuperscript{15} The theoretical reflectance also approaches one as \(n_2\) approaches either zero or infinity. For small refractive index changes, the reflectance varies approximately linearly with respect to the refractive index of the curing material.

An experimental arrangement for the Fresnel reflectometer is shown in Figure 2.3. A broadband source, such as an LED, is used to inject light into one arm of a 2 × 2, bi-directional coupler since such sources typically have less intensity fluctuations. A photodetector is used to interrogate the reflectance from the sensor head as the refractive index of the medium surrounding the sensor changes.
Figure 2.2. The normalized theoretical reflection intensity from an optical fiber with respect to the refractive index of the material in which it is immersed.

Figure 2.3. An experimental arrangement for monitoring the reflectance from a cleaved fiber.

2.1.2 Air Gap Sensors

Figure 2.4 depicts a second example of an amplitude-modulated fiber optic sensing design. The experimental setup is the same as for the Fresnel reflectometer except that the sensing head is composed of an air gap that
Figure 2.4. An intensity-based sensor design based on the reflectance from an air gap sensor.

basically produces two detectable reflections, one from the glass-to-air interface, \( R_1 \), and another from the air-to-glass interface \( R_2 \). Since a broadband light source is used, the two reflected optical waves will combine incoherently. Therefore, the optical phase component of the light waves can be neglected and each reflection intensity will be essentially equal to the square of the reflection coefficient times the input light intensity. However, since the light transmitted through the first interface spreads as it exits the fiber, the second reflection is not completely captured by the input/output fiber. In fact, as the air gap length increases from zero, the amount of light captured by the input/output fiber decreases dramatically at first and then gradually levels off to a constant value that is proportional to the reflectance from the glass-to-air fiber endface.

2.2 PHASE-MODULATED SENSORS

Many reflections are created when light is injected into an optical fiber sensor with a multi-path fiber geometry, such as that in an air gap sensor. When a coherent light source is used, the intensity that is output is dependent on the optical phase difference between the light waves that are created. In this example, the optical phase difference is due to the different path lengths associated with each reflection. Two examples of interferometric sensors
follow that are phase-modulated when the differential path length of some of the reflections from the sensor head are altered. The first example, called the extrinsic Fabry-Perot interferometer, is a relative sensor, meaning that it measures changes in a parameter. The second example is referred to as the absolute extrinsic Fabry-Perot interferometer since it provides actual parameter values. In general, interferometric devices have excellent resolution since their measurements are based on the source wavelength. The main drawback of interferometric-based sensor designs is that they require rather complicated signal processing.

Since the optical phase is dependent on several parameters, phase-modulated sensors can be further classified according to what causes the optical phase difference to be modified. Such modifications can result from a change in the path length, wavelength or frequency content, modal content, and/or polarization of the light. A third sensor design, which implements long-period refractive index gratings as mode couplers, is also included in which the optical phase of a propagation mode is altered as the refractive index of the surrounding medium changes.

2.2.1 The Extrinsic Fabry-Perot Interferometer

For the extrinsic Fabry-Perot interferometer (EFPI), laser light is injected into a bi-directional coupler leading to an input/output singlemode fiber, and a singlemode reflector fiber. These fibers form an air gap that acts as a low-finesse Fabry-Perot cavity, as shown in Figure 2.5. The far end of the second fiber is shattered so reflections do not add to detector noise. The Fresnel reflection from the glass-to-air interface at the front of the air gap (reference reflection, \( R_1 \)) and the sensing reflection from the air-to-glass interface at the far end of the air gap (\( R_2 \)) interfere in the input/output fiber. Although multiple
reflections occur within the air gap, the effect of reflections subsequent to the ones mentioned above are negligible. The reflector fiber is allowed to move longitudinally in the silica hollow core tube so the length of the air gap can increase or decrease. Changes in air gap length lead to changes in the phase difference between the reference and sensing reflection thus altering the intensity of the two interfering light waves at the detected output. The concept of interference is explained in the paragraphs that follow.

Let us once again represent two optical waves as perpendicularly polarized plane waves with electric and magnetic fields, $\vec{E}_{1,2}$ and $\vec{H}_{1,2}$, of the form\textsuperscript{12,13}

$$\vec{E}_{1,2} = \hat{x}\Gamma_{1,2}E_i \exp\left\{j(\omega t - \beta_{1,2}z_{1,2} + \varphi_{1,2})\right\},$$ \hspace{1cm} 2.9

and

$$\vec{H}_{1,2} = \hat{y}\Gamma_{1,2}H_i \exp\left\{j(\omega t - \beta_{1,2}z_{1,2} + \varphi_{1,2})\right\},$$ \hspace{1cm} 2.10
where \( E_i \) and \( H_i \) are the incident electric and magnetic field amplitudes, \( \Gamma \) is the reflection coefficient, \( z \) is the propagation distance, \( t \) stands for time, \( \omega \) is the frequency, and \( \phi \) is the phase constant. The propagation constant, \( \beta \), is

\[
\beta = \frac{2\pi n}{\lambda},
\]

where \( n \) is the refractive index and \( \lambda \) is the wavelength of the optical wave. The subscripts 1 and 2 denote the reference and sensing reflections. Here, we have assumed that the field amplitudes are the same for both reflections. When the two optical waves are superposed, the intensity of the total wave is\(^{14}\)

\[
I_r = \frac{1}{2} \text{Re}(\mathbf{E} \times \mathbf{H}^\star).
\]

Equation 2.7 shows that the reflection coefficient is negative when reflectance takes place in the medium with the lower refractive index. Hence, since \( \Gamma_2 \equiv -\Gamma_1 \), and so substitution of Equations 2.9 and 2.10 into Equation 2.12 yields

\[
I_r = \frac{(\Gamma_1 E_i)^2}{\eta} \left[1 - \cos(\beta_1 z_1 - \beta_2 z_2)\right],
\]

where the substitution \( \phi_1 = \phi_2 \) has been made since the two reflections originate from the same beam of laser light injected into the fiber. Referring to Figure 2.5, we see that the reference reflection (\( R_1 \)) follows a path through the fiber and air gap such that the product, \( \beta_1 z_1 \), is given by

\[
\beta_1 z_1 = -\beta_1 z_{r1} - \beta_1 z_{r2},
\]

whereas the product, \( \beta_2 z_2 \), for the sensing reflection (\( R_2 \)) is

\[
\beta_2 z_2 = -\beta_1 z_{r1} - \beta_a z_a - \beta_1 z_{r2}.
\]

\( \beta_1 \) is the propagation constant of an optical wave in the fiber, \( \beta_a \) is that in air, \( z_{r1} \) is the propagation distance in the fiber from the laser to the sensor head, \( z_a \)
is the length of the air gap, and $z_{12}$ is the distance from the air gap to the photodetector. Therefore, the total field intensity of the two superposed reflections is given by

$$I_t = \frac{2(\Gamma_i E_i)^2}{\eta} \sin^2 \left( \frac{2\pi z_a}{\lambda} \right),$$

where the substitution $n = 1$ has been made for the refractive index in air. The reflectance, $I_r/I$, is equal to the intensity of the two superposed beams divided by the intensity of the incident light wave, $I = E_i^2/2\eta$, which is

$$\frac{I_r}{I} = 4\Gamma_i^2 \sin^2 \left( \frac{2\pi z_a}{\lambda} \right),$$

and so the normalized reflectance is

$$\frac{I_r}{I} = \sin^2 \left( \frac{2\pi z_a}{\lambda} \right).$$

Figure 2.6 shows the variation of the reflectance with respect to the optical phase difference between the two superposed optical waves. Figure 2.7 shows the reflectance variation with respect to the air gap length of the sensor head depicted in Figure 2.5. To find the change in the air gap length that corresponds to one period of intensity variation with respect to the optical phase difference, we set the change in the optical phase difference, $\Delta(\Delta\phi)$, equal to an integer multiple of $2\pi$, or

$$\Delta(\Delta\phi) = \Delta\left( \frac{4\pi}{\lambda} z_a \right) = 2\pi N,$$

where $N$ is an integer that corresponds to the number of periodic revolutions.
Figure 2.6. Variation of the reflectance with respect to the optical phase difference between two interfering light waves.

Figure 2.7. Variation of reflectance with respect to the air gap length of the sensor head depicted in Figure 2.5 for a source wavelength of 1300 nm.

Expansion of the phase difference term results in

$$2 \left[ \frac{1}{\lambda} \cdot \Delta z_a + z_a \cdot \Delta \left( \frac{1}{\lambda} \right) \right] = N.$$  \hfill (2.20)
For EFPI operation, a laser source is used, and so in principle $\Delta(1/\lambda) = 0$. Therefore, the change in air gap length, $\Delta z_z$, of the EFPI sensor head is equal to the number of periodic revolutions, or fringes, multiplied by one-half of the laser source wavelength, as in

$$\Delta z_z = \frac{N\lambda}{2}. \quad 2.21$$

It should be noted that the EFPI is capable of measuring relative changes in the air gap, as opposed to absolute values of the air gap length. This means that if the laser source is turned off, no future measurements can be related back to the previous data. Only changes in the air gap length are measured by the EFPI, not the actual air gap length. Another problem with this design is that changes in direction of the output signal are impossible to detect if the output signal changes direction at a maximum or minimum on the transfer function. So, continuously changing parameters can be fully evaluated through the employment of the EFPI as long as the parameter is only increasing or only decreasing.

2.2.2 The Absolute Extrinsic Fabry-Perot Interferometer

Figure 2.8 depicts the reflectance (defined in Equation 2.18) of two interfering optical waves with respect to wavelength. Suppose now that we alter the arrangement in Figure 2.5 by injecting light from a broadband source, such as a light-emitting diode (LED), and use an optical spectrum analyzer (OSA) to detect the intensity of a broad range of wavelengths. With the substitution, $\Delta z_z = 0$, Equation 2.20 becomes

$$2z_z \cdot \Delta \left(\frac{1}{\lambda}\right) = 2z_z \cdot \left(\frac{1}{\lambda_1} - \frac{1}{\lambda_2}\right) = N, \quad 2.22$$
Figure 2.8. The normalized reflectance of two interfering optical waves with respect to wavelength for an air gap of 2.5 μm.

where \( \lambda_1 \) and \( \lambda_2 \) are the wavelengths of two interference peaks. Thus, the air gap length, \( z_a \), in the sensor head is given by

\[
z_a = \frac{N\lambda_1 \lambda_2}{2(\lambda_2 - \lambda_1)}
\]

where \( N \) is equal to one if we take \( \lambda_1 \) and \( \lambda_2 \) to be the wavelengths of two interference peaks that are directly adjacent to each other.

This arrangement is referred to as the absolute extrinsic Fabry-Perot interferometer, or AEFPI, since the measurements are of the actual air gap length. The consequence of having set the \( \Delta z_a \) to zero is that this sensor can only be used to measure static parameters—not quickly changing measurands. This restriction can be understood when one realizes that the air gap length must be constant while the OSA measures the light intensity that emerges from the sensor for a range of wavelengths.
Figure 2.9 shows the normalized reflectance with respect to the two independent parameters of the phase difference, namely, $z$ and $\lambda$. Notice that for longer wavelengths, the period of the change in reflectance with respect to air gap length is larger.

2.2.3 Long-Period Gratings

Certain optical fiber waveguides, such as germania-doped silica fiber, exhibit the property of photosensitivity. This is the means by which intensity-dependent refractive index changes are photoinduced in the core of fibers upon exposure to ultraviolet (UV) light.\textsuperscript{16,17} External writing techniques are noninvasive, side-exposure techniques by which a periodic spatial variation of high and low intensity is created on the fiber which increases the refractive index of the high intensity regions. Such refractive index gratings are often formed in optical fibers using a phase mask. First, an ultraviolet (UV) light beam is diffracted into the -1, 0, and +1 orders and modulated spatially to form an interference pattern of alternating field intensity along the length of the optical fiber.\textsuperscript{17} Typically, the -1 and +1 orders each contain more than 35% of the diffracted light so the 0\textsuperscript{th} order does not have to be suppressed. This technique is depicted in Figure 2.10.

Partial coupling between two propagating modes of light in an optical fiber occurs whenever the two modes are in phase. If this phase matching condition is met by a multitude of periodic spatial perturbations such as physical deformations or refractive index variations in the fiber, an appreciable amount of power coupling can be achieved between the two propagation modes which have a beatlength equal to the spatial period of the perturbation. Thus, coupling occurs if the optical phase difference, $\Delta \phi$, between the two modes equals an integer multiple of $2\pi$ (i.e., when the two modes are in phase),\textsuperscript{18} as in
Figure 2.9 Variation of reflectance with respect to both wavelength and gap length.
Figure 2.10. Formation of a refractive index grating using diffraction from a mask.

\[
\Delta \phi = \phi_1 - \phi_2 = \beta_1 z - \beta_2 z = 2\pi N.
\]  \hspace{1cm} 2.24

Here, \( \beta_1 \) and \( \beta_2 \) are the propagation constants of the two modes, \( N \) is an integer, and \( z \) is the propagation distance over which partial coupling between the two modes occurs. We can solve for the wavelength at which phase matching between the propagation modes is satisfied by substituting Equation 2.11 into Equation 2.24. This yields

\[
\lambda = \frac{z(\bar{n}_1 - \bar{n}_2)}{N},
\]  \hspace{1cm} 2.25

where the refractive index, \( n \), has been replaced with the refractive index of the propagation mode in the optical fiber, \( \bar{n} \). However, since the modal refractive index is dependent on wavelength, \(^{19}\) strong coupling between the two modes will only occur at one wavelength. Therefore, \( N \) is normally set to 1 and the properties of the diffraction grating determine the period between the maximum refractive index changes in the optical fiber. For optical fiber gratings, \( z \) is equal to the period, \( \Lambda \), of the grating spatial variation in
refractive index, and so we obtain the equation

$$\lambda = \Lambda(\bar{n}_1 - \bar{n}_2).$$  \hspace{1cm} 2.26

Thus, by varying the periodicity of the grating, propagation modes can be coupled (at a specific wavelength) from guided to radiation modes, from one guided mode to another, from one polarization to another, or from forward propagating to backward propagating modes at a particular wavelength.

For the step-index fiber shown in Figure 2.11, the refractive index of guided modes, \(\bar{n}_i\), must be\(^{12}\)

$$n_{\text{core}} < \bar{n}_1 < n_{\text{clad}},$$  \hspace{1cm} 2.27

where \(n_{\text{core}}\) and \(n_{\text{clad}}\) are the refractive indices of the fiber core and cladding, respectively. Likewise, the refractive index of a cladding mode, \(\bar{n}_2\), is\(^{12}\)

$$n_{\text{clad}} < \bar{n}_2 < n_{\text{surr}},$$  \hspace{1cm} 2.28

where \(n_{\text{surr}}\) is the index of the fiber cladding's surrounding environment (usually air). It is apparent that since the refractive index of the cladding mode, \(\bar{n}_2\), is dependent on the refractive index of the surrounding medium, a modification of the refractive index of a material surrounding the fiber will change the waveguiding properties of the light within the fiber such that the wavelength at which mode conversion occurs is shifted. For long-period gratings (LPG's), the two modes that couple together are the original guided mode and one of a discrete number of cladding modes. Since the cladding modes are quickly dispersed into the surroundings and lost, a loss peak in the transmission spectrum of an LPG is observed at the wavelength at which the phase matching condition is satisfied. The periodicity of the refractive index variation for LPG's is typically on the order of hundreds of microns.
Figure 2.11. View of an optical fiber endface.
3 DESIGN OF A MULTI-FUNCTIONAL OPTICAL FIBER SENSOR BASED ON FRESNEL REFLECTIONS

A dual-use sensor design that combines the characteristics of the Fresnel reflectometer with those of the conventional EFPI is evaluated based on its ability to monitor the degree of cure of a polymer matrix composite, as well as the presence of strain post-cure. Figure 3.1 depicts the final sensor design along with the basic experimental arrangement needed for cure monitoring and strain measurement. The experimental setup and sensor design are similar to those discussed in Chapter 2. For cure monitoring, a broadband source is used and so the output intensity of the sensor is amplitude-modulated if the three resulting reflections are varied, and for the measurement of strain, a coherent light source is used so the sinusoidal variation of the output signal is due only to the interference of the two reflections from the air gap.

As a first step toward the final sensor design shown in Figure 3.1, we measured the intensity reflected from an optical fiber endface immersed in a common epoxy resin during its cure. This experimental analysis is discussed in Section 3.1. In Section 3.2, we consider some theoretical details for the optimization of the Fresnel reflectometer/EFPI multi-functional sensor. In particular, the reflection properties of the incoherent light to be launched into the sensor during cure monitoring are analyzed in Section 3.2.1. Based on this analysis, the use of thin-film dichroic coatings as optical filters are considered for the dual-sensor design in an effort to raise the optical power throughput of the air gap as discussed in Section 3.2.2. Finally, Section 3.2.3 discusses how the numerical aperture of the optical fiber as well as the source coherence length are used to determine the optimal air gap length of the sensor.
Figure 3.1 Experimental setup for the Fresnel Reflectometer/EFPI.

3.1 THE FRESNEL REFLECTOMETER

To monitor the reflection intensity from a cleaved optical fiber endface as a function of the refractive index of the medium in which it is immersed, the experimental setup in Figure 3.2 was utilized. First, a function generator was used to intensity modulate a 1300 nm LED at 4 kHz. The light output from this LED was then injected into a 2 × 2 fused, biconical-tapered coupler with a coupling ratio of approximately 50/50. Photodetectors were used to monitor the light intensity at the legs numbered 2 (the sensing signal) and 4 (the reference fiber) of the coupler. Both output signals were then amplified and filtered (a bandpass filter was implemented at 4 kHz) by a lock-in amplifier and subsequently output to an oscilloscope. This arrangement enabled us to
Figure 3.2 Experimental setup used to monitor the reflectance from the cleaved end of a fiber as a function of the refractive index of the oil in which it is immersed.

monitor both the sensing and reference intensity signals at the same time. So that source intensity fluctuations could not affect the data analysis, the sensing signal was divided by the reference signal to obtain the output signal.

The change in output light intensity of the sensing signal was anticipated to be quite small (especially in the dual-sensor design), so some additional precautions were taken to keep the signal-to-noise ratio as high as possible. InGaAs photodetectors were utilized in this experimental setup due to their large responsivity at 1300 nm relative to that of Ge photodetectors. The photodetectors had small active areas (75 μm) so that their dark currents would be small, and an attached lens with a focal length of 1±0.3 mm was used so that light incident on the lens would be focused onto the small active area. Thus, legs 2 and 4 of the coupler had to be cleaved and aligned at an optimum angle and position relative to the photodetector lenses.
Next, leg 3 of the coupler was cleaved and dipped into calibrated, NIST-traceable, refractive index oils made by Cargille Company. Such oils are certified to have an accuracy of ±0.0002. For each oil, the output voltage signal from leg 2 of the coupler was divided by that of leg 4. Normalization of the data yielded the results shown in Figure 3.3 which are quite similar to the theoretical reflectance that was graphed in Figure 2.2. It should be noted that each time the sensing fiber of Figure 3.2 is cleaved, the reflection intensity from its endface changed slightly. Thus, the sensor must be calibrated before each cure test to determine how much reflection occurs with respect to the refractive index of the oil.

![Normalized Reflectance vs Refractive Index](image)

**Figure 3.3** Calibration curve for a cleaved optical fiber as a function of refractive index.

The refractive index of 3M Scotch-Weld™ DP-100 fast setting epoxy adhesive was then monitored during its cure at room temperature for over 80 minutes. The assembly in Figure 3.2 was used once again, except that the cleaved endface on leg 3 was immersed in 50 ml of the curing epoxy resin. To measure any temperature changes that occur within the curing epoxy, thermocouple leads
were run from the curing epoxy to an Omega temperature indicator which had been set to provide an analog output with a linear transfer function such that 0 to 10 V dc output corresponded to about 60 to 350 °F.

By dividing the sensing signal by the reference signal and interpolating between the data points of the calibration curve, the refractive index of the curing epoxy was computed and graphed in Figure 3.4. Initially, the refractive index of the epoxy decreased from approximately 1.54 to 1.53 since the exothermic reaction caused the temperature of the resin mixture to rise from about 76 °F (0.56 V) to 273 °F (7.33 V), but as the heat of the reaction was slowly released to the surroundings, the refractive index increased to above 1.56 at about 106 °F (1.57 V) in approximately 80 minutes. The higher refractive index indicates that crosslinking of the epoxy had lead to a slightly denser composition.

![Figure 3.4 Refractive index and reaction temperature of the epoxy adhesive during its cure.](image-url)
3.2 DESIGN CONSIDERATIONS FOR THE DUAL-SENSOR

In an effort to enhance the ability of the multi-functional sensor to measure the very small changes in refractive index that result during the cure of epoxy resins, both the overall power transmitted through the air gap and the length of the air gap needed to be optimized. In Sections 3.2.1 and 3.2.2, the reflection properties of the incoherent light to be launched into the sensor during cure monitoring are analyzed, and based on this analysis, the use of thin-film optical filters as dichroic coatings is considered for the dual-sensor design in an effort to raise the optical power output of the air gap. Finally, Section 3.2.3 discusses how the numerical aperture of the optical fiber as well as the source coherence length are used to determine the optimal air gap length of the sensor.

3.2.1 Incoherent Reflection

Coherence is defined as "the existence of a correlation between the phases of two or more waves, so that interference effects may be produced between them."\(^{11}\) If an intensity signal is composed of the superposition of two or more reflected optical waves, the coherence of such a signal is weakened if 1) the waves are uncollimated, 2) the difference in the propagation path lengths of the waves is too large, 3) the reflecting surfaces are curved, and/or 4) the reflecting surfaces are not parallel.\(^{14,15}\) Furthermore, the average of coherently interfering field intensities over a broad range of wavelengths yields the same result as the sum of incoherently interfering fields.\(^{15}\) In effect, the optical phase does not need to be taken into consideration for light waves that add incoherently.

Figure 3.5 shows two boundaries with incident, reflected, and transmitted light intensities of \(I_{0}, I_{\text{refl}},\) and \(I_{\text{trans}}\). Since the phase does not need to be considered,
the individual reflectances, $\Gamma_0^2$, can be summed to form an overall reflectance, $\Gamma^2$, as given by\textsuperscript{14}

$$\Gamma^2 = \Gamma_0^2 + (1-\Gamma_0^2)^2 \Gamma_0^2 \left[ 1 + (\Gamma_0^4) + (\Gamma_0^4)^2 + ... \right]. \tag{3.1}$$

Here, multiple reflections have been included, and it has been assumed that there is no absorption in any of the materials that form the boundaries. The geometric series in Equation 3.1 converges, so the reflection intensity becomes

$$I_{\text{refl}} = I_0 \Gamma^2 = I_0 \frac{2 \Gamma_0^2}{1 + \Gamma_0^2}. \tag{3.2}$$

Likewise, the transmitted intensity is

$$I_{\text{trans}} = I_0 (1 - \Gamma^2) = I_0 \left[ \frac{1 - \Gamma_0^2}{1 + \Gamma_0^2} \right]. \tag{3.3}$$

Figure 3.5 Two boundaries with reflectances $\Gamma_0^2$. The incident, reflected, and transmitted intensities are $I_0$, $I_{\text{refl}}$, and $I_{\text{trans}}$.

Figure 3.6 shows three boundaries with incident and total reflection intensities of $I_0$ and $I_{\text{total-refl}}$. Since the two boundaries on the left have an overall reflectance, $\Gamma^2$ (given by Equation 3.2), the total reflectance, $\Gamma_{\text{total}}^2$, is found to be
\[ \Gamma_{\text{total}}^2 = \Gamma^2 + (1 - \Gamma^2)^2 \Gamma_3^2 \left[ 1 + \left( \Gamma^2 \Gamma_3^2 \right) + \left( \Gamma^2 \Gamma_3^2 \right)^2 + \ldots \right], \] 3.4

where \( \Gamma_3^2 \) is the reflectance associated with the third interface. Thus, the total reflection intensity from the three boundaries is given by

\[ I_{\text{total-refl}} = I_0 \Gamma_{\text{total}}^2 = I_0 \left[ \frac{\Gamma^2 - 2\Gamma^2 \Gamma_3^2 + \Gamma_3^2}{1 - \Gamma^2 \Gamma_3^2} \right]. \] 3.5

If the optical loss (due to the spread of light upon exiting the singlemode fiber) in the air gap is neglected, and if a broadband source is used to inject light into the coupler, Equation 3.5 can be used to describe the reflection intensity of the sensor head in Figure 3.1. In this application, \( \Gamma \) is the total reflection coefficient associated with the air gap, and \( \Gamma_3 \) is the reflection coefficient associated with the far-end of the sensor. Furthermore, if the far-end of the sensor is immersed in epoxy, then \( \Gamma_3 \) is related to the refractive index of the epoxy through Equation 2.7.

![Diagram of light reflection](Image)

**Figure 3.6** Three boundaries grouped so that the first two boundaries have an overall reflectance of \( \Gamma^2 \) and the third has a reflectance of \( \Gamma_3^2 \). The incident and reflected intensities are \( I_0 \) and \( I_{\text{refl}} \).

The normalized theoretical reflectance is graphed in Figure 3.7 with respect to
the refractive index of the medium surrounding the far-end of the air gap sensor depicted in Figure 3.1. As the reflectance, $\Gamma^2_\gamma$, from the glass-to-air interface and the air-to-glass interface in the air gap decreases, the minimum of the overall reflectance, $\Gamma^2_{\text{total}}$, shifts down. It is, in part, this minimum reflectance that determines the percentage signal change of the renormalized reflectance within the refractive index range of interest of 3M Scotch-Weld™ DP-100 fast setting epoxy adhesive (1.475 to 1.620).

The renormalized reflectance from the sensor for the refractive index range of 1.475 to 1.620 is depicted in Figure 3.8. Note that each of the curves has been normalized again so that we can compare the percentage of change in the output signal which is not a function of the amount of amplification that would be used experimentally. A normalized curve should be fairly linear and its slope should be close to ±1 in order for the signal output change to be fully maximized with respect to a change in the parameter of interest. Without antireflective coatings, the reflectance, $\Gamma^2_\gamma$ from a cleaved fiber endface is

![Graph](image)

**Figure 3.7** The normalized theoretical reflectance with respect to the refractive index of the medium surrounding the far-end of the air gap sensor depicted in Figure 3.1.
Figure 3.8 The renormalized reflectance for the refractive index range of 1.475 to 1.620 in Figure 3.7.

approximately 3.5%. Figure 3.8 demonstrates that the reflectance from the air gap should be reduced in order to increase the percentage of sensing output signal change that corresponds to a change in refractive index change.

3.2.2 Thin-Film Optical Filters

From the discussion in the previous section, it is apparent that it would be advantageous to evaporate thin-film dielectric materials onto the fiber endfaces for the purpose of creating antireflective glass-to-air and air-to-glass interfaces. Such optical coatings operate by light wave interference created by reflections from the thin-film layers whereby the thicknesses and refractive indices of the layers determine the optical properties of the coating. Here, the concepts of reflection from an interface and the interference of light will be used to explain how multiple thin-film layers of dielectric coatings can be combined to form optical filters.
First, we describe the conditions under which a single layer thin-film is antireflective. Suppose an optical wave is incident from a medium with refractive index \( n_0 \) into a medium with refractive index \( n_2 \). Now suppose a single thin-film is inserted between these two media, as shown in Figure 3.9, with refractive index \( n_1 \) and thickness \( d \). We also stipulate that \( n_1 \) must have a refractive index that is between \( n_0 \) and \( n_2 \).

Let us consider the case when \( n_0 > n_1 > n_2 \). We can describe the interference between the two reflections from the first and second interface in a manner that is very similar to the analysis of the EFPL, except that here the amplitudes of the first and second reflections are not set equal to each other. In short, the reflections are represented by electric and magnetic fields, \( \vec{E}_{1,2} \) and \( \vec{H}_{1,2} \), as given by

\[
\vec{E}_{1,2} = \sqrt{2} A_{1,2} E_i \exp\left[j\left(\omega t + \beta z + \phi\right)\right],
\]

and

\[
\vec{H}_{1,2} = \sqrt{2} A_{1,2} H_i \exp\left[j\left(\omega t - \beta z + \phi\right)\right].
\]

Here, the numerals 1 and 2 denote the reflection from the first and second boundaries, respectively, \( A_1 = \Gamma_1 \) is the amplitude of the electric field reflected from the first boundary, \( A_2 = (1 + \Gamma_1)^2 \Gamma_2 \) is the amplitude of the electric field that is transmitted through the first, reflected from the second, and back through the first boundary, \( E_i \) and \( H_i \) are the incident electric and magnetic field amplitudes, and \( \omega, \beta, \phi \) are defined in Section 2.2.1. Thus, the total intensity, \( I_T \), of the combined two most intense reflected fields is given by

\[
I_T = \frac{1}{2\pi} \left[ A_1^2 + A_2^2 + 2A_1A_2 \cos\left(\frac{4\pi n_1d}{\lambda}\right)\right],
\]
where, $\lambda$ is the wavelength of the incident light and $\eta$ is the intrinsic impedance of the medium in which the two reflected beams are interfering.

There are two conditions which must be satisfied in order for the total intensity to vanish. The first is that the optical phase difference, $4\pi n_1 d / \lambda$, must be an odd integer multiple of $\pi$. The optical thickness of the coating, $n_1 d$, is therefore

$$n_1 d = \frac{\lambda}{4} (2N + 1). \quad 3.9$$

The second condition for destructive interference is that $A_1 = A_2$. For small reflections, $A_1 = A_2$ if $\Gamma_1 = \Gamma_2$, so we can set the reflection coefficients equal to obtain
\[
\frac{n_0 - n_1}{n_0 + n_1} = \frac{n_1 - n_2}{n_1 + n_2},
\]
(3.10)

or equivalently,

\[
n_1 = \sqrt{n_0 n_2}.
\]
(3.11)

Thus, the thinnest thickness for a single-layer antireflective coating should have an optical thickness that is one-quarter wavelength of the incident light wave, and the refractive index should be equal to the square root of the product of the refractive indices of the two media which are joined by the coating.

For the purpose of creating an antireflective coating on a fiber in air, \( n_0 \) is 1.46 and \( n_1 \) is 1. So, \( n_1 \) should be 1.21 in order to satisfy Equation 3.11. Magnesium fluoride is practical for the creation of a thin-film layer and has a low refractive index of 1.37 at 1300 nm\(^{14}\) which produces a reflectance of 0.10% at the first interface and 2.44% at the second interface.

Since it is often difficult to find a material with the refractive index that satisfies Equation 3.11 exactly, and since the reflections from each boundary may not be small enough for the assumption leading to Equation 3.11 to be valid, multi-layer, thin-film optical filters are usually implemented whenever extremely small reflections are required for an application especially if the coating should be antireflective over a broad range of wavelengths. Such coatings can consist of alternating high and low index stacked layers of dielectric material. A more detailed analysis of antireflective filter design techniques is given by H. Macleod.\(^{14}\)

An important consideration when using thin-film optical coatings in an optical fiber sensor design for cure monitoring is that changes in temperature will
cause the thickness of each layer to change by an amount directly proportional to the coefficient of thermal expansion of the layer. Such dimensional changes alter the spectral response of the optical filter which cause fluctuations in the sensor output power that are not a function of the refractive index of the medium surrounding the far endface of the sensor.

3.2.3 Optimization of Air Gap Length

There are two factors that affected our selection of the air gap length—the numerical aperture of the optical fiber and the coherence length of the light source. The numerical aperture dictates how much light will spread as it exits a fiber, and thus it can provide an estimation of the amount of optical loss associated with various air gap lengths. The coherence length of the light source must also be considered before designing the multi-functional sensor since the reflections from each of the interfaces that compose the dielectric stacked-layer coatings must interfere destructively, whereas the overall reflections from both sides of the air gap and from the far end of the sensor should only add incoherently.

The numerical aperture, NA, of an optical fiber is given by

\[ NA = n_0 \sin \theta_{0,\text{max}} \]  

3.12

where \( n_0 \) is the refractive index of the medium surrounding the fiber endface, and \( \theta_{0,\text{max}} \) is both the maximum angle of incidence at which light is accepted into a fiber and the maximum angle at which light exits a fiber. The optical fiber used in the construction of our sensors had a numerical aperture of 0.13 and a core radius of 4.15 \( \mu \text{m} \). Thus, as depicted in Figure 3.10, in order for light exiting one fiber to be completely captured by the other fiber, the air gap length, \( z_\ast \), must be
Figure 3.10 The spread of light upon exiting one fiber before being accepted by another.

\[ z_a \leq \frac{a}{\tan \theta_{0,\text{max}}} = 32 \mu\text{m}. \]  

3.13

Here, we have neglected the possibility of misalignment between the fiber endfaces since the construction of the air gap within a hollow core fiber ensures that misalignments are negligible. Furthermore, the spread of light that takes place in the dielectric coatings has been neglected for two reasons—its overall thickness (< 1 \mu m) is much less than the air gap length, and also the spread of light within the dielectric coating layers is less than that in air since the dielectric materials have refractive indices which match that of the fiber much more closely. The ray optics model has been implemented here for simplicity, and thus it was assumed that the light exits the first fiber from a point instead of from the finite area of the fiber core. Although this model serves the purpose of providing a more intuitive feel for how the gap length of the sensor can affect its performance, it should be noted that a model based on modal and Kirchhoff’s diffraction theory would provide for a better calculation of the spread of light that emerges from the first fiber as well as whether or not the spread light could be accepted by the other fiber.

The coherence length associated with a source can be calculated based on its
spectral characteristics. Since the coherence time, $\tau_{coh}$, can be loosely approximated by the inverse of the effective spectral width of the source, $\Delta v$, we have that\footnote{15}

$$\tau_{coh} \sim \frac{1}{\Delta v} = \frac{\lambda}{v\Delta\lambda} = \frac{\lambda^2}{c\Delta\lambda}.$$  \hspace{1cm} 3.14

where $\Delta v$ is in units of frequency, $v$ is the mean frequency of the source, $\lambda$ is the corresponding mean wavelength, $c$ is the speed of light in a vacuum, and $\Delta\lambda$ is the effective spectral width of the source in units of wavelength. The coherence length, $L_{coh}$, associated with the LED is therefore\footnote{5}

$$L_{coh} = c\tau_{coh} \sim \frac{\lambda^2}{\Delta\lambda}. \hspace{1cm} 3.15$$

If the path length difference, $2z_*$, between two reflections is greater than the coherence length associated with the LED, the two reflections should not interfere. The spectrum of the LED used in our experiments is depicted in Figure 3.11. For our source mean wavelength of approximately 1300 nm and effective spectral width (i.e., the full-width half-maximum value) of about 40 nm, the air gap length, $z_*$, should be greater than

$$z_* \sim \frac{\lambda^2}{2\Delta\lambda} = 21\mu m,$$  \hspace{1cm} 3.16

in order for the reflections from both sides of the air gap not to interfere. It should also be noted that the reflections from the individual thin-film optical layers will interfere, and therefore, operate as an optical filter.
Figure 3.11 Spectrum of the MRED14C015, 1300 ± 20 nm LED made by MRV Technologies, Inc.
4 EXPERIMENTAL PROCEDURES AND RESULTS

In this chapter, we demonstrate the individual measurement of refractive index and strain with the Fresnel reflectometer/EFPI dual-use sensor, and discuss its potential for the measurement of refractive index under embedded conditions.

4.1 MEASUREMENT OF REFRACTIVE INDEX

The first step towards the implementation of the dual-use sensor design for the measurement of the refractive index of epoxy involved incorporating an antireflective thin-film optical filter and evaporating the materials which compose the filter onto the endfaces of optical fibers. An antireflective thin-film optical filter was designed and evaporated onto fiber endfaces by Cascade Optical Coating, Inc. Cascade Optical Coating, Inc. also did a production run to test their optical filter design in which they evaporated a 5-layered coating onto a glass slide. They measured the reflectance from the glass slide to be less than 0.04% for wavelengths ranging from 1240 to 1360 nm. If this reflectance was achieved for the dielectric filters that were evaporated onto the optical fibers by Cascade Optical Coating, Inc., and if the refractive index of the epoxy resin were to change from 1.53 to 1.56, then the normalized change in the output signal would theoretically be larger by a factor of 41 over that observed for a sensor with uncoated fiber endfaces.

Next, the sensing head was constructed by inserting the coated optical fiber endfaces into a hollow-core glass tube so that an air gap was formed. In consideration of the numerical aperture of the optical fiber and the coherence length associated with the light source, the air gap length, $z_a$, should be
between approximately 21 and 32 µm. Three sensors were constructed with air gap lengths of approximately 10, 23, and 31 µm. These sensors are referred to as sensors #1, #2, and #3, respectively. Their gauge lengths were deliberately kept fairly small so that ambient vibrations would not affect the air gap length as much (the gauge lengths were about 5 mm). The far-endface of the reflector fiber was then cleaved several times (for each sensor) while monitoring the amount of reflection to ensure that a good cleave was achieved.

The experimental arrangement and procedure used in this section were similar to that shown in Section 3.1 with two minor exceptions. The multi-functional sensors were fused to leg 3 of the coupler, and leg 4 was wrapped around a post and taped in place to attenuate some of the light so that the power emitted by the LED could be increased without saturating the reference detector. Figure 4.1 depicts this setup. The data obtained from the reference detector was later discarded since the tape that had been used to hold the wound fiber in place was very gradually and consistently losing its grip on the fiber, and thus, the amount of attenuation in leg 4 was steadily decreasing.

To test the multi-functional sensor design, we calibrated the test sensors and submerged the far-endfaces in 3M Scotch-Weld™ DP-100 fast setting epoxy adhesive (the same type used in Chapter 3) during its cure at room temperature. Figure 4.2 depicts the calibration results of the dual-purpose sensors and of the Fresnel reflectometer discussed in Chapter 3. The percentage change in normalized voltage is proportional to the sensitivity of the sensing signal regardless of some experimental parameters such as the amount of amplification or the LED power output. However, other parameters in the experimental setup, such as the amount of reflection from the coupler or any splices must be held constant for all of the experiments in order to compare the data on a fair basis.
Figure 4.1 Experimental setup used to calibrate and test three multi-functional optical fiber sensors.

Figure 4.2 Calibration curves for three dual-use sensors and a cleaved fiber sensor as a function of refractive index.
As expected, the sensors with air gaps are less sensitive due to the added reflections that are not affected by changes in refractive index, and due to the partial air gap loss of the light reflected from the far-endface. In general, larger air gap lengths lead to larger amounts of loss and therefore less sensitivity. However, for air gap lengths that fall within one-half of the source coherence length, interference takes place between the reflections from the air gap. If constructive interference occurs for a particular air gap length, the sensitivity of the sensor to refractive index measurements is decreased since more intensity is subsequently received from the air gap. Likewise, if destructive interference occurs for a particular air gap length, the sensitivity of the sensor to refractive index measurements is increased since less intensity is subsequently received from the air gap. The former explanation is likely the case for the sensor with the 10 μm air gap length, although it is always possible that the cleave on the far-endface was not as good as it could have been. This sensor should also be more susceptible to vibrations since its gap length is well within one-half of the source coherence length.

Figure 4.3 shows the oscilloscope traces (for the experiment using sensor #2) including the sensing (top trace, channel 1) and reference (middle trace, channel 2) voltage signals output from the lock-in amplifiers that were attached to legs two and four of the coupler, as well as the analog voltage signal output from the temperature indicator (bottom trace, channel 3). Although the oscilloscope traces for the experiments involving sensors #1 and 3 are omitted, similar results were achieved.

The refractive index and reaction temperature of all three epoxy cure experiments are depicted in Figures 4.4 through 4.6. As mentioned earlier, the data obtained from the reference detector was discarded since the tape that had been used to hold the wound fiber in place was very gradually losing its grip
Figure 4.3 Oscilloscope traces (for the experiment using sensor #2) of the sensing (top trace, channel 1) and reference (middle trace, channel 2) voltage signals, as well as the analog voltage signal output from the temperature indicator (bottom trace, channel 3).

Figure 4.4 Refractive index and reaction temperature of the epoxy adhesive during its cure (for the experiment using sensor #1).
**Figure 4.5** Refractive index and reaction temperature of the epoxy adhesive during its cure (for the experiment using sensor #2).

**Figure 4.6** Refractive index and reaction temperature of the epoxy adhesive during its cure (for the experiment using sensor #3).
on the fiber. The refractive index data for all three experiments were similar to the results obtained for the Fresnel reflectometer in Section 3.1. Initially, the refractive index of the epoxy decreased since the exothermic reaction caused the temperature of the resin mixture to rise, but as the heat of the reaction was slowly released to the surroundings, the refractive index increased, indicating that crosslinking of the epoxy had led to a slightly denser composition.

An estimate of the resolution for the sensors has been tabulated below. This estimate was calculated as twice the peak-to-peak noise level of the voltage signal times the inverse of the slope of the normalized calibration curve depicted in Figure 4.2. From this curve, it is expected that the sensor with the cleaved endface should have the best resolution, and sensors #2, #3, and #1 should have progressively worse resolutions. Therefore, the sensors are listed in this order in Table 4.1. In addition, sensor #1 should be noisier than sensor #3 because its air gap length is less than one-half the source coherence length.

<table>
<thead>
<tr>
<th>Sensor</th>
<th>Inverted Slope of Calibration Curve ((V^{-1}))</th>
<th>Peak-to-Peak Voltage of Noise ((mV))</th>
<th>Estimation of Resolution</th>
</tr>
</thead>
<tbody>
<tr>
<td>cleaved endface</td>
<td>0.0345</td>
<td>66.0</td>
<td>0.0046</td>
</tr>
<tr>
<td>~23 (\mu m) gap length</td>
<td>0.2778</td>
<td>26.2</td>
<td>0.0174</td>
</tr>
<tr>
<td>(sensor #2)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>~31 (\mu m) gap length</td>
<td>0.4255</td>
<td>34.0</td>
<td>0.0146</td>
</tr>
<tr>
<td>(sensor #3)*</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>~10 (\mu m) gap length</td>
<td>0.3390</td>
<td>25.6</td>
<td>0.0289</td>
</tr>
<tr>
<td>(sensor #1)</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

* This sensor signal was amplified more than the other three before being filtered.
The results of the experimentally measured resolution are in agreement with the calibration data for all of the sensors except the one with an air gap length of 31 \( \mu \text{m} \). However, this sensor signal had been amplified more than the other three before being filtered. The calculated resolutions are 15, 58, 49, and 96% of the expected change in refractive index (which is about 0.03) due to cure. It is expected that the noise level would be reduced considerably if the time constant on the lock-in amplifier were increased from 300 ms to a few seconds since the measurement of cure takes place over relatively long periods of time. Some variation in measurement is expected (as discussed in Chapter 1) and is one reason for developing cure monitors.

4.2 STRAIN MEASUREMENT

The arrangement used to test the Fresnel reflectometer/EFPI for the measurement of strain is depicted in Figure 4.7. This setup is similar to that shown in Figure 2.5 except that the sensor was bonded to a beam with Miller and Stevenson epoxy. A resistive strain gauge was also attached to the same region of the beam so that the strain measurements of the two sensors could be compared. The gauge length of the fiber optic sensor with a 23 \( \mu \text{m} \) air gap length was measured to be approximately 13.5 mm.

According to Equation 2.21, changes in the air gap length of the multi-functional sensor lead to changes in the phase difference between the reference and sensing reflections. This change, in turn, sinusoidally modulates the intensity of the two main interfering light waves at the detected output, which is measured in terms of voltage. Experimentally, we also receive other back-reflections, such as those from the coupler or the fiber endface that is connected to the sensing photodetector. Therefore, Equation 2.21 has been altered in order to describe the experimental signal. Therefore, the voltage signal, \( V \),
Figure 4.7 Experimental arrangement used to test the Fresnel reflectometer/EFPI for the measurement of strain.

Varies with respect to the air gap length, $z_a$, according to the equation

$$V = V_{DC} - V_o \cos \left(\frac{4\pi z_a}{\lambda}\right). \tag{4.1}$$

Here, $V_{DC}$ stands for the DC offset, $V_o$ is equal to one-half of the peak-to-peak amplitude of the sinusoidal variation, and $\lambda$ is the central wavelength of the laser source. Figure 4.8 depicts this theoretical signal in more detail.

Solving for $z_a$, we obtain

$$z_a = \frac{\lambda}{4\pi} \cos^{-1}\left(\frac{V_{DC} - V}{V_o}\right). \tag{4.2}$$

Hence, a change in air gap length is given by

$$\Delta z_a = z_{a2} - z_{a1}. \tag{4.3}$$

Equation 4.1 also shows that each of the periodic variations in the received signal corresponds to a change in the length of the air gap that is equal to one-half of the source wavelength. So, by approximating each 1/2-period of the modulated signal as a linear function, and by multiplying the fraction of the 1/2-period by $\lambda/4$, we can estimate the change in the air gap length. Once the
Figure 4.8 The expected experimentally detected EFPI signal as a function of the air gap length.

change in the air gap length has been calculated, one can calculate the strain present in a specific length of material. The strain, $\varepsilon$, is given by

$$\varepsilon = \frac{\Delta z_a}{GL},$$  

where GL represents the gauge length of the sensor.

A micrometer was used to deflect the beam as much as possible. The micrometer was then released in small increments and the subsequent sinusoidal signal from the photodetector was captured by an oscilloscope. This trace is shown in Figure 4.9. The strain measured with the Fresnel reflectometer/EFPI sensor versus the amount of strain measured with the resistive strain gauge is depicted in Figure 4.10. The strain measurement obtained from the fiber optic sensor was fairly close to that of the resistive strain gauge. Only about 93.5% of the strain imparted by the deflected beam was transferred to the hollow-core region of the fiber optic sensor, i.e., the region of the sensor in which strain causes the air gap to be displaced. In other
Figure 4.9 Oscilloscope trace of the voltage signal as strain was released in increments of about 20 μstrain.

Figure 4.10 Strain measured with the Fresnel reflectometer/EFPI sensor versus the amount of strain measured with the resistive strain gauge.
words, part of the strain was transferred to the optical fiber outside of the hollow-core tube and not to the tube. The strain resolution for the sensor was calculated to be 2.2 με.

4.3 THE POTENTIAL FOR THE MEASUREMENT OF REFRACTIVE INDEX UNDER EMBEDDED CONDITIONS

In this section, we discuss the potential of measuring refractive index with the dual-use sensor when the glass tube is embedded. The experimental arrangement and procedure used to test the sensor under these conditions were the same as described in Section 4.1.

Figures 4.11 and 4.12 show the oscilloscope traces that resulted from this test. As the temperature of the epoxy mixture increased due to the exothermic reaction, the hollow-core glass tube expanded. Thus, the air gap length of the sensor increased, which led to a decrease in the amount of reflected light accepted back into the input/output fiber of the sensor, and subsequently, a decrease in the voltage sensing signal. When the release of heat ceased, the temperature of the curing epoxy began to decrease, leading to an increase in the voltage signal. At some point during this signal increase, however, the epoxy had also become tacky enough to contract the hollow-core tube as it densified. Thus, the air gap length decreased to within one-half of the source coherence length, and the signal exhibited the sinusoidal pattern associated with gap displacement that was discussed in Section 4.2. The author counted approximately 15 fringes. Unfortunately, the voltage signal was not on the screen of the oscilloscope for the remainder of the test, and no further information was gathered. Upon speculation, the chances of this happening again for this sensor are very high since the fringe contrast was so much larger than the amount of signal change that is expected due to a change in refractive index.
Figure 4.11 Oscilloscope traces of the sensing (bottom trace, channel 1) and reference (top trace, channel 2) voltage signals, as well as the analog voltage signal output from the temperature indicator (middle trace, channel 3). Note that the signal contrast, $V$, which is defined as:

$$V = \frac{2(I_1 I_2)^{1/2}}{I_1 + I_2} |g_{12}|$$  \hspace{1cm} (4.5)

increased (i.e., the amplitude of the fringes increased). Here, $I_1$ and $I_2$ are the intensities of the two interfering light waves, and $g_{12}$ is the normalized cross-correlation between the two light waves. Since $I_2$ increases (due to less resultant loss in the air gap) as the air gap length is decreased, $I_1$ becomes more equal to $I_2$ causing the signal contrast to increase. This occurrence further indicates that the air gap length was decreasing during the time that the fringes were observed.
Time (0.1 ks/div)

Figure 4.12 Expanded view of Figure 4.11.
5 FUTURE DIRECTIONS AND CONCLUSIONS

5.1 FUTURE WORK

The refractive index of a curing polymer is modified due to both cure and temperature. Therefore, future experiments testing the abilities of the Fresnel reflectometer/EFPI to monitor cure should be implemented under isothermal conditions and should incorporate a DEA analysis of cure extent for comparison purposes.

Future studies of this multi-functional sensor should involve several modifications to the sensor design. First, the initial air gap length should be larger (about 40 to 50 μm) so that when it decreases during the cure test, it will not decrease to within one-half of the source coherence length. Secondly, the hollow-core glass tube should be encapsulated in some epoxy before the cure test. This modification would ensure that, during the cure test, the air gap length would only be changed as a result of the temperature-induced expansion of the hollow-core tube or epoxy, and not the contraction of the tube as the tacky epoxy densifies.

With the aforementioned modifications in mind, the main weakness of the sensor for cure monitoring would be that the signal would not be large enough and would not change very much as the refractive index of the curing epoxy changes by such a small amount. To overcome this weakness, another improvement to the design of the dual-use sensor should involve depositing a thin-film optical filter onto the far-endface of the sensor. If the filter is properly designed, it would increase the reflectivity and the change in reflectivity with
respect to the refractive index and the change in refractive index, respectively, that occurs during the cure of polymers. It is also anticipated that with coatings composed of many dielectric layers, the resultant broadband filter would not be as sensitive to temperature.

A fourth design alteration would involve polishing the far-endface at an angle. In this way, two polarizations (s and p) would be created with respect to the plane of incidence, which is defined as the plane formed by a vector normal to the reflecting interface and the vector in the direction of incidence. The ratio of the two independent polarization intensities could then be monitored, so source fluctuations would not affect the test results. Furthermore, certain angles would cause the light to be reflected a number of times, and yet follow the same path back into the core of the fiber in the opposite direction.

Once the investigation of the latter two concepts is complete, the feasibility of designing a novel, phase-modulated sensor with respect to the refractive index of the curing polymer should be contemplated. Two independent phase-modulated signals would be created by angling and coating the far-endface of the sensor.

5.2 CONCLUSIONS

Our motivation for this work is based on the need to monitor the cure and in-service health of composite materials. We have reviewed several conventional designs for optical fiber sensing, and have designed a novel, multi-functional sensor with the potential to monitor the state of a composite part “from cradle to grave.”
Furthermore, we have demonstrated the individual measurement of refractive index and strain with the Fresnel reflectometer/EFPI, and have tested it as an embedded refractive index monitor. Our experimental results demonstrate that the refractive index of polymers increases by approximately 2% during the cure process. In addition, the sensor can be used as an interferometer to measure internal composite strains, where the phase difference between consecutive fringe peaks is one-half the wavelength of the source.
REFERENCES


VITA

Mary Kathleen Burford was born in Lynchburg, Virginia on December 10, 1969. She received a Bachelor of Science degree in May of 1993 and completed her Master of Science degree in August of 1996, both from the Bradley Department of Electrical Engineering at Virginia Polytechnic Institute & State University. While in the masters program, she worked at the Fiber & Electro-Optics Research Center under the supervision of Dr. Richard O. Claus. Her research interests include fiber optics and optical design.

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