Fiber Optic Sensors for Cure/Health Monitoring of Composite Materials

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ABSTRACT

The objective of the current program is to develop techniques for using optical fibers to monitor the cure of composite materials in real time during manufacture and to monitor the in-service structural health of composite structures.

Single and multimode optical fibers containing Bragg gratings have been used to perform Near Infrared (NIR) spectroscopy on high refractive index resins and show promise as embedded sensors. In order for chemical spectroscopy to be possible, intimate contact must be achieved between the fiber core and the composite resin. This contact is often achieved by stripping the cladding off of a portion of the fiber, thus making it brittle and easily broken in the composite processing environment. To avoid weakening the fiber to this extent, high refractive index fibers have been fabricated that use a low refractive index acrylate coating which serves as the cladding. This is ideal, as the coating is easily solvent stripped and intimate contact with the glass core can be achieved. Real time resin and composite chemical spectra have been obtained, with possible multifunctional capability using Bragg gratings to assess physical properties such as strain, modulus and other parameters of interest.

Key Words: Fiber optic sensors, Bragg gratings, strain, chemical sensors, composites

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INTRODUCTION

The use of optical fibers for chemical sensing has been extensively reported [1-5]. In order for chemical sensing to take place, intimate contact must be achieved between the fiber core and the material of interest. Using a variety of configurations, most commercially available optical fiber probes contain at least one source and several collection fibers [6-8]. These probes, in general, have the disadvantage of large size and thus cannot be embedded into composite structures without having deleterious effects on their physical properties. Embedded fiber optic sensors that have portions of cladding stripped to achieve intimate contact have also been reported [9-11]. These fibers have the disadvantage of being extremely fragile and are unlikely to survive the high temperatures and pressures associated with composite processing. In addition to these considerations, high performance resins generally have refractive indices (n) greater than that of standard silica glass fibers. In order for light propagation to take place the index of refraction of the core, must be lower than the index of refraction of the material with which it makes contact, following the relationship as shown in Figure 1.

![Figure 1. Light propagation through embedded optical fiber: n₁ > n₂ > n₃](image)

The current research uses novel high refractive index fibers for Near Infrared (NIR) spectroscopy with a geometry that allows intimate contact to be obtained without greatly reducing fiber strength while maximizing light throughput. The goal of this work is to use these fibers not only for chemical spectroscopy, but ultimately for physical property measurements as well, using Bragg gratings written into the fiber. Bragg gratings respond to strain by a shift in wavelength that can be correlated to the amount of strain the sensor experiences. The work proving this multifunctional sensor concept has been reported elsewhere [12, 13]. Using multiple Bragg gratings along a single fiber that can be distributed from 1 cm. to meters apart affords a sensor system that can be tailored to suit many applications and configurations. These same fibers then can also be used as a chemical health monitor capable of sensing such aging effects as oxidation and water absorption and subsequently determining their effects on physical properties such as strength and modulus, all with a single fiber sensor.
EXPERIMENTAL

Several high refractive index glass preforms were obtained by Luna Innovations, Blacksburg, VA. Optical fiber was drawn with a diameter of 107 microns. The fiber was coated with a clear fluorothermoplastic coating, Dyneon 200A. The total fiber diameter, with the coating, was 125 microns. The thermoplastic coating has a refractive index of 1.355 and is easily soluble in ketones, esters and ethers. In this case, the fluoropolymer coating acted as the cladding. Because its refractive index is so low compared to the glass, the numerical aperture of the fiber was quite high, thus reducing bend loss. The refractive index of the resin of interest was approximately 1.8. The refractive index versus wavelength curves for several fibers are given in Figure 2. This paper will report on the results obtained using the F2 and SF6 glasses. The SFL6 was also drawn into fiber, but was too brittle to be used.

![Figure 2. Refractive index versus Wavelength](image)

NIR spectroscopy of both high performance epoxy resin and composites was performed using a Nicolet Nexus 850 spectrometer with a CaF₂ beamsplitter and a Bio-Rad InSb detector. Fiber was prepared by chemically stripping the ends
and an approximately 2 cm. sensing region on the fiber using acetone, where intimate contact was achieved between the fiber and the resin. The fiber was coupled to the spectrometer using FC bare fiber connectors. Resin samples were heated using a Fisher-Johns melting point apparatus. Composite samples, 8 – 10 plies thick, were laid up in stainless steel molds and cured in a press using a standard epoxy cure cycle.

An attempt was made to write Bragg gratings in the SF6 glass, but was unsuccessful due to the limited strength of the fiber. Work is ongoing to obtain more F2 glass fiber, which has much better physical properties.

RESULTS AND DISCUSSION

Fiber optic chemical spectra were obtained using single mode silica fiber containing Bragg gratings. The purpose of this pursuit was to develop a multifunctional embedded sensor. The initial spectrum is shown in Figure 3, using a single mode fiber containing approximately 30 Bragg gratings. While this work was used to prove the concept that the grating do not interfere with spectroscopy, the spectral quality was too poor to be useful. Thus, a different approach was taken to maximize the amount of energy transmitted along the fiber and that allowed evanescent coupling to take place. Lead doped and other high refractive index specialty fibers were obtained from Luna Innovations and examined to determine their ability to achieve good spectral quality and their suitability for writing Bragg gratings.

Figure 3. Single mode fiber optic spectra of epoxy with and without Bragg gratings
High index fibers were examined spectroscopically to determine their transmission characteristics. White light was used as a source and coupled into each of the fibers. Transmission spectra for the F2 and SF6 fibers are shown in Figure 4. The transmission properties of the F2 fiber were considerably greater than for the SF6, in spite of the fact that they had similar geometries, however, the SF6 had a slightly higher cutoff. The single beam intensity spectrum for the SF6 also exhibits more noise.

![Figure 4. Single beam spectra of F2 and SF6 optical fibers (note different scale)](image)

Real time NIR spectra were obtained during the cure of a high performance epoxy resin using the F2 fiber, as shown in Figure 5. The progress of the cure can be monitored using the absorption peak at 1.97 microns that corresponds to the disappearance of the primary amine. As can be seen, the spectral quality is very good. However, this was not found to be the case for the corresponding epoxy composites. The spectra for these samples are quite noisy, although still show some of the salient peaks as seen in Figure 6. For this reason, it was determined to use a higher index fiber, namely the SF6, to aid light propagation and enable a larger sampling area to be stripped.
Figure 5. Real time cure spectra of high performance epoxy resin
In the case of the F2 fiber, the index of refraction of the resin was greater than that for the fiber. With the SF6, the indices between fiber and resin were comparable. This should have resulted in higher quality spectra, however, it did not appear that evanescent coupling took place between the fiber and the resin at all. Figure 7 shows single beam spectra for the fiber and the fiber with the sensing region immersed in resin. As seen, the identical shape indicates that the only spectrum obtained is that of the fiber itself. This phenomenon could be due to complete internal reflection or the chemical composition of the glass itself. Fibers containing metal oxides often are not able to evanescently couple to other materials.
In addition to chemical spectroscopy, optical fibers containing Bragg gratings were embedded into composites and tensile tests were performed. The coupons had strain gages attached to compare with strain data obtained from the fibers. Figure 8 shows excellent correlation between data. This shows data to relatively high strain levels and ultimately, the fiber broke in the grips.

Figure 7. Background single beam spectrum of fiber compared to single beam spectrum of fiber in epoxy resin
CONCLUSION

Chemical spectra were obtained using single mode fibers containing Bragg gratings and high refractive index specialty fiber with a unique geometry. Fibers containing Bragg gratings were embedded in composite coupons and showed excellent strain correlation with strain gage measurements when loaded to high strain levels. Work continues on writing Bragg gratings into large diameter, high index cores to make further strain measurements. Future work will include reading out multimode fiber gratings and improving spectral quality for composites.

REFERENCES