ELSEVIER

Optics and Lasers in Engineering 35 (2001) 91–104

OPTICS and LASERS in ENGINEERING

Fiber optic flow and cure sensing for liquid composite molding

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Received 3 November 2000; accepted 8 December 2000

Abstract

The Polymer Composites group at the National Institute of Standards and Technology has efforts in both on-line flow and cure sensing for liquid composite molding. For our flow program, a novel fiber optic real-time sensor system has been developed that can sense resin at various locations on a single fiber using long-period gratings and a polychromatic source. The sensor operation and characterization will be discussed along with sensor performance during mold filling with various types of reinforcement. The cure sensing program focuses on the interface-sensitive fluorescence response of a dye molecule grafted to a high-index glass fiber. The fluorescence emission of the fluorophore undergoes a blue shift as the resin cures. The fluorescence sensor is made by grafting a silane functional fluorophore onto the surface of the glass with close attention to layer thickness. Fluorescence emission of the grafted fluorophore film is shown to be sensitive to epoxy resin cure, co-silane, and layer thickness. The response of the grafted fluorophore to cure on a high-index fiber is demonstrated. Published by Elsevier Science Ltd.

Keywords: Sensor; Flow; Long-period gratings; Cure; Fluorescence; Fiber optic; Liquid composite molding

1. Introduction

The major advantages of polymer matrix composite products include a high strength-to-weight ratio, corrosion resistance, and thermal and electrical insulating

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0143-8166/01/$ - see front matter Published by Elsevier Science Ltd.
PII: S0143-8166(00)00110-X

properties. However, the cost of polymer matrix composites must be reduced for composites to be competitive against traditional materials such as wood and metal in consumer markets. One strategy is to decrease the cost of manufacturing the composite through process optimization and reduction of scrap. The introduction of fiber optic sensors into the liquid composite molding (LCM) process for flow and cure monitoring provides a non-invasive means of obtaining real time information on mold filling and resin cure for control decisions.

In LCM, the fiber preforms are placed in a steel mold, which is then closed. Resin is injected to saturate the empty pores in the preforms. However, under these typical manufacturing circumstances, it is difficult to detect if the resin is saturating the preform. Embedded sensors can help monitor the impregnation process and many such developments have been attempted [1–4]. For the first time, long period gratings (LPGs) will be used as embedded flow monitoring sensors in LCM to detect the arrival of resin in the mold during filling. LPGs are similar in nature to Bragg gratings, but function differently because they are used to couple light out of the core. LPGs have a grating periodicity of typically 130–150 μm. When the higher index resin covers a LPG sensor, the light that would have been attenuated gets coupled back into the core and, hence, there is no corresponding dip in the transmission spectrum. Resin flow-front information is very useful to composite manufacturers because it alerts them to specific problems that occur during resin injection.

Although the many existing mold-filling simulation packages [5–7] have contributed tremendous insight and understanding of this process, the design optimizations based on them have traditionally been conducted off-line. This poses a difficulty when unanticipated anomalies are encountered during manufacturing. Moreover, the variations in the processing parameters are too numerous to be accounted for in these simulations. A more effective approach would be to monitor the progression of the mold filling as it occurs and implement appropriate corrective measures as necessary. We believe LPGs have the potential to serve in such a sensory capacity.

LPGs possess the usual advantages associated with fiber optic systems, for instance small size and light weight, which allows them to be embedded without compromising the structural integrity of the composite. Furthermore, their inherent immunity to electromagnetic interference gives these systems a clear edge over other flow-sensing systems that are based on the resin dielectric properties [8,9]. Moreover, LPG sensors have an additional advantage over other intensity-based fiber optic systems [1] in that they are robust to fluctuations in light amplitude due to unquantified connection losses and fluctuating light sources [2–4]. As each LPG sensor is uniquely wavelength encoded, several of them can be multiplexed on a single strand of fiber, thereby minimizing the degree of additional heterogeneity introduced into the process.

Grafting a fluorophore to a glass fiber allows a cure sensor that is also interface sensitive to be fabricated. Fluorescent probe molecules have been used to monitor many properties of polymer and composite materials. For example, dimethylaminonitrostilbene (DMANS) was used to measure the glass transition of a cured epoxy resin and to study the physical aging of the resin below the glass transition [10]. Fluorescence monitoring has also been used to study water sorption and diffusion in polymers [11], polymer reaction kinetics [12], and the onset of

gelation during cure of thermosetting resins [13,14]. Some groups are trying to develop practical sensor devices by combining fiber optic technology with fluorescence probe molecules. For example, after doping the resin with trace levels of a fluorescent dye molecule, both distal and evanescent mode fiber optic fluorescence methods have been used to measure the cure of epoxy [14–18] and polyurethane resins [14]. In one study, the position of the fluorescence maximum from the dye shifted during the resin cure [14]. In another work, the intensity changes from a fluorescing dye were monitored, via fiber optics, to follow cure of an epoxy resin system [18].

These and many fluorescence methods for composite monitoring involve dissolving the fluorophore directly into the bulk resin. This can create problems in a manufacturing environment. First, an extra processing step may be required to mix the dye into the resin. Second, a small concentration of dye can drastically change the resin color, which could be undesirable. Finally, since the dye is dissolved in the bulk resin, the fluorescence response comes from the bulk, and the behavior of the fiber/resin interface is ignored. Even if evanescent wave fiber optics are employed to focus on the interface, the evanescent wave may still propagate a micrometer or more into the resin, and much of the sensor response will be from the bulk resin [19].

Immobilizing a fluorescent dye onto fiber optics has been used to make a variety of sensors [20] including oxygen, glucose [21], and calcium [22] sensors. In order to make a sensor that is sensitive to the composite glass/resin interface, a fluorescently labeled silane-coupling agent (FLSCA) has been chemically grafted to glass surfaces. FLSCA is a molecule that contains the DMANS fluorophore tethered to a triethoxy silane-coupling agent. When the fluorophore is covalently immobilized at the glass surface, the fluorescence response of FLSCA can be used to directly study the glass/resin interface. In order to graft the fluorophore to the glass, the ethoxy functionalities on the silane end of FLSCA must be converted to hydroxyl groups. These hydroxyl groups can then condense with hydroxyl groups already present on the glass surface, forming covalent siloxane bonds.

This paper focuses on the development and implementation of both the fiber optic flow and cure sensors. In addition, external factors such as preform architecture and deformation, co-silane, and layer thickness on the response of the sensors are presented. By studying the effects of these external factors, sensor design and function can be optimized.

2. Experimental

2.1. Flow sensing

The LPGs were prepared by F&S Optics (Blacksburg, VA)[1] on communication-grade optical fiber with a cladding diameter of 125 μm. Each strand of fiber used had

[1] Identification of a commercial product is made only to facilitate experimental reproducibility and to adequately describe experimental procedure. In no case does it imply endorsement by NIST or imply that it is necessarily the best product for the experimental procedure.

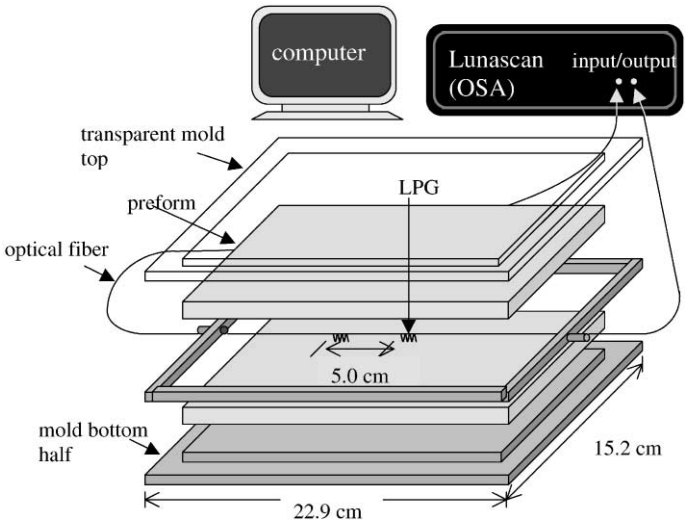

Fig. 1. Schematic of mold and optical fiber assembly and experimental setup.

two, 2.0 cm LPGs spaced 5.0 cm apart. The core refractive index is 1.4515 while the cladding refractive index is 1.444.

These optical fibers are sandwiched between equal numbers of layers of fiber glass preforms and placed inside a flat plaque mold $1.0\,\text{cm} \times 15.2\,\text{cm} \times 22.9\,\text{cm}$ with a transparent top as shown in Fig. 1. The resin was injected from the same location as the optical fiber's point of entry into the mold. A corn syrup–water mixture was used as the simulated resin having viscosities ranging from 0.3 to 0.6 Pa s. The refractive index of the simulated resin was higher than that of the cladding refractive index.

Both ends of the optical fiber are connected to the input–output ports of the Lunascan device using bare fiber adapters. The device is basically an optical spectrum analyzer (OSA) and light source. One port emits light at 1550 nm while the other collects and analyzes the optical spectrum transmitted through the fiber. The spectral output is displayed on a computer and only the spectrum of light between 1495 and 1585 nm is viewable using this system. Approximately three scans are taken by the system every second. Every spectrum that is recorded is the mean of several consecutive scans logged by the OSA. Averaging the scans ensures that any inherent fluctuations in the system are accounted for.

A spectrum from the optical fiber is recorded before the fiber is placed inside the stack of preforms. It is also logged after the mold is closed, before the liquid is injected into the mold, and at various other times during the mold-filling process. This procedure was done for random fiber mats, plain bi-directional weave and two cases of unidirectional fibers. In one case, the reinforcing fibers are parallel to the optical fiber alignment and transverse to it in the other case. Three fiber volume fractions were used for each reinforcing architecture.

2.2. Cure sensing

The structure of FLSCA is shown in Fig. 2. Synthesis of the FLSCA dye and the ethanol-based deposition procedure used to deposit FLSCA with the diluting–coupling agent on the glass surface are described in a previous paper [23]. The diluting–coupling agents used in this work are glycidoxypropyl trimethoxy silane (GPS), isocyanatopropyl triethoxy silane (IPS), aminopropyl triethoxy silane (APS), propyltriethoxysilane (PTS), and octadecyltriethoxysilane (OTS). After coating glass fibers and glass microscope cover slips with a silane layer, they were immersed in epoxy resin. Fluorescence from the grafted FLSCA was measured during the resin cure. The resin system used in this study was a stoichiometric mixture of diglycidyl ether of bisphenol A (DGEBA Tactix 123, Dow Chemical Company) with the amine hardener poly(propylene glycol) bis 2-amino propyl ether (Jeffamine D400, Aldrich Chemical Company) [23].

Fluorescence measurements made on the glass microscope cover slips were taken on a Spex Fluorolog Fluorimeter (Edison, NJ), using 460 nm excitation as described in a previous paper [23]. Fluorescence measurements made on the glass fiber optic were taken from a laser-based fiber optic fluorimeter described in the literature [14]. In this case, the excitation was from an argon ion laser at 488 nm. Unless stated otherwise, the data uncertainty is given as a standard deviation from measurements made on 8 or more samples.

3. Results and discussion

3.1. Flow sensing

The OSA system proved to be a fairly stable system since there was very little fluctuation (less than 2%) in the scans obtained for each case. Fig. 3a shows a typical

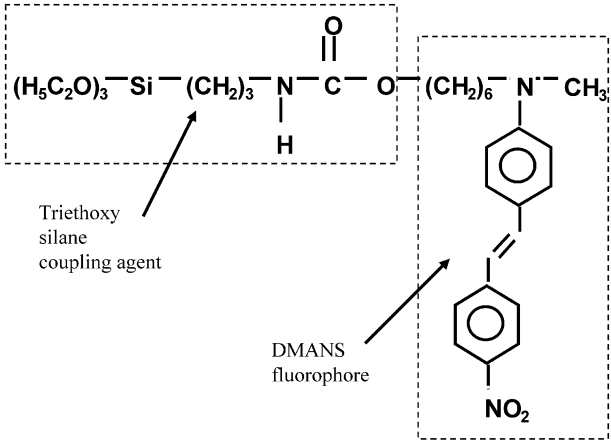

Fig. 2. The dimethylaminonitrostilbene fluorophore is tethered to a triethoxy silane coupling agent, giving the FLSCA molecule shown here. This entire molecule is then grafted to the glass surface, immobilizing the FLSCA molecule at the interface.

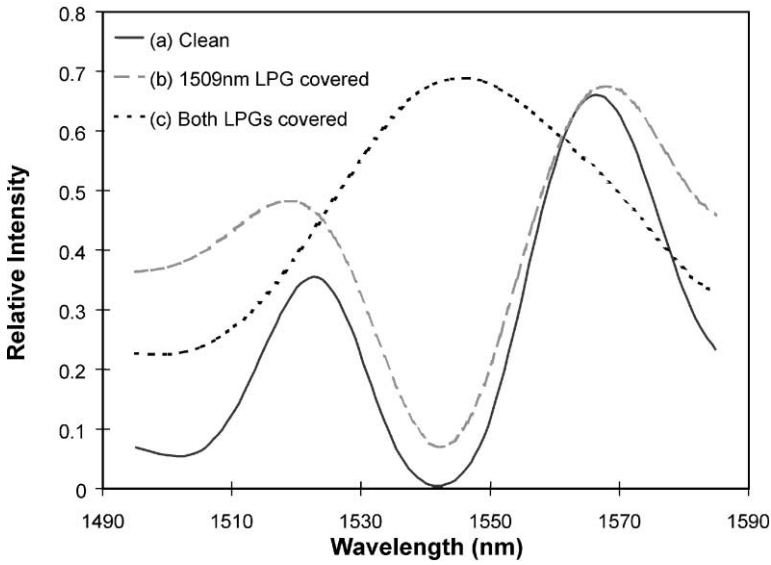

Fig. 3. Spectrum of an optical fiber containing two LPGs written at 1509 and 1549 nm in (a) its clean state, (b) when the 1509 nm LPG is covered by simulated resin and (c) when both LPGs are covered by the simulated resin.

spectrum of an optical fiber with two LPGs written on it. The vertical axis is relative intensity with respect to the maximum intensity detected by the system for that particular scan. In this case, the LPGs were written such that light at 1509 and 1549 nm would be filtered out, as evident from the transmission dips seen in that figure. These dips disappear when the simulated resin covers one sensor (Fig. 3b), coupling the light at 1509 nm back into the core-propagating mode. When both LPGs are covered (Fig. 3c), the spectrum is like that of an optical fiber without any LPGs written on it. Since each of the LPGs is written at distinctly different wavelengths, it is possible to identify where and when the sensors are covered.

As with any optical fiber system, the LPGs are extremely sensitive to microbending when they are compressed between the preform layers inside the mold as can be seen in Fig. 4. It was observed that signal deterioration worsened at higher fiber volume fractions and it was noticeably worst in the random mat case. There was the leastsignal deterioration with the parallel unidirectional reinforcements. Both the transverse unidirectional mat and bi-directional weave cases are between these two extremes, with the signals from the bi-directional weave being slightly more affected than the other.

It is possible that this deterioration in signal is due to a combination of effects. Not only is there excessive microbending on the optical fibers when it is compressed in the midst of the preform stack but the LPGs are also in intimate contact with the fiber glass reinforcement which has a similar refractive index as the buffer and cladding. Moreover, it is likely that compressive stresses are induced in the optical fiber itself.

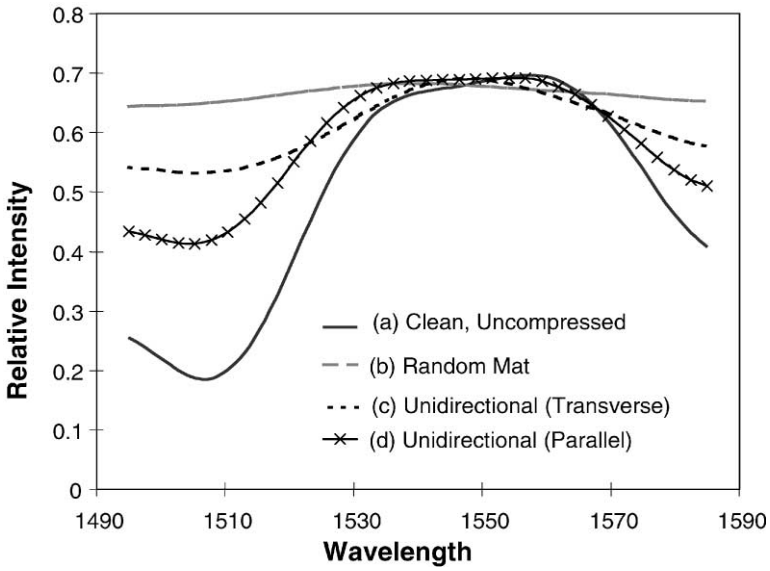

Fig. 4. Spectrum of an optical fiber containing two LPGs written at 1509 and 1549 nm in (a) its clean, uncompressed state, compressed between (b) random mats ($v_f = 17\%$), (c) unidirectional mats with fiber transversely oriented with respect to the optical fiber ($v_f = 60\%$) and (d) unidirectional mats with fibers aligned parallel to the optical fiber ($v_f = 60\%$).

The greatest challenge in these experiments was to retain the integrity of the sensor signals. To overcome this issue, a hollow glass sleeve was placed over the sensors. These protective sleeves have an inner diameter of 445 μm and an outer diameter of 650 μm. They are 4 cm long each, long enough to cover the entire length of the LPG and allow for some slight translational movement along the optical fiber during placement in the mold. These glass sleeves are not permanently fixed onto optical fiber so that liquid may flow inside them and reach the LPGs.

As can be seen from Fig. 5 there is a slight difference between the signals from a LPG compressed inside a stack of random mats but covered by such a sleeve and that of an uncompressed LPG. However, this is a vast improvement over the signals obtained when the sensor is not protected by the glass sleeve and is subjected to the same conditions inside the mold. When the simulated resin is injected into the mold, the dips disappear, as expected, after the simulated resin completely covers each LPG as is shown in Fig. 6. However, there is a delay in the resin reaching the LPG from propagating through the hollow core tube. The hollow core tube is clearly not required for every preform architecture and volume fraction and is only an interim solution until another solution can be found to stiffen the LPG without affecting the resin flow.

3.2. Cure sensing

The results shown in Fig. 7 demonstrate the potential to use the grafted FLSCA dye as an interface-sensitive cure monitoring sensor. Both a fluorescence intensity change

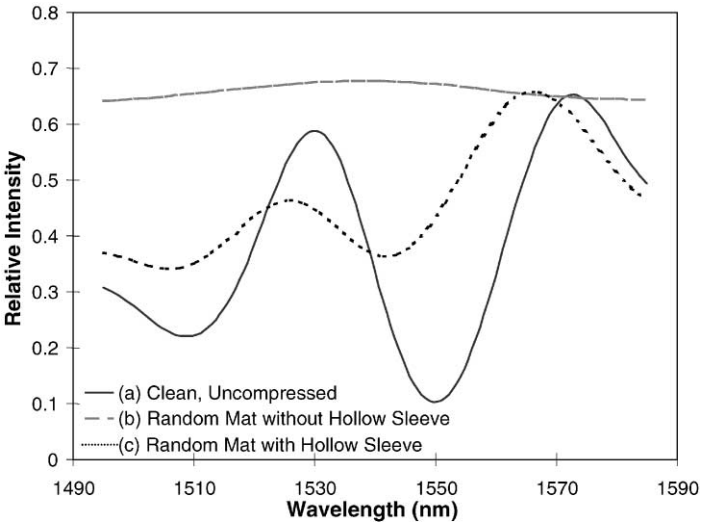

Fig. 5. Spectrum of an optical fiber containing two LPGs written at 1508 and 1549 nm in (a) its clean, uncompressed state, when it is compressed in between (b) random mats ($v_f = 17\%$) without any protective sleeves and (c) random mats ($v_f = 17\%$) with the protective sleeves.

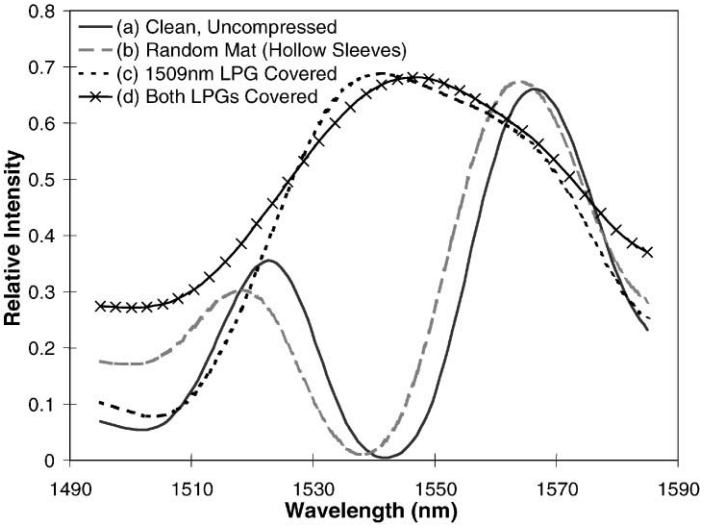

Fig. 6. Spectrum of an optical fiber containing two LPGs written at 1508 and 1549 nm in (a) its clean, uncompressed state, (b) when it is compressed inside the mold with the hollow sleeves in place, when the simulated resin in injected into the mold and covers (c) the 1508 nm LPG and (d) both LPGs.

and a blue spectral shift from the grafted FLSCA can be followed when an epoxy resin cures over the mixed coupling agent layer. For Fig. 7, FLSCA was diluted with the epoxy functional coupling agent, GPS. This mixed silane layer was then grafted to the

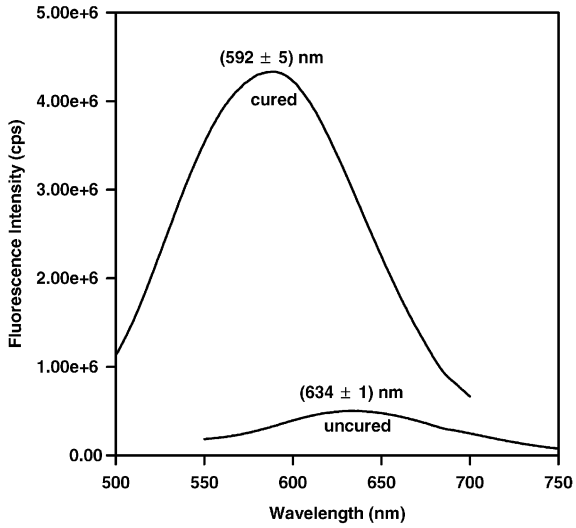

Fig. 7. A blue shift is observed from the grafted FLSCA dye during cure of an epoxy over-layer.

glass microscope cover slip using the ethanol/water based deposition procedure. Scanning electron microscopy showed that the layer thickness was approximately $(0.8 \pm 0.3)\,\mu m$. When the coated cover slip is immersed in uncured epoxy resin, the FLSCA fluoresces with maximum intensity, λ_m, at a wavelength of $(634 \pm 1)\,nm$. While still immersed in the uncured resin, the same cover slip was then put in the oven for 4 h at 100°C to allow the epoxy resin to cure over the GPS/FLSCA grafted layer. The sample was removed from the oven and cooled to room temperature. When fluorescence from this layer was measured in the cured epoxy, λ_m had shifted to $(592 \pm 5)\,nm$. The uncertainties given are standard deviations. This 53 nm blue shift in fluorescence, during the resin cure, gives the grafted dye technique potential to monitor a composite fiber/resin interface during processing. But to make a practical sensor, the magnitude of this spectral shift must be related to structural or mechanical changes in the interphase properties during cure.

To study how the chemical nature of the coupling agent layer affects the FLSCA fluorescence, the probe was diluted with OTS, PTS, IPS, or GPS. These mixed coupling agent solutions were then grafted to glass microscope cover slips. Fig. 8 shows a typical fluorescence emission spectrum from these grafted layers. The control slides showed no fluorescence, and had a background intensity less than 10% of the maximum intensity for the corresponding layer that contained the FLSCA dye. This background is probably due to small amounts of stray light that was reflected or scattered off the control sample into the detector. The background was not subtracted from the spectra shown in Fig. 8 and had no effect on the position of the fluorescence maximum. Because these grafted layers were not immersed in a resin over-layer, the fluorescence response from the immobilized FLSCA is due only to the presence of the diluting coupling agent. When FLSCA is diluted with PTS or OTS, the fluorescence

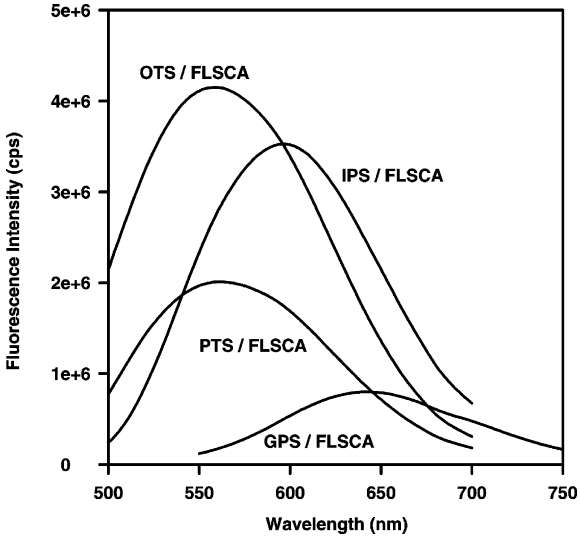

Fig. 8. Grafted FLSCA fluorescence when diluted with various silane coupling agents.

maximum, λ_{max}, is near 560 nm. Because the organic functionality of both PTS and OTS is an alkane chain, the interactions between the dye and coupling agents are similar in these layers, and the position of λ_{max} occurs near the same wavelength. But when the organic functionality of the diluting coupling agent is changed to the more polar isocyanatopropyl group (IPS), the fluorescence emission occurs at longer wavelengths and has a λ_{max} of 593 nm. Even longer wavelength fluorescence is observed for the GPS/FLSCA mixed layer, which has a λ_{max} of 642 nm. This figure shows that the chemical functionality surrounding the FLSCA fluorophore has a significant effect on the position of the fluorescence emission from the grafted layer. As the polarity of the diluting coupling agent increases, the emission from the FLSCA shifts towards longer wavelengths. The intensity differences between these curves could be due to many factors besides the chemical functionality of the diluting coupling agent. For example, thickness variations between the samples or dye surface concentration differences between the layers could also lead to the intensity variations observed in Fig. 8. In addition, the quantum yield of the dye is dependent on the micro-viscosity of the surrounding coupling agent layer. Structural differences between the layers could cause differences in the quantum yield of the grafted FLSCA and lead to intensity variations.

The thickness of the mixed silane layer can also affect the response of the FLSCA molecule. Fig. 9 shows the fluorescence spectrum of a thick ($\sim 10\,\mu m$ based on profilometry) coupling agent layer of FLSCA mixed with IPS. The blue curve shows the layer immersed in uncured epoxy, and has a λ_{max} of 596 nm. But as the epoxy cures, λ_{max} shifts only 4 nm to 592 nm as shown by the red curve. Only a small shift occurs in the thick layer, because most of the FLSCA fluorescence response is due to

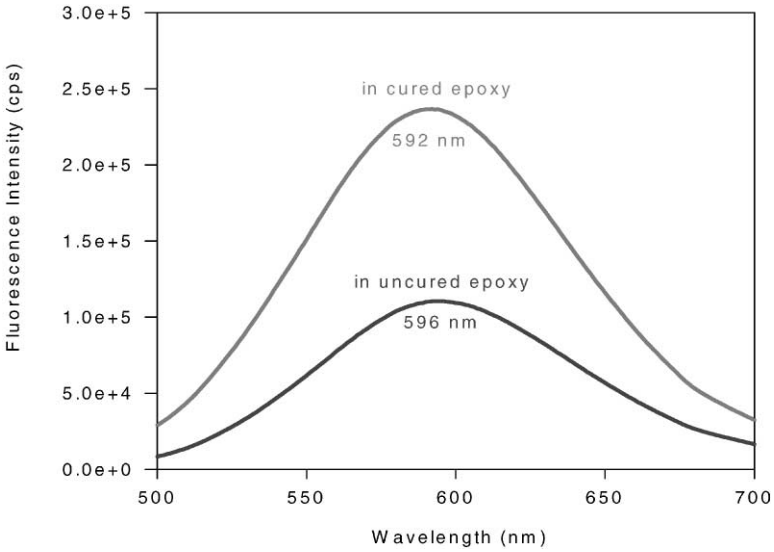

Fig. 9. Fluorescence of a thick FLSCA/IPS mixed silane layer in epoxy resin.

the interaction between FLSCA and the surrounding coupling agent layer. When the layer is thinner, a higher percentage of the FLSCA molecules are able to interact with the epoxy over layer and a larger spectral shift is observed.

Fig. 10 shows fluorescence from the grafted FLSCA/APS layer on a high index, leaded glass fiber. The fiber is not immersed in resin. A typical background spectrum from an uncoated fiber, or a fiber coated only with APS is also shown. This background is significant when compared to the fluorescence from the FLSCA/APS fiber. The small fluorescence signal relative to the background is probably due to the low quantum yield of the dye in polar solvents like APS. The shape of the two curves is very different, clearly indicating that some fluorescence from the grafted FLSCA is being collected by the fiber. In order to generate a fluorescence spectrum, the background spectrum must be accurately subtracted from the spectrum of the FLSCA/APS coated fiber. This is difficult, because variations in connecting different fibers to the laser fluorimeter can cause significant variations in the background intensity. Until a very reliable fiber to fiber connection can be achieved, accurate background subtraction is impossible. We are currently looking at ways to make more reproducible fiber connections.

In order to analyze the small amount of fluorescence collected from the fiber, an initial spectrum is taken immediately after the FLSCA/APS coated fiber is immersed in resin (time zero). This spectrum is then subtracted from all the spectra taken at different times. After this subtraction, the curve for time zero becomes a flat line with zero intensity, as is shown in Fig. 11. If a shift in fluorescence occurs during resin cure, then the shape of the fluorescence spectrum must change. This change in shape can be clearly observed after the initial spectrum at time zero is subtracted. When

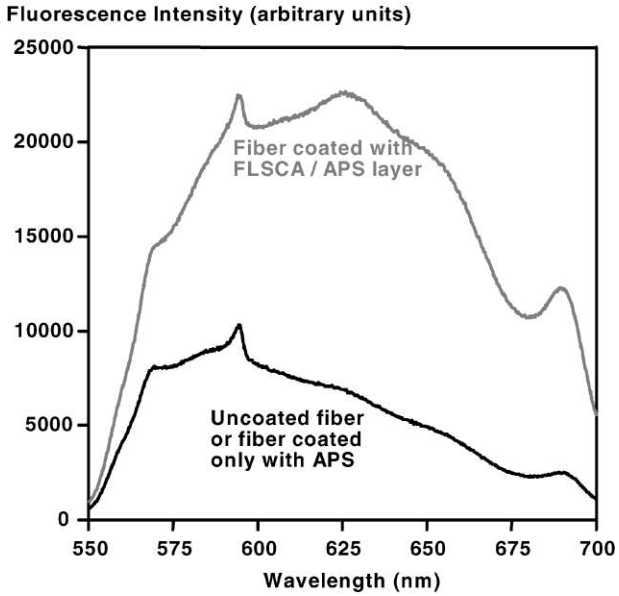

Fig. 10. Fluorescence from a FLSCA/APS layer on a leaded glass fiber optic.

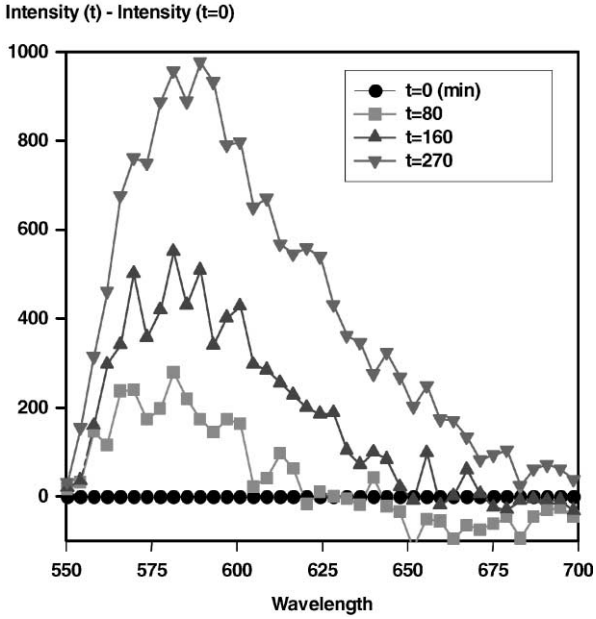

Fig. 11. Intensity grows faster at short wavelengths indicating a blue shift in fluorescence.

a FLSCA/APS layer is grafted to a cover slip and immersed in uncured epoxy λ_{max} occurs near 605 nm. If a blue shift in fluorescence occurs during cure, then the fluorescence intensity at wavelengths < 605 nm will grow faster than the intensity at wavelengths > 605 nm.

4. Conclusions

It has been demonstrated that LPGs can be used as flow sensors in a mold-filling application. Their simple "yes/no" response to the presence of resin at a certain location makes them more robust and less susceptible to losses and fluctuations in light than intensity-based optical fiber sensors. Resin arrival is easily identified since each LPG can be fabricated to uniquely correspond to predetermined wavelengths along the transmission spectrum. This also allows the implementation of wavelength multiplexing along the same strand of fiber. Although, we were able to maintain the most of LPG signals by using the glass sleeves, the sleeves were found to delay the sensor response. Efforts are still underway to find alternative solutions to this problem without increasing the complexity within the mold.

Shifts in the fluorescence spectra from FLSCA grafted to a glass surface can be used to follow cure of an epoxy resin. The magnitude of the shift was dependent upon the polarity of the co-silane and the layer thickness. The FLSCA was successfully grafted to a high-index fiber. Despite the fluorescence background of the fiber, an emission shift was detected through collection of the evanescent response from the grafted FLSCA. We are currently investigating ways of improving the signal-to-noise of the fluorescence from the dye grafted onto the high-index fiber which include optimizing the collection optics and/or FLSCA layer.

Acknowledgements

The authors would like to acknowledge Ms. Paige Furrow and Dr. Mark Jones of F&S Optics for providing the Lunascan and for their fiber-optic expertise. Special thanks to Dr. Steven Pollack, Mr. Kwame Griffith, Dr. Ashebir Fiseha, and Dr. Ryan Cameron from Howard University for aid in the synthesis of the FLSCA dye.

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