An Evanescent Wave Fluorescence Fiber-Optic Flow Sensor for Resin Transfer Molding

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Abstract

An evanescent wave fluorescence-based fiber-optic flow sensor is being investigated. This sensor is based on the interaction of a laser beam in a bare optical fiber with fluorescent probe molecules present in the resin flowing in the direction of the fiber. The electric field of the monochromatic light waves traveling in the fiber by total internal reflection penetrates outside the fiber and is called the evanescent wave field. A fluorescent probe molecule within the depth of penetration gets excited by this field and emits a characteristic fluorescent radiation that is coupled back into the fiber by the principle of reciprocity of optics. If the light at the end of the fiber is filtered for the fluorescent radiation and the intensity is recorded, it gives an estimate of the number of fluorescent probe molecules in contact with the fiber and, hence, the extent to which the fiber is covered with the resin. Preliminary experiments have shown that there is a linear correlation between the peak intensity and the length of fiber in contact with the fluid. A laboratory setup has been assembled at the University of Delaware (UD), using a photomultiplier-tube-based detector, and various experiments have been conducted to assess the effect of covered fiber length on the intensity of fluorescence using the evanescent mode of sensing and on the uses of distal mode sensing of fluorescence for detection of flow.

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1. Introduction

Resin transfer molding (RTM) is a versatile and economic process for the manufacture of polymer composites. RTM is finding increasing acceptance in the fabrication of large and complex parts. Due to their high strength-to-weight ratio, composites have been developed for a number of industrial and aerospace applications as an attractive alternative to conventional metallic parts. However there is a significant need to improve the quality and process control of the RTM processes. The variability of processing conditions and the state of the raw materials cause large part-to-part variations. Thus, there is a need to improve the efficiency and reliability of composite processing.

Modeling and simulation have been used with some success to gain insight into the complex phenomena during flow and cure of resin. The liquid-injection molding simulation software (LIMS) developed at the University of Delaware has been used to predict the occurrence of dry spots occurring when resin flows in molds with complex geometries [1–6].

Another way to improve the efficiency and reliability of a manufacturing process is through the implementation of an on-line sensing-and-control system. This would allow the production of parts with consistently high quality. Hence, there has been considerable work done in the area of on-line sensing techniques. A number of in-situ sensors have been developed for the composite manufacturing processes such as electromagnetic, ultrasonic, spectrometric, and fluorescence sensors [7, 8].

The changes in electromagnetic responses between sensing elements, separated spatially in the mold, as the resin flows and cures have been widely used in many sensors [9–13]. As the resin flows between the elements, it completes an electrical circuit, providing the signal that the resin has reached that point. As the resin cures, there is a change in the permittivity due to decreasing ionic mobility of the adventitious ions, and this can be correlated to the state of the cure.

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Ultrasonic wave-based techniques have been used as tools to detect the cure of epoxies [14, 15]. Ultrasonic waves are passed through the composite part, and the phase velocity and attenuation are measured. As the resin cures the changes in elastic, bulk and relaxation moduli of the resin change, thus changing the speed of propagation, as well as damping of the ultrasonic waves. Some common problems associated with this technique are the low signal-to-noise ratio, dispersion and scattering of the waves, and limitations on the temperature and pressure of operation.

Spectroscopic techniques have been used to detect the cure of composite parts [15–24]. Some of these techniques are ultraviolet (UV)-visible (Vis) and infrared (IR) spectroscopy, fluorescence spectroscopy, and Raman spectroscopy. All of these techniques involve the scattering interaction of a monochromatic beam of light, usually from a laser or a filtered incandescent lamp with the material. The intensity and wavelength of the scattered light yields information about the material under study. In IR spectroscopy, each bond in the resin system has its characteristic peak in the absorption spectrum. Thus, as some bonds form and others get broken during the curing process, the intensities at the corresponding peaks change. In UV-Vis and fluorescence spectroscopy, the light photon excites the molecule to a characteristic energy level. The molecule then de-excites to a lower energy level, emitting a characteristic photon. In a resin system, as the monomer molecules cross-link, the vibrational and translational degrees of freedom get restricted. Thus, the amount of energy available for excitation is higher. This leads to increasing intensity of the emission spectrum, as well as a blue shift as the wavelength of the photons decreases. In Raman spectroscopy, the excitation photon does not have enough energy to excite the molecule to the next energy level. Hence, the energy absorption lifts the photon to a virtual vibrational energy level below the first electronic state. Within a short time, it returns to a stable energetic level in the ground electronic state. If this state is not the same as the original state, then the emission photon has a different wavelength than the excitation light. This shift is called the Raman shift and is characteristic of the molecule. The intensity of the Raman spectrum is directly proportional to the concentration of the chemical species and related to the temperature of the specimen [20]. Hence resin cure can be detected by measuring the decreasing concentration of the epoxide. All these spectroscopic techniques are noncontact; work by direct

molecular-level interaction; and, hence, provide a primary measure of cure. However, the effects of the temperature on the spectra obtained have to be compensated in order to get accurate data.

Fiber optics have been used for remote in-situ spectroscopy [20–36]. The excitation beam is conveyed by the fiber to the region of interest; hence, the fiber optic sensor can be used as both a surface sensor and as an embedded sensor in composites processing. The size of the fiber-optic probe is comparable to the size of the reinforcement fibers in composites. Hence, the interference effects are minimal. The two modes of sensing using fiber optics are (1) the distal mode and (2) the evanescent mode of sensing. In the distal mode, the end of the sensor is in contact in the specimen and is used to sense in a small volume surrounding the tip. In evanescent wave sensing, the interaction of the evanescent electric field of the monochromatic light propagating in the fiber with the surroundings is used for sensing. Thus, the full length of the fiber acts as a probe. It is expected that, as the fiber gets covered, the intensity of the detected signal will increase. In earlier work [35–37], a corresponding blue shift in peak intensity was seen as resin cure progressed. This enables the development of a inexpensive fiber-optic flow and cure sensor, which can be used to investigate the flow into fiber bundles and flow in molds with complex geometry. Thus, fiber-optic evanescent wave sensors have been investigated in an ongoing collaboration between the U.S. Army Research Laboratory (ARL) and the National Institute of Standards and Technology (NIST).

A fiber-optic evanescent wave-based flow sensor is presented here. An argon ion laser was made to interact with a fluorescent dye solution flowing along the length of the fiber optic, and the resultant signal from the fluorescence interaction was monitored using an intelligent photo sensor. An experiment based on distal mode sensing was also conducted.

2. Theory

Optical fibers transmit light by total internal reflection. Light focused on the end face of the fiber intersects the fiber-medium interface at an angle determined by Snell's law. If the refractive index of the medium is less than that of the fiber, then there exists a critical angle of

incidence beyond which the light gets reflected back into the fiber. Thus, light propagating in the fiber at an angle greater than this propagates through the fiber. The electric field amplitude of the guided wave decays exponentially in the medium.

The evanescent wave fluorescent wave sensor is based on the interaction of the fluorescent dye molecules with the evanescent electric field. It is necessary that the refractive index of the sensor be higher than that of the surrounding medium. At the interface between the wave guide and the medium, the light suffers total internal reflection. The evanescent filed arises from the interference between the electric fields of the incoming and the reflected rays. The evanescent wave extends beyond the reflecting interface into the surrounding medium and decays exponentially in amplitude. The distance to which the electric field decays to ($1/e$) its original value is called the depth of penetration. This depth of penetration is given by

$$d_p = \frac{\lambda}{2\pi(\sin^2\theta - (n_2/n_1)^2)^{1/2}}, \tag{1}$$

where λ is the wavelength of the light, n_1 is the refractive index of the fiber, n_2 is the index of the medium, and θ is the angle of incidence of the propagating light at the interface. Values for d_p are of the order of 100–10,000 nm.

We select the angles of incidence at the fiber front to be less than the critical angle so that there are no losses at the face. The amplitude decays exponentially with the distance. The probability of fluorescence is dependent on the product of quantum efficiency of fluorescence and the probability of absorption of the evanescent photon. It is estimated that approximately half the light is emanating from fluorescence at a distance z, where the evanescent wave amplitude is significant. The efficiency of collection and transmission of the fluorescence is dependent on whether the angle in the fiber is greater than the critical angle or not. Almost all the fluorescence in the fiber will be lost for angles less than the critical angle, as can be expected. By the principle of reciprocity of optics, one can expect that, if the emission of the excited molecule itself is evanescent, then the fluorescent photons that get into the fiber will get

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internally reflected and will reach the detector, whereas the fluorescent light that gets refracted into the fiber by the principles of conventional ray optics will lose intensity rapidly with each successive reflection as part of the ray gets refracted into the medium and will therefore be very weak at the detector. Hence, the chief component of the detected fluorescence at the detector end will be the evanescent wave fluorescence. Since the intensity of the electric field of the monochromatic light is approximately the same at every point on the fiber where the laser beam reflects off the fiber-medium interface and nearly all the evanescent wave photons reach the detector, there is a linear correlation between the intensity detected and the length of the fiber in contact with the fluorescent solution. However, there will be a sudden increase in intensity as the conventional fluorescent rays that enter the fiber very near the detector end have not dissipated through losses.

3. Experimental Setup

An optical bench (Figure 1) was constructed for the flow monitoring experiments. The experimental setup consisted of an Ar+ laser, a system of mirrors, beam splitter, 20X microscope objective and a fiber coupler to couple the laser light into the NIST-supplied leaded glass fiber-optic cable.

Figure 1. Schematic of Experimental Setup.

The detection system at the University of Delaware consisted of a laser filter, monochromator, and a intelligent photosensor module (Hammamatsu HC-135 sensor). The wavelength on the monochromator was set using a stepper motor interfaced to the shaft using a flexible coupling. The stepper motor and the intelligent photosensor were interfaced to a PC (P5-166) for control and data acquisition purposes.

The intelligent photosensor combines the sensitivity of a photomultiplier tube with the intelligence of a microcontroller to provide a flexible and sensitive detector that can be interfaced to the computer very easily using a standard RS-232 cable to the serial port. The detector module acquires the signal by counting the number of photons as they enter the input window. This is the most sensitive technique available for light measurement. The photon-counting sensor has a large active area of diameter 21 mm for light gathering. The photomultiplier tube does the photon counting using a series of cascade circuits that generate thousands of electrons for every photon impinging on the light gathering area. The photomultiplier tube is powered by an onboard high-voltage Cockroft-Walton power supply. This is used to limit current consumption and prevent unwanted temperature rise of the assembly. The photosensor is powered by a direct-current (DC) power supply rated at a voltage of 5 V.

The light signal from the photomultiplier tube takes the form of very high current pulses. These pulses are amplified and converted to digital pulses with a high-speed amplifier and discriminator. They are then prescaled by a value of four before counting, which increases the dynamic range without using excessive power. These pulses are counted using by the microcontroller. The HC-135 integrates all the necessary components required for photon counting in a compact cylindrical body of diameter 1.35 in and length of 5 in.

4. Experiments

A fiber-optic flow sensor can be implemented in two modes of sensing. In the first mode, called distal mode sensing, the fiber-optic sensor is a point sensor. The optical fiber acts as a conduit for the laser beam to reach a point in space. When the flow containing fluorescent dye

reaches the tip of the fiber, which is illuminated by the laser beam, the dye molecules get stimulated and emit fluorescent photons. This radiation gets conducted back along the fiber and goes to the detector, which is set up to receive only those photons of the wavelength corresponding to the peak of the spectrum of fluorescence of the dye. The change in signal immediately indicates that the flow has reached that point. In the second mode of sensing, the evanescent mode, the entire length of the optical fiber acts as a sensor. The dye is made to flow along the length of the fiber and the change in signal recorded as the optical fiber gets progressively covered.

A number of experiments were carried out to detect the flow of a solution of Rhodamine B dissolved in ethanol to a concentration of 10^{-6} M. The optical fiber used, that was supplied by the Polymers Division, NIST, had a high refractive index of 1.6. The intensity of background light was measured using the photon counter with the lights off. The "dark count" of the detector (i.e., the photon counts obtained when the detector is closed off to all sources of external light using a cap) was found to be of the order of 150 counts/second. The background and fluorescent photon counts were of the order of 100,000 counts/second. Hence, the dark count was neglected in subsequent calculations. The background count was subtracted from the subsequent readings in order to obtain an accurate estimate of the fluorescent light reaching the detector. The monochromator was set to 570 nm, which is the maximum of the spectrum obtained for the fluorescence of Rhodamine B dye dissolved in ethanol. The laser was turned on, and the background count was recorded to give a baseline for subsequent readings. In the first series of experiments for distal mode sensing, the optical fiber was suspended inside the tube and the fluorescent dye solution was injected from the bottom until it reached the fiber tip. At that point, the change in the counts was recorded. This change in counts is plotted.

A number of experiments were performed to evaluate the evanescent mode of sensing. The optical fiber was run through the tube and clamped at the end. The tip of the fiber extending below the tube was blackened in order to prevent background light from getting conducted to the detector through the fiber. A centimeter scale was attached to the tube. The dye solution was injected along the length of the fiber. At every centimeter graduation, the photon count was

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recorded until the tube filled up completely. The background count was subtracted and the fluorescent count plotted against length of fiber covered.

A few preliminary experiments were conducted at NIST in order to explore use of the fiber-optic flow sensor. A charged coupling device (CCD) camera was used for detection of fluorescent intensities at different wavelengths. The fluorescent spectra were recorded as the fiber length was covered progressively. The spectra are shown in Figure 2. As can be observed, the peak intensity shows an increase. When plotted against the length of fiber covered the peak intensity shows a linear variation with a jump at the end (Figure 3).
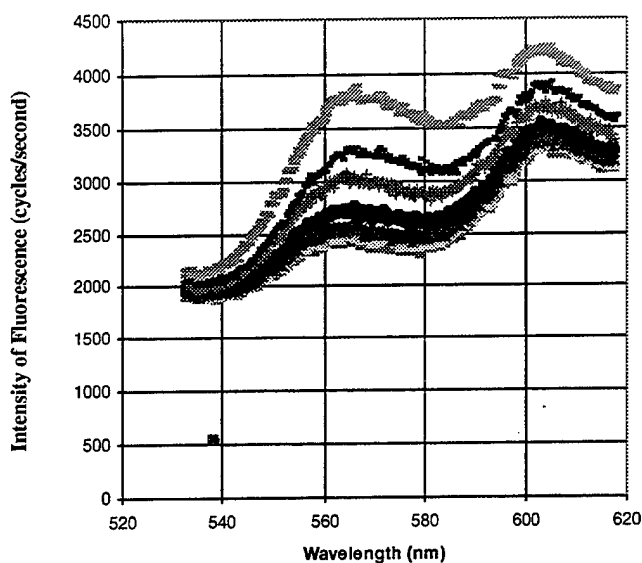
Figure 2. Plot of Intensities of Fluorescence Detected Using a CCD Detector Array With Wavelengths of Light for Different Lengths of Fiber Covered. (Experiment Conducted at NIST.)

5. Results and Conclusions

The results from the various experiments were plotted for both the distal mode (Figure 4) and the evanescent mode experiments. In the experiments with distal mode sensing, a significant increase in signal was observed as the flow reached the tip of the optical fiber. The data from the
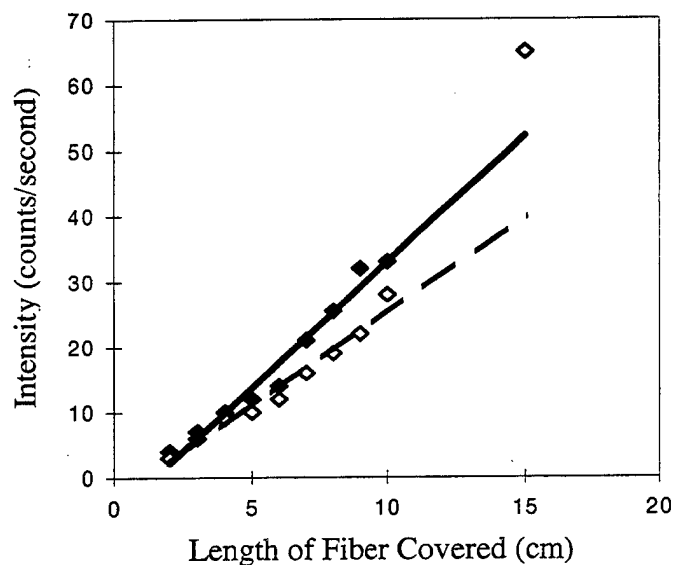
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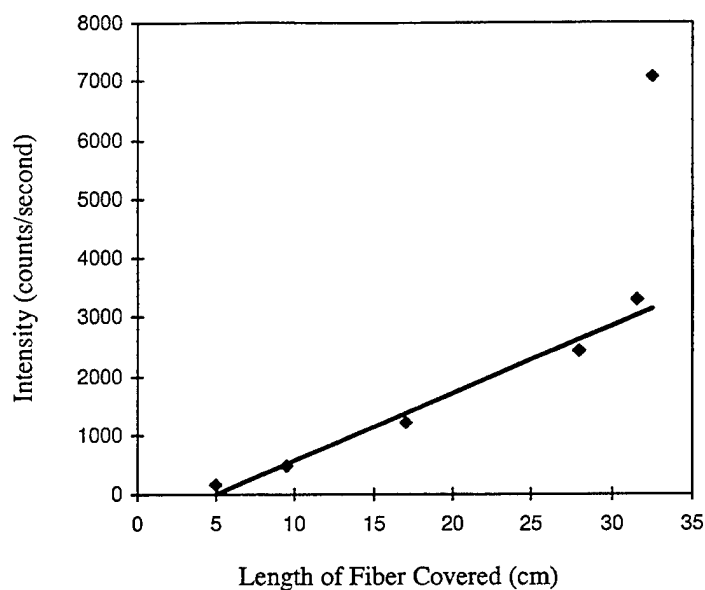
Figure 3. Plot of Peak Fluorescent Intensity at 570 nm Detected Using a CCD Detector at NIST for Different Lengths of Fiber Covered by a Solution of Rhodamine B in Ethanol.
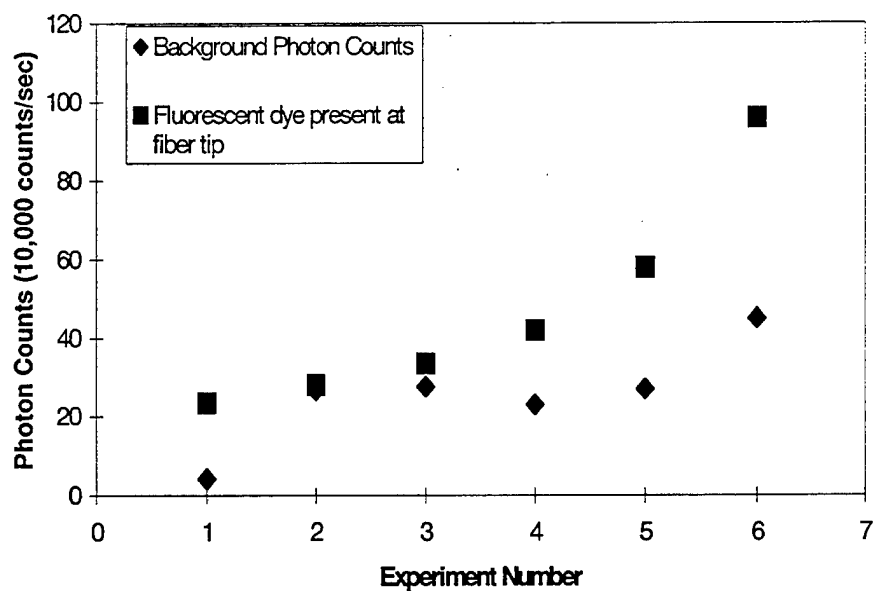

Figure 4. Plot of Change in Intensity Detected as Fluorescent Dye Flows Past the Tip of the Optical Fiber.

experiments demonstrates that a fiber-optic point flow sensor is feasible. During the filling process in mold filling and cure, feedback from a point sensor can provide vital information for on-line control and for triggering decision points.

In the evanescent mode of sensing using optical fibers (Figures 5–7), it was observed that, as the fiber was covered by the dye, there was an increase in signal that was fairly linear in nature. This linearity is predicted by the theory of the evanescent wave fiber-optic sensors. The abrupt increase in signal at the end can be caused by the bulk fluorescence of the dye solution due to leakage of light from the optical fiber. This bulk fluorescence usually dissipates through repeated refractions and reflections at the uncovered part of the surface of the optical fiber and does not reach the detector. As the flow nears the detector, the numerous photons of bulk fluorescence have a smaller distance to travel to register at the detector. Hence, the photon count registers an increase. The intensity of the bulk fluorescence is proportional to the number of dye molecules in the volume surrounding it and, hence, to the length of fiber covered. Thus, the linearity of the signal is still preserved.

Figure 5. **Plot of Peak Fluorescent Intensity at 570 nm Detected Using a Photon Counter for Different Lengths of Optical Fiber Covered by a Solution of Rhodamine B in Ethanol (Experiments 1 and 2).**
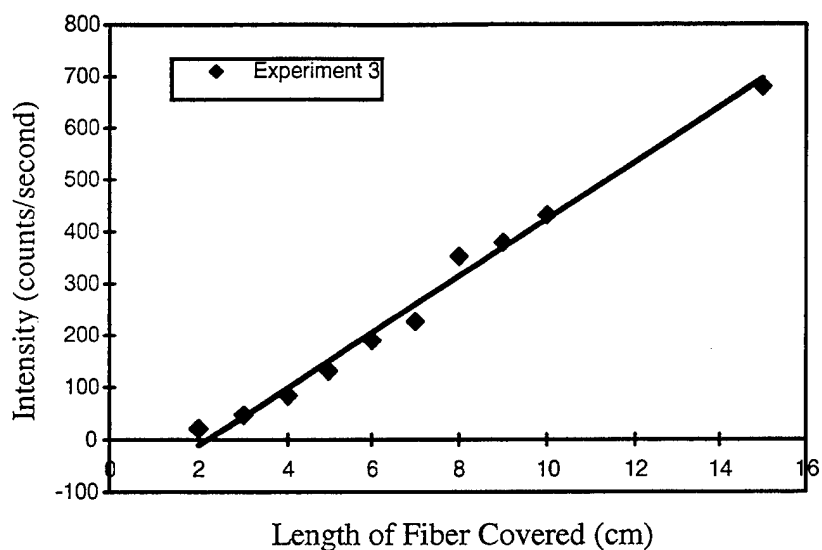
Figure 6. Plot of Peak Fluorescent Intensity at 570 nm Detected Using a Photon Counter for Different Lengths of Optical Fiber Covered by a Solution of Rhodamine B in Ethanol (Experiment 3).
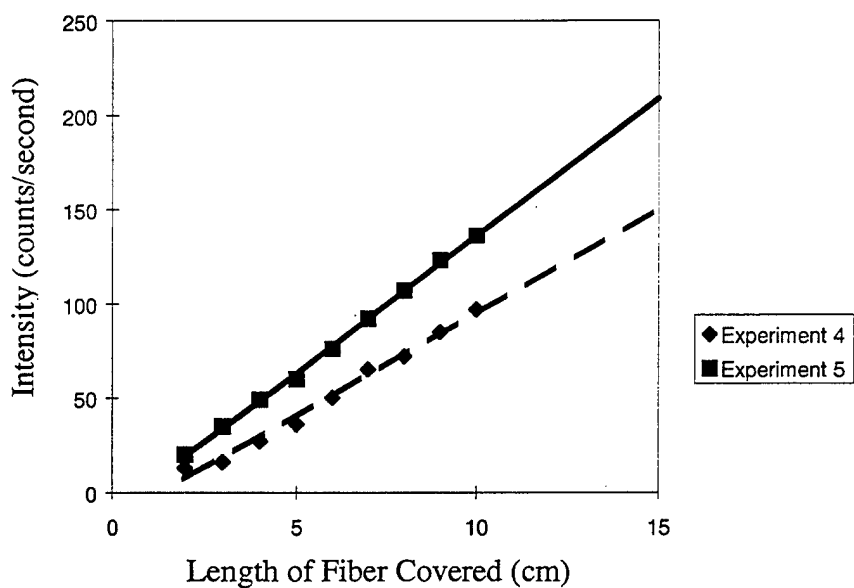
Figure 7. Plot of Peak Fluorescent Intensity at 570 nm Detected Using a Photon Counter for Different Lengths of Optical Fiber Covered by a Solution of Rhodamine B in Ethanol (Experiments 4 and 5).

The experiments show that an evanescent wave fluorescence fiber-optic sensor is a distinct possibility. The linearity of the signal can enable the detection of flow in a mold filling process in any given direction in an "analog" fashion. The reaction time is very small, and the optical fiber may be woven into the preform. This would enable the sensing of flow in complicated molds and in molds for manufacturing thick parts, where the sensors currently in use, which are surface mounted, are inadequate. Further, the optical fiber is of the same size as most preform tows and it would not affect the resin flow. In fact, it may be used for monitoring strains due to loading in the composite part, after manufacture.

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13. ABSTRACT *(Maximum 200 words)*

An evanescent wave fluorescence-based fiber-optic flow sensor is being investigated. This sensor is based on the interaction of a laser beam in a bare optical fiber with fluorescent probe molecules present in the resin flowing in the direction of the fiber. The electric field of the monochromatic light waves traveling in the fiber by total internal reflection penetrates outside the fiber and is called the evanescent wave field. A fluorescent probe molecule within the depth of penetration gets excited by this field and emits a characteristic fluorescent radiation that is coupled back into the fiber by the principle of reciprocity of optics. If the light at the end of the fiber is filtered for the fluorescent radiation and the intensity is recorded, it gives an estimate of the number of fluorescent probe molecules in contact with the fiber and, hence, the extent to which the fiber is covered with the resin. Preliminary experiments have shown that there is a linear correlation between the peak intensity and the length of fiber in contact with the fluid. A laboratory setup has been assembled at the University of Delaware (UD), using a photomultiplier-tube-based detector, and various experiments have been conducted to assess the effect of covered fiber length on the intensity of fluorescence using the evanescent mode of sensing and on the uses of distal mode sensing of fluorescence for detection of flow.

14. SUBJECT TERMS
resin transfer molding, fiber optics, flow sensors

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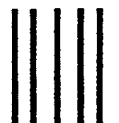**(DO NOT STAPLE)**

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