AN EXPERIMENTAL STUDY ON THE PROCESS MONITORING OF RESIN TRANSFER MOLDED COMPOSITE STRUCTURES USING FIBER OPTIC SENSORS

by

Nazlı Gülsine Özdemir

Submitted to the Graduate School of Sabancı University in partial fulfillment of the

Requirements for the Degree of

Master of Science

Sabancı University

May, 2011

An Experimental Study on the Process Monitoring of Resin Transfer Molded Composite Structures Using Fiber Optic Sensors

APPROVED BY:

Assist. Prof. Dr. Mehmet Yıldız (Thesis Advisor) …..…....…………

Assist. Prof. Dr. Cem Öztürk ...………………..

Prof. Dr. Yusuf Menceloğlu ...…………………

Assist. Prof. Dr. Burç Mısırlıoğlu ………..…...………..

Assist. Prof. Dr. Alpay Taralp …...………………..

DATE OF APPROVAL: ……………………

AN EXPERIMENTAL STUDY ON THE PROCESS MONITORING OF RESIN TRANSFER MOLDED COMPOSITE STRUCTURES USING FIBER OPTIC SENSORS

Gülsine Özdemir

MAT, Master's Thesis, 2011

Thesis Supervisor: Assist. Prof. Dr. Mehmet Yıldız

Keywords: Glass fibers, Curing, Resin transfer molding (RTM), Composites, Optical fiber sensors, Flow monitoring, Cure monitoring

Abstract

This thesis focuses on the research conducted on in situ process monitoring (cure and flow) of resin transfer molded glass fiber reinforced polymer composites using Fiber Bragg Grating (FBG), etched bare fiber optic sensors and Fresnel refractometers.

In this direction, a laboratory scale resin transfer molding (RTM) apparatus was used with the capability of visually monitoring the resin filling process as well as embedding fiber optic sensors. Both FBG and etched fiber sensors are embedded into glass fiber reinforcements in the RTM mold, and are used to monitor the flow front during the resin injection and subsequently the cure cycle. Moreover, the cure cycle of the resin system utilized in this work is studied using an in-house developed Fresnel based refractometer. The data gathered from corresponding cure monitoring experiments have been compared with the results of polymer extraction experiments and excellent correlation between the results was observed.

The embedded FBG sensors also allow the determination of mechanical stresses or loads on the in- service composite parts, through interrogating these sensors. This process is referred to as Structural Health Monitoring, which is not considered within the scope of this thesis work.

REÇİNE İLETİM TEKNİĞİ İLE ÜRETİLMİŞ KOMPOZİT YAPILARIN FİBER OPTİK SENSÖRLERLE PROSES GÖZETİMİ

Gülsine Özdemir

MAT, Master Tezi, 2011

Tez Danışmanı: Ass. Prof. Dr. Mehmet Yıldız

Anahtar Kelimeler: Cam takviye, Kürleşme, Reçine İletim Tekniği, Kompozitler, Fiber Optik Sensörler, Akış Gözetimi, Kürleşme Gözetimi

Özet

Bu tez, reçine iletim tekniği ile üretilmiş cam fiber takviyeli polimer kompozitlerin Fiber Bragg Izgara sensörleri, dağlanmış optik sensörler ve Fresnel optik sensörleri kullanılarak proses izleme tekniği üzerine odaklanmıştır.

Bu doğrultuda reçine dolum prosesini ve fiber optik sensörlerin kompozit yapıya entegrasyonunu sağlayabilecek laboratuar boyutlarına uygun bir reçine transfer kalıplama aparatı kurulmuştur. Fiber Bragg ızgara ve dağlanmış fiber optik sensörler RTM kalıbındaki cam takviyelerin arasına entegre edilmiş ve reçine enjeksiyonu esnasında akış önü gözetimi ve bu işlemi takiben kürleşme işlemi gözetiminde kullanılmıştır. Buna ek olarak reçine sisteminin kürleşme devri Fresnel optik sensörleri ile de doğrulanmıştır. Kür gözetimi deneylerinden elde edilen veriler polimer ayrıştırma deneyleri sonuçları ile mukayese edilmiş ve sonuçların kusursuz uyumu gözlenmiştir.

Bu araştırma aynı zamanda kompozit yapıların servis süreleri esnasında maruz kaldığı mekanik gerilimlerin sağlık gözetimi ile determinasyonunu da içermektedir fakat bu konu bu tezde ele alınmamıştır.

ACKNOWLEDGEMENTS

I would like to take this opportunity to express my appreciation to those who helped me complete this thesis.

I would like to thank all my professors, colleagues and office mates for the encouragement during the period of my study. I would like to express my gratitude to Dr. Mehmet Yıldız and Dr. Cem Öztürk for their supervision.

Special thanks to my parents and friends outside of the school who gave me support and a chance to get away from my studies when I needed to.

The funding provided by The Scientific and Technological Research Council of Turkey (TUBITAK) under the support program of scientific and technological research projects (1001) for the project with the project number of 108M229 is gratefully acknowledged.

TABLE OF CONTENTS

LIST OF FIGURES

LIST OF SYMBOLS

RTM	Resin Transfer Molding
EFS	Etched Fiber Sensors
dBm	Measured Power Referenced to one Milliwatt
n	Refractive Index
n_{eff}	Effective Refractive Index
Λ	Fiber Bragg Grating Pitch
λ_b	Bragg Wavelength
α_Λ	Thermal expansion Coefficient for an Optical Fiber
α_n	Thermo Optic Coefficient for an Optical Fiber
ε_x	Axial Strain
p_ε	Effective Photo elastic coefficient
v	Poisson's Ratio
p_{11}	Components of Strain optic Tensor
p_{12}	Components of Strain optic Tensor
nm	Nano meter
μm	Micro meter
R	Reflection Coefficient
P_i	Incident Light Power
P_r	Reflected Light Power

μW Micro Watt

CHAPTER 1: INTRODUCTION

Due to their strength-to-weight ratios, stiffness, corrosion resistance, and fatigue performance, composite structures offer many advantages over conventional metallic materials. They are utilized in a variety of load bearing structures such as helicopter rotor blades, aircraft fuselage, rib chord, trailing edges, cargo doors and pressure vessel [1]. Considering the manufacture of repeatable products, composites have relatively difficult processing characteristics in compared to metallic materials. For the reliability and the safety of composite components used as primary load carrying structures, it is vital to understand and control the manufacturing process thoroughly to ensure the high quality of manufactured components, which must meet the stringent requirements of relevant standards and specifications for structural composite members.

A number of processing methods exist for continuous fiber reinforced thermoset composite materials. One of the manufacturing processes particularly suitable for manufacturing of structural composite components is resin transfer molding. In this process, fiber preforms (glass or carbon) are placed in a closed mold and resin is injected into the mold to saturate the preform. After the resin cures, the mold is opened and the final composite part is de-molded. The RTM method can produce complex, high quality, near net-shape parts in series production with high fiber volume fractions, a *class-A* surface finish and little post processing. The two most critical processing steps in RTM are the mold saturation (resin injection) and polymerization (cure) stages. Both stages have direct effects on the final quality of the parts since the transfer of loads from the resin matrix to reinforcement (glass fibers) depends heavily on the extent of successful saturation of fibers by resin, and the subsequent cure. Due to a high resistance to resin (a relatively viscous material) flow through the preform (a fairly low permeability material), geometry changes throughout the mold, and a phenomenon known as race tracking (Figure 1a) which creates an unbalanced or uneven resin flow in mold filling process, there might be regions not fully saturated with resin resulting in voids of dry-spots (Figure 1b). Such regions have a significant effect on the structural integrity of the composite part since they are essentially a defect in the material. The closed nature of the RTM manufacturing process hinders visual access to the resin flow since in industry molds are almost always made of non-translucent materials such as opaque composites or metals. Therefore, the RTM process must be fully controlled in order to ensure the production of high quality composite parts in a repeatable and consistent manner within acceptable tolerances.

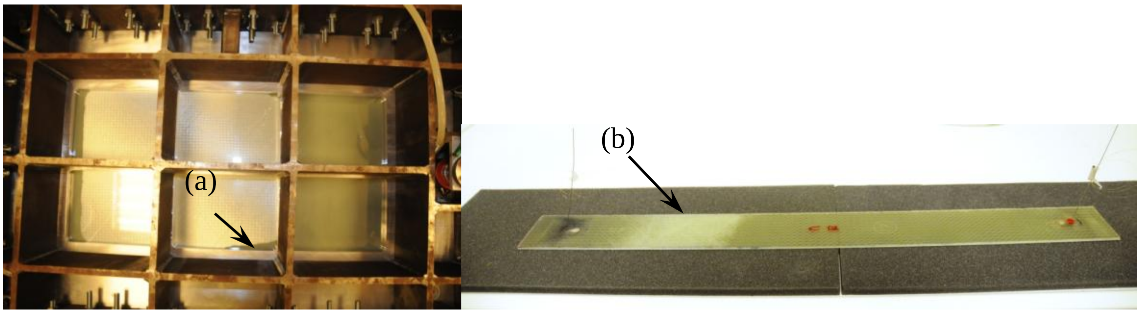


Figure 1. Race tracking phenomena (a), resulting in dry spots (b)

The in-situ cure monitoring is also critically important in processing stages of composite materials since the curing behavior of thermosetting materials such as epoxy resins determines the processing time. For instance, if the composite part is prematurely de-molded, the part may fail to gain its green strength and stiffness and may deform or warp during or after the removal from the mold. Taking the part out of the mold belatedly, on the other hand, results in unnecessarily extended manufacturing time, and over curing of the part, which may adversely affect the desired mechanical performance of the component. In other words, the mechanical properties of fiber-reinforced composites are seriously dependent on the chemorheological process taking place during the cure stage. Hence, the optimum duration of cure cycle is an important processing parameter to optimize the manufacturing process. The term cure for thermosetting polymers refers to complex chemical reactions which take place between resin and hardener, which results in the formation of a cross linked structure. The cure cycle of a resin system consists of two main parts, namely gelation and vitrification. The gelation is an irreversible transition from a viscous material into an elastic gel during which the viscosity of the resin increases. It starts due to the exothermic chemical reactions of the reactive groups in the polymer and continues progressively by forming three dimensional dense polymeric network structures.

When the network density has reached a critical value, the resin becomes a rubber. In the vitrification stage, the resin system undergoes a transition from a rubbery state to solid state where the resin system can no longer flow as a result of the increased crosslink density due to the successive cross-linking polymerization reaction. The cure monitoring process relies on monitoring changes in the various physical or chemical properties of thermoset resin during its transition from liquid state into solid state. More often, composite materials manufacturers rely on the recommendation of resin manufacturers for the cure schedule despite the shape and thickness of material. Therefore, reliable cure sensors are required. In this direction, there are substantial amount of work devoted to the development of techniques required for characterizing the solidification transitions [2].

Towards this end, in literature, various forms of cure sensors have been suggested and developed. Some of them are based on conductometric [3, 4, 5, 6, 7], dielectrometric [8, 9, 10, 11] and ultrasonic [12, 13] techniques. In conductometric or resistive cure monitoring methods, conductor probes (point, lineal or junctions of weaved wires) are used as sensors. Cure monitoring relies on the monitoring of the conductivity change of the resin. In general, the conductivity of resin decreases as it cures. These probes can also be used to determine the resin arrival since electrical resistance between the probes decreases significantly when the resin reaches probes. This method has a significant limitation in that the voltage change between the probes might be rather small and may not be distinguished from electrical noise. Ultrasonic cure monitoring methods are based on monitoring the changes in the characteristics of propagating ultrasound (time of flight and damping of the sound wave both in through-transmission and pulse-echo modes) due to the variation in the physical properties of the curing resin. Nevertheless, they also have some drawbacks associated with the complex device requirements, low signal-to-noise ratio and temperature dependency. Dielectrometric sensors measure the capacitance of the media between the sensor plates. However, they are expensive, sensitive to electrically noisy environment and invasive to the structure. A summary of the currently utilized sensor technologies and methodologies for flow and cure monitoring in composite manufacturing process is provided in [2].

However, these approaches have not received significant acceptance as cure sensors for integration in composite manufacturing processes. In an attempt to find alternative and efficient flow and cure monitoring sensors for composite materials processing, fiber optic sensors have also been considered and studied by various research groups. Fiber optic sensors may be based on the absorption, reflection, refraction or emission of light. Quite a lot of different fiber optic sensor based methodologies have been attempted for cure cycle monitoring of polymeric resins such as transmission spectroscopy [14], evanescent wave spectroscopy [14, 15, 16], refractive index monitoring [14, 17, 18, 19], long period gratings [20], tilted fiber Bragg gratings [20] and Fresnel based refractometer [20, 21, 22, 23]. The main advantages that fiber optic sensors offer in comparison to their previously cited counterparts are increased sensitivity, reduced weight and size, less intrusive in the host structures, immunity to electromagnetic interference, and the multiplexing ability that enables for sensing spatially distributed quantities. The fiber optic based cure sensors proposed in the literature for cure monitoring are not limited to those listed, a concise summary of fiber optic sensors for curing of thermosetting polymers are provided in [24].

The present work distinguishes itself from earlier relevant work reported in literature in the following aspects. Most of the previous works which utilized fiber optic sensors for cure monitoring of thermosetting polymers have focused on showing the capabilities of one or two different sensors for a simple cure monitoring of polymeric resin in a container without considering process monitoring of operational composite manufacturing. Here, an integrated and systematic experimental study has been carried out to determine the effective usability of FBG optic sensors, etched fiber sensors for process monitoring of RTM composite manufacturing system and Fresnel based refractometer sensors for cure monitoring of a polymeric resin system.

Fiber Bragg gratings (FBGs) have attracted attention within the last decade as sensing devices due to their lightweight, small size (less intrusive to host body) and immunity to electromagnetic interference [1, 25, 26]. An FBG sensor is a segment of a single mode optical fiber core with a small portion of periodically varying refractive index in the axial direction.

The grating segment acts as an optical filter by reflecting a specific wavelength back in the direction of the optical source or interrogator, while transmitting other wavelengths. The center wavelength of the reflected spectrum known as the Bragg wavelength, shifts if the FBG is subjected to the strain or temperature variation, thereby making it possible to measure these external effects [27]. They have been used to measure properties such as displacement, strain, temperature, pressure, humidity [27]. Within the scope of this thesis work the critical issue was simultaneously sensing of strain and temperature data in RTM experiments. The corresponding data could be identified with different methods. The simplest method involves bonding one half of a FBG to its host structure and leaving the other half free of mechanical strain (Figure 2). The section of the FBG that is bonded experiences both strain and temperature changes while the free section experiences only temperature changes. This way strain and temperature data could be analyzed simultaneously within the same experiment.

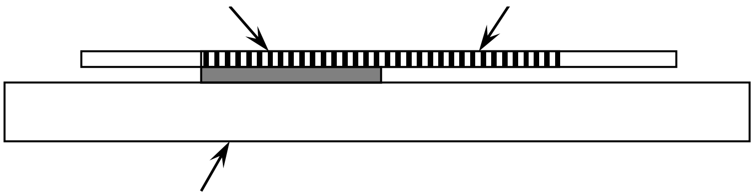


Figure2. Temperature compensated FBG strain sensor in which half of the FBG is strain free and responds only to the local temperature.

Embedded FBGs can be used for three distinct purposes during the life of a resin transfer molded part. An array of multiplexed FBGs in a mold can monitor the mold filling process and can ensure that the mold is completely filled with resin [28]. As stated before, once injected the resin must go through a specific time-temperature cure cycle. In complicated 3-D parts with varying thicknesses and surface areas, different regions of resin cure at different rates and to varying degrees across the part. FBGs have been used to monitor the cure throughout the part [29, 30, 31]. Once in service, the embedded FBGs can be used in a variety of ways to monitor the health of the part thereby reducing maintenance costs and time while at the same time increasing safety.

The second type of optical fiber sensor used in this work is the etched fiber sensor (EFS). This type of sensor is quite simple; a small section of optical fiber is etched to expose the core of the fiber to the environment. When this etched region is surrounded by the resin flowing through the mold, the light power transmitted through the optical fiber changes, thus enabling the sensor to detect fluid. Since both EFS and FBG sensors allow light to pass there is potential for them to be multiplexed on one single fiber. Etched fiber optic sensors can be embedded in geometrically complex regions of composite where resin arrival is a concern. Additionally, with the help of many etched fiber sensors embedded throughout the mold the operator can understand where the resin has reached. Etched sensors can also be used for the cure monitoring process as described in the following sections.

During the life time of a composite structure, the etched sensors may also be used for structural heath monitoring qualitatively such that when interrogated, if there is a disruption or weakening in the transmitted light signal, one can conclude that the sensor location is either excessively loaded or cracked. The third sensor is a Fresnel probe which is also used for cure monitoring through recording refractive index variation of the curing resin.

CHAPTER 2: EXPERIMENTAL SETUP AND PROCEDURES

Resin transfer molding is a subcategory of liquid composite molding. The RTM process involves loading a two sided, fully enclosed mold with a fiber perform usually carbon or glass fiber, closing and clamping the mold, injecting resin into the mold until fully saturated, allowing the resin to cure and removing the molded part. The in-house RTM apparatus used within the scope of the present work has the flexibility of accommodating different mold designs and mold thicknesses, with the feature of a glass viewing window to allow for visual monitoring of resin flow through fiber perform during the injection process and thus to ensure that all dry spots become saturated. Figure 3 shows a schematic of the RTM process together with the lab scale in-house built RTM system.

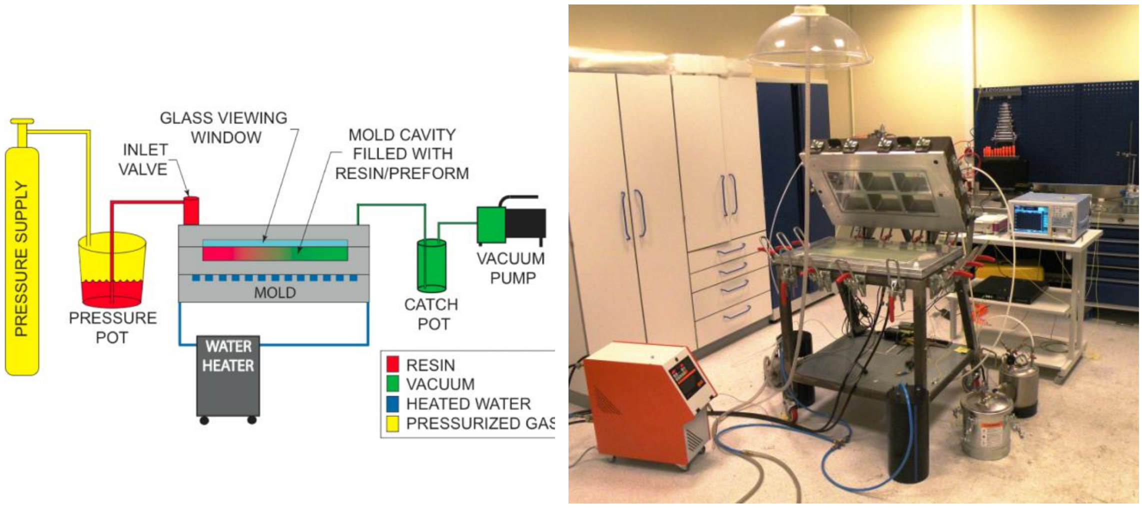


Figure 3. Schematic of the RTM composite manufacturing process (left), and the RTM apparatus together with optical equipments (right).

The RTM apparatus in Figure 3 is a modular clamshell system with the flexibility of accommodating different mold designs and thicknesses. It has one inlet port, two outlet ports, two o-rings around the perimeter to contain the resin (one primary and one auxiliary), locating pins to ensure alignment with the upper half of the mold and nine removable sensors (six pressure, three temperature).

This mold produces a 305mm X 610mm X 3mm panel. In all experiments presented, biaxial glass fiber reinforcements (X 800 E05 800g/m^2) manufactured by Metyx composite are used. In passing, we would like to note that all the techniques proposed and implemented in this work are also applicable to carbon fiber reinforcements. The epoxy resin system used is the mixture of ARALDITE LY 564 resin and XB 3403 hardener, mixed with the ratio of 100 and 36 parts by weight. After mixing the resin and hardener, the mixture is degassed for several minutes inside the pressure pot which is used to inject the resin system into already vacuumed and preheated mold.

The ingress/egress of fiber optics is one of the important issues for embedding fiber optic sensors into composite structures in a repeatable and consistent manner because the optical fiber is fragile at the ingress/egress location, and does not tolerate a sharp bending radius. Connector systems developed by the optical fiber communications industry are available, but they are too bulky for embedding into the composite structures without causing deterioration of the structural strength, or endangering the structural integrity. The closed nature of the RTM process as well as the very fragile nature of fiber optics makes the ingress/egress of FBG sensors into the mold challenging without breaking the sensors. In the literature, most of the studies have considered embedding fiber optic sensors into the composite plate structures produced with hand lay-up, and used the in-plane ingress/egress method. In the in-plane ingress/egress method, the ingress/egress point of an optical fiber is located at the edge of a composite laminate. However, this method can only be applied in the case of an open mold or hand lay-up. To remove a composite part from a mold it must be removed normal to the mold, therefore the embedded fibers must enter and exit through the mold in such a way that upon removal, the fibers are not severed. Towards this end, in the early works of our composite group, a novel through thickness ingress/egress method has been developed, which can overcome the limitations of the in-plane method and be applicable to closed mold processes such as RTM [32]. To protect the fiber with minimal disturbance to the composite material a thin hypodermic tube is placed around the fiber. This protects the fiber through the radius of the bend as well as reinforces the fiber at the ingress/egress point once the part is removed form the mold. A tapered silicone stopper was used to seal around the hypodermic tube. A custom fitting is used to keep the stopper and fiber in place.

Figure 4 shows the visualization of resin flow through a fiber glass reinforcement in RTM, the manufactured composite plates with embedded fiber optics sensors (FBG and etched) by using capillary tubes for ingress and egress, and three loop etched sensors for flow and cure monitoring.

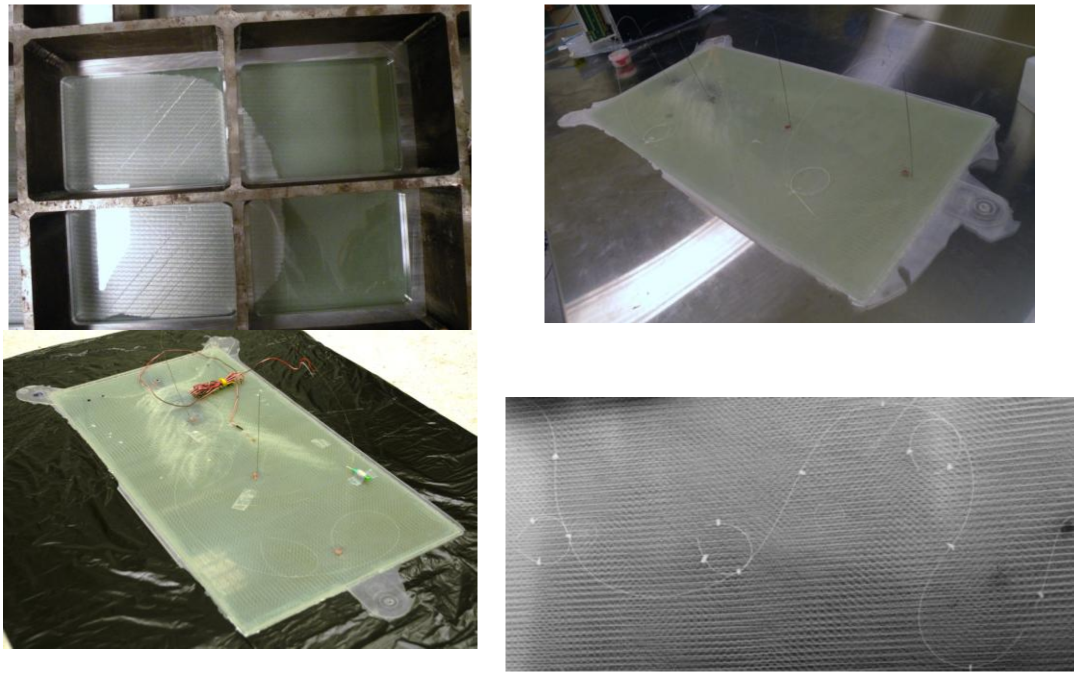


Figure 4. The visualization of resin flow through a fiber glass reinforcement in RTM, the manufactured composite plates with embedded fiber optic sensors (FBG and etched) by using capillary tubes for ingress and egress, and three loop etched sensors for flow and cure monitoring.

As stated in the introduction section, an FBG sensor is a segment of a single mode optical fiber core with a periodically modulated refractive index in the direction of fiber length. The grating segment acts as an optical filter by reflecting a specific wavelength back while transmitting others as schematized in figure 5. The periodic modulation of the refractive index at the grating location will scatter the light traveling inside the fiber core. Out of phase scattered waves will form destructive interference thereby canceling each other and in phase light waves will add up constructively forming a back reflected spectrum with a center wavelength known as the Bragg wavelength.

The Bragg wavelength satisfies the Bragg condition as

$$\lambda_B = 2n\Lambda \tag{1}$$

where λ_B is the Bragg wavelength, n_{eff} is the effective refractive index of the fiber mode and Λ is the pitch of the gratings. The spacing of periodic refractive index modulation is a function of strain and temperature. If an FBG sensor is under a mechanical or thermal load (temperature variation), the spacing between gratings and effective refractive index of the fiber mode will change due to the strain, and thermal expansion, respectively. Since the Bragg wavelength λ_B is a function of the average refractive index and grating pitch, any change in these variables will cause the Bragg wavelength to shift, meaning that the center wavelength of the reflected spectrum changes. Expanding Eq. (1) into a Taylor series yields the differential change in the Bragg wavelength resulting from applied strain and temperature variations as

$$\frac{\Delta\lambda_B}{\lambda_B} = \left(\frac{1}{\Lambda}\frac{\partial\Lambda}{\partial l} + \frac{1}{n}\frac{\partial n}{\partial l}\right)\Delta l + \left(\frac{1}{\Lambda}\frac{\partial\Lambda}{\partial T} + \frac{1}{n}\frac{\partial n}{\partial T}\right)\Delta T \tag{2}$$

where Δl is the change in the length of the grating section. Here, the terms $(\partial\Lambda/\partial l)$ and $(\partial n/\partial l)$ correspond to a change in the grating spacing due to strain, and the strain-optic induced change in the refractive index, respectively. The terms $(\partial\Lambda/\partial T)$ and $(\partial n/\partial T)$ represent the change in the grating spacing due to the thermal expansion and the termo-optic induced change in the refractive index, in the given order. Defining thermal expansion and thermo-optic coefficients for an optical fiber as $\alpha_\Lambda = (1/\Lambda)(\partial\Lambda/\partial T)$ and $\alpha_n = (1/n)(\partial n/\partial T)$, respectively, one may write the shift in Bragg wavelength due to temperature and strain changes respectively as

$$\Delta\lambda_B = \lambda_B(\alpha_\Lambda + \alpha_n)\Delta T\,,\ \Delta\lambda_B = \lambda_B(1 - p_e)\varepsilon_x \tag{3}$$

where p_e is the so-called effective photoelastic coefficient and defined as $p_e = (n^2/2)\left[p_{12} - \nu(p_{11} + p_{12})\right]$ and ε_x is the axial strain. Here, p_{11} and p_{12} are the components of strain-optic tensor, and ν is the Poisson's ratio [33].

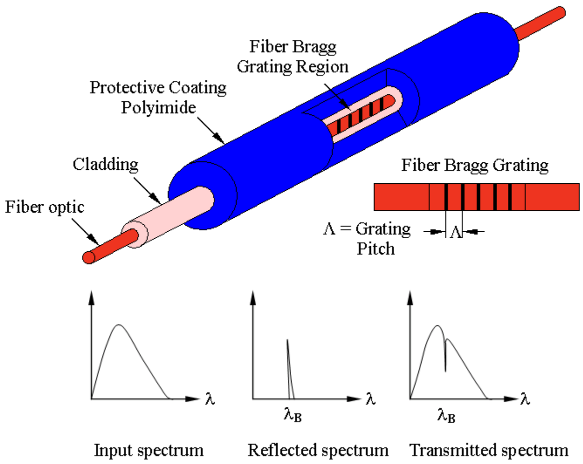


Figure 5. Schematic describing an FBG sensor

Since the center wavelength of Bragg Grating sensors are sensitive to both temperature variations and strain, in sensing applications where only temperature or strain measurement is of interest, the effect of temperature or strain on grating has to be compensated to be able measure only one of these effects. Multiple Fiber Bragg gratings sensors can be easily multiplexed (written) onto a single strand of fiber, thereby forming an array of FBG sensors for sensing spatially distributed quantities over a large distance. The array of FBG sensors minimizes the number of ingress/egress locations during the embedding process in the smart structures.

FBG sensors can be compatibly embedded in fiber-glass reinforced composite structures due to their small size and light weight without compromising the host's structural integrity. In the case of structural strain monitoring, the load on the host is directly transferred onto the grating region through the shear action, hence causing the change in the length of the grating region. As a result, the grating spacing and the refractive index of the grating change, allowing the determination of mechanical properties of the structure through measuring the shift of the Bragg wavelength with respect to a reference value. The presence of the damage or defect formation in a composite structure alters the local strain distribution under structural load.

Hence, damage can be detected when the measured strain deviates appreciably from the value expected of a healthy structure at the sensor location. This allows one to be able to monitor the structural health of composite components under service conditions.

In this work, we have used FBG sensors for both cure and flow monitoring. Since the curing process of polymeric fluid is exothermic in nature, and residual stresses build up inevitably in curing composites, recording the change in the Bragg wavelength due to thermal and mechanical effects reveals the gelation and vitrification behaviors of the composites. Also, injecting the resin into the RTM mold preheated to curing temperature enables one to monitor the resin front in that room temperature resin when reached to sensor location will change the Bragg wavelength. The details of relevant experiments are provided in section 3. In this work, we have shown the promising potentials of FBGs to monitor flow front as well as composite cure mechanism in the RTM system. Since the embedded sensors become an integral part of the final structure after the curing process, these sensors can be used to gather data about mechanical changes that will affect the performance of the structures.

The second type of optic sensors utilized in this work are etched fiber sensors used to monitor both resin flow front, as well as cure behavior of thermosetting polymeric resin. This sensor operates in the transmission mode such that a light source sends light into one end of the fiber optic and an optical spectrum analyzer (OSA) measures the transmitted light intensity at the other end. Etched fiber sensor is a bare optical fiber with discrete 3-4 mm etched regions where the cladding thickness is reduced. In this etched area, the external medium essentially becomes a part of the fiber waveguide. If the refractive index of the external medium is smaller than the core of the fiber, there will only be a small perturbation to the fiber mode, and a low loss single mode will still be supported. On the other hand, if the refractive index of the external medium is larger than that of the fiber, the propagating mode will be a leaky mode. This leaky mode can be viewed as a decaying mode due to optical leakage into the higher refractive index medium. Since the refractive index of the resin is larger than the fiber core, there will be a significant power drop as the resin reaches the etched area, and this property can be employed to monitor resin flow.

The amount of optical leakage (loss) depends on the cladding thickness, etched portion length, and more importantly the refractive index of the external medium. The etched fiber can be modeled using equations in [34], and it was shown that the amount of loss will increase as the refractive index of the external medium approaches the cladding refractive index. Since the refractive index of curing resin changes during the curing cycle, we have demonstrated that it is also possible to monitor the curing of the RTM manufactured composites with the same etched sensors effectively. As a third methodology utilized for cure monitoring, we have designed and implemented fiber optic Fresnel based refractometer whose working principle and experimental configuration are described in coming section.

CHAPTER 3: EXPERIMENTAL IMPLEMENTATIONS AND RESULTS

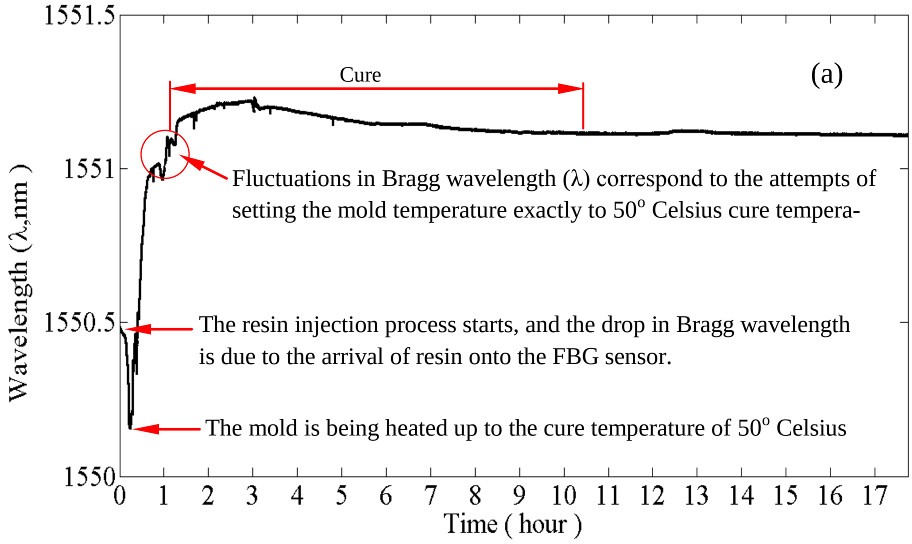
3.1. Flow and cure monitoring with FBG sensors

Several experiments for cure monitoring of RTM manufactured composite plates by using polyamide coated FBG sensors with an outer diameter of 148 μm were performed. The Bragg wavelength shift of the FBG sensor was monitored by a Micron Optics interrogator of the model sm230. Results of three of these experiments are summarized in Figure 6.

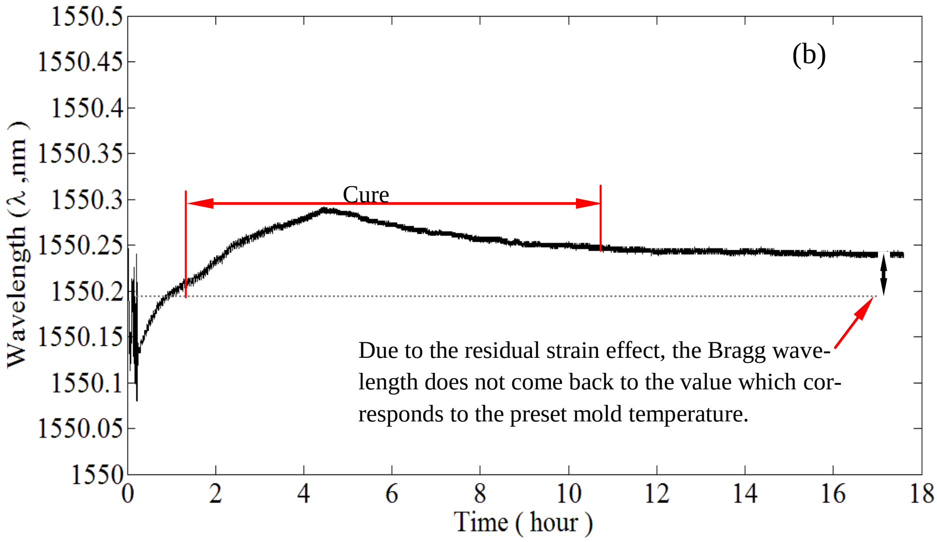
In all three experiments, nine layers of biaxial glass fiber reinforcements are used. FBG sensors are positioned on the fifth ply and fourth ply for the first and the second experiments, and on the seventh ply for the third experiment. In the third experiment three FBG sensors are multiplexed on a single fiber. The center wavelength of FBG sensors are 1549.76, 1549.905, and 1545-1555-1565 nanometers for the first, second and third experiments, respectively. In the first experiment, room temperature resin is injected into the preheated RTM mold (roughly 30 Celsius to remove the moisture). The first sudden drop in Bragg wavelength in figure 6a is due to the arrival of room temperature resin on the FBG sensor, which proves the effectiveness of FBG sensors for resin flow front monitoring. Subsequently, the mold is heated to cure temperature of 50 Celsius, which results in an increase of the Bragg wavelength. The fluctuations in figure 6a correspond to a region where the temperature of the mold was set to the cure temperature. Since the cure process is exothermic in nature, the released heat further increases the center wavelength of the FBG sensors. As the strength of exothermic process diminishes, the water cooling system under the RTM mold attempts to bring the temperature of the curing plate to the preset cure temperature, which is observed in all subfigures of figure 6 as a decrease in the center wavelength of the FBG sensor.

However, given the presence of the residual stress built-up (due to various effects such as thermal gradients, shrinkage, and differentials in thermal expansion coefficients) in the curing composite plate, the Bragg wavelength does not return to level which corresponds to the value observed at the preset mold temperature. This case is much more obvious in figure 6c since the FBG sensor was positioned on the seventh ply (off-neutral axis) while in the first two experiments FBG sensors were placed close to the neutral axis. As the polymerization reaction nears completion, the center Bragg wavelength levels off. The region marked with two vertical lines then corresponds to the curing of the epoxy resin system. The closer the sensor is to the neutral axis, the less strain effect it experiences. Since the strain induced on the structure during the curing process is quite complex in nature, not to preclude the ability of FBG sensors for cure monitoring, FBG sensors should be placed in structures such that it should experience minimum level of residual strain. In the second and third experiments, the resin is injected into a mold heated to 50 degrees Celsius. On the arrival of the room temperature resin to the FBG sensor (at mold temperature), a drop in the wavelength is observed. As the resin gradually reaches the preset cure temperature, the center wavelength of the FBG sensors increases. The horizontal line in figure 6b indicates the preset mold temperature. Regardless of experimental conditions, in all experiments, the tendency of wavelength versus processing time is identical, thereby implying the effectiveness of FBG sensors for cure monitoring for RTM composites.

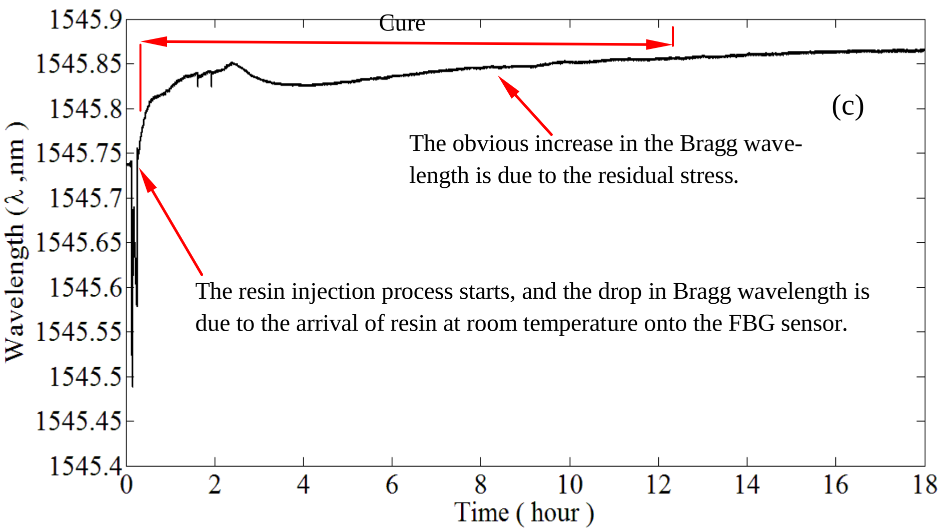
Flow monitoring is achieved with three FBG sensors multiplexed on a fiber optic cable. The arrival of resin is sensed due to the contact of resin at room temperature with FBG sensors at mold temperature. The first FBG was positioned near the inlet the second one towards the middle of the mold, and the last one near the outlet port. Figure 6d indicates the flow front monitoring in RTM system with FBG sensors. To be able to present the changes in the Bragg wavelength of three FBG sensors on the same plot, Bragg wavelength λ_B values recorded during the resin injection process are subtracted from those values $\lambda_{B,o}$ recorded before the injection. The sudden decreases in the normalized Bragg wavelength values (λ_B-$\lambda_{B,o}$) indicate the times when epoxy resin reaches the sensor.



1551.5
1551
1550.5
1550
0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17
Time (hour)
Wavelength (λ,nm)
Cure
(a)
Fluctuations in Bragg wavelength (λ) correspond to the attempts of setting the mold temperature exactly to 50° Celsius cure tempera-
The resin injection process starts, and the drop in Bragg wavelength is due to the arrival of resin onto the FBG sensor.
The mold is being heated up to the cure temperature of 50° Celsius



1550.5
1550.45
1550.4
1550.35
1550.3
1550.25
1550.2
1550.15
1550.1
1550.05
1550
0 2 4 6 8 10 12 14 16 18
Time (hour)
Wavelength (λ ,nm)
(b)
Cure
Due to the residual strain effect, the Bragg wave-length does not come back to the value which cor-responds to the preset mold temperature.



1545.9
1545.85
1545.8
1545.75
1545.7
1545.65
1545.6
1545.55
1545.5
1545.45
1545.4
0 2 4 6 8 10 12 14 16 18
Time (hour)
Wavelength (λ ,nm)
Cure
(c)
The obvious increase in the Bragg wave-length is due to the residual stress.
The resin injection process starts, and the drop in Bragg wavelength is due to the arrival of resin at room temperature onto the FBG sensor.

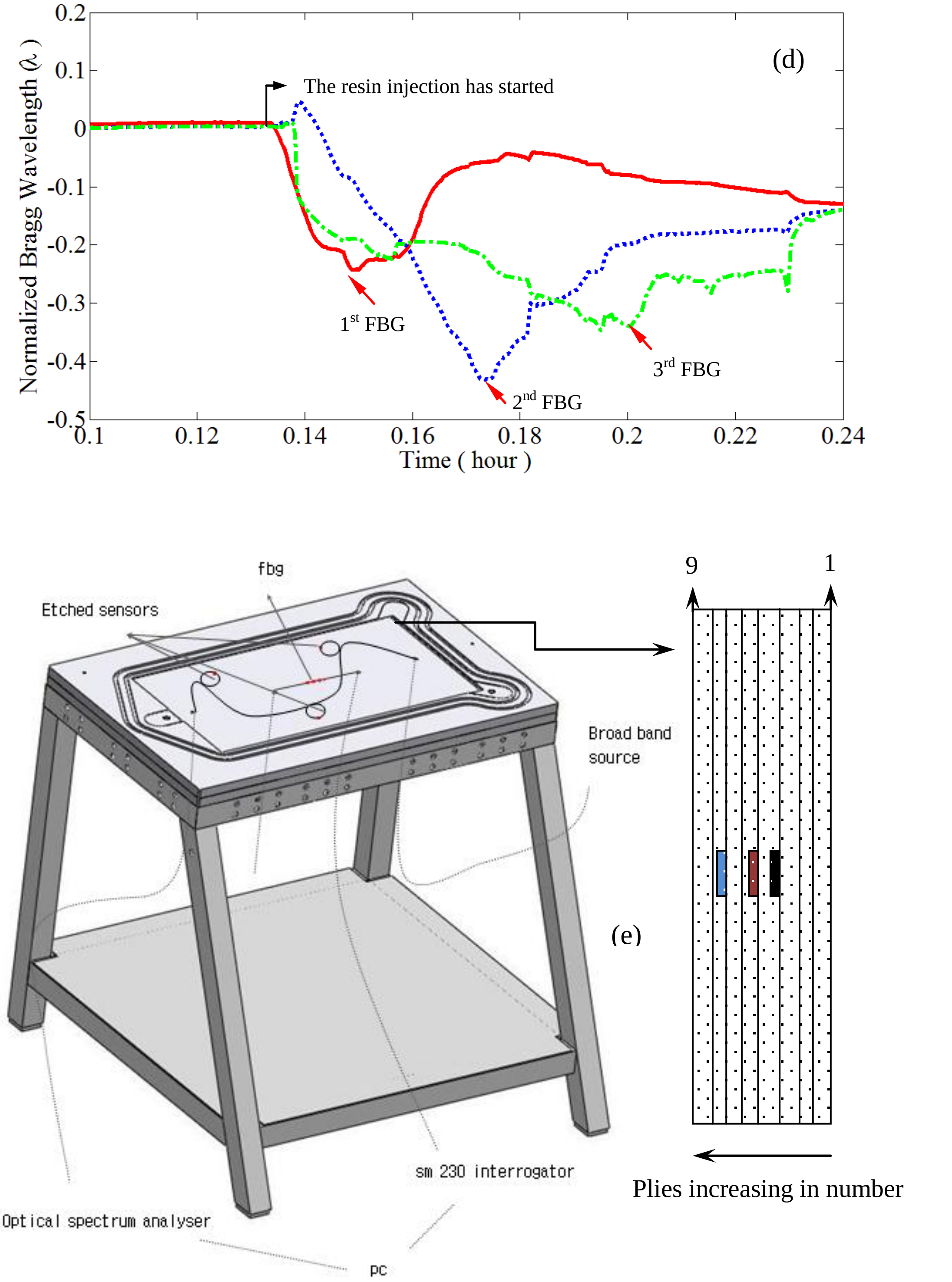


Figure 6. Experimental data for Bragg wavelength versus processing time for cure and flow monitoring by FBG sensors, **a-)** experiment-1, **b-)** experiment-2, **c-)** experiment-3 for cure monitoring, **d-)** experiment-3 for flow front monitoring with three multiplexed FBG sensor array, **e-)** Positions of the FBG sensors for the 1st 2nd and 3rd experiments in 9 plies (Red : 5th , Black : 4th , Blue : 7th experiment), positions of the FBG sensors in the mould

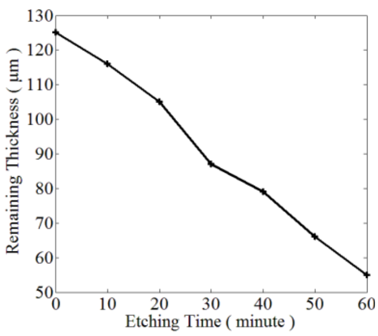
3.2. Flow and cure monitoring with EFS: Fabrication and Characterization

For process monitoring, it is preferable to use a polyamide coated fiber as an etched sensor due to its smaller outer diameter of 148 μm than acrylate coated fiber since it is less intrusive to the composite structure. However, it is more difficult to strip the polyamide coating compared to acrylate. Therefore, for the sake of experimental simplicity in the fabrication of etched sensors, acrylate coated single mode optical fibers (SM-G652D by fujikura, and optomagic) with the coating and cladding diameters of 250 and 125 μm respectively are used. In order to optimize the etched sensor preparation process and characterize the performance of the etched sensors, a number of optical fibers were etched for different time durations. Their remaining thickness and corresponding drop in the transmitted light intensity level when put in contact with the epoxy resin system was measured. Hereafter, the term "*resin system*" is used to refer to the resin-hardener mixture.

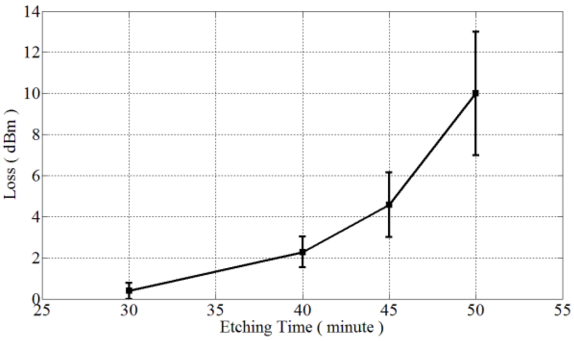
The optical fiber is stripped at sensor locations of interest by 3-4 mm with a mechanical optical fiber stripper. In order to trap light core of the optical fiber(silica) must be surrounded with a smaller refractive index cladding(silica with different ingredients). Generally core of an optical fiber has a refractive index of 1.457 whereas the cladding has a refractive index of 1.455.This difference helps the light travel across the fiber without any loss.When the cladding thickness decreases light can escape from these portions when surrounded by a higher refractive index environment such as resin.This sudden intensity drop can be observed by the optical spectrum and so flow front can be exactly determined.

The stripped section of the optical fiber is looped for two purposes: first, to facilitate the handling process of already fragile optical fiber and second, to increase the bend loss so that light intensity drop due to resin arrival is greater and more easily separated from the noise due to the movement of the sensor . Both ends of the looped sensor are fusion spliced to connectorized pig-tails. The connectorized sensor is connected to a broadband light source at one end and to the optical spectrum analyzer (OSA) (Yokogawa AQ6370B model) at the other end.

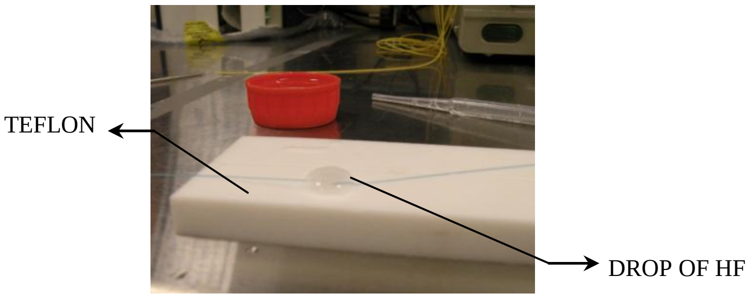
A software interface is written to transfer the data from the OSA to a data acquisition computer. The interface is able to record data continuously at a desired time interval throughout experiments as both the maximum light intensity and total light power. The etching process is conducted on a teflon-jig by forming a small droplet of hydrofluoric acid (38 % HF) on the stripped regions. The teflon-jig has a 3mm diameter spot face that is roughly 5mm deep to contain the HF droplet (Figure 7c). For the quality of the sensor, it should be ensured that the HF droplet encloses the entire volume of stripped sections. During the etching process, the optical fiber is connected to the broad band light source and OSA. Etching process is terminated through washing off the HF with methanol.



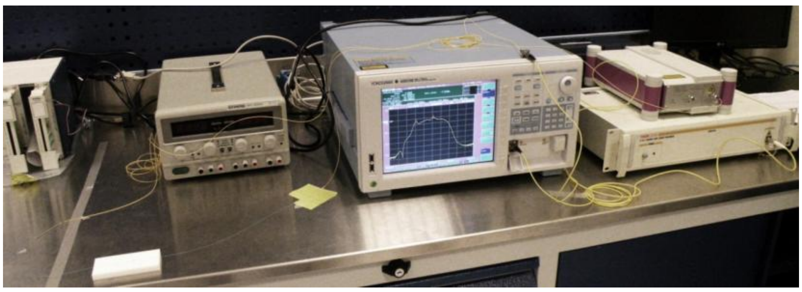
(a)



(b)



(c)



(d)

Figure 7. a-) remaining thickness of the fiber optic cable as a function of etching time, **b**-) the drop in the light intensity of etched sensors as a function of etching time, **c,d**-) optical fiber etching setup

The results of our sensor fabrication and characterization experiments are summarized in figure 7. Figure 7a presents the remaining thickness of the etched fiber as a function of etching time, while Figure 7b shows the drop in the light intensity as a function of etching time. Note that the drop in the light intensity is measured when the etched fiber is inside the epoxy resin system. We have experienced that handling optical fibers without breaking becomes almost impossible if excessive etching is utilized. Therefore, considering figure 7a and 7b together, one can conclude that the remaining fiber thickness of roughly 65-67 μm corresponding to the etching period of 50-55 minutes, which yields an intensity drop of roughly 8-12 dBms is sufficiently large for sensing the resin arrival while still providing handleability. As a result of our experiments to optimize the sensor preparation process as well as characterize the performance of the etched sensors, we have concluded that 50-55 minutes etching time is appropriate. Several etched sensors can be formed (multiplexed) on a length of a single mode optical fiber. There is no restriction on the number of etched sensor that can be created on an optical fiber if the light source is powerful enough. Otherwise, due to the excessive leakage of the light power at sensor locations, the intensity drop might be indistinguishable for each etched sensor out of experimental noise.

To validate the ability of etched sensors for cure monitoring and show that it is a repeatable and reliable process, several hot plate experiments were conducted. The results of four of these experiments are summarized in figure 8. In this direction, several etched sensors are prepared and characterized. In each experiment a single etched sensor is used. The sensor is dipped into resin system in aluminum container at room temperature, and subsequently the resin system is heated to curing temperature of 50 Celsius by the hot plate. The temperature of hot plate is continuously monitored with a thermometer dipped inside the water which is also heated simultaneously on the same hot plate. In one of these experiments, along with monitoring the variation of transmitted light intensity, the thermal history of the curing process is also recorded with a pyrometer as shown in figure 8d.

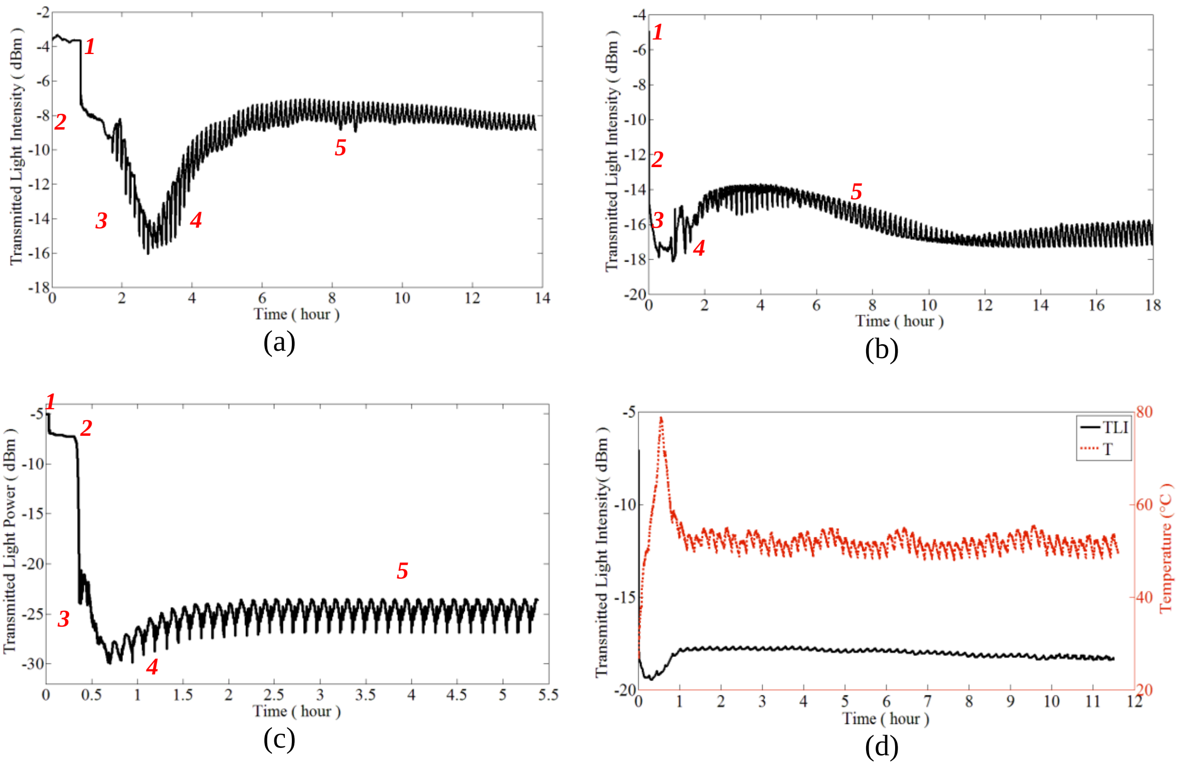


Figure 8. a-d) the variation of transmitted light intensity (TLI) as function of processing time. The regions denoted by numbers in above figures correspond to following stages: **1-)** transmitted light intensity that is recorded when the sensor is in the air. **2-)** the drop in the transmitted light intensity on immersing the etched sensor into the resin-hardener mixture. **3-)** due to the heating of resin to cure temperature, the temperature of the resin system rises up, decreasing the resin refractive index, hence leading to a sustained decrease in the light intensity. **4-)** when the exothermic reaction in curing process starts, the refractive index of the resin mixture starts increasing, hence causing a continuous increase in the transmitted light intensity. **5-)** The transmitted light through the optical fiber decreases gradually because of an increase in the bulk optical loss of the polymerizing resin, which spreads over longer time periods.

As can be seen from figure 8-a, on dipping the etched sensor into a heated resin system by the hot plate, the first intensity drop in the transmitted light is observed due the fact that the etched sensor was put in contact with an environment with a higher refractive index than the core and cladding. Therefore, more light escapes from the etched region. Due to the heating of resin to cure temperature, the temperature of the resin system rises, thereby decreasing the resin refractive index leading to a sustained decrease in the light intensity. It is known that the refractive index of a polymerizing resin changes as the molecular density increases and as the molecular bond number involved in the polymerization processes are altered.

With the initiation of the exothermic curing process, the transmitted light intensity starts increasing due to the increase in refractive index of the resin mixture. As can be clearly seen from figure 8b, the transmitted light through the optical fiber gradually decreases because of an increase in the bulk optical loss of the polymerizing resin, and levels out after roughly 9-12 hours, indicating that cure process is near completion. This cure time is in very good agreement with that observed using FBG sensors. All experimental results support each other. The slight variations between experimental results can be attributed to etched length and thickness of the sensors, and the position of the sensors inside the resin container. The temperature sensitivity of the etched sensors can be noted by looking at the oscillatory behavior of the recorded data. Given that the hot plate used in our experiments has an on and off controller to maintain the preset temperature, these fluctuations in measured data are due to the attempts of the hot plate to adjust the heating power to approach to the preset temperature value. This nature of the hot plate is also confirmed by looking at the pyrometer data in figure 6d which presents the recorded thermal history of the curing process.

To further investigate the ability and effectiveness of etched sensors for resin flow front and cure monitoring in the operational RTM composite manufacturing system, experiments were conducted with 9 layers of biaxial glass fiber reinforcement. Three etched sensors multiplexed on a length of bare optical fiber were located between the fifth and sixth plies in the RTM mold. The sensors are monitored during the resin injection process to detect the presence of resin and possible dry spots. All etched sensors were prepared with the etching time of 50-55 minutes, and each of them is fully characterized during their preparation in terms of the light intensity drop. The first etched sensor is placed near the mold inlet, the second one in the middle of the mold, while the last one was positioned towards the outlet port. For etched sensors, the maximum light intensity was continuously monitored. In the same experiment, an FBG sensor was placed between the same fiber layers in the middle of the mold (see figure 6a).

Figure 9a and 9b indicate the segment of entire the RTM process history recorded by etched optical sensors as given in figure 9c. Recall that the RTM mold in our experimental set-up has a glass window for visual flow monitoring, which is placed on the bottom surface of the top lid of the mold and fixed therein by means of a room temperature vulcanizing aerospace grade silicon. Due to the flexibility of the silicon, when the mold under vacuum, the glass window is sucked towards the mold cavity exerting more force on the etched sensors at their mold ingress and egress points, hence causing a significant drop in the intensity of the light passing through the optical fiber. This situation corresponds to the first drop in transmitted light intensity as shown in figure 9a. At that point, to be able to release forces on the sensors, the vacuuming process is terminated and the air inlet to the mold cavity is allowed. As a result, the transmitted light intensity returns back to its initial level. It should be noted that when the mold is closed after the sensors have been positioned within the mold, the intensity of the transmitted light also drops due to the clamping force which over-bends the optical fiber at its ingress and egress points. On launching the resin injection process, the clamping force that causes a drop in transmitted light intensity is counter-balanced by the positive pressure of the injected resin so that the transmitted light intensity tends to increase slightly.

Once the resin has reached the first etched sensor, the transmitted light intensity drops. As the resin injection proceeds, the clamping force on the optical fiber is further balanced by the pressure of the injected resin, and therefore, the optical fibers are bent less and less at the ingress and egress points. This reflects itself as a slight increase in the transmitted light intensity. When the resin has reached to the second and third sensors, the transmitted light intensity drops again, and then starts increasing again due to the counteracting pressure effect of the resin. Figure 9a clearly shows that it is possible to record the flow process entirely in terms of resin arrival. To be able to create an entire map of the flow process, a number of discrete etched sensors should be used. Having completed the infusion process at room temperature, the curing cycle is launched by heating-up the mold to the curing temperature of 50 Celsius. As concluded from figure 9c, with the data acquisition interface developed, we are able to monitor the composite manufacturing process until it is terminated.

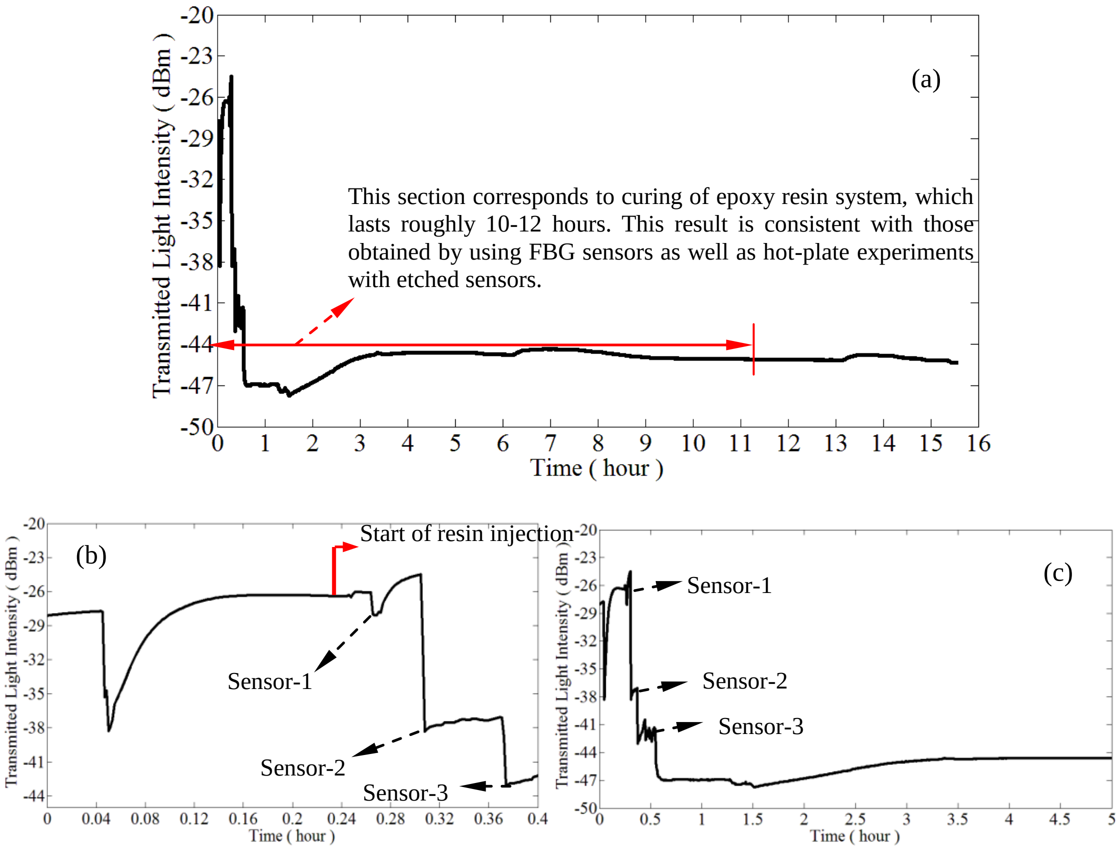


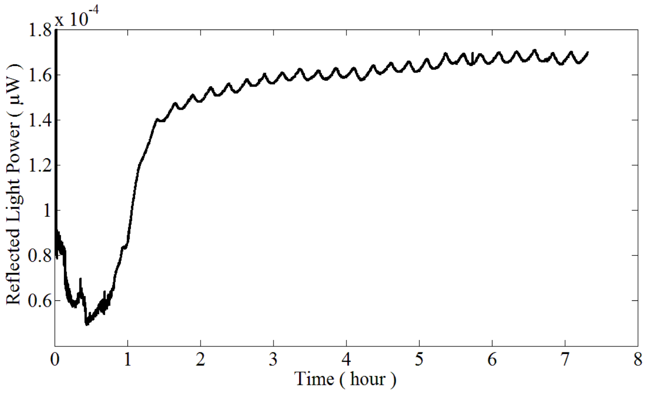
Figure 9. a-) The entire history of the RTM process recorded with etched sensors as a light intensity versus processing time, **b-)** and **c-)** the close-up view for a segment of a light intensity drop versus processing time curve. It should be noted that the drop in the light intensity is sum of those due to three multiplexed etched sensor.

3.3. Cure monitoring with Fresnel Probe

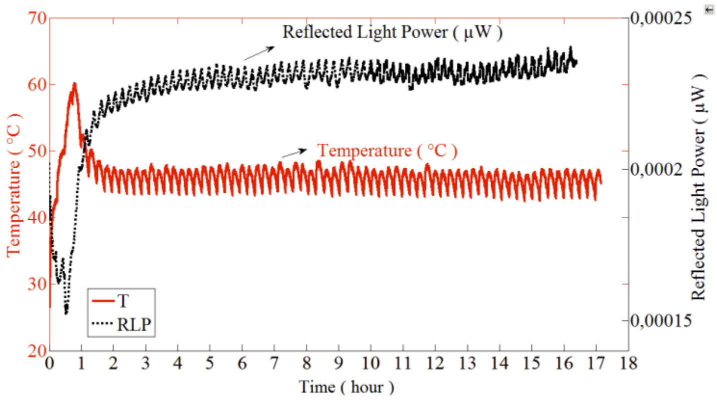
To characterize the refractive index change of resin, hardener, and resin-hardener mixture as a function of temperature as well as monitor the curing of the resin system with a Fresnel probe, a Fresnel reflection based refractometer (FRR) [35] was built. The FRR optic circuit constitutes a broad band light source, an optical circulator and an OSA. A single mode optical fiber connectorized at one end is cleaved at its free end to form a Fresnel probe. The connectorized end is coupled to the second port of an optical circulator with the first and third ports connected to a broad band light source, and OSA, respectively. After inserting the Fresnel probe into the resin-hardener mixture, the mixture is heated to the preset temperature value of nearly 50 degrees Celsius with a simple hot plate without utilizing any specific or constant heating rate. Figure 8 indicates the reflected power as a function of time for two independent experiments. In the experiment given in figure 10b, temperature data was collected concurrently with a pyrometer.

Assuming that the free end of the fiber is cleaved such that the light is at a near-normal incidence to the interface, the reflection coefficient R is given by the Fresnel equation as $R = P_r^s / P_i = \left(n_s - n_f\right)^2 / \left(n_s + n_f\right)^2$, where P_r^s and P_i are powers of the reflected light from the sample ($s = a$ for air, $s = l$ for resin) and the incident light, respectively, and n_s and n_f are the refractive index of sample and fiber in the given order. The refractive index of the liquid medium (resin, hardener, or resin-hardener mixture) can be determined with the above described FRR optic circuit through measuring reflected powers P_r^a, P_r^l when the Fresnel probe is in the air and then in the liquid medium. On writing Fresnel relation for air and liquid medium, and then dividing them to each other, one can write $P_r^l / P_r^a = \left(n_l - n_f\right)^2 \left(n_a + n_f\right)^2 / \left(n_l + n_f\right)^2 \left(n_a - n_f\right)^2$. Taking the refractive index of the air and fiber as 1 and 1.45, respectively, one can compute the refractive index of the sample through the above given relation using measured reflected powers from air and liquid. The experiments revealed that at room temperature refractive index of resin and hardener alone are 1.54 and 1.48 correspondingly while the refractive index of resin and hardener mixture is 1.52 before curing process.

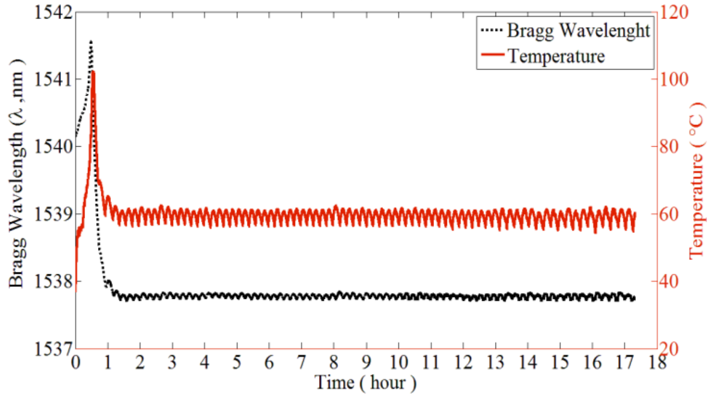
In accordance with the Fresnel relation and taking the refractive index of the fiber as roughly 1.45, when immersing the Fresnel probe into the resin-hardener mixture at room temperature, the reflected power drops significantly from the value measured when the Fresnel probe is in contact with the air to the value in figures 10a and 10b at time zero since $n_l >> n_a$. When heating the resin-hardener mixture with a hot plate, the reflected power gradually decreases. This drop is due to the temperature dependent decrease in the refractive index as the resin system is heated up since at this point the resin and hardener have not mixed thermochemically. Due to the exothermic heating of the resin system, the temperature of the resin system increases more, hence causing additional drop in reflected power.



(a)



(b)



(c)

Figure 10. a-) and **b-)** the record of reflected light power (RLP) and temperature (T) as a function of time. To demonstrate the repeatability of experimental procedure, this experiment was performed several times. Except negligible differences due to experimental origins, results are satisfactorily repeatable. It is to be noted that since we are interested in the tendency of refractive index change due to temperature variation and cross-linking polymerization, measurements are given in terms of reflected power not in terms of real values of the refractive index of the liquid, **c-)** temperature and Bragg wavelength variations as function of time recorded by a pyrometer and an FBG sensor, respectively.

Subsequent to the decrease in the reflected power, there is a rapid increase in the reflected power since the resin system starts experiencing transition from liquid to a rubbery state (gelation). Thus, the sudden increase at the reflected power corresponds to the initiation of exothermic cure reaction. As the exothermic reaction loses its strength, the temperature of the resin system tends to drop down to the preset experimental temperature. The point where ripples have started is considered to be the time when the mixture has nearly reached the 50 degrees Celsius preset temperature. As mentioned previously, these ripples are due to the on-off nature of the hot plate controller. As the cure process continues, the cross-link density of the polymerizing resin increases, resulting in an increase in the reflected power and in turn the refractive index. The point where the graph reaches to steady state is considered as the completion of the curing process to large extent. In view of this, one can see that the steady state has been reached after roughly 9 hours which is in agreement with the cure time measured with both FBG and etched sensors. In one of the experiments, in addition to recording temperature of curing resin with the pyrometer, we have also monitored the temperature variation during curing process with an FBG sensor with a center Bragg wavelength of 1540 nm to validate the occurrence of the sudden rise in temperature of the resin system due to the exothermic curing reaction as shown in figure 10c.

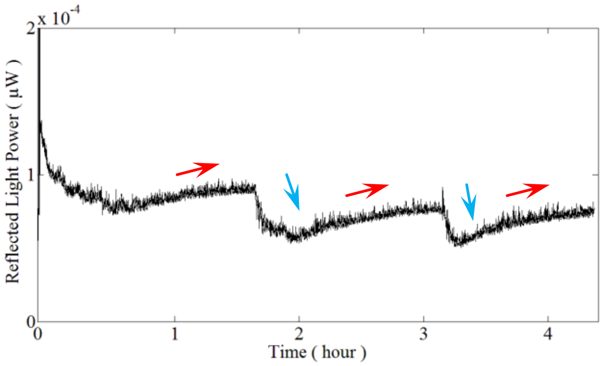


Figure 11. The variation of reflected power for several heating and cooling cycles. Heater is turned off, and the resin is cooling in air (red arrow), then heater is on, resin is heating up (blue arrow). This data shows that the polymer has a negative thermo optic coefficient.

In additional experiments following the above described procedure, the optical behavior of the resin alone has been investigated by measuring reflected power as a function of time corresponding to increase in the temperature of the liquid medium.

Having obtained the significant drop in reflected light power on immersing Fresnel probe into the epoxy resin, the heater was turned on. The heater was preset to 75 degrees Celsius. As the resin temperature increases the reflected power decrease, implying that the refractive index of the resin decreases with an increase in temperature. As shown in figure 11, this experiment is repeated for several heating and cooling cycles by turning off the heater when the preset temperature of 75 Celsius is reached and subsequently turning on again as the temperature decreases down to 30 degree Celsius. In figure 11, the slope of the reflected power versus time graph is shallow when the reflected power is recorded in the cooling mode in the air since heating rate is much bigger than the cooling rate in the air. The refractive index is inversely proportional to temperature for the resin alone.

To validate the cure monitoring abilities of three sensors studied in this work, polymer extraction experiments were performed to determine the degree of cure as a function of process time. Therefore, several samples of the epoxy resin system are prepared and cast in a Teflon mold. These samples are then cured at 50 degree Celsius for different time durations of 2 to 12 hours. Having removed a sample from the mold, its mass is measured and subsequently wrapped with tissue paper, and put into the solution of tetrahydrofuran solvent. The solvent removes the uncured epoxy resin over a period of 24 hours, leaving the fully cured part behind. After taking the remaining sample out of the extraction machine, it is dried inside an oven for some time. The final mass of the sample is measured to determine the percentage of cure. The results of the extraction experiments are summarized in figure 12. From this figure it could be concluded that this specific epoxy resin system completes its cure period in almost 9-12 hours. This result is consistent with the data obtained with the optical sensors.

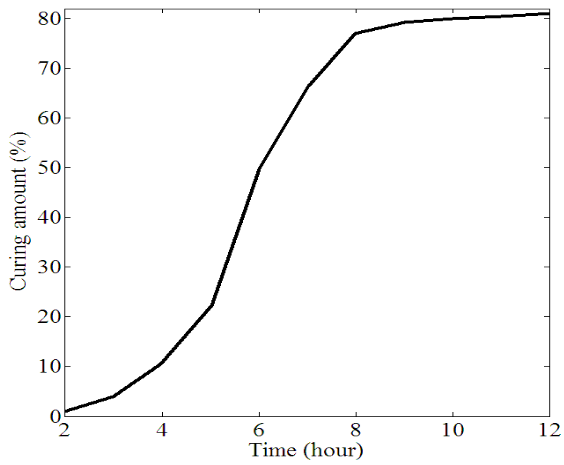


Figure 12. The degree of cure as a function of time.

CHAPTER 4: Conclusion and Future Work

4.1. Conclusion

FBG sensors possess several advantages as flow, cure and subsequently structural health monitoring sensors in composite structures because of their small size, simple structure, light weight, corrosion resistivity, immunity to electromagnetic field interference, multiplexing capability, and long service life. They can be embedded into the structure and act like a part of the system without changing any mechanical characteristics. Etched fiber sensors provide data about the resin flow front ensuring that the mold is fully saturated. With the help of many etched fiber sensors embedded throughout the mold the operator can understand where and where not the resin has reached. In addition to being used for resin flow monitoring in the mold filling process, these sensors can be used for determining the degree of cure, which is required to find out the optimal time for de-molding. In this work the systematic experimental studies for process monitoring (cure and flow monitoring) for RTM manufactured composite materials is presented. The ability of etched sensors for resin flow front monitoring as well as cure monitoring in operational RTM process is demonstrated. Since the refractive index of the resin-hardener mixture is a function of temperature, and the degree of cross-linking, the transmitted light intensity through fiber with etched sensor varies depending on the environment surrounding the etched segment of the fiber, thereby enabling one to monitor the curing process of polymeric resin. This conclusion has been correlated with cure monitoring results from FBG sensors as well as a Fresnel probe. It has been demonstrated that FBG sensors can also be effectively used for tracking resin flow front. In conclusion, etched and Fresnel sensors can be used as a simple, cost effective alternative to FBG sensors for process monitoring in composite materials manufacturing. The experimental results presented show the potential and the efficiency of the three optic sensors for providing real time information about the processing stages of RTM composite manufacturing systems.

4.2 Future Work

In light of the research presented in this thesis work, followings are issues which can be addressed as a continuation of the current project.

- The etched fiber sensor preparation should be automatized for better response from the sensors
- The ingress and egress of optical sensors into composite parts produced by closed mold process should be improved for industrial level usage.
- The cost of EFS is quite low when compared to FBG sensors. Hence, EFS could be used in other polymer processing industries such as rubber industry in order to determine the optimal curing time of different polymeric compounds and monitor the over all-curing process.
- Fresnel refractometers could be used to gather data about the refractive indices of different materials. Like EFS, they could also be used in industry to monitor curing process of polymeric materials.

APPENDIX A

RTM EXPERIMENTS

No	Objective of the Experiment	Types of Sensors	T	Notes
1	Optimization of the parameters of the mould	-	50°C	High amount of wash out
2	Integration of Electrosprayed Carbon nano tubes into the structure	Bare Optical Fibers	50°C	Homogenous distribution of CNTs
3	Integration of Bare OFs and electrospun CNT into the RTM mold without damage	Bare Optical Fibers	50°C	CNTs were stuck on optical fibers
4	Integration of Bare Optical fibers into the Mould	Bare Optical Fibers	50°C	One of the OFs was broken
5	Optimization of the Ply number	-	50°C	7 Biaxial Plies resulted in no wash out
6	Flow Monitoring	3 Multiplexed ES	50°C	ES broken
7	Flow Monitoring	3 Multiplexed ES	50°C	ES broken
8	Flow Monitoring	3 Multiplexed ES	50°C	Intensity Data gathered
9	Flow and Cure Monitoring	3 Multiplexed ES , FBG	50°C	Simultaneosly ES data and interrogator data gathered
10	Flow and Cure Monitoring	Etched Sensor , FBG	49°C	Fibers broken
11	Flow Monitoring with FBG s	FBG, Strain gages	50°C	Residual Strain effect observed on FBG sensors

References

[1] Raymond M. Measures. Structural monitoring with fiber optic technology, Academic Press, 2001.

[2] Bekir Yenilmez, E. Murat Sozer. A grid of dielectric sensors to monitor mold filling and resin cure in resin transfer molding. Composites: Part A 40 (2009) 476–489

[3] Lawrence JM, Hsiao KT, Don RC, Simacek P, Estrada G, Sozer EM, et al. An approach to couple mold design and on-line control to manufacture complex composite parts by resin transfer molding. Compos A: Appl Sci Manuf 2002;33(7):981–90.

[4] Danisman M, Tuncol G, Kaynar A, Sozer EM. Monitoring of resin flow in the resin transfer molding (RTM) process using point-voltage sensors. Compos Sci Technol 2007;67(3–4):367–79.

[5] Barooah P, Berker B, Sun JQ. Lineal sensors for liquid injection molding of advanced composite materials. J Mater Process Manuf Sci 1999;6(3):169–84.

[6] Fink BK, Walsh SM, DeSchepper DC, Gillespie Jr JW, McCullough RL, Don RC, et al. Advances in resin transfer molding flow monitoring using SMART weave sensors. In: Proceedings of ASME, international mechanical engineering congress and exposition. San Francisco, CA, 1995;69(II):999–1015.

[7] Bradley JE, Diaz-Perez J, Gillespie Jr JW, Fink BK. On-line process monitoring and analysis of thick-section composite parts utilizing SMARTweave in situ sensing technology. In: Proceedings of international SAMPE symposium and exhibition; 1998. p. 254–67.

[8] McIlhagger A, Brown D, Hill B. The development of a dielectric system for the on-line cure monitoring of the resin transfer molding process. Compos A: Appl Sci Manuf 2000;31(12):1373–81.

[9] Hegg MC, Ogale A, Mescher A, Mamishev AV, Minaie B. Remote monitoring of resin transfer molding processes by distributed dielectric sensors. J Compos Mater 2005;39(17).

[10] Kim HG, Lee DG. Dielectric cure monitoring for glass/polyester prepreg composites. Compos Struct 2002;57(1–4):91–9.

[11] Bang KG, Kwon JW, Lee DG, Lee JW. Measurement of the degree of cure of glass fiber epoxy composites using dielectrometry. J Mater Process Technol 2001;113(1–3):209–14.

[12] Rath M, Doring J, Stark W, Hinrichsen G. Process monitoring of moulding compounds by ultrasonic measurements in a compression mould. NDT&E Int2000;33(2):123–30.

[13] Schmachtenberg E, zur Heide Schulte J, Topker J. Application of ultrasonics for the process control of Resin Transfer Moulding (RTM). Polym Testing 2005; 24(3):330–8.

[14] G R Powell, P A Crosby, D N Waters, C M France, R C Spooncer and G F Fernandok. In-situ cure monitoring using optical fibre sensors-a comparative study. Smart Mater. Struct. 7 (1998) 557–568.

[15] Woerdeman DL, Parnas RS. Model of a fiber-optic evanescent wave fluorescence sensor. Applied Spectroscopy 2001;55:331–7.

[16] Woerdeman DL, Spoerre JK, Flynn KM, Parnas RS. Cure monitoring of the liquid composite molding process using fibre optics sensors. Polymer Composite 1997;18:133–50.

[17] Afromowitz MA. Fiber optic polymer cure sensor. Journal of Lightwave Technology 1988;8:1591–4.

[18] Lam KY, Afromowitz MA. Fiber optic epoxy composite cure sensor: I. Dependence of refractive-index of an autocatalytic reaction epoxy system at 850 nm on temperature and extent of cure. Applied Optics 1995;34:5635–8.

[19] Lam KY, Afromowitz MA. Fiber optic epoxy composite cure sensor: II. Performance-characteristics. Applied Optics 1995;34: 5638–44.

[20] S J Buggy, E Chehura, S W James and R P Tatam. Optical Fibre Grating Refractometers for Resin Cure Monitoring. Journal of Optics A: Pure and Applied Optics, Volume 9, Number 6, June 2007 pg S60)

[21] Liu YM, Ganesh C, Steele JPH, Jones JE. Fiber optic sensor development for real-time in-situ epoxy cure monitoring. Journal of Composite Materials 1997;31:87–102.

[22] Chailleux E, Salvia M, Jaffrezic-Renault N, Matejee V, Kasik I. In situ study of the epoxy cure process using a fibre-optic sensor. Smart Materials and Structures 2001;10:194–202.

[23] Cusano A, Breglio G, Giordano M, Calabro A, Cutolo A, Nicolais L. Optoelectronic characterization of the curing process of thermoset-based composites. Journal of Optics A: Pure and Applied Optics 2001;3:126–30.

[24] Chensha Li, Maosheng Cao, Rongguo Wang, Zhengping Wang, Yingjie Qiao, Libing Wan, Qiu Tian, Haitao Liu, Deqing Zhang, Tongxiang Liang, Chenhe Tang. Fiber-optic composite cure sensor: monitoring the curing process of composite material based on intensity modulation. Composites Science and Technology 63 (2003) 1749–1758

[25] Raman Kashyap . Principles of Optical Fiber Grating Sensors. Fiber Bragg Gratings (Second Edition), 2010, Pages 441-502.

[26] J. A. Guemes, J. M. Menéndez. Response of Bragg grating fiber-optic sensors when embedded in composite laminates. *Composites Science and Technology 62 (2002) 959–966*

[27] R.M.Measures. Structural Monitoring with Fiber Optic Technology. Academic Press, 2001

[28] Dunkers J.P., Lenhart J.L., Kueh S.R., Van Zanten J.H., Advani S.G., Parnas R.S. Fiber optic flow and cure sensing for liquid composite molding (2001) Optics and Lasers in Engineering, 35 (2), pp. 91-104.

[29] Dweynter-Marty, V., et al, (1998). Embedded fiber bragg grating sensors for industrial composite cure monitoring, J. Intell. Mater. Syst. Struct., Vol. 9.

[30] Murukeshan, V.M., Chan, P.Y., Ong, L.S., Seah, L.K., (2000). Cure monitoring of smart composites using fiber Bragg grating based embedded sensors, Sens. Actuators. A Phys., 79, 153-161.

[31] Jung, K., Kang, T.J., (2007). Cure monitoring and internal strain measurement of 3-D hybrid braided composites using fiber Bragg grating sensor, J. Compos. Mater., V. 41, No. 12.

[32] Casey J. Keulen, M. Yildiz and A. Suleman: “Fiber Optic Sensors for Flow and Health Monitoring of Resin Transfer Molded Composite Structures”, Composites

2009, 2nd ECCOMAS Thematic Conference on the Mechanical Response of Composites, 1-3 April 2009, Imperial College London, UK

[33] R.M.Measures. Structural Monitoring with Fiber Optic Technology. Academic Press, 2001.

[34] Anurag Sharma, Jacintha Kompella, And Prasanna K. Mishra. Analysis of Fiber Directional Couplers and Coupler Half-Blocks using a New Simple Model For Single-Mode Fibers. Journal Of Lightwave Technology, Vol. 8, No. 2, February 1990.

[35] Kim C B and Su C B 2004 Measurement of the refractive index of liquids at 1.3 and 1.5 micron using a fibre optic Fresnel ratio meter *Measurement Science and Technology* **15** 1683-86