


Applicability of fiber Bragg grating sensors for cure monitoring in resin transfer molding processes

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Abstract

This article examines the use of fiber Bragg grating sensors for cure monitoring purposes in resin transfer molding processes. Within a resin transfer molding test series a thermoset epoxy-amine resin system was used in combination with a woven flax fiber reinforcement. Particular attention was paid on the location of the optical fiber sensor and its sensitive Bragg grating element inside the mold cavity. Three different installation approaches were tested and the correlation of the corresponding strain response with the actual cure state of the resin system was investigated at 50°C and 70°C isothermal cure temperature, respectively. We could demonstrate that characteristic, conspicuous strain changes are directly related to the sol–gel conversion of the thermoset polymer, which was analyzed considering different approaches for the gel-point detection based on rheological measurements. With the installation of the sensor inside a controllable, capsuled resin volume, we could achieve the most reliable strain response that provides capabilities to give in-situ information of the cure state beyond the gelation point.

Keywords

Fiber Bragg grating (FBG) sensor, thermosetting resin, cure behavior, resin transfer molding (RTM)

Introduction

The manufacturing of fiber-reinforced composites using liquid composite molding (LCM) processes has been established as one of the main techniques for the production of resilient structural parts.¹ Such processes are often influenced by interconnected process inputs affecting the impregnation of the reinforcing textile structure and the irreversible cross-linking reaction of the thermoset polymer. Unsuitable process parameters and defective process control result in potential quality problems for the final composite, like an incomplete impregnation, a poor surface quality, an under-cured resin or unnecessarily excessive cycle times.² Such sophisticated process technologies can be improved in terms of quality and reliability when meaningful in-situ data can be obtained during the process. Various in-line sensor technologies and applications have already been developed and discussed for monitoring applications.^{3,4} One group of sensors is represented by optical fibers, which uses in general optical interferometric effects. One of the most cited and used optical fiber sensors for measurement and monitoring tasks in

connection with polymer composites is the fiber Bragg grating (FBG) sensor.⁵ That is likely due to specific advantages, as they provide a simple design, a small size and are suitable for integration into composite materials with minimal perturbation to the reinforcing fibers.⁶ FBG sensors are coated, optical fibers with a usual total diameter of 100–200 µm. A Bragg grating means physically a pattern of alternating fiber sections with high and low refractive indices typically within a length of 1–20 mm, which reflects a defined wavelength

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of a light spectrum guided into the fiber.^{7,8} An optical interrogator is used as light source and measurement device for the reflected wavelengths. Applied strain in the FBG sensor leads to an elongation or compression of the grating pattern and therefore to a wavelength shift. Thus, the FBG sensor is suitable for measuring the actual strain at the Bragg grating locations that is caused by mechanical external loads or by thermal expansion or contraction due to temperature changes. Doyle et al.⁶ presented in their study an overview of optical fiber sensors not just in terms of their capability for process monitoring tasks but also for a condition monitoring of the composite afterwards. The use of FBG sensors for structural health monitoring (SHM) of fiber-reinforced composites is well established yet and their potential has been shown in numerous investigations.^{9–13} The additional use of the signal response of the same optical fiber sensors during the processing of the composite part in terms of process control can be seen as a relevant added benefit. Therefore, the use of the FBG strain response in terms of cure monitoring in resin transfer molding (RTM) processes is investigated within this study.

The application of FBG sensors for in-situ data acquisition in LCM processes was focused in several previous studies,^{14–20} primary with the aim to investigate correlations of the signal response with the resin cure state, to measure the coefficient of thermal expansion, to detect process-induced residual strains in the final part or to prepare the sensor-integrated composite for subsequent SHM purposes. In the majority of the studies, the strain responses of the FBG sensors are mainly affected by temperature changes, due to external heat/cool cycles or exothermal reaction heat. In contrast, the present study focuses on the strain development due to the chemical transition of the polymer from liquid to solid in isothermal conditions and analyze the capabilities for providing online information of the actual cure state during RTM processing based on the acquired strain data. This is done by using different strategies for the sensor installation considering the variation of the immediate surrounding of the Bragg grating from a direct contact with the textile reinforcement to a location of neat resin and inside a capsuled resin volume, which allows a direct comparison of the related strain responses in terms of reliability and reproducibility.

Within the studies from Harsch et al.,^{21–24} FBG sensors played a major role in the analysis of cure-induced stresses in filled and unfilled epoxy systems. A fiber optical sensor method was developed based on a laboratory test setup to measure the strain development in the cross-linking polymer. Antonucci et al.²⁵ did similar investigations to analyze the cure-induced strain build-up inside an epoxy resin using FBG sensors. A good

agreement between the acquired strain data and accompanied simulation results was observed. Another study with an abstracting experimental setup was conducted by Karalekas et al.²⁶ who investigated residual stresses and strains inside a cylindrical epoxy sample with embedded FBG sensors resulting from subsequent thermal cycles. These studies demonstrated the capability of fiber optic sensor in providing valuable information on the resins cure state, but outside of any production environment.

The use of FBG sensors for RTM process monitoring was part of the investigations from Chiesura et al.^{17,16} and Yildiz et al.²⁷ Within these studies FBG sensors were embedded in exemplary RTM processed parts and used as in-situ sensors for process monitoring during injection, curing and post-cure processes. The capability for detecting the resin flow during injection, temperature changes, and residual strains inside the part were shown, but a reliable tracking of the cure state based on the FBG signal could not be provided due to inconsistencies of the strain responses.

The present study examines, whether the cure-induced strain measurement with FBG sensors within an RTM process can be conducted in a manner to give valuable in-situ feedback of the actual cure state of the resin system under process conditions by analyzing different installation approaches for the optical sensor. This was done with particular attention to the reproducibility of the signal behavior as well as to their generality by varying the isothermal cure temperature within our series of RTM experiments.

Experimental

Materials

The reinforcement fabric used for the composite manufacturing was a woven flax-fiber textile (Biotex Flax 400 g/m² 2/2 Twill) supplied by Composites Evolution (Chesterfield, UK). The matrix system for the RTM test series was a two-component diglycidylether of bisphenol A (DGEBA) epoxy system for infusion processes (Epinal LR 80-A2.01) provided by bto-epoxy GmbH (Amstetten, Austria). With the given diamine-based hardener (Epinal LH 80-B2.00) the resin system provides a pot life of 60 min at room temperature and cures within a recommended temperature range between 25°C and 90°C. The mixing ratio for the resin and hardener component is 100:25 by weight according to the manufacturer specification.

Sensors

Sylex FFA-01 FBG sensors (Sylex, Bratislava, Slovakia) were used for the strain measurement which

provides a measurable strain range of $\pm 3000 \mu\epsilon$. The sensor configuration was chosen with a total fiber length of 3 m, a single Bragg grating located in the middle of the optical fiber and a defined wavelength within the range between 1510 and 1590 nm. In general, there is also the possibility to apply multiple Bragg gratings in an array design along the optical fiber to enable the strain measurement at different positions at once. The sensors used for the test series are usually used for direct gluing on top of the measured surface. According to the supplier's recommendations epoxy and cyanoacrylate adhesives systems are suitable for the mounting of the polyimide-coated optical fiber. In the present case, the embedding of the fiber in the epoxy resin system was done without any pretreatment of the fiber surface, since pretests with stripped off coatings around the Bragg gratings did not show any quality improvements of the measured strain data but significantly increased the probability for sensor failures. The active Bragg grating length in the present case is $< 5 \text{ mm}$. The data acquisition was conducted by a Sylex S-Line Scan 800 interrogator with an acquisition rate of 1 Hz.

Additional temperature measurements inside the mold were done using the data acquisition system TC-08 from Omega Engineering (Deckenpfronn, Germany) in combination with type-K thermocouples (TT-KI-40) with outer wire dimensions of $0.6 \text{ mm} \times 1.0 \text{ mm}$.

RTM process setup

The woven textile layers were cut and dried in a convection oven (Model FDL 115, Binder GmbH, Germany) at 120°C for approximately 2 h directly before the processing. Afterwards, the textile stack, consisting six individual layers, was positioned in the preheated RTM mold with a cavity dimension of $270 \text{ mm} \times 270 \text{ mm} \times 4 \text{ mm}$. In addition, the FBG

sensors and additional thermocouples were installed, which is described in detail in the following section. The mold carrier used for the RTM test series was a laboratory press (LZT-OK-80-SO) from Langzauner (Lambrechten, Austria). After the preparation of the preform and the sensors, the two components of the thermoset epoxy-amine system were mixed and subsequently degassed for approximately 15 min using a desiccator and a vacuum pressure of 50 mbar at room temperature. The injection of the resin was done using a pressure pot with an injection pressure of 2 bar. The RTM mold provides a resin distribution channel across the cavity width at the inlet side and two vents at the opposite side, which were open during the resin injection. After the mold was filled and excessive resin reached the outlet, the venting tubes were closed and a post pressure of 2 bar was maintained by the pressure pot during the curing process. The injection and the curing step were performed under isothermal conditions at 50°C and 70°C , respectively. After a suitable cure state was reached, the composite plate with the integrated FBG sensors was demolded. During the manufacturing process, starting with the closing of the mold until the demolding step, the signals of the FBG sensors and additional thermocouples were recorded using an acquisition rate of 1 Hz.

Approaches for the FBG sensor integration

Particular attention was paid regarding to the FBG sensor location inside the mold and the local surrounding next to the sensitive Bragg grating element. Three different methods were evaluated, which are illustrated in Figure 1 and described in the following:

Approach A. Within this installation approach, the FBG sensor is placed inside the preform between the 5th and 6th layer of the laminate and represents the most

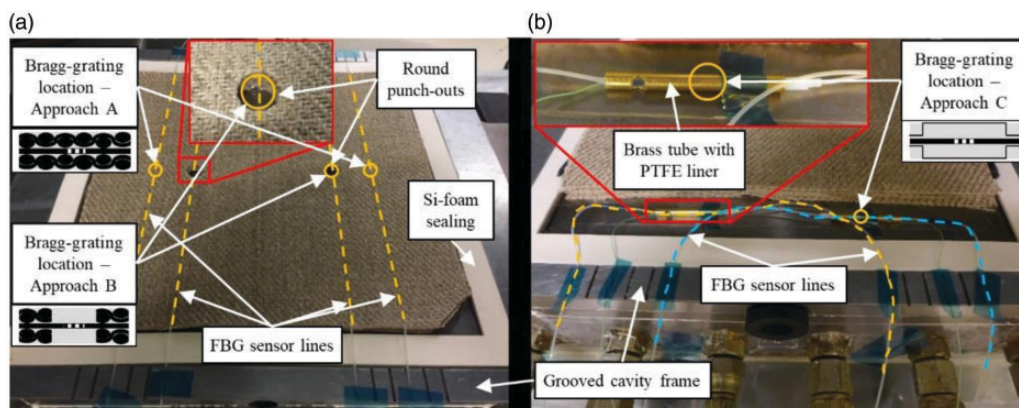


Figure 1. View into the RTM mold cavity: three different approaches for the installation of the FBG sensor (approaches A and B in experiment setup (a), approach C in experiment setup (b)).

common way for embedding optical fibers which is then an intrinsic part of the composite structure. Inside the textile reinforcement the FBG sensor serves as an integrated strain sensor, which can be used for the purpose of condition and health monitoring of the final composite structure. But for monitoring tasks during the production process the interaction between the textile reinforcement and the optical sensor can cause detrimental effects in terms of measurement accuracies due to the undefined conditions of the sensor-reinforcement-contact and the higher complexity of the cure-induced strain development due to the presence of the fibrous phase in the immediate surrounding.

Approach B. To avoid any preloads on the sensor due to compaction mechanisms during the mold closure and to examine the influence of the textile reinforcement on the strain development, the Bragg grating is positioned in locations, which were not directly in contact with the reinforcing component. Therefore, the textile preform was prepared with round punch-outs in which the Bragg grating is positioned afterwards. Those spaces were filled with neat resin material during the injection process. The advantage of this approach is the absence of direct textile related effects on the sensing Bragg grating location. Since the local removal of the textile reinforcement to create a neat resin area would not be acceptable within the composite component, such Bragg grating must be positioned, e.g. in locations which are trimmed afterwards or in neat resin areas, e.g. near the resin inlet(s) and outlet(s). Thus, as no intrinsic part of the composite structure the use of the sensing Bragg element as strain sensor afterwards is not

possible in this case. For the experiments in this study the punch-outs are located beside the Bragg gratings from approach A to create comparable conditions in terms of resin arrival times and temperatures.

Approach C. The third approach goes one step further with the objective to obtain better control of the local surrounding next to the Bragg grating location. Therefore, the sensitive part of the optical fiber is encapsulated in a small brass tube with an integrated PTFE liner. In Figure 2 the corresponding setup is depicted schematically. During the injection process, the empty space of the tube-shaped control volume is filled with resin, which enters the tube through the inlet hole and flows along the resin outlet tube. After excessive resin reaches the end of the outlet tube, which is located outside of the mold, the tube is clamped with a hose clamp. This sensor design aims to obtain an improved reproducibility of the cure-induced strain development by providing a defined sensor position inside a controlled resin volume and a non-adhesive PTFE liner as contact surface for the thermoset material with the immediate surrounding. A type-K thermocouple is positioned inside the control volume to check the compliance of isothermal conditions during cure. Analog to approach B the Bragg gratings which are located inside the tube have the single purpose for cure monitoring during the process. This is reasonable, since higher accuracy and reliability may be expected for the task of cure-monitoring. The application of the FBG sensor for health monitoring tasks afterwards can be obtained for approaches B and C by using sensors with multiple Bragg gratings, which allows additional positions

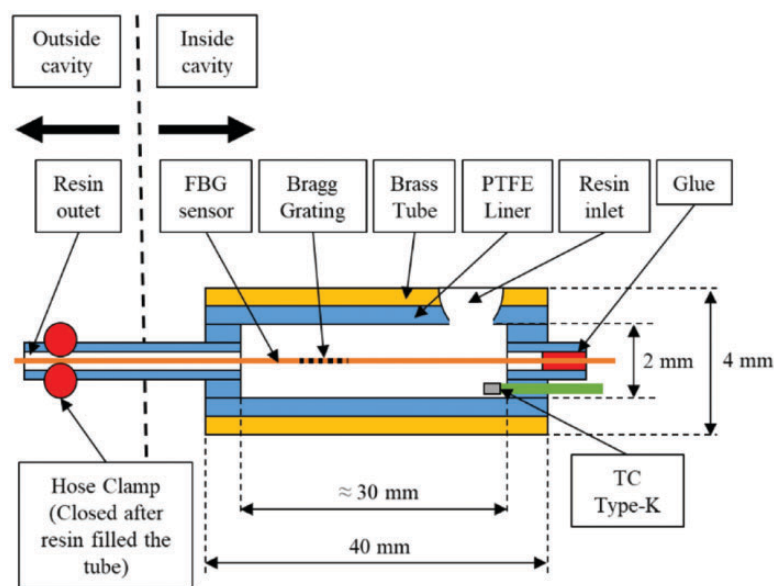


Figure 2. Schematics of the FBG sensor design of approach C.

inside the composite structure. In connection with the sensor installation, the bigger dimensions compared to the single optical fiber must be considered. In many RTM processes, suitable locations are provided at resin inlet and outlet areas or at locations of the composite part which are trimmed afterwards similar to approach B.

In the course of the RTM process preparation, the optical fibers and additional thermocouple wires are guided into and out of the mold, using a metal cavity frame with 1 mm × 1 mm sized grooves, as can be seen in Figure 1. The sealing is maintained by using two silicone foam cuttings between the grooved frame and the cavity. To protect the FBG sensor from any damage a PTFE tube is used to cover the optical fiber where it is not an intrinsic part of the composite plate. The FBG sensor strain was set to zero after they were installed. Afterwards the mold is closed, accompanied by the compaction of the textile preform, and the resin is subsequently injected.

Cure characterization of the epoxy-amine system

To get detailed insight regarding the actual properties of the thermoset matrix system during the cure process, rheometric and calorimetric measurements were carried out and analyzed. The results allow the comparison of the FBG sensor data, acquired during the process monitoring, with the associated cure degree and characteristic points associated with the sol–gel transition.

Rheological analysis

Rheological measurements were done on an MCR 501 rheometer (Anton Paar GmbH, Graz, Austria) using a parallel plate configuration with a plate diameter of 50 mm and a gap height in the range between 0.727 and 1.597 mm. In isothermal conditions, an oscillatory mode was used with an angular frequency of 10 rad/s and a strain amplitude of 30%. Temperatures of 50°C and 70°C were used, respectively, to provide analogous conditions compared to the process temperatures used in the RTM experiments. Additional measurements at

55°C, 60°C, and 65°C allow an accurate evaluation of the temperature dependent behavior. Due to the variety of existing approaches to define the gelation point of a curing thermoset system based on rheological measurement data, special attention was put on the selection procedure in this study. Therefore, the most common criteria, which can be found in literature, were applied and discussed.

Based on the resulting curve progressions for the storage modulus G' , the loss modulus G'' , the loss factor $\tan\delta$ or the normal force F_n , the following criteria for the determination of the gelation point have been established:

- (i) Intersection point of the storage modulus G' and the loss modulus G'' ^{28,29}
- (ii) Peak of the curve of the loss factor $\tan\delta$ ^{30–32}
- (iii) Onset point of the increase of the storage modulus G' ³³
- (iv) Onset point of the normal force drop F_n ^{22,34}
- (v) Point of $\tan\delta$ characterized by frequency independence^{35,34}

With the exception of the latter, which could not be applied due to the defined test procedure using a fixed frequency, the detected times for the gel points depending on the criteria (i) to (iv) are listed in Table 1.

The corresponding curves for the storage modulus G' , the loss modulus G'' , the loss factor $\tan\delta$, the normal force F_n and the dynamic viscosity η_{dyn} are depicted in Figure 3, exemplarily for an isothermal cure temperatures of 50°C. The vertical lines mark the evaluated time points for the gelation based on the underlying criteria. The experiments at 50°C and 55°C showed a sudden signal drop after 87.4 and 62.6 min, respectively, before the $G' - G''$ crossover point was reached.

The comparison of the time values indicates that the gelation times based on criteria (ii) and (iii) result in an earlier time range than the time values based on criteria (i) and (iv) within the tested temperature range. The shortest gelation times for all tested temperatures result from the $\tan\delta$ -peak criterion (ii), while the

Table 1. Gelation times depending on different determination criteria. (Time values in brackets were detected after a sudden signal drop appeared during test.)

		Cure temperature (°C)				
	Criteria	50	55	60	65	70
Gelation time (min)	(i)	(87.6)	(64.4)	44.7	33.6	23.9
	(ii)	60.0	45.1	33.8	26.6	19.9
	(iii)	61.4	46.0	34.4	27.1	20.3
	(iv)	83.3	61.3	43.0	32.5	23.5

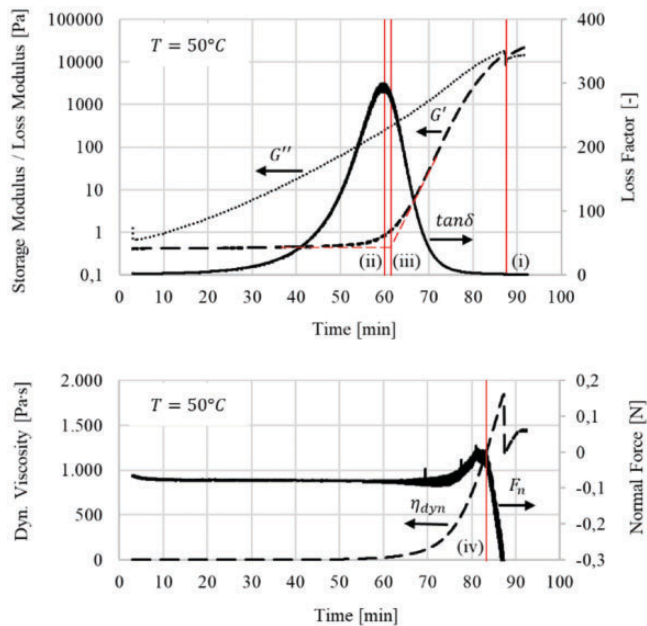


Figure 3. Rheological measurement results for an isothermal cure temperature of 50°C and detected gelation times based on criteria (i) to (iv).

highest values correspond to the G' – G'' -intersection criterion (i).

Dynamic scanning calorimetry (DSC) analysis

DSC experiments were carried out on a Q2000 device from TA Instruments (New Castle, DE, USA). An isothermal test approach at 50°C and 70°C was chosen, to represent the cure situation in the RTM process. The measurement times were set to 24 and 14 h for the isothermal cure at 50°C and 70°C, respectively. All measurements were conducted under nitrogen atmosphere using a N_2 gas flow of 50 ml/min. Based on the corresponding heat flow, the cure degree over time was evaluated using equation (1)

$$\alpha = \frac{\Delta H(t)}{\Delta H_T} \quad (1)$$

where ΔH_T is the total reaction heat and $\Delta H(t)$ the partial reaction heat from experiment start until time t . The total reaction heat was evaluated based on the results from isothermal measurement at 50°C and a subsequent dynamic heat–cool–heat cycle within the temperature range from 25°C to 250°C and a ramp of 10°C/min to calculate the residual reaction heat of the sample. The sum of both parts was then defined as the total reaction heat ΔH_T as follows in equation (2)

$$\Delta H_T = \Delta H_{iso} + \Delta H_{res} \quad (2)$$

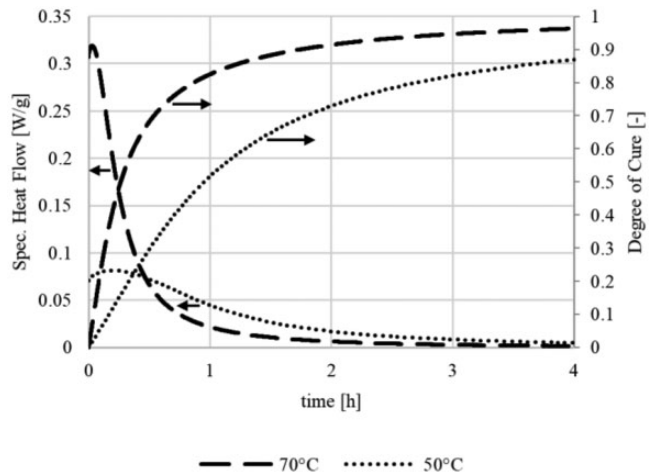


Figure 4. DSC measurements: specific heat flow and corresponding cure conversion for isothermal cure at 50°C and 70°C, respectively.

The corresponding values were $\Delta H_{iso, 50^\circ C/24h} = 450.3 \text{ J/g}$ and $\Delta H_{res, 50^\circ C/24h} = 26.1 \text{ J/g}$. The isothermal reaction heat at 70°C was $\Delta H_{iso, 70^\circ C/14h} = 472.6 \text{ J/g}$. Based on these values the final cure degrees after the isothermal measurements were determined as 94.5% and 99.2% for 50°C and 70°C, respectively. The diagram in Figure 4 shows the specific heat flow over time under isothermal conditions and the corresponding cure degree conversion for 50°C and 70°C, respectively. The heat flow decreases asymptotically to values close to zero at times long before the total experiment time. Ninety-nine percentage of the final cure degree was reached after 8.4 and 6.5 h during the DSC measurement at 50°C and 70°C, respectively. Therefore, it can be assumed that the practically achievable maximal degree of cure, corresponding to the specific cure temperature, was reached at the experiment end after 24 h (50°C) and 14 h (70°C), respectively.

Discussion of the considered gel point criteria and corresponding gelation times

Since the time range of detected gelation times based on the used criteria is remarkably large, particular attention was put on the selection of the most representative criterion and time values, correlating with defined conditions at the gel point. In general, the gel point marks the transition of a material from liquid to solid (sol–gel transition) and correlates with the growth of a connected molecular structure.³⁶ For a thermoset polymer system, this state is defined by the initial formation of an infinite polymer network as a consequence of the chemical cross-linking reaction.^{36,37} By definition, the degree of conversion at the gel point is constant for a thermoset polymer system, independent

of the cure temperature.³⁸ That property was used in the present work by evaluating the cure degrees correlating to the different gelation times. Figure 5(a) depicts the cure degree values at the evaluated gelation times based on criteria (i) to (iv). The comparison shows a significant difference of the cure conversion at the gel point using the $\tan\delta$ -peak criterion (ii) or the G' -onset criterion (iii). The corresponding cure degrees are in the range of 52% and 57% for cure temperatures of 50°C and 70°C, respectively. A more constant degree of cure, independent from the cure temperature, results with the G' - G'' -intersection criterion (i) and especially with the F_n -drop criterion (iv). With the latter, the corresponding cure degrees at the gel point are in the range of 62%. As a complement to the cure degree values, the evaluation of the corresponding dynamic viscosities at the gel points can be seen in Figure 5(b). While the dynamic viscosity corresponding to criteria (ii) and (iii) are in a range between 14 and 31 Pa·s, the gelation times based on criterion (iv) correspond with higher dynamic viscosity values from 1204 Pa·s at 50°C to 309 Pa·s at 70°C with a plausible temperature dependence. Due to the signal drop during the rheological measurements, as mentioned before, the dynamic viscosity for the rheological experiments at 50°C and 55°C could not be evaluated for the G' - G'' -intersection criterion (i).

A comparison with former results based on DGEBA/diamine thermoset systems supports the validity of the F_n -drop criterion (iv) for the gel-point detection. López et al.³⁹ studied the gelation and vitrification process at three different epoxy systems using dynamic mechanical analysis. The DGEBA epoxy resin with a cycloaliphatic diamine-based hardener system (amine/epoxy ratio = 1) showed a mean cure conversion of 62.4% based on five measurements at 60°C, 70°C, 80°C, 90°C, and 100°C, but with a relatively high distribution of the single results in a range between 58% and 65%. In the study of Cadenato et al.³¹ a DGEBA epoxy system with an aromatic

amine hardener was characterized and the cure degree at the gel conversion for isothermal cure between 50°C and 105°C was determined based on thermo mechanical analysis and dynamic mechanical thermal analysis in combination with subsequent DSC tests. They determined a constant gel conversion of 0.62 ± 0.03 within the tested temperature range and confirmed with their results the isoconversional character of the gelation phenomenon. Núñez-Regueira et al.⁴⁰ also used a DGEBA epoxy resin with a metaxylylenediamine curing agent for their characterization of the gelation process. Based on rheometric measurements the mean degree of conversion at the gel point was determined as 0.60 within the tested temperature range between 50°C and 100°C. The theoretical value of the cure conversion at the gel-point based on the Flory theory is 57.7% in case of stoichiometric DGEBA/diamine systems.^{37,31} The slightly higher values which are determined experimentally can be explained by effects like different reactivities of the reactive groups and intramolecular ring formation, which are not considered in this theory.^{31,41}

Due to the key criterion of an isoconversional character of the gelation process and supported by the validity check, that confirmed the plausibility of the cure degree at the gel point, the F_n -drop criterion (iv) can be stated as the most representative method for the detection of the gelation time, considered within this study. As might be expected, the time-temperature relationship of the gelation kinetics is in good agreement with the Arrhenius equation as can be seen from Figure 5(c). The corresponding activation energy is 58.8 kJ/mol.

Results and discussion

Figure 6 shows the measured FBG strain signals during the RTM process with isothermal cure temperatures of 50°C and 70°C, respectively. From top to bottom the strain curves belong to the FBG sensors installed regarding the above-mentioned approaches A, B and

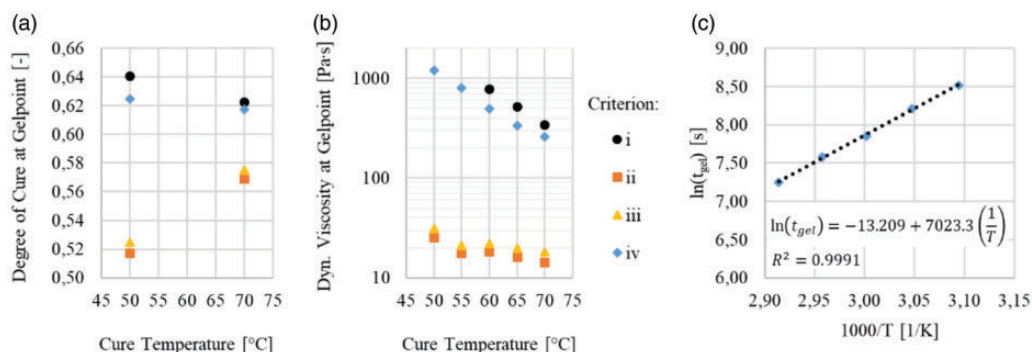


Figure 5. Cure degree (a) and dynamic viscosity values (b) for the evaluated gelation times. Arrhenius plot based on the results of criterion iv (c).

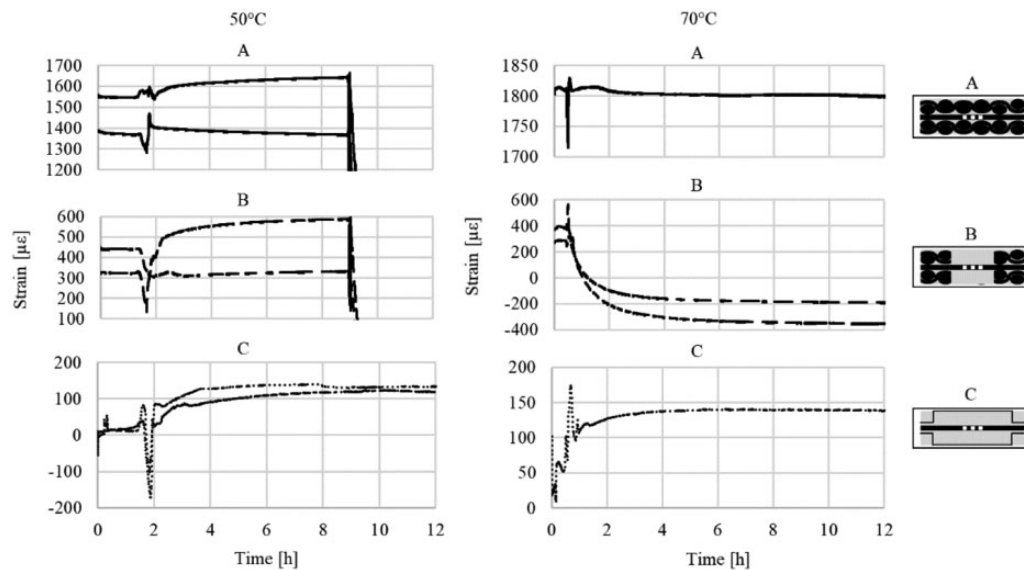


Figure 6. FBG strain data during RTM process monitoring with isothermal cure conditions at 50°C and 70°C, respectively.

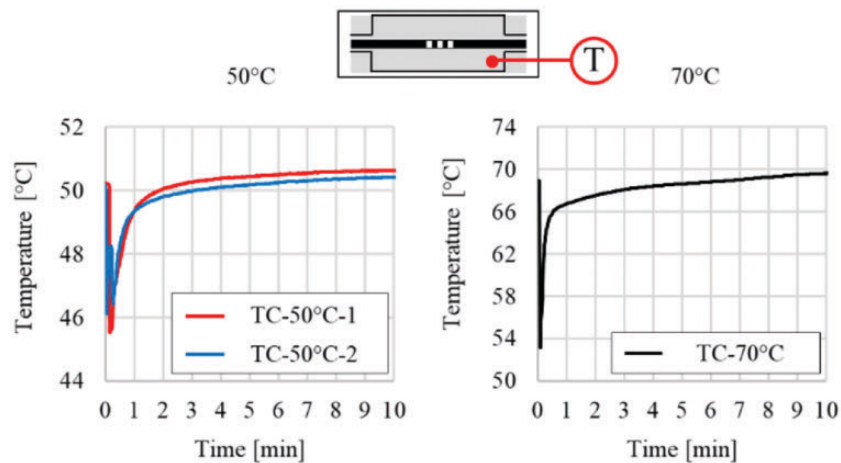


Figure 7. Temperature development during the initial phase after resin injection measured with the integrated thermocouples of approach C.

C, respectively. The resin injection starts at time zero. After the resin reached the outlet, the venting tubes were clamped and the injection pressure of 2 bar at the inlet was maintained during the curing process. The signal drop of the strain data at 50°C for approaches A and B is due to the demolding process and subsequent temperature drop to room temperature, which was done after 9 h in this experiment. While it was aimed to record two FBG sensors in each experiment for each installation approach, two sensor failures along with a signal loss appeared within the two experiments at 70°C. Therefore, approaches A and C at 70°C are represented by just one signal curve, respectively.

Since the FBG sensor position of approach C is located right at the resin inlet, the detected strain signal during the initial phase was influenced by temperature changes, due to the temperature differences of the injected resin (RT) and the preheated mold (50°C, 70°C). Therefore, the data were corrected according to the provided temperature correction formula with the given calibration parameter for the corresponding FBG sensor. The actual temperature next to the Bragg grating element could be measured accurately with the integrated type-K thermocouple. The temperature development during the first minutes after injection is depicted in Figure 7. The first contact with the resin is characterized by a temperature drop, which

converged quickly within the first minutes with the target temperature. For approaches A and B the situation is different, since the corresponding Bragg gratings are located in the middle of the mold and the resin flow path. The resin arrived that position during injection after about 60 and 30 s in the 50°C and 70°C experiment, respectively. When the resin arrived the Bragg grating element it already reached a temperature close to the isothermal target temperature. Furthermore, exothermic reaction heat is not crucial in the present case. The resin temperature did not exceed a temperature of 71°C inside the mold when cured at 70°C. Therefore, a strain data correction due to temperature changes was not necessary for approaches A and B.

As can be seen from the diagrams in Figure 6, the curves show similar curve characteristics in the first phase of the cure reaction. After an initial time period with an almost constant strain progression, the signal changes rapidly in tension or compression direction. This conspicuous strain decrease or raise appears in later times in case of a cure temperature of 50°C compared to the experiments at 70°C. After a certain time period, which is characterized by alternating changes in tension and compression direction, the signal stabilizes again and shows in most cases a monotonous progression either in positive or negative direction. In some cases, this latter stage is interrupted by smaller signal steps, as can be seen in the bottom curve of approach B and in the two curves of approach C at 50°C.

The corresponding curves of approach A show the highest strain levels due to the applied strain during compaction. The Bragg grating is located between two layers of the textile stack and in direct contact with the fiber reinforcement. The nesting process between two textile layers during compaction is a dominant mechanism for woven fabrics^{42,43} and forces the optical sensor in between in a more undulated and tension loaded state. The curves show the before mentioned sudden strain change in the first stage but with further progression the behavior varies. In later times, the strain shows an increasing, decreasing or constant behavior. This unreproducible and unpredictable behavior was also present in the FBG strain curves from Chiesura^{17,16} and Yildiz et al.,²⁷ who placed the Bragg gratings inside the reinforcing textile structure. Similar observations were made by Tsai et al.,⁴⁴ who concluded that the cure induced shrinkage inside a prepreg laminate could not be measured accurately with the embedded fiber optical sensor, due to the influence of the reinforcing fibers. Referred to our results, the undefined preloaded state after compaction and the influence of the reinforcement structure besides

the grating could be the reason for the observed variation of the strain progression during cure.

The results based on approach B confirm the assumption of the crucial influence of uncontrollable boundary conditions next to the Bragg grating. While the strain curves based on the measurements at 50°C show a progression in tension direction, the curves at 70°C run asymptotically in the opposite direction. The preload caused by the textile compaction could be significantly reduced, but the reproducibility of the strain progression could not be achieved.

Approach C was conducted to improve the reproducibility of the cure-induced processes in close proximity of the Bragg grating element, by measuring the strain inside a capsuled controllable resin volume. Therefore, the corresponding measurements were not influenced by the textile compaction since the sensor is positioned inside a brass tube as described above. While a reproducible signal progression could not be achieved with the integration approaches A and B, the more controllable surrounding besides the Bragg grating led in this case to a strain development in tension direction with an asymptotic behavior at later cure times in all measurements.

We assume that differences between the experiments, like locations of first detachments between the resin and the cavity surface and local inhomogeneities of the cure conversion during the solidification, determine the resulting local stress and strain conditions inside the cross-linking resin system, thus the acquired strain data measured by the FBG sensor. Although the cross-linking process in the epoxy system is associated with a chemical shrinkage, the cure-induced deformation can lead to elongation of the optical fiber due to deflections out of the initial fiber axis which lead to a tensional strain progression as observed in the majority of our measurements.

FBG strain response near the gel point

Detailed sections of the acquired strain data in early cure stages are shown in the diagrams of Figure 8. The red vertical lines mark the evaluated gelation time based on the previously discussed F_n -drop criterion (iv) for both cure temperatures, respectively. As can be seen, the before mentioned characteristic strain changes appear right after the gelation state is reached. Little differences between the gelation time and the start of the signal change can be explained by a delayed cure reaction in the RTM experiments due to the time required for heating the matrix system from room temperature to the adjusted tool temperature during injection (see Figure 7).

The sudden signal steps in the curves corresponding to approach C before the gelation time was reached

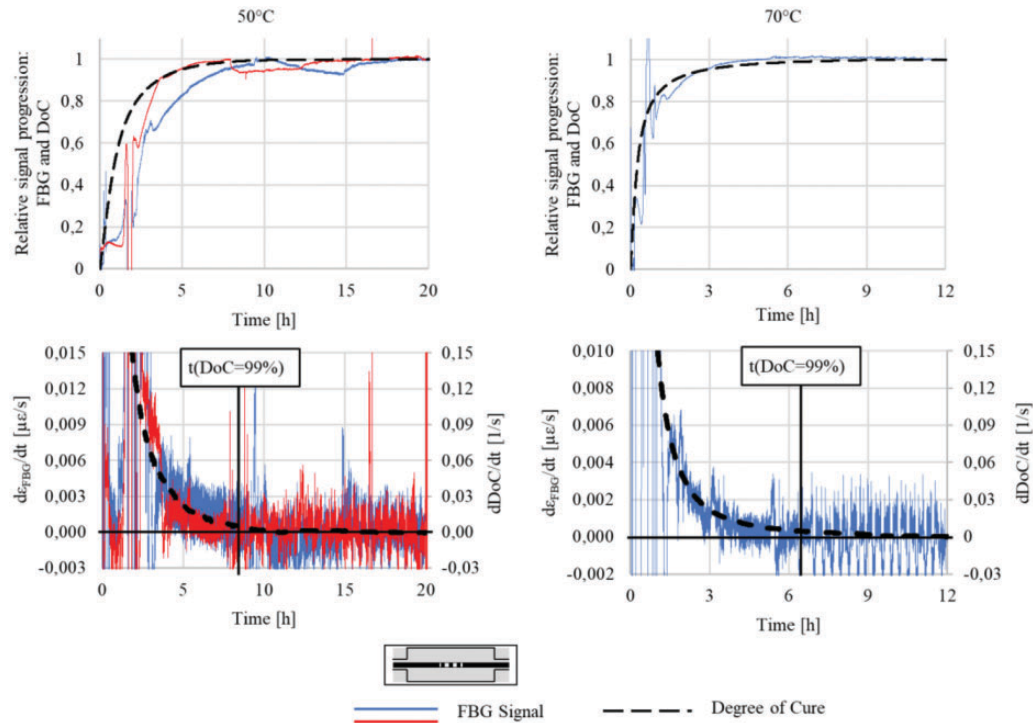


Figure 9. Relative signal progresses and (I) derivations considering the FBG strain of approach C and the cure degree development over time at 50°C and 70°C, respectively.

remain constant. Based on the acquired FBG data a quantitative reliable analysis, that can be applied as online monitoring, requires a higher consistency of the strain response. Therefore, further work should be conducted to analyze suitable conditions to ensure a high reproducibility of the measurement data with minimal disturbance. A significant improvement was achieved with the positioning of the sensor inside a controlled resin volume. The acting boundary conditions, especially the sensor–resin interface and the contact surfaces between and resin and adjacent components should be explored in future to further increase the reliability.

Based on our results we can state that under suitable conditions the FBG sensor gives valuable feedback in connection with the cure state even in advanced cure stages. Future investigations should be adapted to optimize the approach considering the sensor preparation, installation, and online data processing.

Conclusion

In this study, FBG sensors were used within the scope of cure monitoring in RTM processes. Particular attention was paid on the preparation and location of the Bragg grating location inside the mold cavity. Three different approaches: inside the reinforcing structure (A), inside a neat resin location (B), and inside a

controlled resin volume (C), were tested and analyzed. The experiments were conducted under isothermal cure conditions at 50°C and 70°C, respectively. The accompanying variation of the cure rate allowed us to verify the reproducibility and reliability of the acquired strain data and their correlation with the actual cure state. We can state, that the FBG sensor response enables the detection of the characteristic gel point of the thermoset epoxy-amine system during the conversion from liquid to glassy state. Therefore, it can be concluded that the strain detection inside the curing resin using FBG sensors delivers valuable information and enables an in-situ feedback of the actual cure state of the matrix system in that early cure stages. With increasing curing times, the reproducibility of the FBG strain progression and thus the correlation between the cure degree and the strain response significantly depend on the location of the Bragg grating inside the process. The highest reliability was achieved with the Bragg grating located inside a tube-shaped resin volume (approach C). We expect that further optimizations of the sensor design and installation, which provide an even better control of the boundary conditions in the near surrounding of the sensitive Bragg grating, will lead to significantly better reproducibility of the FBG strain signal.

From a practical point of view, it is likely to use optical fiber sensors with multiple Bragg gratings

along the fiber length. This allows the positioning of the sensing Bragg gratings inside the reinforcing fabric according to approach A but offers also the possibility to follow the strategies of approaches B and C for a more accurate cure monitoring at the same time. If the FBG sensor with multiple Bragg gratings is used for precise cure monitoring and as an integrated sensor inside the composite structure, combined with a SHM during its service life, the full potential of this fiber-optical sensor technology can be exhausted.

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