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Monitoring the temperature during thermoplastic composites assembling with fibre Bragg gratings: Validation using a rheometer

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Abstract. For many years, thermoplastic composites have been conquering the technological market in commercial applications in civil and military structures, with particular emphasis on aircraft. One of their most attractive advantages is the potential to be repaired and welded in a short time. However, due to the lack of knowledge on the impact of process parameters (time, pressure, temperature) bonding thermoplastic composites remains a challenge. The combined action of force and temperature leads to intimate contact between the substrates, which is the prerequisite for macromolecular interdiffusion and welding. A polymer film named Energy Director (ED) can be set up at the interface between substrates to enable the process and to ensure the strength of the joint. This leads to the need for a temperature measurement technique to better understand the physics responsible for the best bonding parameters. The use of Fibre Bragg Grating (FBG) seems to be suitable for obtaining real-time temperature data during the joining. The present work is challenging this technique by means of a rheometer as a device for results validation. The considered bonded materials are carbon fibre-reinforced polyetheretherketone (CF/PEEK) and polyetherimide (PEI). The integration of PEI film on top of the CF/PEEK composite parts is studied. The FBG sensors respond to both temperature and longitudinal strain. Then two kinds of measurements are examined under the rheometer: the first response of the fibre as a function of temperature in static mode during processing and the second one as a function of torque loading. Wavelength shifts and spectral form changes induced by the contraction of the composite during cooling were observed. The use of a rheometer is suitable for the simulation of CF/PEEK composites joining processes such as welding. FBG method has a higher potential for temperature measurement than incorporated thermocouples (TC) as studied during ultrasonic (US) welding [1]. Its advantage is the possibility to allow several measurement points along the optical fibre. The ultimate goal is to provide temperature mapping in the welding zone. The results from both techniques: optical fibre sensors and thermocouples were compared.

Introduction

Temperature monitoring remains a challenge, especially for high-frequency phenomena like US welding, however, optical fibre sensors may propose many important advantages for industrial uses. They are small with a diameter below 0.25 mm, highly sensitive to temperature and strain, multiplexable, immune to electromagnetic perturbation, and resistant to corrosion and fatigue. The fibres are flexible, and coated with a polymer-like polyimide that can readily be installed in industry without significantly disturbing the measurement environment [2]. The optical fibre

Bragg gratings have been developed since the 90s to offer new sensing systems. The measurement for pressure, strain and temperature by the FBG system is extensive since the propagation of light within an optical fibre reflects particular wavelengths of light and transmits all others.

Three common principles of fibre optic temperature measurement were studied and compared by Hoffmann et al. [3] considering fibre Bragg gratings, Raman scattering and interferometric point sensors. They resumed that FBG is the most suitable where many temperature measurement points are required or where strain measurements are carried out.

Progress in applications of FBG sensors to large composites and concrete structures was investigated by Rogers [4] and Rao [2]. Systems based on fibre Bragg gratings, with particular reference to the measurements of pressure, strain and temperature in industrial environments have already been installed and become commercially available in the electrical power industry, medicine and chemical sensing. Since then, multimode FBG using correlation signal processing methods has been developed [5]. Strain testing has been done from $0 \mu\epsilon$ to $10000 \mu\epsilon$ and temperature testing from $-40 \text{ }^\circ\text{C}$ to $80 \text{ }^\circ\text{C}$ for various applications. For use in aerospace extreme environments, the oxygen-chemical composition grating exhibited a response similar to the original FBG sensors at temperatures up to $1000 \text{ }^\circ\text{C}$. The sensors would allow temperature measurement to be made within 3 % of the reference S-type thermocouple. Also, long-term survivability over 400 h of thermal cycling to $750 \text{ }^\circ\text{C}$ and 7 h of thermal cycling to $1000 \text{ }^\circ\text{C}$ has been demonstrated [6]. Then, Habisreuther et al. [7] by using sapphire FBG sensors showed the temperature sensing up to $1900 \text{ }^\circ\text{C}$.

The method based on the measurements of the fibre Bragg grating phase obtained by combining optical low coherence interferometry and inverse scattering algorithm was proposed by Chapeleau et al. [8]. The authors performed non-uniform strain distribution and temperature gradient measurements using uniform or chirped FBG sensors. Recently, Pereira et al. [9] proposed a method to obtain independent strain and temperature measurements using embedded FBG in polymeric tensile test specimens, which is also promising for polymer fatigue test specimens. Generally, FBG sensors have a strain and temperature cross-sensitivity, making them inaccurate for tests where both change at the same time. An additional strain is created by temperature variation in the specimen, caused by thermal expansion. A method to decouple this cross-sensitivity, enabling independent and accurate measurement of strain and temperature, was developed. This technique can be accomplished by using two single mode FBG sensors embedded in the material.

In this work, the thermal analysis conducted on CF/PEEK composites using the PEI layer on the top is displayed. The fibre Bragg grating sensors were chosen to monitor temperature during assembly. The experiments present measurements where temperature profiles are analyzed through optical fibre sensors. FBG responded to changes in the material state. Finally, the Bragg wavelengths registered during assemblies of CF/PEEK with $250 \mu\text{m}$ PEI layer are correlated to the thermal profiles of thermocouples.

Materials and Methods

Substrates are laminates from Carbon fibre-reinforced polyetheretherketone composite fabrics, named PEEK from Porcher Industries and the energy director is a polyetherimide, named Ultem 1000 from Sabic. The CF/PEEK composite glass transition temperature (T_g) and melting point (T_m) are $143 \text{ }^\circ\text{C}$ and $343 \text{ }^\circ\text{C}$, respectively. Prepreg fibre areal weight is $456 \text{ g}\cdot\text{m}^{-2}$ and the resin weight content is 38 wt.%. The T_g of PEI is $220 \text{ }^\circ\text{C}$.

The composite plates were produced under the thermo-compression device from Pinette Emidecau Industries with frames of appropriate thickness. Two types of plates were manufactured: (1) 2 mm thick CF/PEEK plates and (2) the same plates with an additional $250 \mu\text{m}$ PEI layer integrated on top. 7 plies of CF/PEEK fabric were stacked to create a mirror symmetry lay-up [0/90]. Plates were first consolidated at $390 \text{ }^\circ\text{C}$ under an equivalent force of 192 kN on the laminate

for 10 min. A second thermo-compression cycle at 350 °C under the force of 117 kN for 10 min was applied for PEI integration. The plates were removed at 250 °C to avoid shrinkage and deformation. 25 mm diameter disc samples were then cut. Before experiments, materials were dried in the oven for at least 24 h at 130 °C.

The FBG are 10 mm long and placed in the middle of the Ø25 mm specimen. Their orientation is perpendicular to the carbon fibres. In the case of CF/PEEK with integrated PEI on top, the FBG without coating were previously inserted inside the PEI layer of the bottom specimen. The insertion procedure consisted of depositing the FBG on the PEI layer of the composite and covering it with treated Polyimide film and a 5 kg metal mass. The assembly was then placed in an oven at 290 °C for 40 min. Next, the FBG was extended to connect to a SmartScan interrogator.

Temperature measurements were performed using the oven of ARES rheometer from Rheometrics and thermocouple K-type from RS connected to the midi data recorder from Graphtec Corporation. The samples were clamped between 2 aluminium discs of 25 mm diameter in parallel plate configuration.

The experimental set-up shown in **Figure 1** has been provided to create a carefully controlled temperature gradient during composite assemblies. Two rheometer procedures were successively applied during temperature and wavelength measurements: holding plates (1) in static mode position and (2) in dynamic mode, meaning subjected to mechanical torque for 15 min with deformation of 0.1% and frequency of 1 Hz.

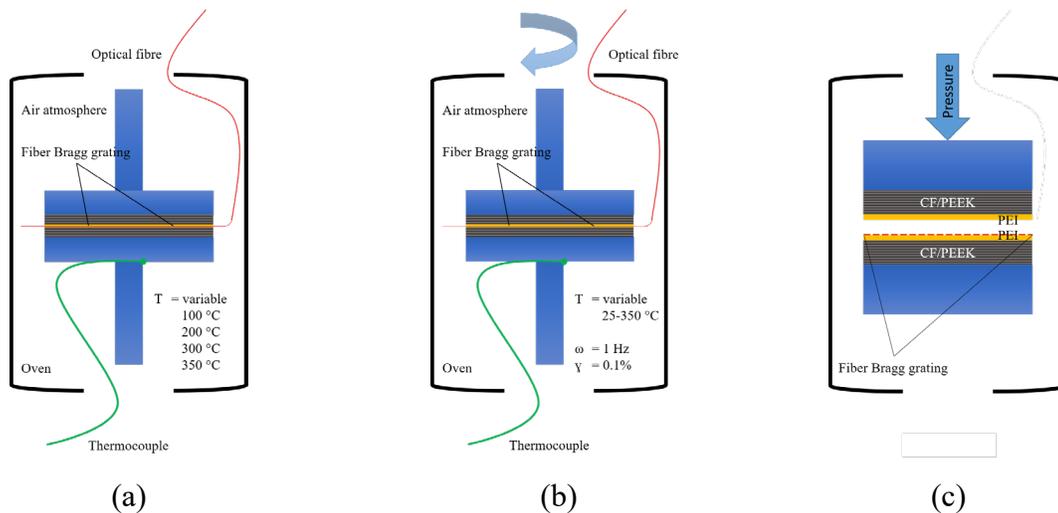


Figure 1. Experimental set-up of rheometer procedure in (a) static mode (b) dynamic mode (c) zoom of assembly

Temperature measurement and calibration

The fibre Bragg grating is made of a core with alternating refractive indices and a constant periodicity. The central wavelength of back-reflected light from a uniform Bragg wavelength, λ_{Bragg} , is given by the following equation:

$$\lambda_{Bragg} = 2\Lambda n_{eff} \quad (1)$$

where Λ is the period of the grating and n_{eff} is the effective index of the fundamental mode of the fibre. Both Λ and n_{eff} are affected by the strain and temperature of the grating through elongation and thermal expansion effects of the material. This formula can be extended to include the effect of strain and temperature on the effective refractive index of refraction and periodic spacing between the grating planes, so the Bragg wavelength is shifted.

$$\Delta\lambda_{Bragg} = \lambda_0[(1 - P_\epsilon)\epsilon + (\alpha + \xi)\Delta T] \quad (2)$$

where λ_0 is the initial value of Bragg wavelength, P_e is the effective photo-elastic constant, ε is the strain applied to the gratings, α and ξ stand for the coefficient of thermal expansion and the fibre thermo-optic coefficient of the grating material, respectively, and ΔT is the temperature variation. If no stress is applied to the FBG during the experiments, strain measurements of the FBG can be neglected. The corresponding Bragg wavelength shift is given by:

$$\Delta\lambda_{Bragg} = \lambda_0[1 + (\alpha + \xi)\Delta T] \quad (3)$$

Figure 2 shows the amplitude reflection coefficient of the FBG before the measurement. A temperature change causes a shift in the Bragg wavelength. The peak position strongly depends on temperature, and the reflected intensity from the FBG is sufficient for further signal processing. Only the peak wavelength is recorded by the software as a function of time.

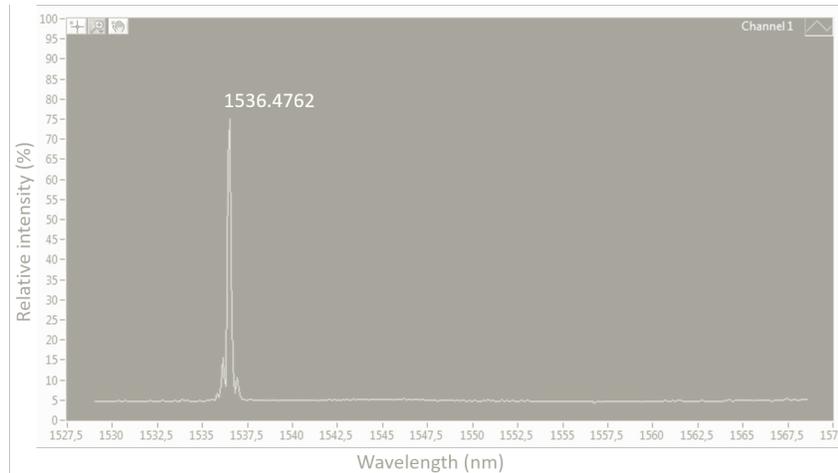


Figure 2. Amplitude reflection coefficient of FBG

The thermal profile characterization was performed with temperatures ranging from room temperature to 350 °C. A short cycle of 2 min at 100 °C and 5 min at 350 °C in static mode was first tested to display the interrogator's response with an assembly of CF/PEEK with loose PEI layers. This thermal cycle was insufficient to observe temperature holding levels, but adequate to form a cohesive welding interphase incorporating the FBG with no risk of degradation. A unified interphase, which is created assembly joint, is also a way to limit the strain induced by the set-up for further calibration tests.

The calibration cycle was then performed in static mode: three isothermal temperatures (100, 200 and 300 °C) were applied for 15 min each to obtain a constant temperature, and the last one at 350 °C was kept until stabilization to avoid too long degradation of the material, **Figure 3.a**. The Bragg wavelength was centred at 1540.69 nm at room temperature. This calibration aims to determine the trend curve correlated between the Bragg wavelength shift and thermocouple temperature. Thermocouple measurements on the set-up are not reliable for accurately indicating the interphase temperature, but stabilizing the oven temperature guarantees a usable value. The oven's dimensions are small enough to ensure a uniform temperature around the grating and the reference thermocouple. The first observation is that stabilization time in a rheometer needs at least 5 min at the requested isotherm.

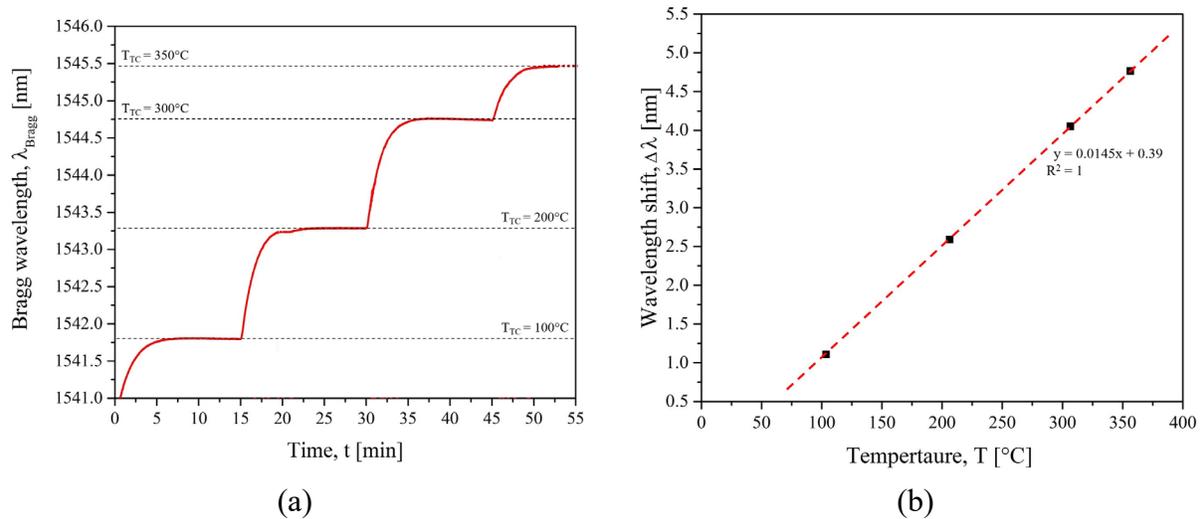


Figure 1. Bragg wavelength versus time (a) and correlation of Bragg wavelength shift versus temperature (b)

Based on the accumulation of data for the isotherms, the responses of the sensors recorded as changes in wavelength with increasing temperature are shown in **Figure 3.b**. The FBG shows a linear response to temperature over the ranges from 25 °C to 350 °C. All four isotherms exhibited very similar performance characteristics. The results show a linear relationship between Bragg wavelength shift and temperature: all gratings shifted approximately 1.5 nm per 100 °C. A correlation factor of 1 indicates there is no stress during this experiment, as wavelength shift is only affected by temperature. The linear fit which represents the calibration of the optical fibre sensors can be given by the following equation:

$$\Delta\lambda_{Bragg} = 0.0145 * T + 0.39 \quad (4)$$

where $\Delta\lambda_{Bragg}$ is the wavelength shift of the initial peak at 25 °C and T is the temperature [°C], at which the measurement is taken. From this formula, the parameter, $d\Delta\lambda/dT$, can be calculated and hence for this FBG the temperature sensitivity can be determined to be 0.0145 nm·°C⁻¹ over the temperature range of 25-350 °C. Then, the temperature gradient for all measurements can be calculated from the following equation:

$$T = 69.081 * \Delta\lambda_{Bragg} + T_0 \quad (5)$$

where T_0 is the beginning temperature during the measurements [°C].

Typical uncertainty in the temperature measurements was ± 5 °C [10].

Experimental results and validation

As mentioned before, two optical fibre temperature measurements were examined: the response of the FBG as a function of temperature in static mode and subject to mechanical stresses. Moreover, the influence of PEI and FBG left loose on top of CF/PEEK, as well as, PEI and FBG integrated on CF/PEEK surface were studied. The results combining gradient temperature from FBG sensors and thermocouples are presented in **Figure 4** and **Figure 5**. The measurements were taken from ambient temperature up to 350 °C with an isotherm for 15 min to ensure thermal stabilization. The chosen temperature did not affect the T_m of PEEK noticeably, however, overcame the T_g of both, PEEK and PEI. Then, the cooling rate was registered for 10 min. The temperature results were taken by a thermocouple during assembly with a measurement interval of 1.00 s, while the shift of Bragg wavelength may be more accurate with 0.04 s.

Comparing the experimental temperature profiles from FBG sensors and thermocouples, a slight mismatch can be highlighted. It was expected, as the results of fibre Bragg gratings were

recorded at the interface of PEI while the thermocouple was located under the bottom plate of the experimental set-up. Despite this, the curves are well correlated. The faster heating and cooling rates are observed for TC as the temperature in the oven affects them directly. Temperature stabilization around 350 °C in the oven still takes at least 5 min. During this time, no specific changes are observed on TC curves.

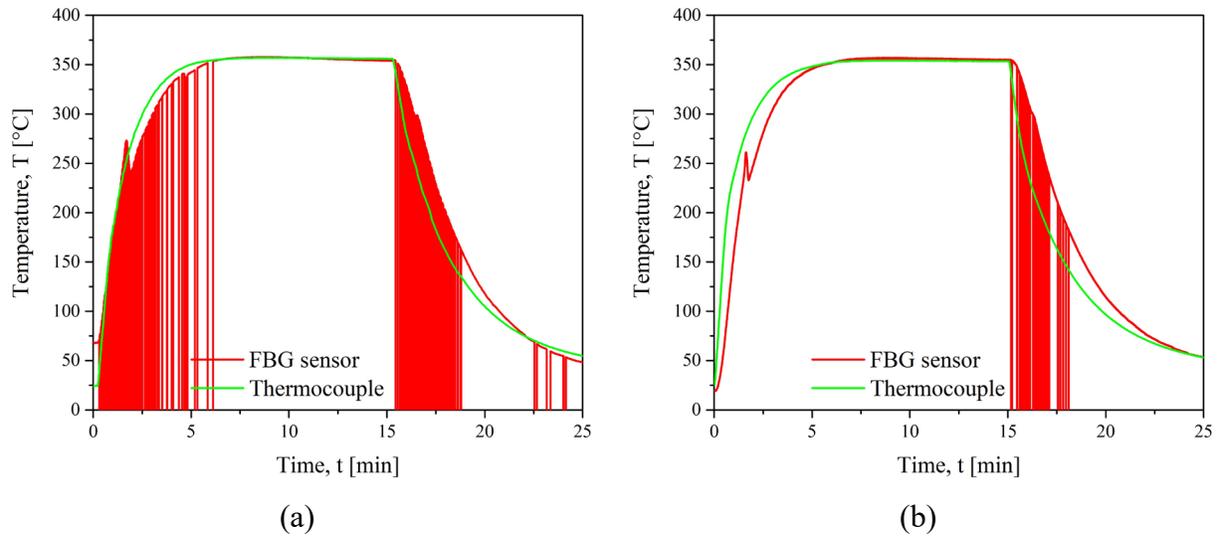


Figure 2. Static temperature measurement with loose PEI layers (a) and integrated PEI layers (b)

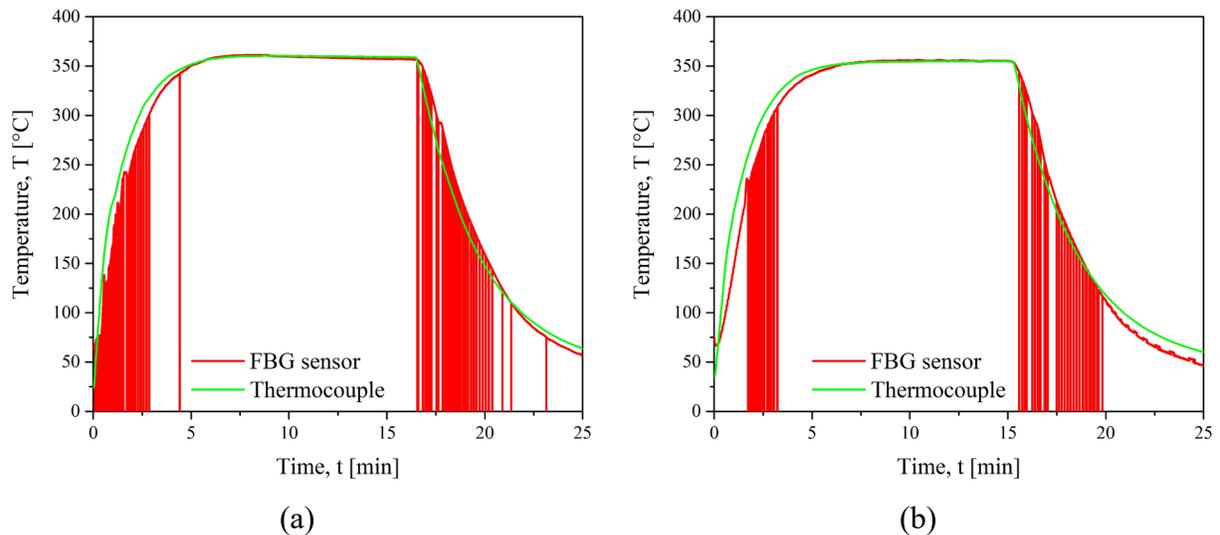


Figure 3. Dynamic temperature measurement with loose PEI layers (a) and integrated PEI layers (b)

In the case of fibre Bragg gratings, a regular disconnection is apparent during thermal transitions (vertical red lines), whereas none is observed when the temperature is stabilized. This is attributed to the vertical mechanical action of the rheometer to maintain the assembly under constant pressure during its thermal expansion. We remind that an excess of curvature or micro-curvatures may reduce the light power inside the optical fibre and then the reflected peak is no longer recognized. This also indicates that the vertical loads have a greater impact on the wavelength intensity than the shear loads applied by the rheometer. While heating, PEI begins to flow as a result of nucleation (viscoelastic heating mechanism controlled by surface heating) and the growth of hot spots. The flow of the energy director after reaching its T_g of 220 °C causes a peak registered by FBG sensors. For loose PEI the peak reached 273 °C at 1.7 min while for integrated PEI it was 261 °C at 1.6

min. The cooling rate of FBG is slower than TC. Indeed, the welding interphase takes more time to cool down than the aluminium set-up.

The FBG responses to mechanical stresses show small differences compared to static mode. The thermal profiles obtained by optical fibre sensors and thermocouples are almost identical. However, Bragg wavelength disconnections are more pronounced for loose ED configurations. In dynamic mode, the flowing of PEI after reaching its T_g causes a less intense peak registered by FBG sensors, but closer to the glass transition temperature of material. Thus, for loose PEI and integrated PEI at 1.7 min, the peak reached 235 °C and 240 °C, respectively. The cooling rate seems to be similar for both methods (FBG and TC). It means that material after the application of mechanical stresses organizes the chemical structure to be cooled down about $5\text{ °C}\cdot\text{min}^{-1}$ faster than material in static mode.

After the first temperature measurements by fibre Bragg gratings, the samples in static and dynamic mode were subjected to a second measurement, as presented in **Figure 6**. In this case, CF/PEEK composite samples are already assembled by a unified PEI interphase and the FBG is embedded inside. In each case, the first experiment caused partial deconsolidation of the composite, reducing its conductive properties, which led to a reduction in the heating and cooling rates recorded by the optical fibre sensors in the second measurement. Also, the peak previously recorded after T_g of PEI is not apparent during the second experiment. It could suggest an optical change in the fibre due to the environmental transition from the glassy to the rubbery state, but fully embedded fibres should be affected. Therefore, a stress release on the fibre is more likely: the slight pressure of the rheometer set-up squeezes the optical fibre, but when the T_g of PEI is reached, it softens, and the fibre returns to its original shape.

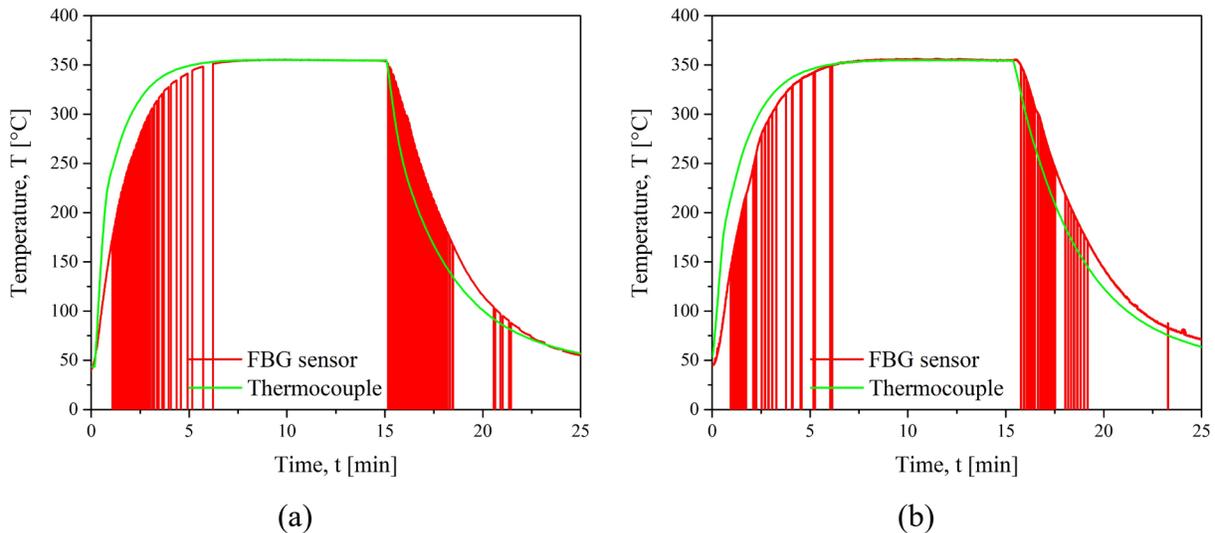


Figure 4. Second temperature measurement passes (a) static mode and (b) dynamic mode

The fibre Bragg grating wavelength is affected by different environmental conditions such as temperature, strain, and coupling with the host material. The agreement between the results from optical fibre sensors and thermocouples demonstrated the FBG method's validity.

Summary

This study focused on the temperature profiles during the assembly of CF/PEEK specimens. CF/PEEK parts with 250 μm PEI layers were joined together to obtain the response of the FBG sensors as a function of temperature in static mode and subject to mechanical stresses. Both configurations with loose and integrated PEI layers on top of the composite sample were studied. The FBG considered as temperature sensors up to 350 °C showed linear Bragg wavelength shift with temperature. The optical fibre sensors are then able to monitor high temperatures in the

assembly interphase, as correlated to thermocouples. Temperatures above the T_g of the materials reduced fibre strain, and therefore its impact on wavelength shift.

It is a promising technique to quantify temperature distribution inside molten polymers. This measurement would lead to an accurate characterization of thermal or even mechanical behaviour where classical techniques are only able to provide surface measurements.

The method has been validated and can be applied for CF/PEEK composite joining processes such as welding. FBG method has great potential for temperature gradient measurement and could be less intrusive than incorporated thermocouples. Further work will focus on using FBG sensors during ultrasonic welding of CF/PEEK parts with PEI as energy director.

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