MONITORING NON-ISOTHERMAL POLYMERISATION AND CRYSTALLISATION OF CYCLIC BUTYLENE TEREPHTHALATE COMPOSITES MANUFACTURED BY RTM

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Introduction

Cyclic butylene terephthalate (pCBT) composites obtained by isothermal polymerisation have shown to be brittle and to need long cycle times. Non-isothermal routes, instead, allows tougher thermoplastic matrix but their low energy-efficiency and long cycle times related to the high thermal inertia of the moulds limit their implementation for industrial applications.
Monitoring mould filling, polymerisation and crystallisation of such a complex system is essential in order to optimise the RTM process, since extrapolation of materials characterisation results (DSC, SEC…) to real process is not trivial. Several monitoring techniques have been proved to be useful to study the polymerisation, crystallisation and mould filling of pCBT, but only some of them can be integrated in RTM and are suitable for high temperatures. Fibre Optic Sensor characteristics allow low intrusion sensor integration. Distributed sensing capabilities will be explored, measuring strain and temperature distribution over the full laminate and the crystallization event during the curing.

Experimental Method

The present research work evaluates the sensitivity of two monitoring systems to the physical and chemical variations of CBT 160 matrix during RTM process; a Direct Current (DC) based electric sensor placed in the mould close to the resin inlet, and a distributed optical sensing technique based on Rayleigh backscattering measured by an Optical Backscatter Reflectometer (OBR). The RTM equipment allows controlling the temperatures at different places (melting device, injection machine, runner system and mould). Therefore, the cycle time and microstructure can be optimised. Figure 1a shows the resin temperature measured during the process where the main events are identified by numbers in bracket; melting (1), resin arrival to the mould (2), polymerisation (3), cooling down (4) and crystallisation (5-6). The polymerisation degree at each instant (figure 1b) has been estimated by the model proposed by Steeg et al. [1]. Furthermore, the crystallisation behaviour of pCBT at real cooling rate has been characterised by DSC (figure 1c).
OBR allows measuring the optical losses all along the fibre. The Rayleigh backscatter losses of a bare optical are proportional to variations of external stimulus, as temperature or strain variations. Furthermore, Optical losses are also proportional to transversal stresses over the optical fibre in every point.

Results

Based on temperature, conversion degree and crystallisation evolutions, the DC sensor signal (figure 1d) can be interpreted. The arrival of the resin to the location of the DC sensor induces a sudden resistance drop. During the polymerisation of CBT the resistance increases slightly and stays constant until cooling step starts. DC signal is affected by temperature, since resistance increases as temperature decreases, but when crystallisation takes place the resistance variation is even more pronounced. Thus it could be concluded that resistance is also influenced by the crystallisation of the pCBT, and consequently DC sensor is useful to detect it. As Optical losses are proportional to the transversal stresses in every point of the fibre, it is possible to detect the resin shrinkage due to crystallisation event (T<200 ºC) in different sections of the fibre (Figure 2a). Applying the distributed sensing technique [2], it is also possible to obtain the strain distribution all along the surface during the curing, and determine residual stresses distribution and mould filing area (Figure 2b).

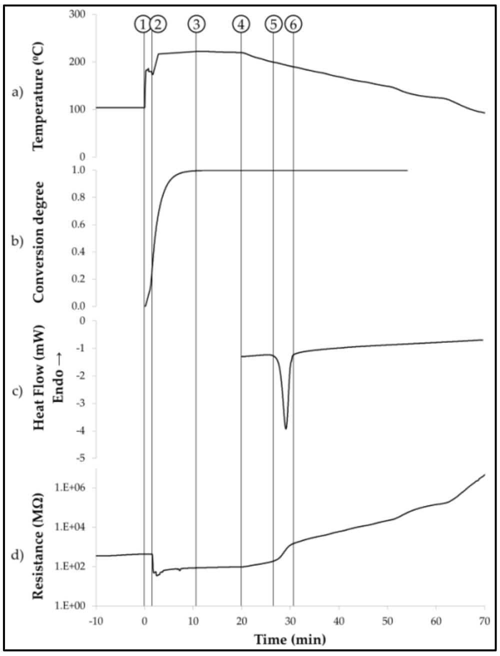

Figure 1: *Synchronized data of resin temperature (a) cure degree (b), DSC Test (c) and electric resistance (d) for the RTM process.*

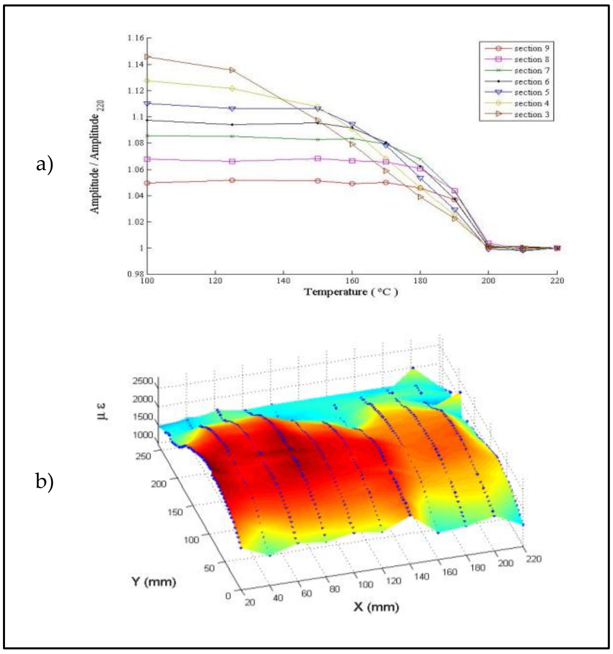

Figure 2: *Fibre optic loses at different fibre sections (a) and the deformation field measured by the fibre optic sensor in filling stage (b).*

The main conclusion of this work is that the electric sensor is able to perceive changes in conversion degree and crystallinity despite the influence of the temperature on resistance. In addition, Fibre Optic Distributed technique is useful for monitoring the mould filling, detecting dry spots and crystallisation. So both sensors are necessary in order to monitor the whole RTM process of pCBT composites.

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References

[1] M. Steeg, P.Mitschang, P.Chakraborty, T. Hartmann, Modeling the viscosity and conversion of in-situ polymerizing PBT using empirical data, In: *17th ICCM*, Edinburgh, UK, 2009

[2] A. Guemes, A. Fernandez-Lopez, B. Soller, Optical Fiber Distributed Sensing – Physical Principles and Applications, *Structural Health Monitoring* Vol 9(3): 233–13