Isothermal and non-isothermal thermoset cure and induced-strains monitored by optical fibre sensors

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Introduction

Thermosetting-based composites are generally used as structural materials because of their excellent specific strength. However, they can suffer for sensitivity to damage and impact, and their properties are not always well predicted. This is caused partially by residual internal stresses and flaws, originating from the elaboration process. This latter can be monitored by including adequate high performance in-situ sensing systems, allowing a better knowledge of the process cycle and its related parameters.

One of the key parameter is the degree of conversion and the temperature history experienced by the matrix resin, that ensure partly the quality of the composite part. However, as thermosetting reactions are exothermic, purely isothermal curing is often not strictly reached and temperature should be measured and highly controlled. If this is not the case, this can result e.g. for thick parts, in matrix degradation or incomplete curing. Secondly, conceptually the process and the materials in presence induce inevitably the development of cure residual stresses (CRS) that can affect the final material properties. CRS have three main origins: chemical (cross-linking shrinkage), thermal (mismatch between the thermal expansion of all the components at different scales), and mould-laminate interaction origins. The knowledge of the development, characterisation and amplitude of these CRS is thus necessary to reduce their effects thanks to an optimisation of the cure cycle (by adjusting the process parameters) and materials.

Cure monitoring assessment

In this study, we first report on the cure monitoring of a thermosetting resin usually used in liquid composite moulding processes (RTM6), by using simultaneously a multi-instrumentation based on a Fresnel refractometer sensor together with a dielectric analyze (flexible PI interdigitated electrodes sensor). The measurements were compared and a high correlation between dielectric and optical signals was shown. It allowed to base our analyses of the curing only on the signal from the weak-intrusive optical sensor. In a second step, Fresnel signal was calibrated with the predictions of a classical thermo-kinetic model of the RTM6 resin cure, assuming parameters values from the literature, and temperature measurements for inverse identification of the heat exchange coefficient between the resin and its environment.

Two mould materials were used to enhance or reduce the exothermic phenomenon, leading to isothermal and non-isothermal cures. In both cases quantitative values of the degree of conversion were deduced from the experimental Fresnel signal together with the numerical model of the cure. Under isothermal conditions, results showed that the Fresnel signal can be directly exploited to obtain qualitatively the degree of conversion. This was also demonstrated in non-isothermal conditions by correcting the Fresnel signal from its temperature dependence. If the initial degree of conversion is known, its quantitative assessment can be achieved using the Fresnel and temperature measurements.

Cure-induced strains by fibre Bragg grating sensor

The different curing experiments were also monitored using fibre Bragg grating (FBG) sensors and thermocouples to assess the cure-induced strains, the evolution of the degree of conversion of the resin being known. For all experiments (isothermal or non-isothermal), at the onset of stress transfer to the optical fibre, the degree of conversion ranged between 0.63 and 0.68 (slightly higher than the degree of conversion at gel point). It was also reported that the FBG deformed under chemical shrinkage with an amplitude widely lower than the expected chemical strain, showing that the FBG signal was not directly related to the actual chemical shrinkage. However, once the resin was cured, the FBG provides directly the coefficient of thermal expansion of the resin, which was calculated for the different experiments as a function of both the temperature and degree of conversion. Finally, glass temperature transition and debonding from the mould were also clearly detected by the FBG sensor.