PACKING AND PERMEABILITY PROPERTIES OF E-GLASS REINFORCEMENTS FUNCTIONALISED WITH CAPSULES FOR SELF-HEALING APPLICATIONS

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Introduction

The development of capsule-based self-healing fibre-reinforced polymers [1-4] implicates the embedment in the matrix of micron-size capsules, constituted of a protective but brittle shell and a core containing the healing agent or a trigger for the healing phenomenon. The main type of damage that these healing systems aim at repairing, for composites, is the inter-ply failure that occurs within the resin pockets. Introduction and survival of the healing system is one of the major issues in the manufacturing of such composites, and this requires the characterisation of the packing and permeability properties of the stack in the presence of these capsules.

Materials and methods

The 125-250 μm healing capsules are produced by oil-in-water emulsion, their core is a solution of 2.5 vol% of bisphenol A diglycidyl ether in ethyl phenylacetate (EPA) with red dye, and their shell is constituted of urea-formaldehyde. The composite reinforcement consists of 16 plies $[(+45/-45)/(0/90)]_{4s}$ of a 390 g/m^2 woven twill 2x2 E-glass reinforcement. The longitudinal and transverse permeability measurements are performed with a commercial silicon oil and solution of polyethylene glycol (35'000 molar mass) in distilled water, respectively. The composite plates produced through Vacuum Assisted Resin Infusion Moulding have a matrix of low viscosity bisphenol F diglycidyl ether resin, mixed with diethylenetriamine in a 100:12 weight ratio; they are cured for 24 h at room temperature and post-cured for 24 h at 35°C.

Thermogravimetric analysis, optical microscopy and X-ray micro-computed tomography are used in order to characterise synthesised capsules and capsule-functionalised fabrics. A standard compression machine is used for quantifying the packing behaviour of the dry fabric stack. A Resin Transfer Moulding-based apparatus is used for the longitudinal permeability tests, whereas a specifically made system is used for the transverse measurements.

Results and discussion

The produced capsules presented a number-length average diameter of 201 ± 22 μm, a diameter-independent [5] shell thickness of 223 ± 42 nm and a typical thermal behaviour, namely a temperature onset of the gradual weight loss at about 100°C. Bursting forces varied between 4 and 6 mN depending on the diameter [5].

Optical imaging of the fabric demonstrated inter-bundle interstices in the range 150-200 μm (width), in which particles with 125-250 μm diameter can thus fit; it is believed that capsules, manually sieved onto the reinforcement fabrics, are in general prone to fall down, due to gravity, within the interstices, if their size is suitable; nevertheless, some of them lay on the top of warp/weft yarns.

The presence of capsules (overall volume concentration varying between 1.25 and 3.75 vol%) modified the linear packing behaviour of the glass reinforcements tested in dry conditions (Figure 1), especially when lower pressures were applied and the capsules were still intact; at higher pressures, the packing behaviour followed a power law as for plain samples, probably because capsules burst or moved further into the interstices. The change in compressibility properties also means that lower fibre volume fractions and higher overall stack thicknesses are achievable at equal applied pressure if capsules are present.

In terms of longitudinal unsaturated permeability, it was found that the presence of capsules increased by a factor of 6 the permeability as compared to plain samples, without any specific trend depending on their content. This increment was related to the higher porosity that capsules induce, as demonstrated with compressibility experiments. Moreover, the phenomenon can be amplified by the presence of driving capillary forces, correlated with capsule presence and possible bursting. Transverse saturated permeability properties, however, were not influenced by the capsule presence.

For a given fabric architecture, only a limited amount of capsules can be introduced and nest in the interstices, thereby being protected during processing. Moreover, manufacturing parameters (i.e. degree of vacuum and matrix content) need to be adapted in order to get comparable plain and capsule-containing samples as well as to avoid premature capsule rupture. Impregnation kinetics tend to be enhanced in the longitudinal direction, making the process time shorter, whereas they remain similar in the through-thickness direction.

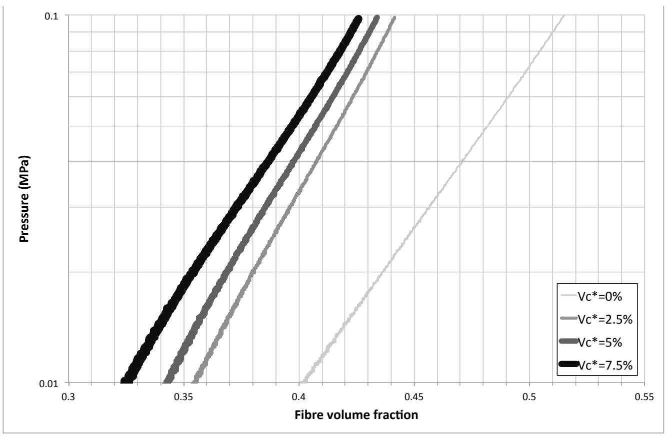

Figure 1: *Compressibility of plain and capsule-functionalised fabrics in terms of fibre volume fraction over loading.*

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