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Experimental assessment of dual-scale resin flow-deformation in composites processing

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ABSTRACT

In this paper we are concerned with the assessment of sub-models within a two-phase continuum mechanical FE framework for process modeling of composites manufacturing. In particular, the framework considers the inclusion of two deformation dependent models describing resin flow related to: (1) meso-scale wetting and compaction of individual plies and (2) overall preform deformation and macroscopic Darcian flow. Using micro-mechanical modeling, we model the physics of these sub-processes in relation to the recently developed Out-Of-Autoclave (OOA) prepergs. The models are placed in context with a compression–relaxation experiment, employed to study the preform deformations considered separated from other sub-processes. Finally, calibrations and model validations are carried out against the relaxation experiment to relate the FE framework to the mechanical response of the preform. Therefore, using the above experiment, parameter values out of the literature and those estimated from micrographs gave a fair agreement between the simulation and experiments.

1. Introduction

1.1. OOA-processes summary

Partially impregnated prepregs in out-of-autoclave (OOA) or vacuum-bag-only (VBO) processing consists of both dry and resin-rich areas in their microstructure. The dry areas forming a relatively permeable network will be infiltrated by the surrounding resin [1]. This technology is used to promote air removal during consolidation as well as to keep the semi-product flexible [2,3]. In the most efficient OOA-processes subsequent de-bulking and molding operations are performed simultaneously: liquid resin penetrates into plies (local wetting), the entire mixture deforms into its desired shape (forming) and resin is displaced relative to the plies (macroscopic drainage), all in one shot [4]. Since many processes occurring simultaneously on different scales it is a considerably challenging task for material characterization as compared to considering the elementary processes stand-alone.

1.2. Background

In the context of consolidating composite prepregs, there are at least four relevant processes: elastic deformation of individual fiber plies (meso-scale), elastic deformation of the entire preform, i.e. of the network of fibers (macro-scale), infiltration of individual fiber plies (tow impregnation) and macroscopic Darcy's flow. Today, these sub-processes are reasonably well understood when they are considered isolated from each others. In the overall consolidation problem during autoclave processing of thermoset matrix composites, macroscopic seepage appears to be the rate determining step of the overall process. Hence, this problem has received considerable amount of attention [5–10]. Typically, these models treat the composite as a deformable fiber bed saturated with a curing resin.

Material parameters to be incorporated into the constitutive models when handling integrative phenomena in OOA-processing are required. There are number of previous research on tow impregnation phenomena, mostly in the context of liquid composite molding (LCM) or of composites based on commingled yarns [11–17]. In such processes, flow commonly occurs under isothermal conditions and relatively high driving pressures. However, it has been established that impregnation in OOA processing may occur in non-isothermal conditions, such as during the initial temperature ramp, as well as during the isothermal dwell [18]. More importantly, the effect of material properties and process parameters for the compaction and saturation of partially impregnated thermoset prepreg has been the focus of many studies [19–23]. For example, Thomas et al. [24] used ultrasonic imaging to measure the flow rate of an epoxy resin film penetrating through the

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thickness of a single layer of woven carbon fabric in a VBO-process in order to calculate the permeability of the porous material via Darcy's law. In that sense the interaction between the sub-processes in different scale, prior to curing in this study, are interesting to be modeled using the material parameters obtained experimentally.

In this context, the resin flow relative to the fiber bed is governed by Darcy's law and it is considered to be coupled to the compaction behavior of the fiber bed. Compaction responses have been studied by several investigators for carbon fiber beds [10,25–30] and glass fiber fabrics [31,32]. Kim et al. [33] also did numerous experiments on dry and lubricated glass and carbon fiber beds. Toll [34] derived a general model that fits all the fiber bed compaction curves found in the literature. He emphasizes that this model is valid only if the fiber bed configuration is not modified by the condition of the experiment. A few examples of modeling the physically coupled sub-processes exists, e.g. Larsson et al. [35] where a generic two phase continuum framework is developed aimed for thermoplastic composite manufacturing. In the context of material characterization, there is a principal experimental difficulty to separate the fiber bed response from the fluid response, since the resin plays a significant part in the total response and it is difficult to determine the fluid pressure. Therefore, the fiber bed is typically tested in a dry form or it is impregnated with silicone oil after dissolving out the polymer matrix, cf. [33,34]. However the act of dissolving the resin out of the prepreg can change the fiber arrangement and therefore could affect the fiber bed compaction behavior.

1.3. Objectives

Concerning the simulation of OOA and VBO-processes for consolidation of thermoset composites, with all its complexity, a multi-phase continuum formulation is required in order to incorporate constitutive models with characterized material parameters experimentally for the relevant mechanisms in a unified way.

In the present paper, we adopt an experimental procedure that allows for the measurement of the compaction behavior directly from the actual prepreg. This technique allows the measurement of the material as it is, without any steps that could alter the fiber arrangement in the matrix. In addition, the technique is quite simple, and it requires little sophisticated instrumentation or equipment. In this development we exploit the recently developed generic two-phase continuum mechanical framework capable of handling the coupled two-phase porous media problem, coupling two different temporal and spatial scales at the same time, cf. Ref. [4]. Here, we consider the sub-models for the consolidation of partially impregnated prepreg based composites. The complex theory adapted here, as a distinct difference with similar works mentioned in the survey from modeling point of view, is an specialization of the theory of porous media. This framework was based on the work in [35,36] and was also adapted for modeling the consolidation of thermoplastic composites in [37]. The simulation mimics the experiment set up in terms of boundary conditions and multi-step loading situation. The model assessment is done against an experimental method and the comparison between the two is presented and discussed in detail in the final section of the paper. Our results are obtained for a commonly used composite system, followed by a series of calibration–validation simulations against the experimental data for 'HexPly® M21 T'.

2. Continuum modeling of wet-out in composites processing

To describe the continuum model for the considered category of composite manufacturing processes, such as compression molding and OOA-processes, we summarize in this section the key steps of a compressible solid phase and fluid phase continuum formulation, developed in [4,35]. Central to this formulation is the consideration of the phases with respect to their *volume fractions*, cf. Fig. 1, where the macroscopic volume fractions $n^\alpha[\boldsymbol{x},t]$ as the ratio between the local constituent volume and the bulk mixture volume. To ensure that each control volume of the solid is occupied with the solid–fluid mixture, we have the saturation constraints

$$n^s + n^f = 1, \quad 0 \leqslant n^s \leqslant 1 \quad \text{and} \quad 0 \leqslant n^f \leqslant 1 \tag{1}$$

The phases s,f relate to different reference configurations, whereby s,f-particles define the current configuration in Fig. 1 via individual deformation maps with the associated velocity fields $\boldsymbol{v} = \boldsymbol{v}^s = \dot{\boldsymbol{\varphi}}[\boldsymbol{X}]$ and $\boldsymbol{v}^f$, respectively. In particular, the mapping $\boldsymbol{x} = \boldsymbol{\varphi}[\boldsymbol{X}]$ characterizes the motion of the solid fiber network and $\dot{\bullet}$ denotes the material derivative with respect to the solid reference configuration B_0, cf. [4]. Related to this we consider the deformation gradient $\boldsymbol{F}$ defined by $\boldsymbol{F} = \boldsymbol{\varphi} \otimes \nabla$ with the Jacobian $J = \det[\boldsymbol{F}] > 0$. To define the liquid transport relative to the solid we introduce the Darcian velocity $\boldsymbol{v}^d = \phi^l \boldsymbol{v}^r$ with $\boldsymbol{v}^r = \boldsymbol{v}^f - \boldsymbol{v}$.

As alluded to in Fig. 1 the volume fractions of the phases are subdivided in terms of *constituents* of the meso-structure so that

$$n^s = \phi^p + \phi^v, \quad n^f = \phi^l \tag{2}$$

where ϕ^p is the fiber particle volume fraction, ϕ^v is the void space and ϕ^l is the volume fraction of the liquid constituent. Due to the process of void exclusions in the liquid infiltration of the fiber bed induced by microscopic wet-out the solid phase *densifies*. This is described by the logarithmic densification measure ϵ defined as

$$\epsilon = \log\left[\frac{\rho_0^s}{\rho^s}\right] \Rightarrow \dot{\epsilon} = -\frac{\dot{\rho}^s}{\rho^s} \tag{3}$$

where ρ^s is the variable solid density. Clearly, $\dot{\epsilon} < 0$ for a densification $\dot{\rho}^s > 0$, whereas the fluid density $\rho^f = \rho^l$ is considered incompressible, i.e. $\dot{\rho}^f = \dot{\rho}^l = 0$. It appears that the densification of the solid phase may be formulated in terms of the (reversible) fiber content φ in the dry region indicated in Fig. 2a and the irreversible wetting variable $0 \leqslant \xi \leqslant 1$ as

$$\frac{\rho_0^s}{\rho^s} = \frac{\varphi_0}{\varphi}\frac{1-\xi(1-\varphi)}{1-\xi(1-\varphi_0)}(1-\xi(1-\varphi_0)) \quad \text{with} \quad \varphi_0 = \frac{\rho_0^s}{\rho^p} \tag{4}$$

where ρ^p is the density of the (fiber) particles. Taking the logarithm of (4) yields the densification ϵ of the solid phase characterized as an additive decomposition into reversible and irreversible components as

$$\epsilon = \epsilon^e + \epsilon^p \quad \text{with} \quad \epsilon^e = \log\left[\frac{\varphi_0}{\varphi}\frac{1-\xi(1-\varphi)}{1-\xi(1-\varphi_0)}\right] \quad \text{and}$$
$$\epsilon^p = \log\left[1-\xi(1-\varphi_0)\right] \tag{5}$$

The reversible component ϵ^e is essentially related to the fiber content $\varphi > \varphi_0$, whereas the irreversible wetting densification ϵ^p is related to the saturation degree ξ.

2.1. Balance equations of mixture with a compressible solid phase

The *conservation of mass* for the two-phase material is expressed considering the conservation of mass for the individual phases in terms of the solid and fluid contents $M^s = Jn^f\rho^f$ and $M^f = Jn^s\rho^s$. The mass conservation is then expressed in view of Eq. (1) as

$$\dot{M}^s = 0, \quad \dot{M}^f + J\nabla\cdot\left(\rho^f\boldsymbol{v}^d\right) = 0 \rightsquigarrow J\boldsymbol{\nabla}\cdot\boldsymbol{v} - n_0^s e^\epsilon\dot{\epsilon} = -J\nabla\cdot\boldsymbol{v}^d \tag{6}$$