

## EXPERIMENTAL DETERMINATION AND NUMERICAL MODELLING OF PROCESS INDUCED STRAINS AND RESIDUAL STRESSES IN THICK GLASS/EPOXY LAMINATE

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### Abstract

In this work, a cure hardening instantaneous linear elastic (CHILE) model and a path dependent (PD) constitutive approach are compared, for the case of modelling strain build-up during curing of a thick composite laminate part. The PD approach is a limiting case of viscoelasticity with path dependency on temperature and cure degree. Model predictions are compared to experimentally determined in-situ strains, determined using FBG sensors. It was found that both models offer good approximations of internal strain build-up. A general shortcoming is the lack of capturing rate-dependent effects such as creep.

### 1. Introduction

In the manufacture of large commercial wind turbine blades, usually multiple blade components such as the blade root, shear web, suction- and pressure shells, are initially moulded separately then joined together using epoxy adhesives to form the complete wind turbine blade [1]. A general challenge involves ensuring precise dimensional accuracy of the various parts, such that the final assembly time needed is minimal. In large composite structures with thick laminate sections, avoiding large thermal and cure gradients through the thickness during processing is cumbersome. These through-thickness thermal and cure gradients are known to induce shape distortions and residual stresses [2]. Other mechanisms responsible include, the mismatch in thermal expansion of the constituent composite materials, laminate lay-up, curing temperatures, resin chemical shrinkage and tool-part interface interaction (see [3-7]). In large wind turbine blades, laminate thicknesses can be as much as 150mm at the blade root section in 40+ meter long blades. Processing of these thick composite laminates requires long cure cycles at elevated temperatures. Generally, E-glass/epoxy composites are the material of choice for laminates in commercial blades. Thermosetting epoxy resins are known to exhibit viscoelastic effects at elevated temperatures [8]. Hence creep and stress relaxation can occur during processing. Different viscoelastic (VE) numerical process models have been proposed for modelling curing of composites, see e.g. [4, 9-10]. These models offer good accuracy as they capture the matrix material behaviour well during curing. However, VE models require extensive material characterization in order to determine discrete relaxation times, weighting terms and temperature-time-cure shift functions. Furthermore, VE models are computationally heavy due to the added need of storing internal state variables. The number of these variables is directly dependent on the size of the Weichert- or Prony series used and their goodness of fit to experimental data – usually better fit is equal to larger series. Alternatives to VE models

include the well known Cure Hardening Instantaneous Linear Elastic (CHILE) approach, originally proposed by Bogetti & Gillespie (1991)[2], and the path-dependent (PD) model, proposed by Svanberg (2002)[11]. The PD model is a limiting case of linear viscoelasticity where rate dependency is replaced by a path-dependency on cure degree and temperature history. Both these approaches require less material data and are computationally fast. In this work, we investigate how adequate the above-named non-viscoelastic approaches are, at predicting process-induced internal strains in slow curing thick laminates. Model strain predictions are compared to experimentally determined process strains from a 52ply UD E-glass/epoxy laminate (app.46mm thick) using embedded Fibre Bragg Grating (FBG) sensors.

## 2. Constitutive model formulation

The main differences in the analysed constitutive approaches lies within their respective incremental matrix material stiffness expressions. Thermoset epoxy resins exhibit temperature- and cure degree dependent phase transformation within the liquid, rubbery and glassy states during curing [8, 12]. In the subsequent sections, a brief description of the two modelling approaches used to capture the mechanical behaviour of the epoxy matrix is given. In both approaches, the self-consistent field model micromechanics approach for a unidirectional continuous fibre reinforced composite was used to calculate effective laminate thermal expansion and chemical shrinkage.

### 2.1 CHILE approach

The CHILE approach is a further development of the incremental linear elastic model originally proposed by Bogetti & Gillespie (1992) [2]. In the CHILE approach, the epoxy matrix material is cure- and temperature dependent and expressed as:

$$E_m = \begin{cases} E_m^0 & T^* < T_{C1} \\ E_m^0 + \left( \frac{T^* - T_{C1}}{T_{C2} - T_{C1}} \right) (E_m^\infty - E_m^0) & T_{C1} \leq T^* \leq T_{C2} \\ E_m^\infty & T^* > T_{C2} \end{cases} \quad (1)$$

where cure dependency is included in the temperature:  $T^* = (T_g^0 + a_{Tg} * \alpha) - T$  (2).

In the equations above,  $E_m^0$  and  $E_m^\infty$  are the fully uncured (rubbery) and fully cured (glassy) resin modulus respectively. The critical temperatures  $T_{C1}$  and  $T_{C2}$  mark the bounds determining the linear variation of the modulus with  $T^*$ , which is cure dependent. The coefficients used in Eq. (1) and (2) are fit to the temperature dependent elastic response provided by the resin supplier [13]. In essence, Eq. (1) and (2) express a two-step temperature- and cure degree dependent resin modulus. This approach also allows softening of the fully cured resin if the cure temperature  $T$  is large enough to allow  $T^*$  to be smaller than  $T_{C2}$ . Physically this represents increasing the temperature of the vitrified resin resulting in transition back from glassy to rubbery state.

### 2.2 PD approach

The path-dependent constitutive material model approach is a limiting case of viscoelasticity. Linear viscoelastic materials, such as epoxy resins, are most commonly described using Weichert or Maxwell-Zener spring-damper models. These models are adequate because thermosetting polymers exhibit a bound in stress relaxation due to unbreakable cross-links in the polymer structure. Svanberg's simplified approach involves replacing the rate dependence by a path dependence on the strain, temperature and cure degree. In effect this results in an instantaneous stress relaxation at temperatures above  $T_g$ :

$$\sigma_{ij} = \begin{cases} C_{ijkl}^r \cdot (\varepsilon_{kl} - \varepsilon_{kl}^E) & , T \geq T_g(\alpha), \eta \rightarrow 0 \\ C_{ijkl}^g \cdot (\varepsilon_{kl} - \varepsilon_{kl}^E) - (C_{ijkl}^g C_{ijkl}^r) \cdot (\varepsilon_{kl} - \varepsilon_{kl}^E) & , T < T_g(\alpha), \eta \rightarrow \infty \end{cases} \quad (3)$$

where  $C_{ijkl}^r$  and  $C_{ijkl}^g$  are the rubbery and glassy state stiffness tensors,  $\varepsilon_{kl}$  and  $\varepsilon_{kl}^E$  are the mechanical and expansion (chemical + thermal) strains. The cure dependent glass transition temperature is determined using the DiBenedetto equation. In order to allow numerical implementation, incrementalization of the constitutive model is necessary, resulting in the following equation system:

$$\Delta\sigma_{ij} = \begin{cases} C_{ijkl}^r \cdot \Delta(\varepsilon_{kl} - \varepsilon_{kl}^E) - \beta_{ij}^l(t) & , T \geq T_g(\alpha) \\ C_{ijkl}^g \cdot \Delta(\varepsilon_{kl} - \varepsilon_{kl}^E) & , T < T_g(\alpha) \end{cases} \quad (4)$$

where the internal state variable  $\beta_{ij}^l(t)$  is updated in each time increment as:

$$\beta_{ij}^l(t + \Delta t) = \begin{cases} 0 & , T \geq T_g(\alpha) \\ \beta_{ij}^l(t) + (C_{ijkl}^g - C_{ijkl}^r) \cdot \Delta(\varepsilon_{kl} - \varepsilon_{kl}^E) & , T < T_g(\alpha) \end{cases} \quad (5)$$

Through the equation above, the stress history of the resin matrix is stored at temperatures below  $T_g$  and fully relaxed above  $T_g$ . For model requirements that must be fulfilled, see [11]. To sum up, the presented constitutive approaches estimate the resin stiffness behaviour as a function of temperature and cure degree. This is shown for the different models overlaid the fully cured resin modulus from the resin supplier in Fig 1. The main difference between the different constitutive models is the ability of the PD approach to relax stresses instantaneously once in glassy state once the cure temperature is larger than  $T_g$ .

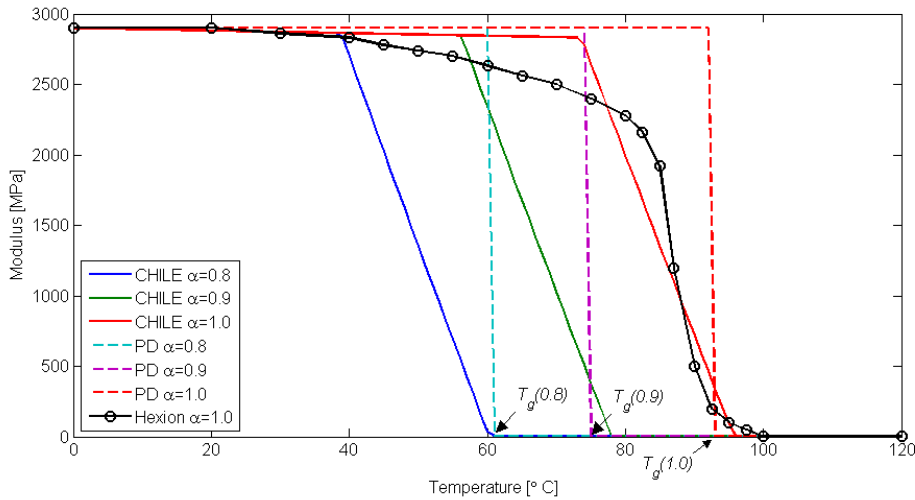


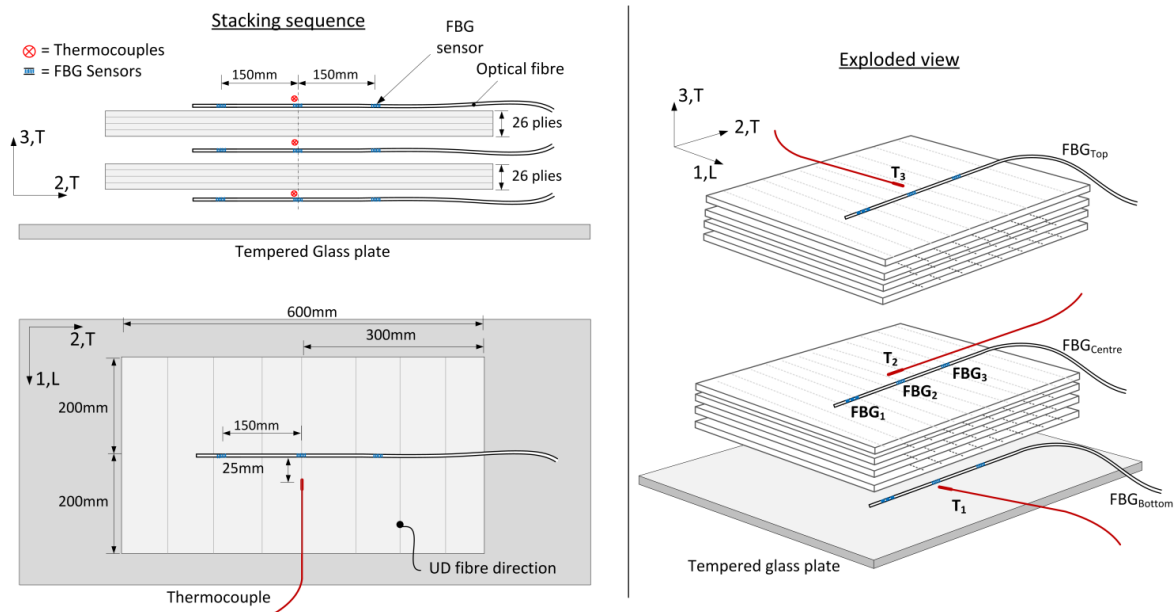
Figure 1. CHILE and PD approach resin modulus temperature and cure degree dependence

### 3. Experimental in-situ strain measurements

#### 3.1 Experimental setup

A glass/epoxy laminate plate measuring 400x600mm consisting of 52-layers (app. 46mm thick) UD E-glass fiber mats was vacuum infused on a 10mm tempered glass plate. Epikote RIM R 135/R 137 epoxy resin/hardener is used, which is a slow curing resin system developed for the wind turbine industry. J-type (Fe-CuNi) 2x0.5mm thermocouples and three optical fibres, consisting three FBG sensors each, were placed at the bottom-, mid- and top planes of the laminate plate, see Fig. 2. The optical fibres were placed perpendicular to the longitudinal reinforcement fibre direction. Data from the thermocouples were used for the de-

convolution of the thermal strains in order to obtain the mechanical strains measured by the wavelength shifts in the FBG sensors, for more details see Nielsen *et. al.* 2012 [14]. The entire layup (laminate and 10mm thick tempered glass plate) was placed on an electric heating plate with a digital temperature control unit, kept at a constant prescribed temperature of 40°C. During the entire process, data was logged from the mid-plane FBG sensors and all three thermocouples. The main goal with the experiment was to achieve a high exothermic peak temperature at the laminate centre with large temperature and cure degree gradients through the thickness. Furthermore, a long temperature hold was chosen such that viscoelastic creep or stress relaxation behaviour would be visible.



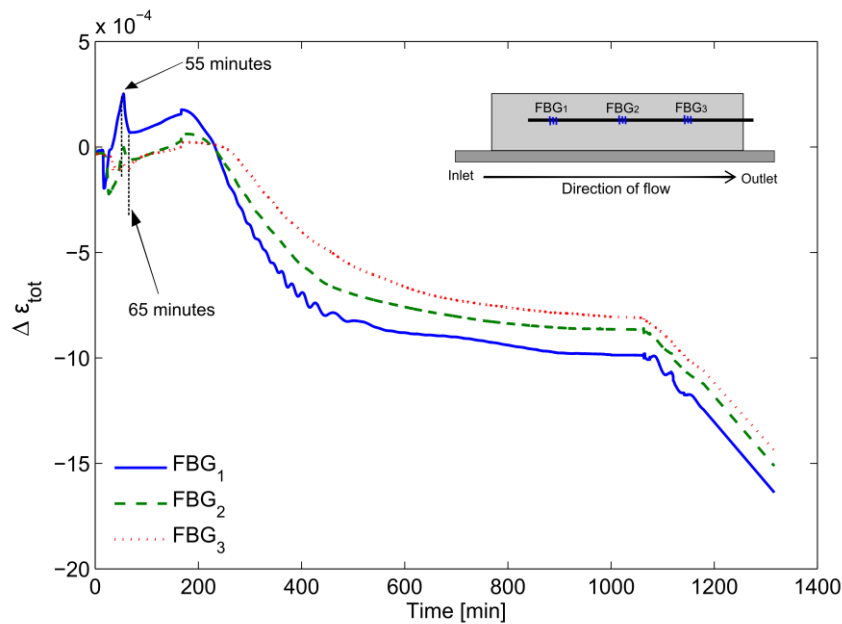
**Figure 2.** Laminate plate stacking sequence and sensor placement schematic

### 3.2 Experimental results

The total transverse strain variation  $\Delta\varepsilon_{tot}$  during infusion at the laminate middle plane is seen in Fig. 3. The fluctuations seen at the beginning of the process mark the instants when the resin flow front reaches the FBG sensors. After app. 55 minutes into the infusion process, the resin flow front reaches the outlet hose, marked in Fig. 3 by the sudden decrease in strain measurements as flow is drastically reduced. Finally, after app. 65 minutes the vacuum pressure is reduced to 60% to allow complete impregnation of the reinforcement fibres. The variations in strain measurements have mainly been driven by to resin flow. In the early stages of infusion prior gelation, caution should be taken in evaluating the measured strains, as perfect bonding between the host material and optical fibres does not yet exist. After gelation, temperature changes, resin chemical shrinkage effects and tool/part interaction primarily drive the changes measured. After the exothermic reaction takes place, compressive strains are measured, showing a difference in strains dependent of the FBG placement. During the constant temperature hold (i.e. between 500 and 1050minutes) a slight slope of the strain curves is seen. As app. 60% of chemical shrinkage is known to occur in epoxies prior gelation [15], it is mostly likely that the slope is due to viscoelastic creep of the polymer matrix.

Also seen in Fig. 3 are the fluctuations which arise during cooling. The build-up of high shear stresses formed at the tool/part interface is known to cause a stick-slip effect or separation, usually experienced during cooling [15,16].

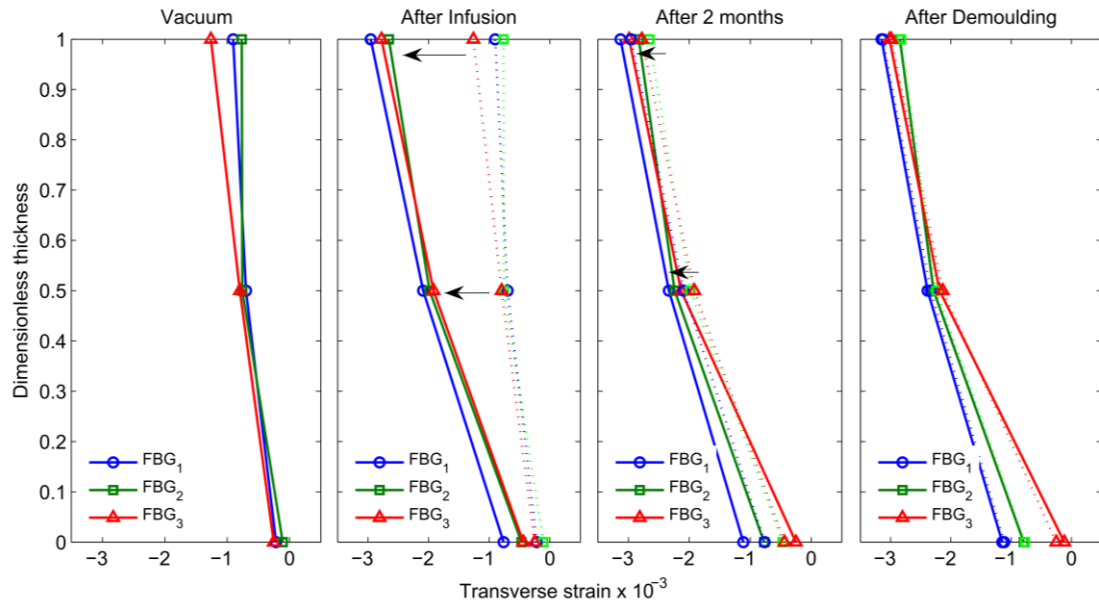
Finally, the total transverse strain from all three optical fibres ( $FBG_{Bottom}$ ,  $FBG_{Centre}$  and  $FBG_{Top}$  in Fig. 1) at various intervals is seen in Fig. 4. These measurements were carried out to further verify any viscoelastic behaviour. The intervals shown include: (i) prior infusion with the vacuum pressure applied, (ii) after infusion, (iii) after 2 months (iv) after demoulding. At each interval the laminate was at ambient temperature. In the figure, the strain state from the previous graph (dotted lines) are overlaid the current strains. The largest changes in strains occur during infusion, visible by the increase in compressive strains after infusion. A slight increase in strains is also seen after 2 months in ambient temperature, possibly due to creep or separation at the tool/part interface. Finally after demoulding, no significant change in strains is experienced.



**Figure 3.** Variation in process-induced total transverse strains at the laminate mid plane during infusion

#### 4. Numerical model predictions

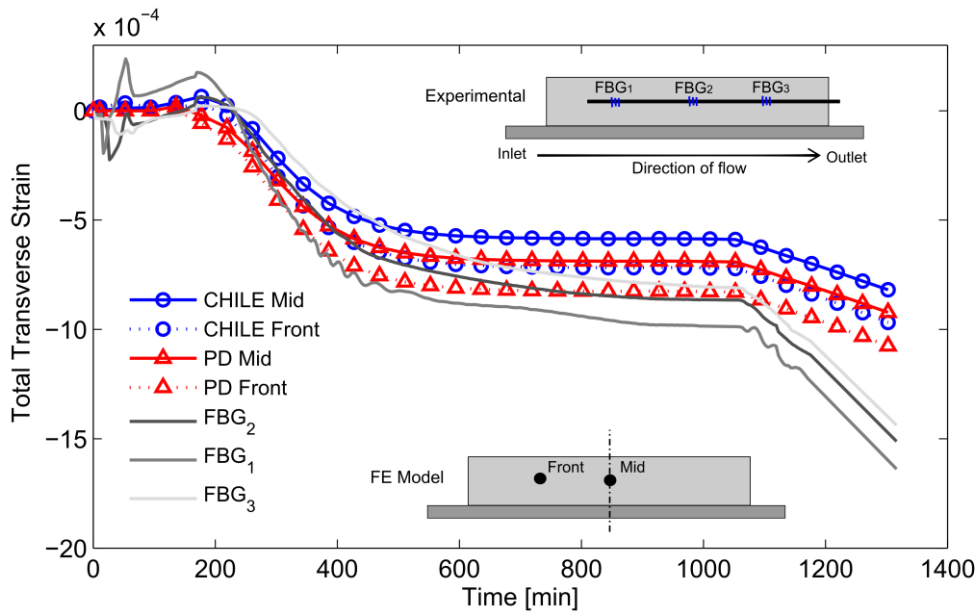
A 46[mm] thick glass/epoxy laminate plate is modelled using a sequentially coupled thermomechanical analysis setup. In the thermal step, all thermochemical aspects of the problem are calculated, i.e. the temperature, cure degree evolution and volumetric heat generation. In the mechanical step, chemical shrinkage, thermal expansion and the instantaneous laminate mechanical property evolution is calculated leading to predictions of process strains and stresses. The same thermal step is used for the CHILE and PD approaches. Further information on model settings and material parameters used is summarized elsewhere, see [14]. The different constitutive model total transverse strain predictions are seen in Fig. 5 compared to experimental data. As one can see, both models offer good predictions of the strain history during infusion. The largest compressive strains are measured at the model node corresponding to the  $FBG_1$  position in the experiment. This coincides well with the experimental data, although in real life the strain measurements show some dependency to resin flow. In the models, the larger strains away from the laminate mid region are a result of the part contraction during cooling and as chemical shrinkage progresses.



**Figure 4.** Total transverse strains from all embedded FBGs at various intervals during manufacturing. The dotted lines show the previous strain state overlaid the current strains at the measured time

When comparing the results from the different constitutive approaches, slightly larger compressive strains are achieved when using the path dependent (PD) approach. This is not due to stress relaxation but rather due to the fact that the  $T_g$  dependent transition from rubbery to glassy state occurs later than in the CHILE approach, resulting in a softer material for a longer period. The reason no stress relaxation occurs is because the cure temperature is not higher than the resin  $T_g$  at any time after gelation occurs. Hence even though the simplified approach taken in the PD model accounts for stress relaxation in its constitutive relations, the need to further capture creep-type behaviour, as described earlier, still exists when considering processes where low temperatures are mainly used. Fig. 6 illustrates the temperature evolution at the laminate centre for the initial 500minutes of the process, comparing the PD approach curing temperature  $T_{Mid Ref}$  to  $T_g(\alpha)$  and the CHILE approach  $T^*$  to the bounding temperatures  $T_{c1}$  and  $T_{c2}$ . From the figure, one can see that the PD approach has a transition from rubbery to glassy state later than in the CHILE model.

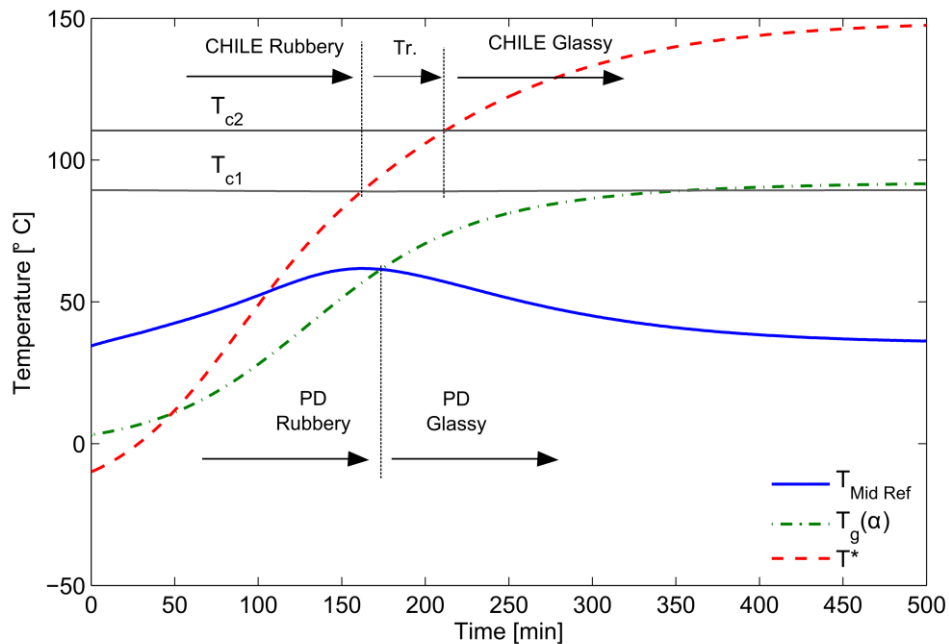
In the FE models, a tied mechanical boundary condition at the tool/part interface was used, which physically represents perfect bonding between the surfaces. This is not entirely descriptive of the tool/part interaction taking place during infusion where sliding and sticking conditions exist at various phases of the infusion process, governed by the resin material phase transition and thermal expansion, as shown in [17].



**Figure 5.** FE CHILE and PD model predictions of total transverse strains during infusion compared to experimental FBG measurements

### 5. Conclusion

In the present work, two different constitutive approaches used to model curing of composite laminates and the resulting process induced residual stresses and strains build-up were investigated. As a case, a 52 ply thick laminate plate was modelled and compared to experimentally determined in-situ strain measurements. Regarding the cure hardening, instantaneous linear elastic model approach, generally good predictions of the process strains were achieved, though the model underestimates the compressive strains that build up during infusion.



**Figure 6.** Comparison of the transition temperature governing the resin modulus stiffness evolution in the PD and CHILE constitutive approaches. “Tr.” Denotes the CHILE approach transition zone, see also Fig. 1.



Regarding the path dependent model, which is a limiting case of linear viscoelasticity, slightly better strain predictions were achieved. This was however not due to calculation of transition dependent stress relaxation. I.e. for slow curing infusion processes, where cure temperatures are not that high, time dependency is needed to accurately predict process strains. Furthermore, a more accurate model of the tool/part interface is needed, taking sliding and sticking contact behaviour into account, if trustworthy modelling results are to be achieved.

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#### References

- [1] F. Jensen, "Ultimate Strength of a Large Wind Turbine Blade", PhD Thesis, Risø National Laboratory for Sustainable Energy, Roskilde, Denmark, 2008.
- [2] T. Bogetti and J. Gillespie "Process-induced stress and deformation in thick-section thermoset composite laminates". *Journal of Composite Materials*, **Vol. 26- 5**, pp 626-660, 1992.
- [3] J. Favre "Residual Thermal Stresses in Fibre Reinforced Composite Materials – A Review". *Journal of the Mechanical Behaviour of Materials*, **Vol. 1- 1-4**, pp 37-53, 1988.
- [4] Y. Kim and S. White "Stress Relaxation Behaviour of 3501-6 Epoxy resin During Cure". *Polymer Engineering and Science*, **Vol. 36-23**, pp 2852-2862, 1996.
- [5] J.M. Svanberg "An Experimental Investigation on Mechanisms for Manufacturing induced Shape Distortions in Homogeneous and Balanced Laminates". *Comp: Part A: Vol 32*, pp.827-838, 2001.
- [6] Johnston, A. "An integrated model of the development of process-induced deformation in autoclave processing of composite structures". PhD Thesis, University of British Columbia, Vancouver, Canada, 1997.
- [7] Twigg G., Poursartip A. and Fernlund G. "Tool-part interaction in composites processing. Part II: Numerical Modeling". *Composites Part A; Vol.35*, pp.135-141, 2004.
- [8] J.D. Ferry, "Viscoelastic Properties of Polymers" John Wiley & Sons, inc., 2<sup>nd</sup> Edition, 1961.
- [9] S.L. Simon, et. al. "Modeling the evolution of the dynamic mechanical properties of a commercial epoxy during cure after gelation". *Journal of Applied Polymer Science*, **Vol. 76**:pp.495–508, 1999.
- [10] M.A. Zocher, S.E. Groves and D.H Allen " A three-dimensional Finite element Formulation for Thermoviscoelastic Orthotropic Media". *Int.Journal for Numerical Methods in Engineering*, **Vol. 40**:pp.2267–2288, 1997.
- [11] J.M. Svanberg "Predictions of Shape Distortions Part I: FE Implementation of a Path Dependent Constitutive Model ". *Composites: Part A: Vol 35*, pp.771-721, 2004.
- [12] J. K. Gillham. Formation and Properties of Thermosetting and high Tg Polymeric Materials. *Polymer Engineering and Science*, Volume:**Vol.26**:p.1429–1433, 1986.
- [13] Momentive Speciality Chemicals Inc. Epikote RIMR 135 Technical Data Sheet, 2006.
- [14] M.Nielsen, J.Schmidt, J.Hattel, T.Andersen and C. Markussen "In-situ measurement using FBGs of process-induced strains during curing of thick glass/epoxy laminate plate: Experimental results and Numerical modelling". *Wind Energy*, **Submitted** April 2012.
- [15] L. Khoun, R. de Oliveira, V. Michaud and P. Hubert, " Investigation of process-induced strains development by fibre Bragg grating sensors in resin transfer moulded composites" *Composites: Part A*, **Vol. 42**, pp.274-282, 2011.
- [16] R. de Oliveira, S. Lavanchy, R. Chatton, D. Costantini, V. Michaud R. Salathe and J.-A.E. Manson, "Experimental Investigation of the effect of the mould thermal expansion on the development of internal stresses during carbon fibre composite processing" *Composites: Part A*, **Vol. 39**, pp.1083-1090, 2008.
- [17] Twigg G., Poursartip A. and Fernlund G. "Tool-part interaction in composites processing. Part I: Experimental investigation and analytical model". *Composites Part A; Vol.35*, pp.121-133, 2004.