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## Towards optimisation of load-time conditions for producing viscoelastically prestressed polymeric matrix composites

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#### A R T I C L E I N F O

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#### ABSTRACT

A viscoelastically prestressed polymeric matrix composite (VPPMC) is produced by applying a tensile creep load to polymeric fibres, the load being released before the fibres are moulded into a polymeric matrix. The viscoelastically recovering fibres induce compressive stresses within the matrix, which can improve mechanical properties by up to 50%. This study investigates the feasibility of reducing the creep loading period for VPPMC production. By using nylon 6,6 fibres, we have demonstrated that the previously adopted viscoelastic creep strain, requiring 330 MPa for 24 h, can be achieved over a shorter duration,  $t_n$ , using increased creep stress. Thus  $t_n$  was 92 min at 460 MPa and 37 min at 590 MPa. Subject to avoiding fibre damage however, it may be possible to reduce  $t_n$  further. From the three creep settings, elapsed recovery strain values were similar, as were the Charpy impact test data from corresponding VPPMC samples; i.e. there were no significant differences in impact energy absorption, these being ~56% greater than their control (unstressed) counterparts.

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#### 1. Introduction

Previous publications have demonstrated that viscoelastically prestressed polymeric matrix composites (VPPMCs) provide improved mechanical performance relative to counterparts without the prestress. These improvements are most evident for Charpy impact toughness [1-8] and flexural moduli [8-10], in which increases of typically 30-50% have been obtained; also tensile tests have demonstrated modest increases in strength (>15%) [11]. The VPPMC production process involves two stages: (i) polymeric fibres are stretched under a constant load for a period of time so that they undergo viscoelastic creep; (ii) the fibres are released from the load and subsequently moulded into a resin matrix (e.g. polyester or epoxy). The previously strained fibres continue to attempt viscoelastic recovery after the matrix has solidified, and this produces compressive stresses in the matrix, which are counterbalanced by residual tension within the fibres. It has been suggested that four mechanisms, resulting from prestress effects, may contribute towards the observed improvements in mechanical properties [5]; i.e. (i) matrix compression impedes crack propagation from external tensile forces; (ii) matrix compression attenuates dynamic overstress effects, reducing probability of fibre fracture outside the immediate area of impact; (iii) residual fibre tension causes the fibres to respond more collectively and thus more effectively to external loads; (iv) residual shear stresses at the fibre—matrix interface regions promote (energy absorbing) debonding over transverse fracture.

A more conventional approach to producing prestressed PMCs is to exploit elastic recovery. Here, fibres (e.g. glass or carbon) are stretched elastically within a mould whilst the surrounding resin matrix solidifies. The resulting elastically prestressed PMCs (EPPMCs) can provide similar mechanical property improvements to those offered by the VPPMC approach, in the form of laminates [12–14] and unidirectional fibre-reinforced composites [15–19]. VPPMC methodology requires the use of polymeric fibres with appropriate viscoelastic properties and most of the research to date has involved nylon 6,6 fibres [1–6,9,11]. Clearly, these fibres are, in terms of strength and stiffness, mechanically inferior to the fibres that can be used for EPPMCs, although performance enhancement has been recently demonstrated with nylon 6,6 fibres (for prestress) commingled with Kevlar fibres [8]. Moreover, VPPMCs using viscoelastically generated prestress from other reinforcements have been successfully demonstrated, i.e. UHMWPE fibres [7,10] and bamboo [20].

Since the fibre stretching and moulding operations are decoupled, the two-stage approach used in VPPMC production





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offers great flexibility. A creep load can be applied to a fibre tow with relatively simple equipment. Also, following release of the load, the fibres can be chopped to any length and placed in any orientation within any mould geometry that can be filled with a matrix resin. To date however, all VPPMC-based studies within our laboratory have utilised a creep loading period of 24 h [1–11]. Although this is a convenient period for research purposes, such a lengthy duration would be less practical for VPPMC production in a commercial environment. The purpose of this paper is to consider the first steps towards process optimisation by significantly reducing the creep loading period for VPPMC production. As nylon 6,6 is the most established fibre reinforcement for VPPMCs, this will be the material under investigation.

#### 2. Background

Fig. 1 shows schematically, the strain—time characteristics of a polymeric creep-recovery cycle, with time-dependent components represented by functions based on the Weibull or Kohl-rausch—Williams—Watts function [21]. For creep,  $\varepsilon_{ctot}(t)$  is the total strain at time *t*, under an applied constant stress:

$$\varepsilon_{\text{ctot}}(t) = \varepsilon_{\text{i}} + \varepsilon_{\text{c}} \left[ 1 - \exp\left( - \left( \frac{t}{\eta_{\text{c}}} \right)^{\beta_{\text{c}}} \right) \right]$$
(1)

Here,  $e_i$  is the instantaneous strain from initial application of the stress and the  $e_c$  function is the time dependent creep strain where  $\eta_c$  is the characteristic life and  $\beta_c$  is the shape parameter. Following removal of the creep stress and the instantaneous recovery  $e_e$ , the remaining recovery strain,  $e_{rvis}(t)$  is:

$$\varepsilon_{\rm rvis}(t) = \varepsilon_{\rm r} \left[ \exp\left( - \left( \frac{t}{\eta_{\rm r}} \right)^{\beta_{\rm r}} \right) \right] + \varepsilon_{\rm f}$$
(2)

The  $\varepsilon_r$  function is the time dependent recovery strain with  $\eta_r$  and  $\beta_r$  being the Weibull parameters analogous to Eq. (1). The (non-recoverable) strain from viscous flow is represented by  $\varepsilon_f$ .

Clearly, in order to reduce the creep time applied to polymeric fibres for VPPMC production, the applied stress must be increased from the 'standard' 24 h creep stress of ~340 MPa [4–9,11] applied to nylon 6,6 fibres. Using published creep data [22], nylon 6,6 fibre has shown approximately linear viscoelastic properties up to ~50 MPa creep stress over a period exceeding 1000 h but there is increasing deviation from linear viscoelasticity below 100 h [21].



Fig. 1. Schematic tensile creep-recovery strain cycle for a polymeric material.

Thus attempting to predict the required creep stress to achieve similar results in a much shorter time than the 24 h creep cycle may be unreliable. Other factors to consider are whether a much higher creep stress (i) increases the risk of failure from fibre fracture during the creep cycle and (ii) causes unwanted changes to the fibre properties. In terms of (ii), the standard 24 h creep stress has been demonstrated to show no adverse effects on the fibres, such as surface damage or changes in short-term tensile test parameters [11].

By considering the above points, an empirical approach is adopted and Fig. 2 illustrates the basic principle. Eq. (1) is used to fit a curve to strain data from the standard run at 24 h, so that after instantaneous strain  $\varepsilon_{i1}$ , the time-dependent strain value,  $\varepsilon_c(24)_{std}$ , can be found. Subsequent runs, performed at stress values,  $\sigma_n$ , higher than the standard run, will also provide from Eq. (1), strain values  $\varepsilon_c(t_n)$  equal to  $\varepsilon_c(24)_{std}$ , where  $t_n < 24$  h. Again,  $\varepsilon_c(t_n)$  excludes the corresponding instantaneous strain,  $\varepsilon_{i2}$ . Therefore, a value for  $t_n$  which approaches the shortest practical creep time,  $t_{min}$ , can be determined, consistent with other factors (no fibre damage) outlined above.

The next step is to compare measurements of recovery strain as a function of time from a run subjected to creep up to  $t_n$ , with those obtained from a standard creep run. It may be expected that fitting the data to Eq. (2) should reveal similar parameter values from both runs.

The final step is to validate the effectiveness of VPPMCs produced under the  $t_n$  creep conditions. Since Charpy impact testing has been used for the majority of investigations into the performance of nylon fibre-based VPPMCs [1–6,8], this is the most appropriate evaluation method. Thus batches of VPPMC samples using  $t_n$  can be compared with similar batches produced under standard (24 h) creep conditions.

#### 3. Experimental

#### 3.1. Fibre evaluation

In contrast with previous VPPMC studies using nylon 6,6 fibre [1–6,8,9,11], the fibre used in this study was obtained from an industrial supplier, Ogden Fibres Ltd, UK. Both new and old (i.e. previously studied) fibre materials were continuous untwisted multifilament yarns of ~94 tex; however, scanning electron microscopy (SEM) was used to compare samples of new and old yarns,



**Fig. 2.** Reducing the fibre creep time from 24 h to  $t_n$  by equalising the creep strain from a higher stress,  $e_c(t_n)$ , with  $e_c(24)_{std}$ .

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