# VISCOELASTICALLY PRESTRESSED COMPOSITES – WHERE NEXT?

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**Abstract**
Producing a viscoelastically prestressed polymeric matrix composite (VPPMC) involves (i) subjecting polymeric fibres to tensile creep, (ii) releasing the creep load, then (iii) moulding the loose fibres into a matrix. Following matrix solidification, the viscoelastically recovering fibres exert compressive stresses in the matrix, thereby improving mechanical properties by up to ~50%. This paper provides a brief overview of VPPMC research, followed by recent evidence of an electric charge phenomenon, resulting from viscoelastic recovery mechanisms, which may benefit fibre-matrix interactions. Potential applications are discussed, which include impact-resistant structures, shape-changing (morphing) structures, dental materials and prestressed precast concrete.

## 1. Introduction

Although prestressed concrete is a familiar material, interest in using prestress within polymeric matrix composites, to improve mechanical properties, is comparatively recent. Elastically prestressed polymeric matrix composites (EPPMCs) exploit prestressed concrete principles, as fibres within the composite are stretched to maintain an elastic strain during matrix curing. Compressive stresses are produced in the matrix (after curing), which are counterbalanced by residual fibre tension. Research with unidirectional glass fibre and carbon fibre EPPMCs has shown increases in impact resistance, strength and stiffness of 25–50% compared with control (unstressed) counterparts [1-5]. These improvements can be explained by the residual stresses (i) impeding or deflecting propagating cracks and (ii) reducing composite strains resulting from external bending or tensile loads [1-3]. Thus elastic prestressing within a PMC can offer significant benefits; however there are potential drawbacks. First, fibre tension must be applied during matrix curing, which may impose restrictions on fibre length, orientation and spatial distribution, thereby compromising mould geometry [6]. Also, for laminate production, it is said that stretching rig design with appropriate fibre clamping can be technically challenging [7, 8]. The second drawback arises from the matrix being a polymeric material: elastically generated prestress will encourage localised matrix creep to occur near fibre-matrix interfaces, which could cause a gradual deterioration in prestress [6].

The use of viscoelastically generated prestress offers an alternative approach to EPPMC methodology. This paper summarises a recently published overview [9] of research into viscoelastically prestressed PMCs (VPPMCs), with updated information. This is followed by a review of our latest findings on some fundamental aspects of VPPMCs, leading to discussions on future directions.

## 2. VPPMC Principles

VPPMC production involves the following stages: (i) high-strength polymeric fibres are stretched over a period of time, so that they undergo (viscoelastic) creep; (ii) the creep load is subsequently released, so that they are in a loose state; (iii) the (unconstrained) fibres are then moulded into a matrix. Following matrix solidification, these previously strained fibres continue to attempt contraction through viscoelastic recovery. This fibre recovery occurs gradually; hence a prestress state comparable to an EPPMC can be achieved. In contrast with EPPMCs however, VPPMCs offer flexibility in production, since fibre stretching and moulding operations are decoupled. Thus relatively simple equipment can be used for applying a creep load to fibre tows. As fibres are in a loose state before moulding, they can be cut to any length and positioned in any orientation within any shape of mould that can be filled with a matrix resin.

VPPMCs also offer benefits in terms of product longevity. Localised matrix creep at the fibre-matrix interface regions can be expected to occur in EPPMCs, leading to some deterioration in prestress with time; an effect that has (to some extent) been observed by Mostafa *et al* [10]. Although this can also be expected to occur within a VPPMC, the effect would be offset by active responses from longer term viscoelastic recovery mechanisms within the polymeric fibres [6]. Nevertheless, a potentially major limitation with VPPMCs is that viscoelastic activity is temperature-sensitive. Thus viscoelastically generated prestress could deteriorate or become ineffective if a VPPMC is subjected to high-temperature curing cycles or long-term exposures to hot ambient conditions. This aspect is addressed in Section 3.2.

Essentially, VPPMC principles exploit the basic creep-recovery strain cycle for a polymeric material [11], as shown in Fig. 1. When a creep load is applied, an instantaneous strain $\varepsilon_i$, occurs, followed by a time-dependent creep strain $\varepsilon_c(t)$, until the load is released. Elastic recovery $\varepsilon_e$, occurs immediately on load release. Subsequent viscoelastic recovery $\varepsilon_r(t)$, is of vital importance to viable VPPMC production, in both magnitude and timescale. Thus any contribution from viscous flow $\varepsilon_f$ (due to permanent molecular slippage from creep), should be minimal, as this will reduce the contribution from $\varepsilon_r(t)$.

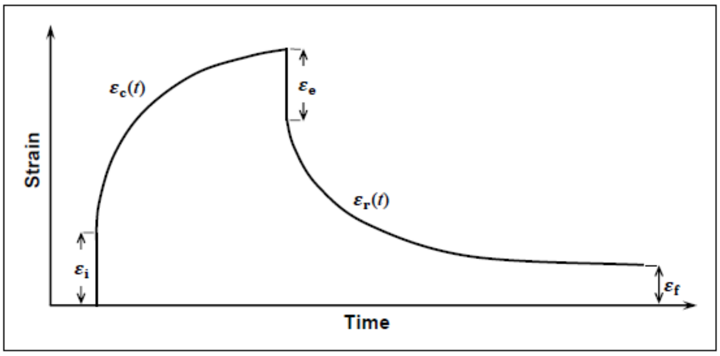


**Figure 1.** Schematic of the tensile creep-recovery strain cycle for a polymeric material [11].

Early work [12, 13] focused on determining the feasibility of VPPMC principles. Nylon 6,6 was selected for study, as it is a readily available, high strength polymeric fibre. It was found that subjecting annealed nylon tows to a 24 h creep load of ~330 MPa, the magnitude and timescale of the viscoelastic recovery strain was suitable for VPPMC production. Later work [14] demonstrated that annealing (150°C for 0.5 h) had no detrimental effect on fibre strength and stiffness. To demonstrate whether viscoelastic recovery mechanisms could provide a recovery force within a matrix material, Fig. 2(a) shows the result of an early experiment [13]. Here, nylon 6,6 monofilament was annealed and then subjected to creep, before being moulded into a thin, transparent polyester resin matrix. As Fig. 2(a) shows, a (compressive) stress pattern can be seen under polarised light in the 'test' (VPPMC) sample, in contrast with the 'control' (unstressed) counterpart. Later work [9, 15] demonstrated that a substantial recovery force could be developed within a composite matrix. Here, annealed nylon 6,6 tow was subjected to creep and following removal of the creep load, the loose tow was transferred to a bespoke force measurement rig and allowed to contract to a fixed strain (~2%) within a short time $\Delta t$, to become taut. This enabled the resulting viscoelastic recovery force to be monitored. As shown in Fig. 2(b), the force was found to increase with time and was predicted to reach a limiting value of 15.4 MPa (i.e. 4.8% of applied stress).

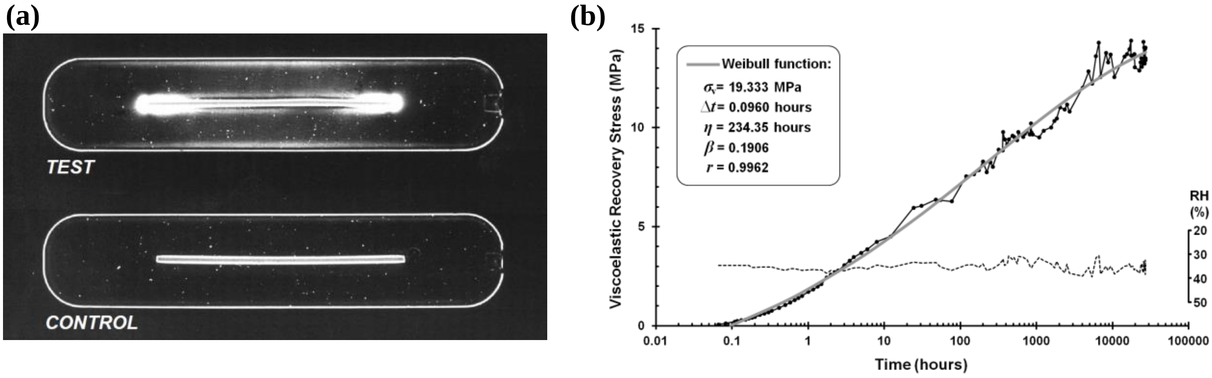


**Figure 2.** (a) nylon 6,6 monofilaments (1.6 mm diameter) moulded in polyester resin samples (150 × 30 × 2 mm) under cross-polarised light: a stress pattern can be clearly seen in the 'test' sample due to viscoelastic recovery (from 60 MPa 24 h creep), in contrast with the (unstressed) 'control' sample [13]; (b) viscoelastic recovery stress output (force exerted across fibres) from nylon 6,6 yarn (320 MPa 24 h creep): the curve represents a Weibull-based equation fitted to the data [9].

## 3. VPPMC Mechanical Performance and Durability

### 3.1. Mechanical Tests

Since their inception, the principal method for assessing VPPMC mechanical performance has been to produce batches of unidirectional fibre composite samples for Charpy impact testing. Each batch was produced by open casting two strips of polyester resin, one strip embedded with a continuous length of 'test' (previously annealed then stretched) nylon 6,6 fibres, the other with 'control' (annealed, not stretched, but otherwise identical) fibres. Stretching rig capacity limited the fibre volume fraction, $V_f$, to ~2%, but this facilitated post-fracture analysis. Results from several studies have consistently demonstrated that the VPPMC test samples absorb typically 25–30% more impact energy than their control (unstressed) counterparts, with increases of 50% or more in some cases [6, 12-19]. Fig. 3 shows typical test and control samples after impact testing; the impact-induced fibre-matrix debonding in the test sample is clearly greater than the control. A triggering effect was considered to be the principal mechanism [17]. Here, prestress enhances shear stresses between fibres and matrix and, during impact, these stresses are triggered to promote fibre-matrix debonding, in preference to transverse fracture of the composite sample. The energy absorbed by debonding is greater than that needed for transverse fracture, hence the increase in energy absorption by the VPPMC samples. This triggering mechanism had also been proposed in earlier work with glass fibre EPPMCs [2].

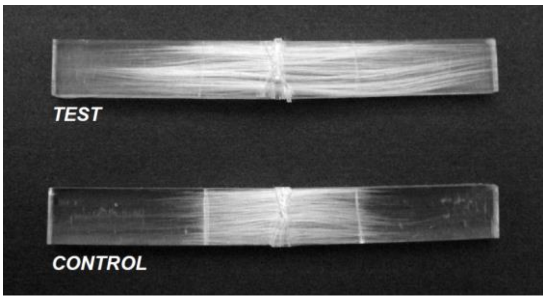


**Figure 3.** Typical appearance of test (VPPMC) and control (unstressed) samples following impact testing; note the greater region of fibre-matrix debonding in the test sample [9].

To evaluate flexural stiffness [20], batches of nylon fibre-based composite samples (200 × 10 × 3.5 mm) were produced by open casting, similar to Charpy testing. Samples were tested using three-point bending, under conditions similar to ASTM D790M recommendations in terms of support pin dimensions and a span/thickness ratio of ~30. The flexural modulus, $E$(t), was determined from deflections measured at $t$ = 5 s (representing elastic deformation) and 900 s (short-term creep). For both measurements, $E$(t) was found to increase by ~50% due to viscoelastically generated prestress.

Tensile testing [14] required the use of closed moulding, as composite samples of only 1 mm thickness were required (i.e. 200 × 10 × 1 mm), to meet appropriate test standards. A wide range of $V_f$ values was evaluated (16–53%) and, as expected, strength and stiffness improved with increasing $V_f$ (e.g. tensile strengths were 130 MPa at 16% and 420 MPa at 53%). Prestress-induced increases were observed in these parameters, but only at intermediate $V_f$ values. An optimum $V_f$ value (~35–40%) was indicated, at which the benefits from prestressing were maximised; the increases for strength, modulus and strain-limited toughness exceeding 15, 30 and 40% respectively. This optimum $V_f$ is attributed to competing roles between fibres and matrix. At lower $V_f$, too few fibres results in less compressive stress; at higher $V_f$, there are too many fibres, thus reducing the matrix cross-sectional area available for compression.

### 3.2. Durability

As highlighted in Section 2, fibres within a VPPMC must possess long-term viscoelastic recovery characteristics, to (i) offset the potential for deterioration in prestress from localised matrix creep, especially at fibre-matrix regions and (ii) maximise resistance to elevated ambient temperatures. This capability was evaluated through recovery strain measurements on nylon 6,6 fibres after they were subjected to the creep loading conditions used for VPPMC production. Viscoelastic activity was successfully demonstrated through real-time recovery strain measurements up to 4 years and accelerated ageing (using time-temperature superposition) up to 100 years at 20°C [16]. Beyond this however, strain measurements became impractical and instead, Charpy impact test (and control) samples were subjected to predetermined accelerated ageing conditions. Impact testing results have demonstrated no deterioration in impact performance for at least 25 years at a constant ambient temperature of 50°C [18]. Clearly, this suggests that VPPMC technology is viable for most practical applications. Moreover, although VPPMC processing with high temperature matrix curing cycles could be restricted, several hours exposure to a moderately raised curing temperature of (for example) 80°C should be feasible, whilst maintaining an acceptable (subsequent) duration of operation at lower ambient temperatures.

VPPMC performance at different ambient temperatures is another aspect of durability. A recent study [21] of VPPMC Charpy impact behaviour from -25°C to 45°C demonstrated that impact energy absorption by VPPMC samples was greater than their control counterparts over the full temperature range, the increases being ~40% at ≥ 20°C, reducing to ~20% at lower temperatures. Although energy absorption from VPPMC samples was principally from fibre-matrix debonding, resin impact toughness decreased at lower temperatures to promote matrix cracking, this being more prominent in the control samples (prestress impeding the effect). Thus control sample performance was improved.

### 3.3. Mechanical Performance of Alternatives to Nylon Fibre-Based VPPMCs

Nylon 6,6 fibre VPPMCs have been used as the principal research vehicle; however, polymeric fibres with mechanically superior properties to nylon could be utilised, provided they have appropriate viscoelastic properties. For example, ultra-high molecular weight polyethylene (UHMWPE) fibre-based VPPMCs have shown increases of 20–40% in flexural modulus [22] and Charpy impact energy absorption [23]. Another option is to exploit the use of fibre commingling. Thus nylon 6,6 fibres, used for creating viscoelastically generated prestress, could be commingled with glass or carbon fibres. Other fibres may include aramid (Kevlar) fibres, which are stronger and stiffer than nylon 6,6 fibres. An initial study of nylon 6,6–Kevlar fibre hybrid composites by Charpy impact and flexural stiffness testing [24] has demonstrated that (i) hybrid composites (with no prestress) absorb more impact energy than Kevlar fibre-only composites, due to the nylon fibre ductility; (ii) prestress further increases impact energy absorption in the hybrid case by up to 33% and (iii) prestress increases flexural modulus by 40% in the hybrid composites. Eco-friendly VPPMCs based on plant fibres are also a possibility. Cui et al [25] investigated VPPMCs based on bamboo slivers and found that flexural toughness could be increased by 28%. Our latest studies have involved investigations into regenerated cellulose/viscose continuous fibres for VPPMC production [26]. We found that the VPPMC samples demonstrated an increase of up to 20% in tensile strength and modulus and a comparable improvement in flexural properties.

## 4. VPPMC Fundamental Aspects – Recent Findings

### 4.1. Towards Process Optimisation

Although a creep stress applied for 24 h is convenient for research purposes, this would be unsuitable for commercial VPPMC production and recent work has focused on reducing the creep time by increasing the applied creep stress. By using nylon 6,6 fibres, it was found that our 'standard' viscoelastic creep strain, requiring 330 MPa for 24 h, could be achieved over a shorter time; i.e. 92 min at 460 MPa and 37 min at 590 MPa. Charpy impact test data from corresponding VPPMC samples showed no significant differences in impact energy absorption, these being ~56% greater than their control counterparts. If fibre damage can be avoided, it may be possible to reduce the creep time down to several minutes [19].

### 4.2. The VPPMC Fibre-Matrix Interface and Polymer Fibre Molecular Phenomena

Although we have made notable progress in understanding viscoelastic recovery forces, the fibre-matrix interactions within VPPMCs has been poorly understood. We recently addressed this by studying composite samples with the scanning electron microscope mirror effect (SEMME). SEMME has been used by others to investigate the dielectric behaviour of fibre-reinforced PMCs; e.g. glass fibre/epoxy resin [27]. Here, fibre/matrix interface regions were found to play a major role in the trapping or diffusion of charges, charge diffusion being associated with high interface strength. SEMME involves irradiating an insulating sample in an SEM with a high voltage (10s of kV). This causes negative charges to become trapped and stabilised within the sample, producing an electric field in the SEM sample chamber. The sample can then be observed with a lower energy electron beam (100s of volts), which is reflected by the electric field. The set-up is thus comparable to a convex mirror in visible light.

By using SEMME and comparing results from VPPMC samples with control counterparts, ~30% fewer trapped negative charges were found in the former, implying that the VPPMCs possess higher fibre-matrix interfacial strengths. Also, tensile test results on similar samples supported these findings. It was suggested that viscoelastic activity within the fibres could influence electric charge interactions at the fibre-matrix interface regions [28]. Mechanical stresses, when applied to an insulating material, lead to an injection of electric charges, so this may be expected to occur when fibres are subjected to creep for VPPMC production. The proposed explanation was that viscoelastic recovery mechanisms within the fibres release these charges and, in a VPPMC, interactions with these charges may lead to reducing the influence of defects at the interface regions. As reported in Section 3.2, viscoelastic recovery from these fibres is a long-term phenomenon; it is suggested to occur through the time-dependent triggering of molecular jumps, with longer term activity being represented by sites triggered through very long time constants [11, 29]. Thus it was speculated in [28] that the dielectric and viscoelastic properties of a polymer correspond to the same movement of molecular chains; hence the release of trapped charges may concur with the triggering of molecular jumps during viscoelastic activity.

Since viscoelastic recovery is a long-term mechanical phenomenon, fibres within a VPPMC should release electric charges over the same timescale. Not only might this improve fibre-matrix adhesion, as identified in [28], fibre hydrophobicity could also be affected. For insulating materials, there is a strong correlation between the presence of electric charge and loss of hydrophobicity [30]. Thus viscoelastically recovering fibres could be more hydrophilic than their control counterparts. Fig. 4 presents recent (previously unpublished) findings, using water droplets on nylon monofilaments and fibres. Both indicate reduced hydrophobicity as a result of viscoelastic recovery effects. This suggests that such fibres may adhere more strongly to water-based matrix materials, such as cement (Section 5.4).

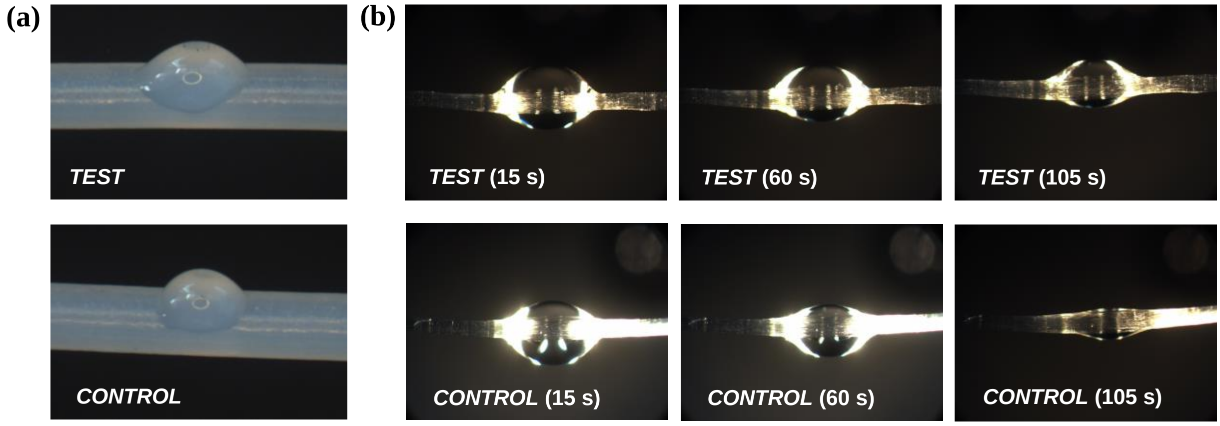


**Figure 4.** (a) Nylon 6,6 filament (1.6 mm diameter), with water droplets: the test sample was previously subjected to 54 MPa, 20 h creep and, compared with the (unstressed) control, shows lower contact angle, indicating reduced hydrophobicity; (b) nylon 6,6 yarn (140 filaments, 27 µm diameter), with water droplets: the test sample was previously subjected to 330 MPa, 24 h creep and, compared with the (unstressed) control, shows slower wicking, suggesting reduced hydrophobicity.

## 5. VPPMCs – Where Next?

### 5.1. VPPMCs for Crash Protection – Would They Have an Impact?

As highlighted in Section 3.1, VPPMCs have successfully demonstrated increases in impact energy absorption. Nevertheless, they have only been assessed by Charpy testing; i.e. at a low impact velocity (3.8 $ms^{-1}$). Energy absorption under higher impact velocities has not been evaluated and, if mechanisms during impact are similar to those of EPPMCs [31], then the benefits from prestress beyond low velocity impact may be limited. VPPMCs could also be produced with randomly distributed discontinuous fibres; though as demonstrated by Fig. 2(a), fibre ends will produce stress concentrations, which could be detrimental to mechanical performance. For a random fibre VPPMC however, the compressive stresses imparted by neighbouring fibres, located in the vicinity of a fibre end, may reduce this effect. Also, it is clear that the effect would be reduced by using longer discontinuous fibres in VPPMC production.

### 5.2. VPPMC Morphing Structures – Changing the Shape of Things to Come?

In aerospace, shape-adaptive (morphing) composite structures offer opportunities for improved aerodynamic performance and functionality but avoiding increased mass and complex construction. Here, the use of EPPMCs has been successfully demonstrated [32]. The simplest morphing structures are those which are bistable; i.e. they can 'snap through' between one of two states. We have recently developed a VPPMC-based bistable structure, using VPPMC strips bonded to the sides of a thin, flexible resin-impregnated fibre-glass sheet [33, 34]. Pairs of strips were orientated to give opposing cylindrical configurations within the sheet, thereby enabling the sheet to 'snap-through' between two states.

### 5.3 Viscoelastically Prestressed Dental Materials – A Bite Beyond Conventional Tooth Fillings?

As viscoelastic prestressing has been successfully demonstrated with conventionally sized fibres (10–30 µm in diameter), applications involving VPPMCs based on nanofibres may be possible. Dental restorative materials, such as direct-filling composites (wear-resistant inorganic filler particles in acrylic-based resin) could be of interest. Although these have replaced dental amalgams, they have lower strengths and tend to have shorter lives. Short life has been attributed to masticatory stresses being transmitted to filler particles projecting from the occlusal (biting) surface; the submerged regions of these particles provide stress concentrations which enable small cracks to propagate into the (softer) matrix

[35]. Clearly, matrix crack propagation could be impeded by compressive prestress, and a study based on unidirectional glass fibre EPPMCs has been published [4]. Alternatively, VPPMCs based on nanofibres, such as UHMWPE, could hold promise for such a small-scale application in a biological environment; the technology would allow these fibres to be randomly distributed throughout the composite filling, which may be stored refrigerated (to retard viscoelastic recovery) as prepreg, prior to in-situ curing.

### 5.4 Viscoelastically Prestressed Concrete – Cracking Down on Cracking?

Fibre-reinforced concrete (FRC) contains randomly oriented fibres to impede cracking and polymer fibres are routinely employed [36-38]. Polypropylene fibres are most commonly used, though nylon fibre-based FRC has been found to sustain higher flexural stress levels [37]. Thus VPPMC principles may offer further opportunities for increasing crack resistance; the polymeric fibres could be processed (i.e. annealed, stretched, then chopped to size) and, if required, stored under refrigerated conditions, before being mixed on site. Also, prestressed, pre-cast concrete components with complex shapes could be factory-produced. A further benefit of viscoelastic prestressing, is that fibre hydrophobicity would be reduced, as discussed in Section 4.2; i.e. fibre adhesion to the (cement) matrix should be improved.

## 6. Conclusions

When compared with elastic prestressing, the use of viscoelastically generated prestress within a composite structure provides benefits of increased flexibility in manufacture and, for polymeric matrices, the probability of greater longevity in service. With appropriate interest and support from industry, opportunities could exist for a wide range of commercial developments, including impact-resistant structures, shape-changing (morphing) structures, dental materials and prestressed precast concrete.

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