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PRESTRESSED POLYMERIC COMPOSITES: AN ALTERNATIVE APPROACH

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Summary: *Although prestressed concrete is an established structural material, interest in the use of prestress within polymeric matrix composites, to improve mechanical properties, is comparatively recent. Elastically prestressed polymeric matrix composites (EPPMCs) exploit prestressed concrete principles, in that fibres within the composite are stretched to maintain an elastic strain during matrix curing. On matrix solidification, compressive stresses are produced within the matrix, which are counterbalanced by residual fibre tension. Research with unidirectional glass fibre EPPMCs has shown increases in impact resistance, strength and stiffness of 25–50% compared with control (unstressed) counterparts. Although such improvements can be achieved without increasing section dimensions or weight, the need to apply fibre tension during matrix curing can impose limitations on processing and product geometry. Also, as the matrix is polymeric, fibre-matrix interfacial creep may cause the prestress to deteriorate. An alternative approach is to consider viscoelastically prestressed polymeric matrix composites (VPPMCs): here, polymeric fibres are subjected to tensile creep, the applied load being removed before the fibres are moulded within a matrix. Following matrix curing, the strained fibres impart compressive stresses (through viscoelastic recovery) to the surrounding matrix. Since fibre stretching and moulding operations are separate, VPPMC production offers great flexibility; also, any potential for deterioration through fibre-matrix creep would be offset by active responses from longer term viscoelastic recovery mechanisms. Research has shown that VPPMCs can be produced from fibre reinforcements such as nylon 6,6, UHMWPE and bamboo. Compared with control (unstressed) counterparts, these VPPMCs have shown improvements in mechanical properties comparable to those from EPPMCs. Of major importance however, is longevity: accelerated ageing techniques have demonstrated that VPPMCs (based on nylon fibre) show no deterioration in impact performance over a duration equivalent to ~25 years at 50°C ambient. Potential applications include crashworthy and impact-resistant structures, dental materials, prestressed precast concrete and shape-changing (morphing) structures.*

1 INTRODUCTION

Although the use of prestressing in structural materials such as concrete is a familiar concept, an awareness of possible benefits from producing fibre-reinforced polymeric matrix composites (PMCs) with (compressive) prestress seems to be comparatively recent. In fact, residual stresses within composite mouldings are normally considered to be an unfortunate consequence of differential shrinkage from the processing route [1]. If stress is applied intentionally during composite processing, it is usually for improving fibre alignment in filament-wound structures [2, 3]. Studies into exploiting prestress for enhancing the mechanical properties of PMCs seem to be relatively uncommon, despite such improvements avoiding any need to increase mass or section thickness within a composite structure.

Elastically prestressed PMCs (EPPMCs) are comparable to prestressed concrete, in that fibres (e.g. glass) are stretched to maintain an elastic strain during matrix curing. After releasing the applied load, compressive stresses are created within the solidified matrix, which are balanced by residual fibre tension. Early EPPMC studies focused on laminates [4, 5], though later investigations with unidirectional glass fibre EPPMCs have shown increases in tensile strength of ~25% and elastic modulus of ~50% [6], compared with unstressed counterparts. Impact resistance, flexural stiffness and strength have also been found to increase by up to 33% [7, 8]. Such 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 [6-8]. Investigations within the last few years have included unidirectional glass fibre EPPMCs as potential dental materials, with prestress-induced increases in flexural strength of ~30% [9] and unidirectional carbon fibre EPPMCs, with impact toughness being increased by ~30% [10]. There has also been interest in the exploitation of EPPMCs for use as shape-adaptive (morphing) composite structures, either as prestressed laminates [11] or unidirectional fibre prestressed structural elements [12].

Although elastic prestressing within a PMC would seem to offer significant benefits, there are two potential drawbacks. First, the need to apply fibre tension during matrix curing, may impose restrictions on fibre length, orientation and spatial distribution, thereby compromising mould geometry [13]. It is also reported that stretching rig design with appropriate fibre clamping can be technically challenging [11, 14]. The second drawback arises from the matrix being a polymeric material: it can be expected that the elastically generated prestress will encourage localised matrix creep to occur at fibre-matrix interface regions, which could cause this prestress to deteriorate progressively with time [13]. This paper provides an overview of research into an alternative approach to EPPMC methodology, which is based on viscoelastically generated prestress. The principles are covered, followed by mechanical properties and long-term performance aspects. Future directions are also discussed.

2 PRESTRESS BASED ON VISCOELASTICITY

2.1 Principles

Viscoelastically prestressed PMCs (VPPMCs) do not require simultaneous fibre stretching and moulding operations. Instead, high-strength polymeric fibres are stretched over time, so that they undergo (viscoelastic) creep; the creep load is then released before the fibres are moulded into a matrix. Following matrix solidification, the previously strained fibres continue to attempt viscoelastic recovery which produces compressive stresses in the matrix, counterbalanced by residual tension within the fibres. Thus a prestress state comparable to an EPPMC can be achieved. In contrast with EPPMCs however, the fibre stretching and moulding operations are decoupled; hence there is potential for considerable flexibility in VPPMC production. Relatively simple equipment is needed for applying a creep load to fibre tows and, on releasing the load, the fibres can be cut to any length, then positioned in any orientation within any shape of mould capable of being filled with a matrix resin.

In addition to the potential for production flexibility, a significant advantage offered by

VPPMCs is longevity. Although localised matrix creep at the fibre-matrix interface regions is expected to occur as in EPPMCs, this would be offset by active responses from longer term viscoelastic recovery mechanisms within the polymeric fibres [13]. There is however, a potentially major limitation, since viscoelastic activity is temperature-sensitive. Therefore, the prestress could deteriorate or be rendered ineffective by high-temperature curing cycles or long-term exposures to hot ambient conditions. This aspect is addressed later in the paper.

2.2 Proof of concept

Figure 1 shows the basic creep-recovery strain cycle for a polymeric material [15]. Clearly, to produce a viable VPPMC, the viscoelastic contribution within the recovery phase, $\varepsilon_r(t)$, is of vital importance, both in magnitude and timescale. Also, any viscous flow effects (due to permanent molecular slippage from creep), ε_f, should be minimal. To determine the feasibility of VPPMC principles, nylon 6,6 was selected, as it is a readily available, low-cost, high strength polymeric fibre. Early experiments revealed that as-received nylon 6,6 fibre, after being subjected to a 24 h creep load of ~330 MPa, gave a viscoelastic recovery strain that approached zero at 1000 h (6 weeks), an unacceptably short timescale [16, 17]. It was found however, that annealing the fibres prior to creep increased the magnitude and timescale of viscoelastic recovery significantly. Based on the work of others [18, 19], the annealing conditions for subsequent nylon 6,6 fibre processing were set to 150°C for 0.5 h.

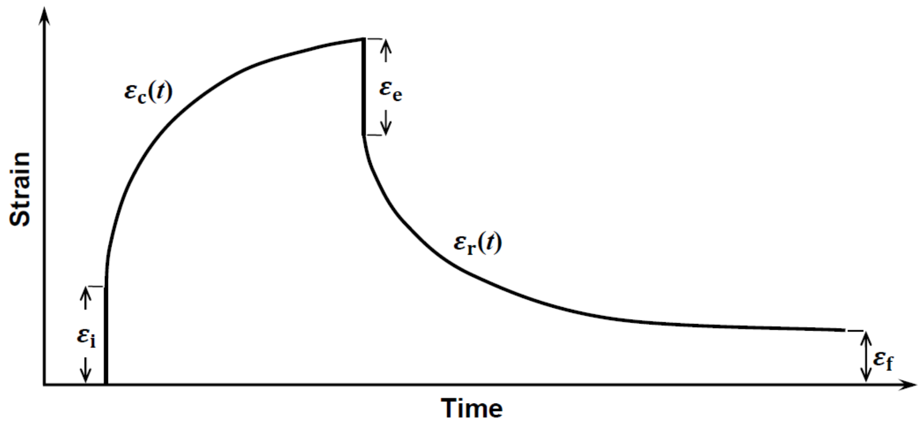

Figure 1: Schematic tensile creep-recovery strain cycle for a polymeric material.

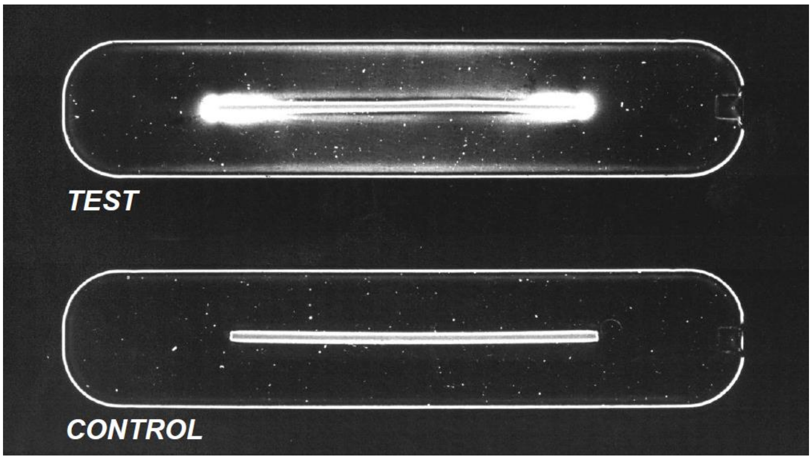

Figure 2: Nylon 6,6 monofilaments (1.6 mm diameter) in polyester resin samples (150 × 30 × 2 mm) under cross-polarised light. Note the stress pattern from viscoelastic recovery in the 'test' sample, compared with the (unstressed) 'control' sample. Image first published in Ref. [17].

In addition to magnitude and timescale aspects, evidence of a recovery force output was required. To demonstrate the presence of a viscoelastic recovery force, Figure 2 presents the result of an early experiment [17]. Here, nylon 6,6 monofilament was annealed and then subjected to a 24 h creep stress, before being moulded into a thin, transparent polyester resin matrix. As Figure 2 shows, a (compressive) stress pattern can be clearly seen under polarised light in the ‘test’ (VPPMC) sample, compared with the ‘control’ (unstressed) counterpart.

2.3 Principal mechanical evaluation – impact tests

Since the earliest studies, the most straightforward method for determining 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 polyester resin strips from the same resin mix, 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. In both cases, identical aluminium moulds with polished channels were used and the nylon yarns were brushed out into flat ribbons immediately prior to moulding. Following sufficient curing, each resulting strip was cut into five lengths ($80 \times 10 \times 3.2$ mm) so that a batch consisted of five test and five control samples, ready for impact testing.

After several studies involving Charpy testing, results have consistently shown that the VPPMC test samples absorb typically 25–30% more impact energy than their control (unstressed) counterparts, with some samples achieving increases of 50% or more [13, 16, 17, 20-23]. Figure 3 shows typical test and control samples after impact testing. The region

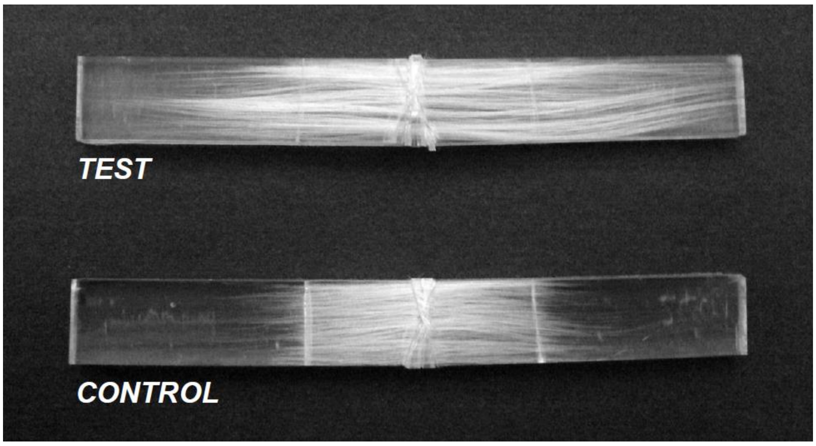

Figure 3: Typical appearance of test (VPPMC) and control (unstressed) samples after impact testing; note the greater region of fibre-matrix debonding in the test sample.

of fibre-matrix debonding resulting from impact is greater in the test sample and this has been consistently observed for all batches studied. Similar increases in debonded area have been observed with EPPMC samples subjected to Charpy impact testing, compared with unstressed counterparts [7], which provides further evidence of prestress in VPPMCs.

Earlier studies led to the conclusion that prestress-induced increases in impact energy absorption could arise from four mechanisms [21]: (i) matrix compression impedes crack propagation, (ii) matrix compression attenuates dynamic overstress effects, (iii) residual fibre tension creates a more collective response to external loads, and (iv) residual shear stresses at the fibre-matrix interface regions promotes energy absorbing fibre debonding over transverse fracture. Recent work however [22], suggests that (iv) is the principal mechanism; thus prestress-enhanced residual shear stresses between fibres and matrix are triggered to promote fibre-matrix debonding (in preference to transverse fracture) when subjected to externally imposed shear stresses resulting from the impact event. This triggering mechanism has also been observed with glass fibre EPPMCs [7].

2.4 Other mechanical tests

The success achieved with Charpy impact testing led to investigations of other basic mechanical characteristics, i.e. the flexural stiffness and tensile properties of VPPMCs. With the exception of one study [22], all Charpy impact investigations utilised composite samples with a low fibre volume fraction, V_f, of 2–3%. This had originally resulted from restrictions in the quantity of fibre that could be stretched for VPPMC sample production. Subsequent design and construction of improved equipment enabled the fibre stretching capacity to be increased by an order of magnitude [24].

In a flexural stiffness study [25], samples were produced by using the open casting method outlined in Section 2.3. In this case however, the samples had higher V_f values (8–16%) with an epoxy resin matrix. Although the epoxy resin had lower viscosity (to facilitate moulding), room temperature gel time at ~15 h was much longer than those of the polyester resins (15–20 min.) previously used and a release film was required for successful de-moulding. The resulting composite strips were cut to produce two test and two control samples per batch, each sample being $200 \times 10 \times 3.5$ mm. Samples were subjected to three-point bend tests using a freely suspended load, the geometry for testing being similar to ASTM D790M recommendations in terms of support pin dimensions and span/thickness ratio of ~30. The flexural modulus, $E(t)$, was determined from deflections measured at t = 5 s (elastic deformation) and 900 s (short-term creep): it was found, over the range of V_f values studied, that $E(t)$ for both time values was increased by ~50% due to viscoelastically generated prestress.

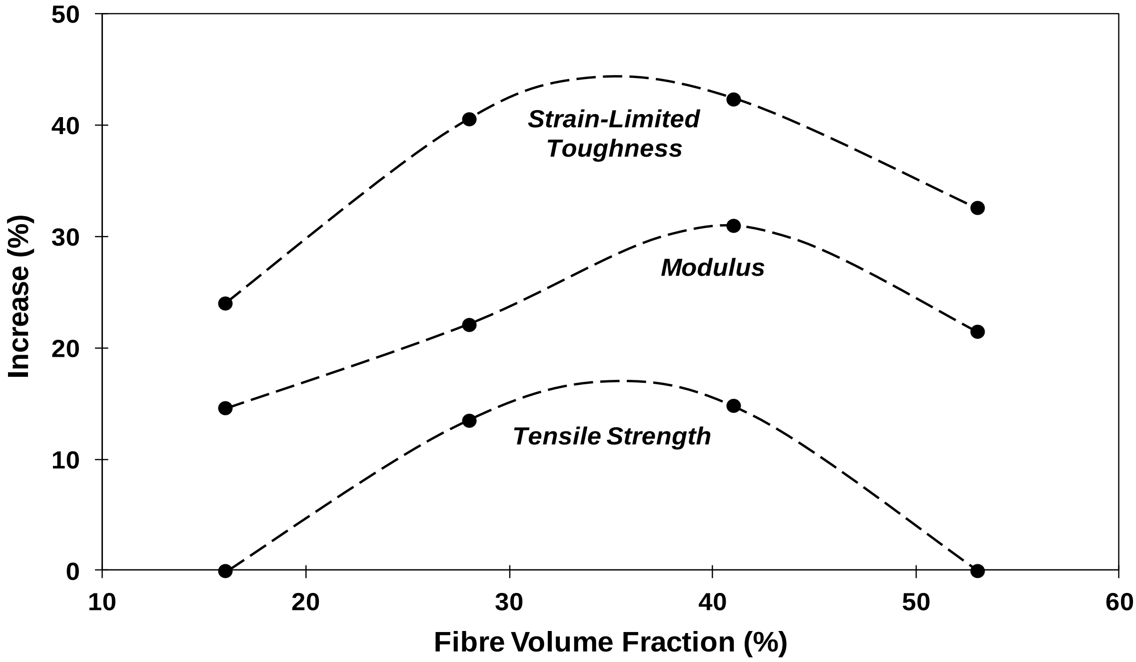

Figure 4: Effect of fibre volume fraction on the measured tensile properties of test (VPPMC) samples relative to their control counterparts. Strain-limited toughness represents energy absorbed/unit volume to a fixed strain (0.25), from area under the stress-strain curve. Redrawn from Ref. [26].

In a tensile testing study [26], composite samples of only 1 mm thickness were required, to meet appropriate test standards. The required thickness accuracy could not be achieved by open casting; hence a "leaky mould" method was adopted, based on principles from Ladizesky and Ward [27]. This was a closed channel moulding technique, which enabled excess resin to escape from the (open) channel ends. As for flexural testing, epoxy resin was used and two test and two control samples per batch were produced, each sample being 200 × 10 × 1 mm. Batches with a wide range of V_f values were evaluated (16–53%), to determine how the tensile properties were affected by V_f. Properties such as strength and stiffness increased with increasing V_f (e.g. tensile strengths at 16% and 53% were 130 and 420 MPa respectively); however, it was also observed that there were prestress-induced increases in these parameters, but only at intermediate V_f values, as shown in Figure 4. The curves in Figure 4 indicate an optimum V_f value (~35–40%) at which the benefits from prestressing are maximised, the increases for strength, modulus and strain-limited toughness exceeding 15, 30 and 40% respectively. This optimum V_f can be attributed to the competing roles between fibres and matrix: at lower V_f, less compressive stress will be produced as there are too few fibres; at higher V_f, there are too many fibres, which therefore reduces the matrix cross-sectional area available for compression.

3 LONGEVITY OF VPPMCS

3.1 Long-term viscoelastic activity

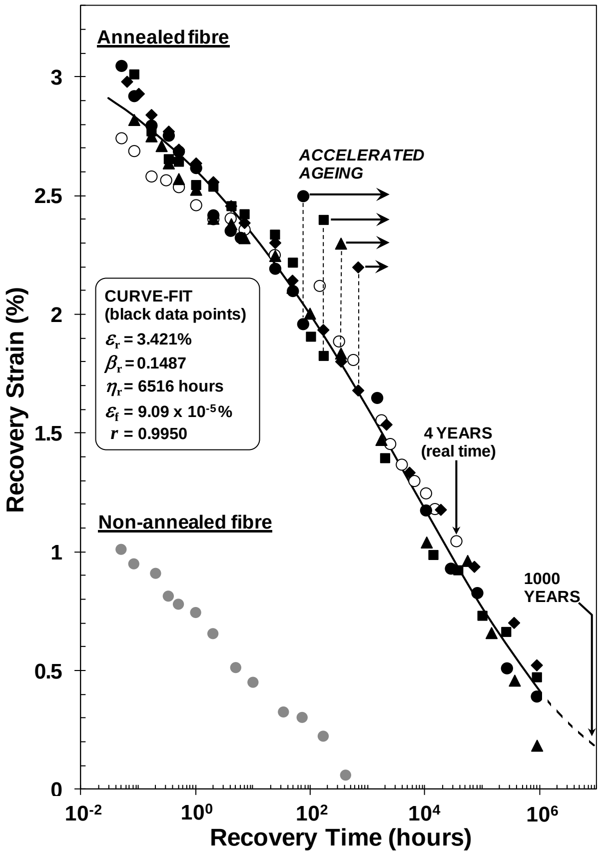

Figure 5: Recovery strain data at 20°C from nylon 6,6 yarn after being subjected to 24 h creep at 342 MPa. For fibre annealed prior to creep, white data points were measured in real time; black data points are from four samples subjected to periods of accelerated ageing, with curve and parameters from Eq. (1), where r = correlation coefficient. After Refs. [20, 21].

Referring to Section 2.1, fibres within a VPPMC should be capable of long-term viscoelastic recovery, to offset the potential for deterioration in prestress from localised matrix creep, especially at fibre-matrix regions. This capability can be determined from polymeric fibres by taking recovery strain measurements resulting from the creep loading conditions used for VPPMC production. Figure 5 shows recovery strain data for nylon 6,6 fibre in the form of untwisted continuous yarn. Full details have been previously published [20, 21], but main points are summarised here. For non-annealed (i.e. as-received) fibre, recovery strain approaches zero within 1000 h of releasing the creep stress; but fibre annealing (150°C for 0.5 h) prior to creep causes viscoelastic recovery to remain active over a considerably longer timescale, as stated in Section 2.2. The white data points in Figure 5 show strain measurements taken in real time, up to 4 years. Beyond this however, accelerated ageing methods are needed, and these were used for obtaining the black data points, up to an equivalent age of 100 years at 20°C. Figure 5 clearly shows good agreement between data from real-time and accelerated ageing, and the curve, fitted to the black data points, represents the following equation for recovery strain:

$$\varepsilon_{\mathrm{rvis}}(t) = \varepsilon_{\mathrm{r}}\left[\exp\left(-\left(\frac{t}{\eta_{\mathrm{r}}}\right)^{\beta_{\mathrm{r}}}\right)\right] + \varepsilon_{\mathrm{f}} \qquad (1)$$

Eq. (1) comes from the Weibull or Kohlrausch-Williams-Watts function, in which polymeric deformation can be described by a mechanical model comprising time-dependent latch elements [15, 28]. As recovery time t approaches ∞, there is a residual (permanent) strain, ε_{f}, resulting from viscous flow effects. For time-dependent viscoelastic recovery, the ε_{r} function depends on the Weibull shape parameter, β, and characteristic life, η. Parameter values from the curve-fit are shown in Figure 1 and since ε_{f} is predicted to be close to zero ($<10^{-4}$%), virtually all the available recovery is indicated to be viscoelastic, suggesting that viscous flow has an insignificant influence on the viscoelastic prestressing mechanism. From Eq. (1), extrapolation of the curve to 8.766×10^{6} h (1000 years), predicts that $\varepsilon_{\mathrm{rvis}}(t)$ will be 0.185%. This clearly suggests that viscoelastic activity, under the conditions considered here, is a long-term phenomenon.

Although long-term viscoelastic activity is demonstrated by Figure 5, there is no information on the force output associated with such fibres when constrained within their VPPMC matrix. The force-time relationship was however obtained from a separate study [29]. Here, annealed nylon 6,6 yarn was subjected to a 24 h creep stress of 320 MPa and following removal of the creep load, the loose yarn was allowed to contract to a fixed strain, enabling the resulting recovery force to be monitored. In Ref. [29], the viscoelastic recovery force was found to increase with time, this being predicted to reach a limiting value of 12 MPa (i.e. 3.8% of applied stress) as t approached ∞. Continued monitoring to 25000 h has demonstrated that the force output progresses in accordance with this trend [21].

Recovery strain measurements from accelerated ageing, as shown in Figure 5, become impractical beyond the equivalent of 100 years at 20°C. Also, even if accelerated ageing techniques could be applied to viscoelastic recovery force experiments, the results would not necessarily relate to the long-term behaviour of an actual VPPMC, since gradual changes in the characteristics of a real matrix are not accounted for. The only alternative therefore, is to subject VPPMC samples (together with control sample counterparts) directly to accelerated ageing. Subsequently, these can be evaluated by Charpy impact testing to determine whether there is any deterioration in performance with age.

3.2 Time-temperature superposition

If a polymeric fibre has been subjected to creep, the resulting viscoelastic recovery rate will increase if the temperature is raised, thus time-temperature superposition principles can be considered. For many polymeric materials, these principles enable accelerated ageing methods to be implemented, if the appropriate shift factor, α_T, is known. Thus α_T equates an elevated temperature to a shift in time, i.e. ageing. Previous studies [20, 21] have evaluated α_T at 60°C relative to 20°C, thus by subjecting samples of viscoelastically recovering nylon 6,6 yarn to periods of 60°C, this α_T value was used to produce the accelerated aging data in Figure 5. Moreover, VPPMC samples (with control sample counterparts) were also subjected to long-term exposures to 60°C (months) and, following Charpy impact testing at 20°C, no deterioration in impact performance was observed, even at an equivalent age of 1000 years at 20°C [21].

3.3 The VPPMC time-temperature boundary

A recent study has successfully demonstrated that nylon 6,6 VPPMCs can be subjected to accelerated ageing at 70°C [23]. Here, viscoelastic activity would be 76300 times faster at 70°C, relative to 20°C. Three batches of composite samples (i.e. 15 test and 15 control) were produced and stored at room temperature (19–22°C) for 336 h (2 weeks) before being subjected to a constant 70°C for 2298 h (3.2 months). The samples were then stored at room temperature for a further 336 h before undergoing Charpy impact testing. This, at least in terms of time-temperature superposition, resulted in the samples being aged to the equivalent of 20000 years at 20°C.

The mean (± standard error) impact energy absorption from the VPPMC samples was 47.5 ± 3.3 kJm^{-2} and, with the control samples giving 34.1 ± 1.3 kJm^{-2}, the increase in impact energy absorbed due to viscoelastically generated prestress was ~40%. Although ageing to an equivalent of 20000 years clearly demonstrates the longevity of these VPPMCs, this result

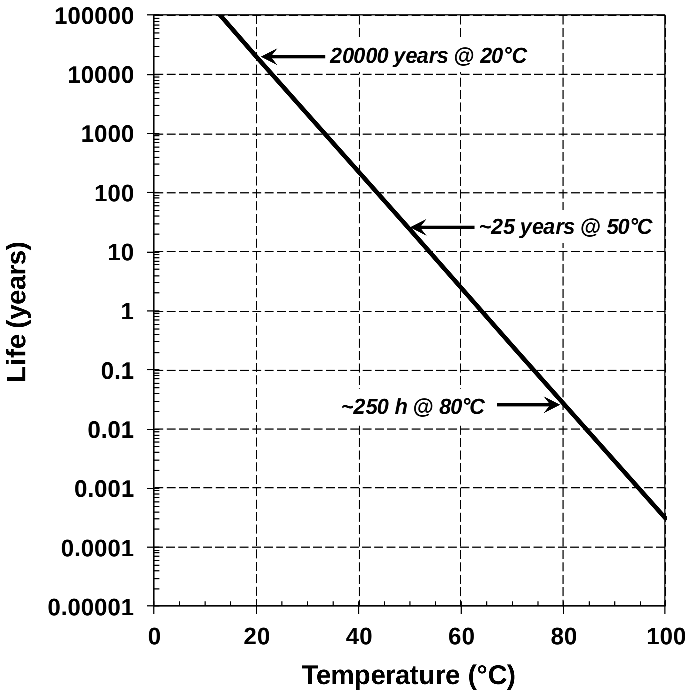

Figure 6: VPPMC life as a function of ambient temperature, based on a time-temperature equivalent of 20000 years at 20°C. Redrawn from Ref. [23].

does not provide a useful measure of practical life. Ambient temperatures greater than 20°C will reduce VPPMC life (in relation to viscoelastic activity), hence longevity must be quantified by temperature as well as time. This requirement is met by Figure 6. Here, the time-temperature boundary indicates that these VPPMCs should, for example, show no deterioration in impact performance after ~25 years for a constant ambient temperature of 50°C. Clearly, this suggests that VPPMC technology is viable for most practical applications.

Figure 6 also indicates that VPPMC processing involving high temperature matrix curing cycles is somewhat restricted. Nevertheless, several hours exposure to a moderately raised curing temperature of 80°C (for example) should be feasible, whilst maintaining an acceptable duration of operation at lower ambient temperatures. In this context, it is worth mentioning that for applications such as aerospace, low temperature curing resins are of interest, since they would enable autoclave-free curing and lower cost tooling [30, 31]. Moreover, the performance of EPPMCs at elevated ambient temperatures is open to speculation: although EPPMC production can involve curing at high temperatures (as prestressing loads are maintained), elevated temperatures in service may exacerbate any fibre-matrix creep effects, thus reducing the useful life of EPPMCs.

4 FUTURE DIRECTIONS

4.1 Alternatives to nylon fibre VPPMCs

Although nylon 6,6 fibre VPPMCs have been the principal research vehicle, other fibres may have the potential for creating viscoelastic prestress. For example, eco-friendly VPPMCs based on plant fibres, are a possibility. An investigation by other researchers into VPPMCs based on bamboo has demonstrated that flexural toughness increased by 28% [32]. Our own recent research has focused on VPPMCs using ultra-high molecular weight polyethylene (UHMWPE) fibres, which are ~4 times stronger and >20 times stiffer than nylon 6,6 fibres. Here, we found increases of 20–40% in flexural modulus [33] and Charpy impact energy absorption [34].

A further alternative is to exploit commingled fibres in VPPMCs. Thus for example, nylon 6,6 fibres, used for creating viscoelastically generated prestress, can be commingled with Kevlar fibres which have superior strength and stiffness. An initial study of these hybrid composites by Charpy impact and flexural stiffness testing [35] has demonstrated that (i) hybrid composites (with no pre-stress) absorb more impact energy than Kevlar fibre-only composites, due to ductility of the nylon fibres; (ii) pre-stress further increases impact energy absorption in the hybrid case by up to 33% and (iii) pre-stress increases flexural modulus by 40% in the hybrid composites.

It is evident here, that going beyond basic nylon 6,6 fibre VPPMCs could open up various commercial opportunities, where improvements in mechanical properties are required, especially impact toughness and flexural stiffness, without the need to increase mass or section sizes. Thus potential applications include crashworthy (vehicular) and other structures requiring impact resistance, such as aerospace applications, wind turbine blades and protective apparel. Moreover, the decoupling of fibre stretching and moulding operations in VPPMC production facilitate the manufacture of complex composite structures and, if required, previously stretched fibre could be stored as refrigerated prepreg material (to retard viscoelastic recovery) for fabrication at alternative sites.

4.2 Nanofibre-based VPPMCs

Since the viscoelastic prestress technique has been successfully demonstrated with conventionally sized fibres (i.e. 10–30 µm in diameter), then applications involving VPPMCs based on nanofibres may be considered. One area of interest could be dental restorative materials (DRMs), such as direct-filling composites (wear-resistant inorganic filler particles in acrylic-based resin). These have been widely accepted as replacements for traditional dental amalgams [36]. Nevertheless, acrylic-based DRMs have lower strengths (80–120 MPa) and life (~5–7 years) compared with amalgams (>400 MPa, >15 years) [37, 38]. 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 enabling small cracks to propagate into the (softer) matrix [36, 37].

Clearly, matrix crack propagation could be impeded by compressive prestress, and in this regard, a study based on unidirectional glass fibre EPPMCs has been published [9]. 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 could be stored as a refrigerated prepreg prior to in-situ curing.

4.3 Viscoelastically prestressed ceramic matrix composites (VPCMCs)

Fibre-reinforced concrete (FRC) has been developing since the early 1960s [39]. FRC contains randomly oriented fibres to impede cracking and polymer fibres are routinely employed [39-41]. Polypropylene fibres are the most commonly used, though nylon fibre-based FRC has been found to sustain higher flexural stress levels [40]. Therefore, VPPMC principles may offer further opportunities for increasing crack resistance; the polymeric fibres could be processed (i.e. annealed, subjected to creep, then chopped to size) and, if required, stored under refrigerated conditions, prior to being mixed on site. This technology would enable prestressed, pre-cast concrete components to be produced with complex shapes.

4.4 Shape-changing (morphing) structures based on VPPMC technology

As outlined in Section 1, there has been interest in the exploitation of EPPMCs for use as shape-adaptive (morphing) composite structures. These offer opportunities for improved aerodynamic performance and functionality without the need for increased mass and complex construction. Thus for example, morphing aerofoils can facilitate camber and twist changes without the need for conventional actuation mechanisms [12]. The simplest morphing structures are those which are bistable; i.e. they can ‘snap through’ between one of two states. Recently, we have developed a bistable structure, based on VPPMC technology; this consists of prestressing strips bonded to the sides of a thin, flexible resin-impregnated fibre-glass sheet [42]. Pairs of strips are orientated to give opposing cylindrical configurations within the sheet, thereby enabling the sheet to ‘snap-through’ between two states.

5 CONCLUSIONS

In comparison with elastic prestressing, the use of viscoelastically generated prestress within a composite structure offers 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.

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