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# Durability of viscoelastic fibre prestressing in a polymeric composite

**Xueqi Lin [1,2], Bing Wang [1,2,3*], Chenmin Zhao [1,2], Walter Nsengiyumva [1,2], Shuncong Zhong [1,2], Hui Chen [1] and Dianzi Liu [4]**

[1] Fujian Provincial Key Laboratory of Terahertz Functional Devices and Intelligent Sensing, School of Mechanical Engineering and Automation, Fuzhou University, Fuzhou, 350108, P.R. China
[2] Institute of Precision Instrument and Intelligent Measurement & Control, Fuzhou University, Fuzhou, 350108, P.R. China
[3] School of Engineering, University of Hull, Hull, HU6 7RX, UK
[4] Engineering Division, Faculty of Science, University of East Anglia, Norwich, UK
* Correspondence: b.wang@fzu.edu.cn

**Abstract:** Viscoelastic fibre prestressing (VFP) is a promising technique to counterbalance the potential thermal residual stresses within a polymeric composite, offering superior mechanical benefits for structural engineering applications. It has been demonstrated that the time required for desirable creep strain can be significantly reduced by implementing higher creep stresses, while its long-term reliability is still unknown. Here, we developed the prestress equivalence principle, and investigated the durability of viscoelastic fibre prestressing within a composite, in order to further enrich the prestress mechanisms. The effectiveness of the prestress equivalence principle was refined through Charpy impact testing of prestressed samples with various prestrain levels. The durability was investigated by subjecting samples to both natural aging (up to 0.5 years) and accelerated aging (by using the time-temperature superposition principle). It is found that the prestress equivalence principle offers flexibilities for viscoelastically prestressed polymeric matrix composite (VPPMC) technology; the impact benefits offered by VFP are still active after been accelerated aged to an equivalent of 20,000 years at 20°C, inferring long-term reliability of VFP-generated fibre recovery within a polymeric composite. These findings demonstrated that both materials and energy consumptions could be conserved for advanced composites. Therefore, they promote further steps of VPPMC technology towards potential industrial application especially for impact protections.

**Keywords:** Durability; Polymeric composite; Viscoelasticity; Prestress; Impact.

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## Abbreviations

| | |
|---|---|
| EEST | Elastic energy storage site |
| EFP | Elastic fibre prestressing |
| EPPMC | Elastically prestressed polymeric matrix composite |
| PMC | Polymeric matrix composite |
| SE | Standard error |
| TTM | Taut-tie molecule |
| TTSP | Time-temperature superposition principle |
| VEST | Viscoelastic energy storage site |
| VFP | Viscoelastic fibre prestressing |
| VPPMC | Viscoelastically prestressed polymeric matrix composite |
| WLF | Williams-Landel-Ferry |

## 1. Introduction

Polymeric matrix composites (PMC) have been widely used in aerospace, automotive, biomedical, as well as sustainable engineering [1,2]. It is known that thermal residual stress levels within a PMC can be significantly affected by the fibre prestressing technique, depending on the fibre and matrix combinations, as well as the laminate stacking sequences [3–5]. This can be achieved through (i) elastic fibre prestressing (EFP) [6–8] and (ii) viscoelastic fibre prestressing (VFP) [9,10]. For (i), tension is applied to long fibres embedded in an uncured polymeric matrix; the prestress load is released on solidification of the resin to produce an elastically prestressed polymeric matrix composite (EPPMC); whilst for (ii), creep tension is applied to long fibres, then the load is released prior to mould the fibres into a resin; following curing of the matrix, a viscoelastically prestressed polymeric matrix composite (VPPMC) is manufactured [4].

It has been demonstrated that both (i) and (ii) techniques can improve the mechanical properties of polymeric composites without increasing their mass or structural dimensions [10], as well as reducing process-induced deformation [11]. These benefits are induced from improved internal stress levels by either EFP-generated recovery (for EPPMCs) or VFP-generated recovery (for VPPMCs) along the prestressed fibres. Although fibre prestressing technique is promising, its applications depend on the mechanical properties of the prestressed fibres [12,13]. Commonly, EFP is applicable to brittle (elastic) fibres such as carbon fibres and glass fibres [14–17], whilst VFP is more suitable for tough fibres such as semi-crystalline thermoplastic fibres [18]. As for structural applications, it is demonstrated that VPPMCs are superior than EPPMCs in terms of product geometry and longevity [19,20]. To produce an EPPMC, the tension load is maintained throughout the curing process which significantly restricts the product geometry, and equipment designs for simultaneously stretching and moulding can be technically challenging [21–23]; whilst for VPPMC, fibre prestressing and moulding are decoupled, providing total flexibility in product geometries [24,25]. As for longevity, VFP-generated fibre recovery has been demonstrated to be a long-term activity [26], while EFP-induced benefits will deteriorate with time due to localised matrix creep [19,27]. Previous works have shown that the VFP within a PMC can improve the tensile strength by ~15% [20], impact toughness by ~50% [28–31], and flexural stiffness by ~50% [32]; the VFP-induced benefits have also been applied to produce morphing (bistable) structures [33,34], green composites [35], and shown great potential for viscoelastically active sutures [36].

The fibre prestressing technique is effective in improving the mechanical properties of PMCs, the prestress mechanisms have also been introduced. The prestressed fibre recovery generates compressive stresses in the fibre/matrix interface, which in turn interacting with the thermal residual stress [37] to adjust the in-plane stress levels [38]; the fibres are pre-stretched before embedded into a matrix, which is effective in destroying the defective fibres in advance, in order to reduce the energy impacts from the stress waves generated by their premature failures on adjacent fibres [39,40]; the fibre prestressing process improves the straightness of the fibre bundles, thus increases the number of effective fibres when loaded in service, and improves the load-bearing capacity of a cured composite [15]; impact of a prestressed PMC triggers the interfacial shear stress caused by fibre prestressing, which promotes the interface debonding between fibre and matrix to absorb energy, and thus improves the transverse impact resistance [41–43]; the compressive stress generated by the prestressed fibres shifts the neutral axis of the composite under bending, which interacts with the applied stress on the tension surface, hence improves the flexural stiffness of the composite [32].

Although it has been demonstrated that the same VFP benefits could be achieved by exploiting a higher fibre creep stress over a shorter term [30], the long-term reliability of the optimised VFP within a PMC is still unknown. In this research, we develop a prestress equivalence principle, and investigate the durability of viscoelastic fibre prestressing within a polymeric composite, aiming to further enrich the underlying prestress mechanisms. Since prestress benefits within a VPPMC depend on the viscoelastic recovery of the prestressed fibres, VFP levels can be represented by viscoelastic creep strain values or 'prestrain' levels, *i.e.* the effect of elastic strains is subtracted from the total creep strains. The Weibull-based creep and recovery model allows the prediction of viscoelastic creep strain at each optimised creep condition, and this shows opportunities to achieve prestress equivalence between different creep stresses, offering further flexibilities for VPPMC technology. The durability of VFP within the PMC is then investigated through both natural aging (up to 6 months) and accelerated aging (using the time-temperature superposition principle), in order to reveal further insight into the fundamental VFP mechanisms.

## 2. Theoretical

### *2.1. Prestress equavilence principle*

When a polymeric fibre is subjected to creep stress (below yield stress), it undergoes both elastic and viscoelastic deformation. The viscoelastic behavior of creep and recovery can be represented by a number of Voigt elements connected in series. Figure 1 (a) shows the strain evolution during creep and recovery cycle of a polymeric material. The time-dependent components represented by functions are based on the Weibull model [44]. For creep, $\varepsilon_{ctot}(t)$ is the total strain at time $t$, under an applied constant creep stress:

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

Here, $\varepsilon_i$ is the instantaneous strain from applied stress which is time-independent; $\varepsilon_c$ 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, the elastic deformation is recovered, as represented by $\varepsilon_e$ in Figure 1 (a), and the remaining recovery strain $\varepsilon_{rvis}(t)$ is:

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

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

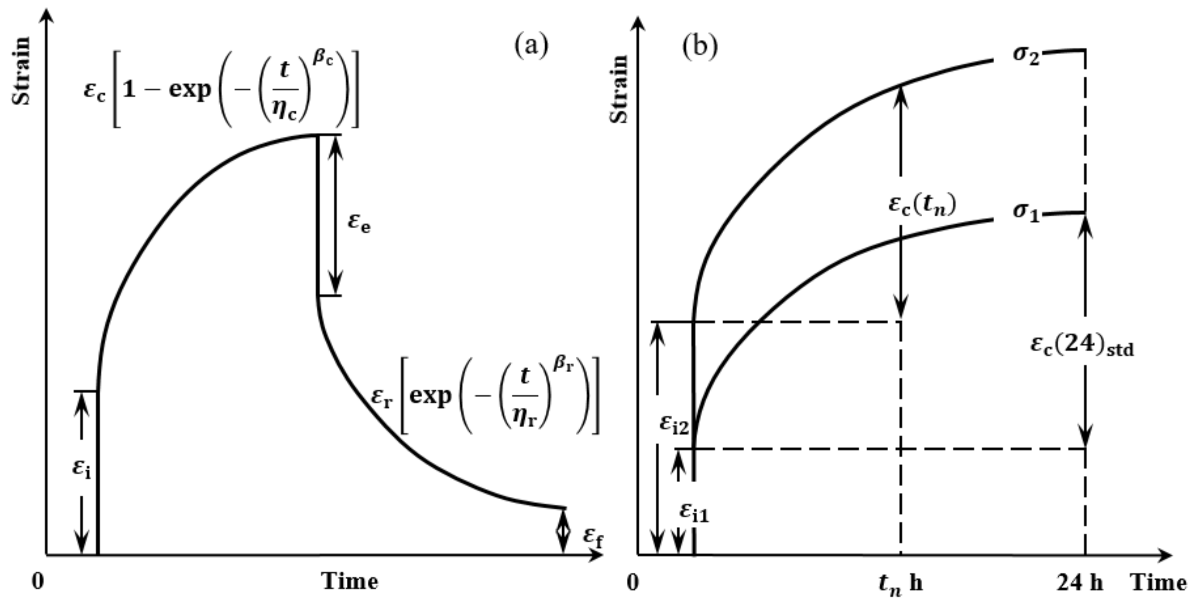


**Figure 1.** Schematic of the strain-time behaviour of a polymeric fibre under constant creep stress, showing (a) creep-recovery strain cycle; and (b) the prestress equivalence principle.

To get the similar prestress benefits, the same prestrain level can be achieved through shorter term via higher creep stress, and the equivalence principle is illustrated in Figure

1 (b). Here, fibre creep tension is applied for 24 h for convenience. Since creep strain curve is represented by Eqn 1, the instantaneous strain $\varepsilon_{i1}$ and the time-dependent strain $\varepsilon_c(24)_{std}$, can be determined experimentally under stress $\sigma_1$. The prestrain level is defined as $[\varepsilon_c(24)_{std} - \varepsilon_{i1}]$. To achieve the same prestrain level, the subsequent run is performed at higher stress value, $\sigma_2$ (> $\sigma_1$). For strain value at $\varepsilon_c(t_n)$ to be equal to $\varepsilon_c(24)_{std}$, $t_n$ will be < 24 h. Note, $\varepsilon_c(t_n)$ excludes the instantaneous strain $\varepsilon_{i2}$. Therefore, a value for $t_n$ which approaches the shortest practical creep time, $t_{min}$, can be determined to give similar VFP benefits. Thus, the same prestrain level can be achieved through shorter term via higher creep stress level, and the prestress equivalence principle is mathematically:

$$\sigma^{-1} = a \ln t_{\mathrm{n}} + b \quad (3)$$

where, *a* and *b* are constants depending on the prestrain level, which can be determined experimentally.

To validate the prestress equivalence principle, VPPMC samples are produced under the $t_n$ creep conditions. Thus, batches of VPPMC samples using $t_n$ is compared with similar batches produced under standard (24 h) creep conditions to evaluate the VFP benefits. Viscoelastic force, $\sigma(t)$, under $t_n$ creep conditions is also measured and compared to the standard 24 h runs, in order to provide direct experimental evidences and reveal the fundamental VFP mechanisms, which follows [45]:

$$\sigma(t) = \sigma_{\mathrm{v}} \left[ \exp\left( -\left(\frac{\Delta t}{\eta}\right)^{\beta} \right) - \exp\left( -\left(\frac{t}{\eta}\right)^{\beta} \right) \right] \quad (4)$$

where, the $\sigma_v$ function represents VFP generated time-dependent stress, as determined by the characteristic life $\eta$ and shape $\beta$ parameters.

### *2.2. Durability prediction through TTSP*

The time-temperature superposition principle (TTSP) has been commonly used to generate the master curve for tensile creep [46–48], flexural creep [49], dynamic tensile modulus [50], stress relaxation [50,51], or predict the long-term viscoelastic polymeric fibre recovery [26]. The TTSP is based on the free volume theory [52,53], and adopted here to investigate the durability of VFP within a polymeric composite. VPPMC samples under $t_n$ creep conditions are subjected to accelerated aging, and impact tested to determine the long-term effectiveness of the VFP, to reveal the fundamental mechanisms. These are achieved through transferring an elevated temperature into a time scale shift in terms of free volume [54], which is mathematically expressed as [55]:

$$\log \frac{\eta(T)}{\eta(T_0)} = \log \alpha_{\mathrm{T}} = -\frac{B}{2.303 f_0} \left( \frac{T - T_0}{f_0/a_{\mathrm{T}} + T - T_0} \right) \quad (5)$$

where, $\alpha_T$ is defined as the temperature shift factor; $T_0$ is arbitrarily chosen as the reference temperature and $T$ is any other temperature; $a_T$ is the thermal expansion coefficient of the free volume fraction; $f_0$ is the free volume fraction at reference temperature $T_0$. This yields the well-known Williams-Landel-Ferry (WLF) equation with $C_1$=$B/(2.303f_0)$, $C_2$=$f_0/a_T$.

The TTSP in Eqn 5 infers a non-linear relationship between temperature and the shift factor log $\alpha_T$; however, nylon 6,6 fibre shows approximately linear viscoelasticity within the yield creep strain, and the linear superposition principle holds [56]. This corresponds with the following: (i) Howard and Williams [46] applied the TTSP to the creep of oriented nylon 6,6 fibre under anhydrous conditions when a low range of creep stress (10-51 MPa) was adopted, and results show that the shift factor log $\alpha_T$ was linear to temperature; (ii) Murayama et al [51] investigated the applicability of TTSP to the stress relaxation of nylon 6,6 fibre, and a linear relationship between shift factor log $\alpha_T$ and temperature was obtained; (iii) similarly, a linear curve was also obtained by Dunell et al [50] through the investigation into superposition of stress relaxation and dynamic tensile modulus of nylon 6,6 monofilaments at temperatures between 10 and -100°C; (iv) the linear relationship was also found with unoriented nylon 6,6 filaments [57] when subjected to small creep strain values. Therefore, rather than the non-linear relation as represented in Eqn 5, a simpler linear TTSP was established for oriented nylon 6,6 fibre, which is based on the published data in terms of creep [46] and stress relaxation [51].

The linear regression gives a gradient of 0.09765°$C^{-1}$ [54]; this is comparable to 0.093 as determined by Williams and Bender [57] through the investigation of unoriented nylon 6,6 filaments. This enables log $\alpha_T$ to be determined at 70°C relative to 20°C, and the resulting value is -4.8825. Hence, viscoelastic activity would be 76,300 times faster at 70°C relative to 20°C, *i.e.* if samples are subjected to 70°C for 2,298 h, the prestress effect from viscoelastic recovery mechanisms will be aged to an equivalent of 20,000 years at 20°C, as well as to be ~25 years at 50°C ambient temperature.

## 3. Experimental

### *3.1. Sample preparation and durability evaluation*

Composite sample preparation followed the procedures from previous studies using nylon 6,6 fibres and polyester resin [31]. To concentrate on the prestress effects, a $V_f$ of ~2.0% was adopted for all composite samples and subjected to Charpy impact testing [30]. Durability of VFP within the composite was evaluated through (i) naturally aged short-term, and middle-term impact performance, as well as (ii) long-term impact resistance through accelerated aging. For (i), five batches of VPPMC samples were produced for each creep condition and were either stored to 336 h (2 weeks for short-term) or 4392 h (6 months for middle-term) at room temperature (19-22°C), and then impact tested; for (ii), samples were stored at room temperature for at least 2 weeks, and then subjected to accelerated aging prior to the impact tests.

A calibrated fan-assisted oven was used for accelerated aging, with a long-term temperature stability of ± 0.5°C. Batches of samples with fibres previously subjected to creep at 590 MPa for $t_n$ h were aged, together with the standard VPPMC sample batches for comparison. Owning to limitation in capacity of the fan-assisted oven, three batches of VPPMC samples fabricated with each of the two creep conditions were evaluated (*i.e.* six batches in total). Heat treatment was maintained at a constant 70°C for 2,298 h (3.2 months), which is equivalent to an exposure of 20,000 years at 20°C in terms of viscoelastic recovery within the nylon fibres following the TTSP in Section 2.2. Samples were placed as a single layer on the tray, and Figure 2 (a) shows the arrangements of samples before aging, with individual test and control samples in alternating positions. This ensured all the batches were subjected to the same heat conditions. Figure 2 (b) shows the samples after accelerated aging. Samples were removed from the trays and stored in polythene bags at room temperature for a further 336 h prior to impact tests.

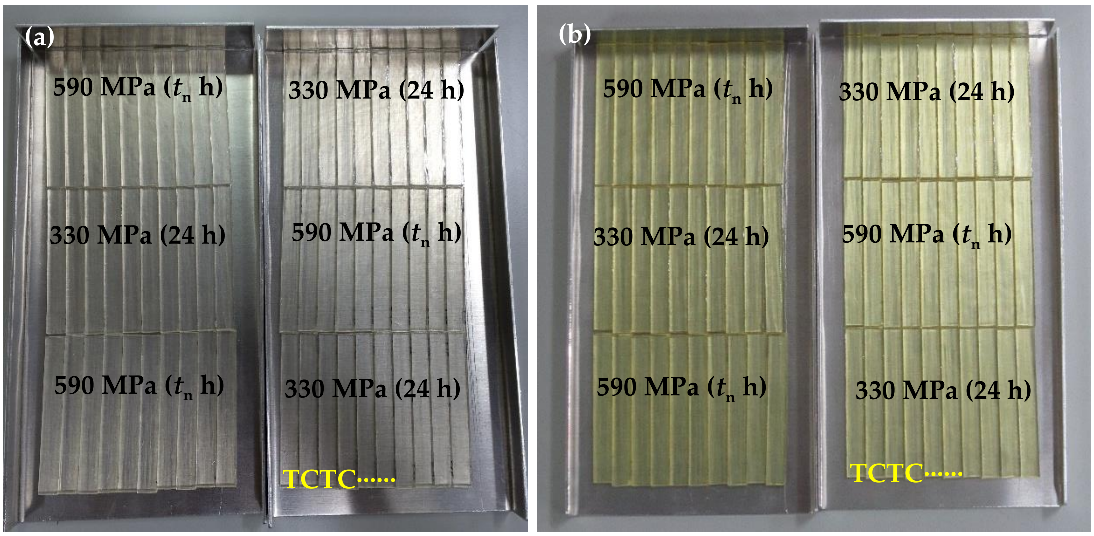

**Figure 2.** Arrangements of samples within a tray (a) before and (b) after being subjected to the accelerated aging; "T" stands for test sample, and "C" is the control (non-prestressed) counterpart.

### *3.2. Viscoelastic recovery force*

Previous studies into the force output-time characteristics of viscoelastically prestressed fibres have provided useful evidences into fibre recovery [31,45]. Here, effectiveness of prestress equivalence principle is studied in terms of recovery force induced by fibre prestressing within the nylon fibre, in order to provide further insight into prestress mechanisms. Procedures followed those detailed in [31]. Here, the generated recovery force from up to $t_n$ creep condition was evaluated and compared to the standard 24 h creep run. Since elastic deformation was fully recovered after load removal, the recovery force induced by the same prestrain level, *i.e.* either from the standard 24 h or $t_n$ h (higher creep stress) run, was expected to be at similar level.

### *3.3. Statistical analysis*

As stated above, multiple sample batches were tested for various optimised creep conditions for repeatability, which were then compared to the standard 24 h creep run to evaluate the effectiveness of the prestress equivalent principle. Thus, statistical analysis is essential to evaluate the significance level of the data variances. These were performed by following the standard procedures of the two-tailed hypothesis testing at a significant level of 5% [58].

## 4. Results and discussion

### *4.1. Short-term effectiveness*

Creep and recovery strain-time data of the nylon fibre have been characterised by using the Weibull-based curve-fits from Eqn 1, the $\varepsilon_c(24)_{std}$ value (330 MPa) was found to be 3.39%. Thus, for $\varepsilon_c(t_n)$ to be equal to $\varepsilon_c(24)_{std}$, the $t_n$ values (at ~3.4% strain level) from the 395 MPa, 460 MPa, 525 MPa and 590 MPa creep data are found to be 420 min, 92 min, 75 min, and 37 min, respectively. Figure 3 shows the applied creep stress vs $t_n$ values with fitted logarithmic relationship following Eqn 3 at a prestrain level, $\varepsilon_c(t_n)$, of ~3.4%. It offers an opportunity to predict the required creep stress for a designated $t_n$ value.

The effectiveness of the trend was experimentally evaluated. This was achieved by selecting a point on the extended dashed curve in Figure 3. Batches of Charpy impact samples were made with the obtained creep condition, and benefits from prestress were investigated through impact testing. The Eqn 3 indicates that stress values of 797 MPa and 715 MPa correspond to 10 min and 15 min stretching respectively; however, initial attempts on creep stress under these conditions rapidly led to fibre fracture due to stress concentration effects. Since it is known that nylon fibre could sustain a 665 MPa creep stress for more than 1 h, this value was adopted here, which requires a corresponding 20 min stretching time to achieve a prestrain level of ~3.4% in Figure 3. Thus, batches of composite samples were made and Table 1 shows the impact results (tested at 336 h, *i.e.* 2 weeks after manufacture). The five batches of samples give an absorbed energy increase of 55.78 ± 3.77%. Two-sided hypothesis testing at 5% significance level shows that the increase is equivalent to the benefits (54.14 ± 7.52%) from the standard runs (330 MPa for 24 h) [30]. Referring to the creep strain-time data under 665 MPa [59], the strain value $\varepsilon_c$ for $t_n$ = 20 min was found to be 3.49% using Eqn 1, which is comparable to the $\varepsilon_c(24)_{std}$ value at 330 MPa, *i.e.* 3.39%.

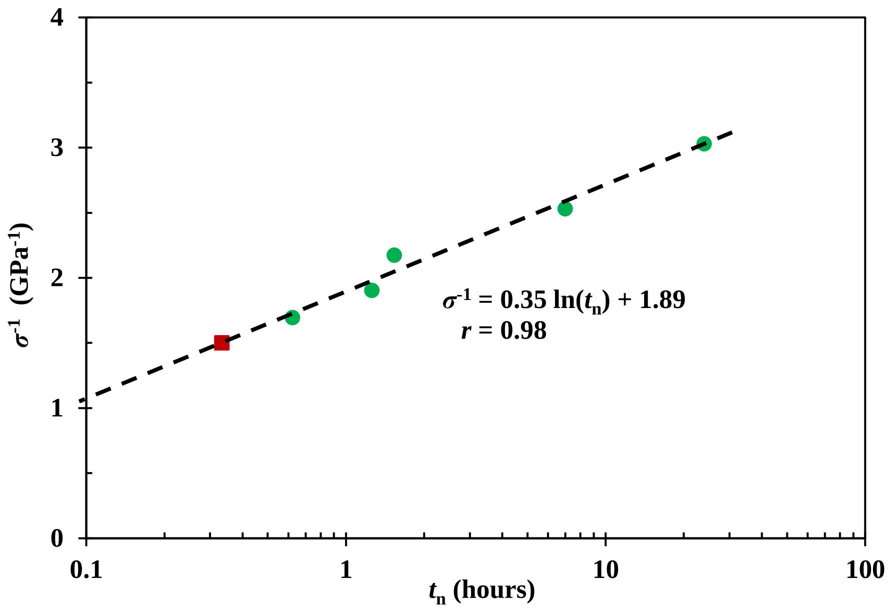


**Figure 3.** Relationship between applied creep stress $\sigma$ and required $t_n$ for creep loading to get the same prestrain level. Round data points are calculated using the prestress equivalence principle in Figure 1 (b); the square point predicts the creep stress requirement for a stretching period of 20 minutes; curve is fitted following Eqn 3.

These results indicate that ~3.4% viscoelastic creep strain would give an increase of ~56% in absorbed impact energy. In terms of industrial application, this improvement in mechanical properties could be achieved within a few minutes of fibre stretching as shown in Figure 3, if stress concentration effects can be avoided. It is worth noting that the prestress benefits within a composite may also be related to the stiffness ratio between fibre and matrix, which infers different in-plane stress transfer mechanisms [59].

**Table 1.** Charpy impact results for VPPMC samples produced with a creep condition of 665 MPa for 20 min; tests were performed at 336 h after manufacture; SE is the standard error.

| Batch | Natural age (h) | Mean impact energy ($kJ\ m^{-2}$) | | Increase in energy (%) |
|---|---|---|---|---|
| | | Test ± SE | Control ± SE | |
| 330 MPa (24 h) | 336 | 33.93 ± 3.14 | 23.78 ± 1.48 | 42.64 |
| | | 37.02 ± 1.78 | 20.71 ± 0.63 | 78.76 |
| | | 35.36 ± 1.71 | 25.03 ± 0.96 | 41.26 |
| | | 36.71 ± 2.89 | 25.60 ± 1.15 | 43.37 |
| | | 35.58 ± 1.96 | 21.61 ± 1.13 | 64.69 |
| Mean ± SE | | 35.7 ± 1.0 | 23.4 ± 0.6 | 54.1 ± 7.5 |
| 665 MPa (20 min) | 336 | 34.74 ± 1.18 | 22.54 ± 1.16 | 54.14 |
| | | 37.32 ± 1.44 | 24.00 ± 0.59 | 55.54 |
| | | 38.42 ± 1.78 | 22.73 ± 0.45 | 68.99 |
| | | 38.74 ± 2.46 | 25.03 ± 0.59 | 54.79 |
| | | 34.00 ± 1.69 | 23.38 ± 0.55 | 45.46 |
| Mean ± SE | | 36.65 ± 0.82 | 23.53 ± 0.35 | 55.78 ± 3.77 |

### *4.2. Recovery force equivalence*

A further evidence of the prestress equivalence discussed above is to evaluate the recovery force generated by the prestressed fibres. Since the optimised condition in Figure 3 has shown effectively the same mechanical benefits for VPPMCs, the recovery force with the 590 MPa for 37 min condition was investigated, and compared to the standard creep run (330 MPa for 24 h). Two runs were tested for each creep condition (for repeatability), and these were monitored up to 1000 h. Figure 4 shows the resulting data. Although there is variation in data points, the 590 MPa condition shows a slightly higher recovery rate below 10 h, which corresponds with previous findings [31]. This may due to the quick response of taut-tie molecules (TTMs) upon load removal, which would dominate for several hours. Beyond 10 h however, the 590 MPa condition shows similar recovery force values.

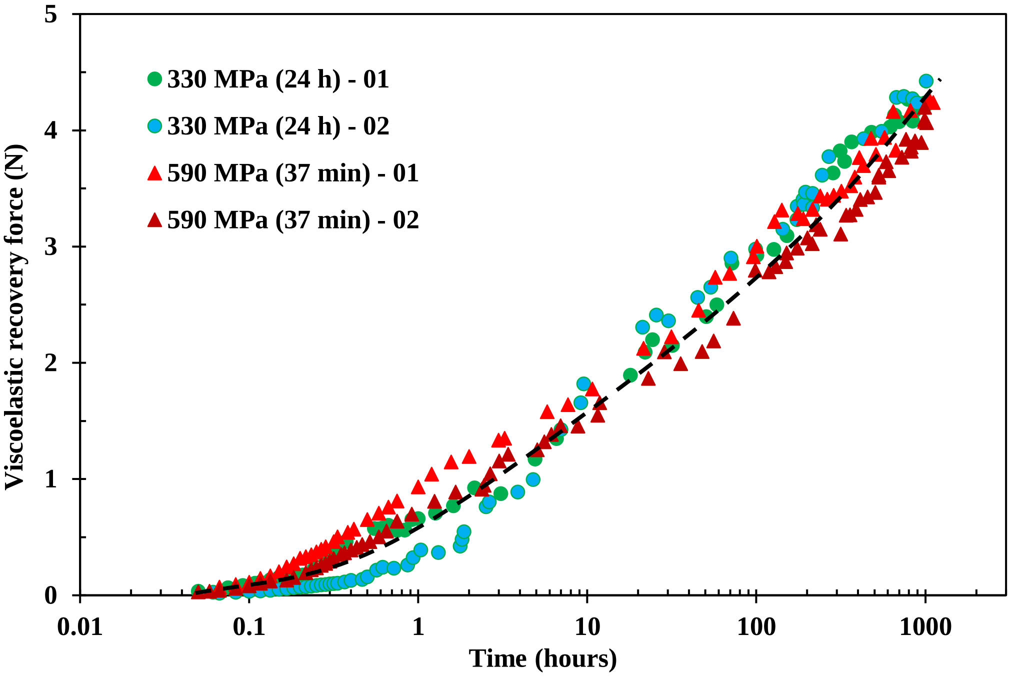


**Figure 4.** Recovery force measurements from nylon 6,6 fibre after being subjected to the standard creep condition (330 MPa for 24 h) and the 590 MPa 37 min condition.

The Weibull-based model represented by Eqn 4 was fitted to the experimental data and corresponding parameter values are presented in Table 2. This enables prediction of recovery force values at 336 h after creep load removal, which are expected to directly relate to the absolute values of recovery force within a VPPMC at the same age. The resulting data give 3.76 ± 0.06 N, and 3.46 ± 0.14 N for 330 MPa and 590 MPa conditions, respectively, and two-tailed hypothesis tests show that these two forces are the same at a significance level of 5%. Thus, the recovery force generated from the two creep conditions are equivalent within the experimental error. Therefore, it is concluded that recovery force from the reduced time creep (up to 590 MPa creep stress) gives a similar value to the standard creep conditions at an equivalent viscoelastic creep strain level.

**Table 2.** Summary of the viscoelastic recovery force parameter values using Eqn 4; *r* is the correlation coefficient.

| Creep condition | Recovery force | | | | |
|---|---|---|---|---|---|
| | $\sigma_v$ (MPa) | $\Delta t$ (h) | $\eta$ (h) | $\beta$ | $r$ |
| 330 MPa-24 h -01 | 5.2028 | 0.0414 | 9.6148 | 0.3382 | 0.9989 |
| 330 MPa-24 h -02 | 5.0470 | 0.1632 | 59.778 | 0.3831 | 0.9968 |
| 590 MPa-37 min-01 | 11.744 | 0.0676 | 4135.6 | 0.1337 | 0.9989 |
| 590 MPa-37 min-02 | 14.527 | 0.0751 | 116460 | 0.1548 | 0.9988 |

Although viscoelastic recovery force and short-term impact tests (336 h) provide evidences of the prestress equivalence, the long-term endurance of prestress effects does not necessarily relate to that of a VPPMC, due to the possible matrix creep effects [19]. Thus the middle-term and long-term effect of VFP are further investigated and discussed below.

### 4.3. Middle-term effectiveness

Furthermore, batches of VPPMC samples with their control counterparts were produced and allowed to age in real time to 4392 h (0.5 years). Again, batches corresponding to the 590 MPa, 37 min creep condition were evaluated, together with standard 330 MPa, 24 h batches for comparison. Table 3 shows the Charpy impact results. Of particular interest, is that the increase in impact energy in both cases is comparable, and this is verified through a two-tailed hypothesis testing at a significance level of 5%. Therefore, the viscoelastic recovery mechanism from nylon 6,6 fibre within these VPPMCs is still functional, and there is no deterioration in prestress benefits up to 0.5 years in real time aging.

**Table 3.** Summary of Charpy impact results, tested at 4392 h (0.5 years) after manufacture. Five sample batches (5 test and 5 control samples in each batch) were tested for each viscoelastic fibre prestressing condition; SE is standard error.

| | Natural | Mean impact energy (kJ $m^{-2}$) | | Increase in |
|---|---|---|---|---|
| Batch | age (h) | Test ± SE | Control ± SE | energy (%) |
| 330 MPa (24 h) | 4392 | 40.00 ± 2.40 | 25.77 ± 0.64 | 55.21 |
| | | 35.50 ± 3.12 | 20.32 ± 0.63 | 74.72 |
| | | 37.71 ± 1.44 | 23.43 ± 0.81 | 60.96 |
| | | 34.97 ± 3.71 | 23.13 ± 0.38 | 51.20 |
| | | 34.31 ± 3.01 | 25.05 ± 1.14 | 36.95 |
| Mean ± SE | | 36.50 ± 1.22 | 23.54 ± 0.47 | 55.81 ± 6.17 |
| 590 MPa (37 min) | 4392 | 44.14 ± 1.82 | 25.17 ± 1.27 | 75.33 |
| | | 44.26 ± 3.11 | 24.46 ± 0.50 | 80.98 |
| | | 39.76 ± 2.70 | 25.51 ± 0.69 | 55.85 |
| | | 35.34 ± 1.74 | 23.96 ± 1.78 | 47.49 |
| | | 34.06 ± 1.90 | 22.90 ± 0.35 | 48.71 |
| Mean ± SE | | 39.51 ± 1.24 | 24.40 ± 0.50 | 61.67 ± 6.94 |

### 4.4. Long-term effectiveness

When subjected to accelerated aging, apart from the sample colour (yellowish), there is no significant difference visually between the sample batches, see Figure 2 (b). Table 4 summarises the impact results from tests two weeks after the heat treatment. By subjecting the VPPMC samples to accelerated aging, the increase in impact energy absorption shows that viscoelastically generated prestress remains active for an equivalent of 20,000 years at a constant 20˚C with both creep prestressed samples, *i.e.* as verified through a two-tailed hypothesis testing (at 5% level), the mean increase value of 52.81 ± 3.09% agrees with the impact result (54.14 ± 7.52%) aged for 336 h. Compared to the optimised creep condition, *i.e.* 590 MPa for 37 min, the benefits from VFP are also similar. Hypothesis testing shows that the increase of 60.43 ± 9.52% is not different to 52.81 ± 3.09% at a 5% significance level. Hence, there is no deterioration in increased energy absorption.

**Table 4.** Summary of Charpy impact results. Samples were subjected to accelerated aging to an equivalent of 20,000 years at 20˚C. Each prestressing condition is represented by three batches, with 5 test and 5 control samples in each batch; SE is standard error.

| | Exposure | Age equivalent | Mean impact energy (kJ $m^{-2}$) | | Increase in |
|---|---|---|---|---|---|
| Batch | to 70˚C | @ 20˚C | Test ± SE | Control ± SE | energy (%) |
| 330 MPa (24 h) | 2,298 | 20,000 | 34.51 ± 2.07 | 22.30 ± 1.11 | 54.73 |
| | | | 33.74 ± 1.63 | 22.99 ± 0.77 | 46.76 |
| | | | 34.77 ± 2.32 | 22.15 ± 0.96 | 56.94 |

| | | | | | |
|---|---|---|---|---|---|
| Mean ± SE | | | 34.34 ± 1.09 | 22.48 ± 0.52 | 52.81 ± 3.09 |
| 590 MPa (37 min) | 2,298 | 20,000 | 31.78 ± 0.81 | 22.44 ± 0.84 | 41.64 |
| | | | 32.65 ± 0.20 | 18.93 ± 0.47 | 72.47 |
| | | | 35.91 ± 1.74 | 21.48 ± 0.33 | 67.18 |
| Mean ± SE | | | 33.45 ± 0.76 | 20.95 ± 0.51 | 60.43 ± 9.52 |

### *4.5. Durability of viscoelastic fibre prestressing*

Durability of VFP within the composite is plotted in Figure 5 in terms of Charpy impact resistance. It is clear that the 590 MPa, 37 min creep conditioned VPPMC samples show comparable increase in absorbed impact energy with the standard 24 h creep condition (330 MPa), in terms of both natural aging and accelerated aging. This demonstrates that the same prestrain level achieved through higher creep stress via a shorter term remains active, and there is no deterioration in increased energy absorption in either naturally aged samples up to 0.5 years or accelerated aged to an equivalent of 20,000 years at room temperature. For a VPPMC produced with a viscoelastic creep strain level of ~3.4%, the prestress effects can be expected not to deteriorate for at least ~25 years at a constant 50˚C. This would lead VPPMCs to many practical industrial applications.

Therefore, effects of nylon fibres prestressed through 590 MPa for 37 min is broadly the same with the 330 MPa, 24 h creep condition within a composite. That is to say, VFP processing time could be reduced from 24 h to tens of minutes by using higher creep stress (below failure stress) with no detriment to impact performance. It is also extrapolated that the logarithmic regression shown in Figure 3 is effective in long-term. Thus, it is concluded that the prestress equivalence principle can be achieved by controlling the viscoelastic creep strain values, *i.e.* the same prestrain level would generate equivalent mechanical benefits within a polymeric composite.

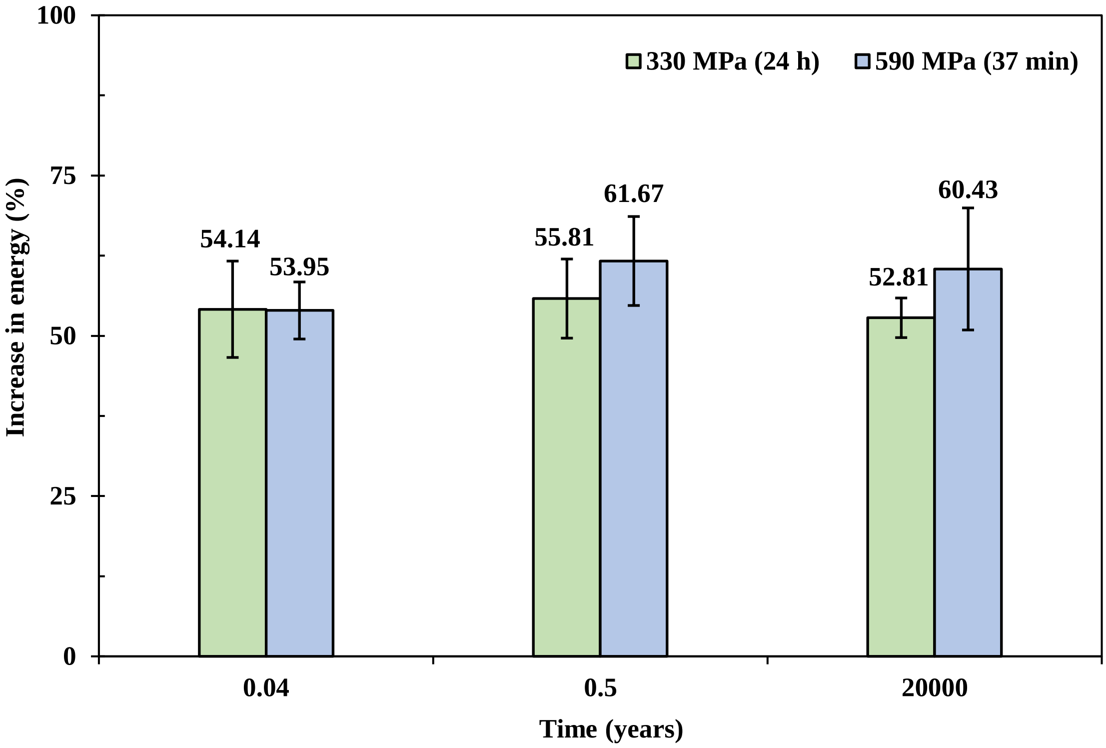


**Figure 5.** Increase in impact energy against aging time for VPPMC samples produced under different viscoelastic fibre prestressing conditions.

### *4.6. Towards full potential of viscoelastic fibre prestressing*

It has been demonstrated that the benefits from VFP is maximised when fibres are subjected to 460 MPa for 24 h, and the $\varepsilon_c(24)$ value (460 MPa) is found to be 4.03% [31]. Thus, further optimisation in load-time conditions could be achieved by applying the prestress equivalence principle in Section 2.1. The prestress benefits can be explored towards the full potential by applying the same principle to a higher viscoelastic creep strain, *i.e.* ~4.0%. Since it is known that nylon 6,6 can sustain a 665 MPa creep stress for more than 1 h, then for $\varepsilon_c(t_n)$ equals to $\varepsilon_c(24)$ at 460 MPa, the $t_n$ values for creep at 590 MPa and 665 MPa were found to be 134 min and 48 min, respectively. Data from corresponding Charpy impact results are summarised in Table 5. For reference, batches subjected to 460 MPa for 24 h from Ref [31] are also presented.

The resulting mean data from Table 5 are shown in Figure 6 for comparison. Again, the optimised conditions are in good agreement with the 24 h creep runs in terms of increase in impact energy absorption. Although there is a slight decrease in increased impact energy over the three creep conditions, from 79% to 73%, little difference was observed in impact energy absorption (in absolute terms). In fact, two-tailed hypothesis testing results show that there is no significant difference in absorbed energy increase, *i.e.* they are similar. Compared to the creep strain level at ~3.4%, raising the viscoelastic creep strain level to ~4.0% gives a mean increase in energy absorption of ~75%. Thus, an 18% increase in prestrain level results in a further increase of ~34% in prestress benefits.

**Table 5.** Summary of Charpy impact test results, tested at 336 h and 4392 h after manufacture. Five sample batches were tested for each prestressing condition with 5 test and 5 control samples in each batch; SE is standard error.

| | Natural age | Mean impact energy (kJ m$^{-2}$) | | Increase in |
|---|---|---|---|---|
| Batch | (h) | Test ± SE | Control ± SE | energy (%) |
| 460 MPa (24 h) | 336 | 39.63 ± 2.22 | 24.08 ± 0.74 | 64.60 |
| | | 37.30 ± 0.54 | 22.28 ± 0.88 | 67.39 |
| | | 38.57 ± 1.35 | 19.46 ± 0.15 | 98.21 |
| | | 39.36 ± 0.95 | 22.59 ± 0.66 | 74.23 |
| | | 44.81 ± 1.86 | 23.41 ± 0.59 | 91.36 |
| Mean ± SE | | 39.93 ± 0.81 | 22.36 ± 0.42 | 79.16 ± 6.66 |
| 590 MPa (134 min) | 336 | 38.73 ± 1.76 | 20.79 ± 0.62 | 86.26 |
| | | 39.38 ± 1.19 | 23.18 ± 0.68 | 69.90 |
| | | 38.35 ± 1.95 | 23.70 ± 0.51 | 61.82 |
| | | 42.00 ± 3.15 | 23.25 ± 0.74 | 80.69 |
| | | 41.75 ± 3.39 | 24.06 ± 1.49 | 73.53 |
| Mean ± SE | | 40.04 ± 0.92 | 22.99 ± 0.35 | 74.44 ± 4.24 |
| 665 MPa (48 min) | 336 | 42.07 ± 4.99 | 23.91 ± 0.49 | 75.95 |
| | | 43.29 ± 1.86 | 23.87 ± 0.66 | 81.23 |
| | | 37.57 ± 1.53 | 22.81 ± 0.60 | 64.67 |
| | | 39.29 ± 2.10 | 23.66 ± 0.72 | 66.05 |
| | | 44.07 ± 2.84 | 25.16 ± 0.09 | 75.17 |
| Mean ± SE | | 41.26 ± 1.30 | 23.89 ± 0.28 | 72.61 ± 3.15 |
| 590 MPa (134 min) | 4392 | 37.65 ± 2.09 | 26.05 ± 0.74 | 44.54 |
| | | 38.57 ± 2.44 | 25.14 ± 0.63 | 53.38 |
| | | 36.16 ± 3.16 | 22.82 ± 0.46 | 58.48 |
| | | 40.19 ± 3.49 | 22.09 ± 0.66 | 81.95 |
| | | 42.26 ± 4.60 | 23.20 ± 0.28 | 82.17 |
| Mean ± SE | | 38.96 ± 1.21 | 23.86 ± 0.42 | 64.10 ± 7.66 |

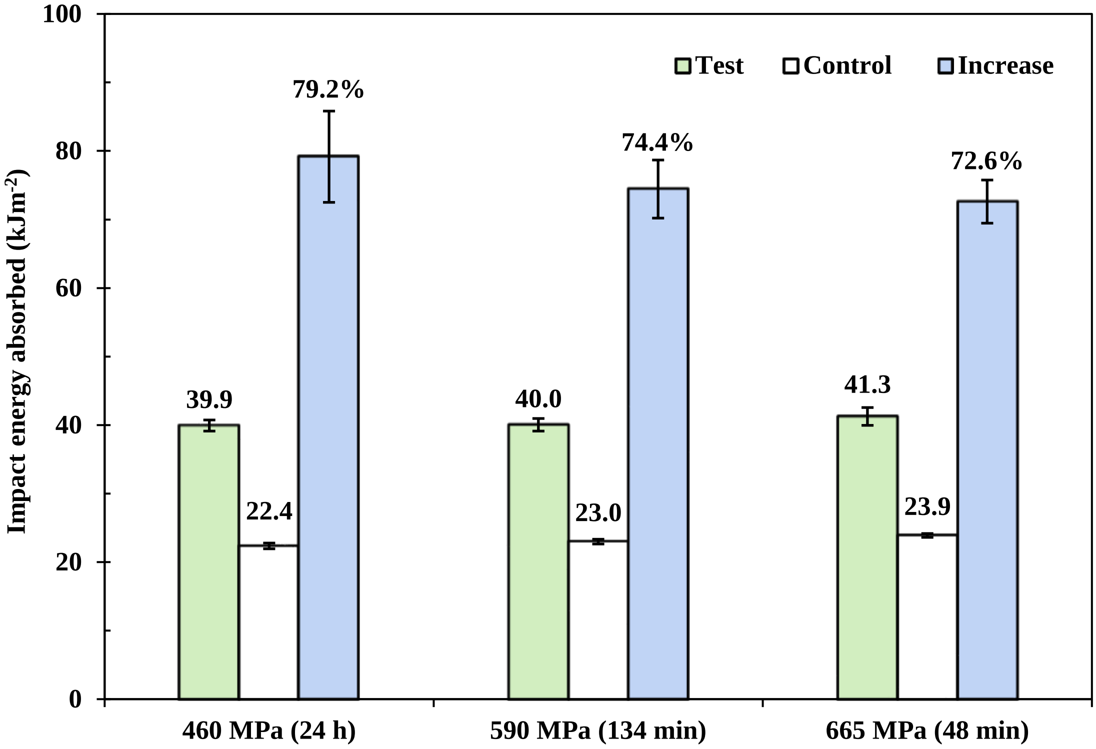


**Figure 6.** Charpy impact test results, the three creep conditions are equivalent in terms of final viscoelastic creep strain for $\varepsilon_c(24)$ at 460 MPa, *i.e.* ~4.0%; error bars represent the standard error.

A further five batches of VPPMC samples with their control counterparts, produced with the 590 MPa, 134 min creep condition were stored for 4392 h (0.5 years) prior to impact testing to investigate stability in prestress benefits. The results are also shown in Table 5. Again, all batches show effectively the same mechanical benefits from VFP (at 5% significance level). Therefore, it is concluded that there is no deterioration in impact performance of VPPMC samples at ~4.0% creep strain level for up to 0.5 years in real time.

### *4.7. Viscoelastic fibre prestressing mechanisms*

Figure 7 shows the relationship between applied stress $\sigma$ and $t_n$ at a prestrain level of ~4.0% following the principle described in Figure 1, together with the curve at ~3.4% prestrain level from Figure 3, and fitted by Eqn 3. It shows that the two curve-fits meet at ~0.1 h with a creep stress of ~920 MPa, which infers that the fibre breaks after very short time loading. Since the breaking strength of nylon fibre is up to 900 MPa, again, Eqn 3 indicates reasonable predictions on prestress equivalence.

Here, two viscoelastic creep strain levels were adopted, where a ~3.4% (Section 4.1) prestrain level represents the standard creep condition (330 MPa for 24 h), and ~4.0% represents the strain when the maximum mechanical benefits were observed for all the prestress conditions under investigation. For ~3.4%, the impact benefits from the optimised VPPMC samples are broadly the same with the standard prestressed samples (330 MPa for 24 h). Also, there is no detriment to impact behaviour in samples either naturally aged to 0.5 years or artificially aged to an equivalent (in terms of TTSP) of 20,000 years at a constant 20°C. This infers the long-term reliability of VFP in a PMC. Confirmation of effectiveness is also demonstrated at a viscoelastic creep strain (prestrain) of ~4.0%, where prestress equivalence is observed from the impact performance of VPPMC samples naturally aged up to 0.5 years in real time. The effect of viscoelastic creep strain level has shown a decrease in benefits when fibres are subjected to 590 MPa for 24 h, *i.e.* ~4.8% in viscoelastic creep strain [31]. These findings further developed the understanding of the viscoelastic prestress mechanisms within the composite.

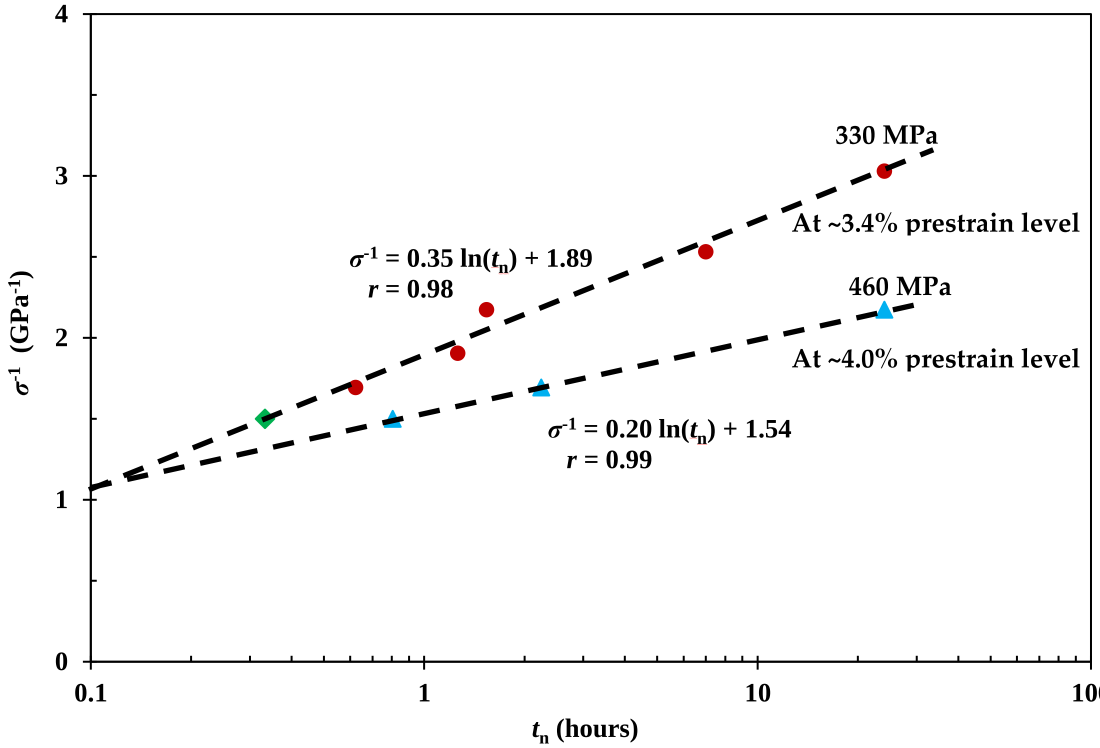


**Figure 7.** Relationship between applied creep stress $\sigma$ and required $t_n$ for loading at two viscoelastic creep strain levels; dashed lines are fitted by using Eqn 3.

The VFP mechanisms are closely related to the microstructures of the prestressed fibres. For a viscoelastic solid, the time-dependent deformation can be represented by a series of springs and dashpots. These are treated as consist of elastic energy storage sites (EESTs) and viscoelastic energy storage sites (VESTs) [59]. For a semi-crystalline polymeric fibre, instantaneous elastic deformation is mainly determined by the crystalline regions, *i.e.* EESTs; whilst viscoelastic deformation is dominated by the amorphous regions, *i.e.* VESTs. As implied by the free volume theory and the TTSP, various prestrain levels can be achieved under the same creep stress for different durations. Figure 8 shows the increase in impact energy under a creep stress of 590 MPa, versus the VFP processing time. When subjected to a constant creep stress, VESTs are progressively triggered to store energy. As the creep stretching progresses over longer periods, an optimum level for prestress benefits from stored energy releasing was observed. Thus effectively, increasing the creep stress or increasing the creep time has similar effects on VESTs, which controls the mechanical performance of VPPMCs, as demonstrated in Figure 7. This also provides a theoretical foundation for the fact that time-stress superposition principle could be well fitted to the strain-time data of nylon 6,6 fibre [47], *i.e.* stress effects can be represented by time duration at a constant stress value.

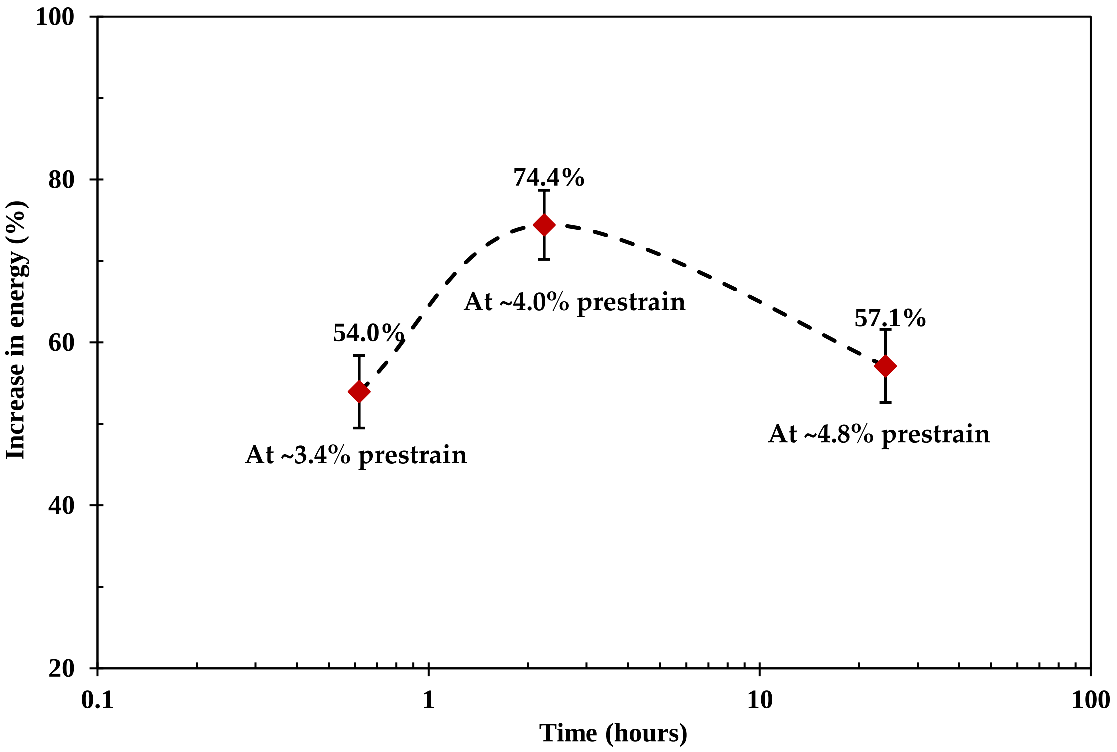


**Figure 8.** Summary of increase in impact energy from VPPMC samples under a creep stress of 590 MPa. Viscoelastic creep strain levels are achieved through creep exposure, *i.e.* 37 min for ~3.4%, 134 min for ~4.0% and 24 h for 4.8%.

## 5. Conclusions

This research develops the prestress equivalence principle, and investigated the durability of viscoelastic fibre prestressing within a composite, in order to achieve towards the full potential of prestress benefits for VPPMC technology, and further enrich the prestress mechanisms. The effectiveness of the prestress equivalence principle was refined through Charpy impact testing of prestressed samples with different prestrain levels; durability was analysed by subjecting VPPMC samples to both natural aging (up to 0.5 years) and accelerated aging (by applying the time-temperature superposition principle). The VFP mechanisms are then proposed. The main findings include:

(i) The durability of viscoelastic fibre prestressing in a polymeric composite is evaluated through both natural aging (up to 0.5 years) and accelerated aging. It is found that the impact benefits are still active after been accelerated aged to an equivalent of 20,000 years at 20°C, inferring long-term reliability of VFP-generated fibre recovery as in a composite.

(ii) The developed prestress equivalence principle shows a logarithmic relationship between applied creep stress $\sigma$ and $t_n$, allowing prediction of the $t_n$ value required for a given $\sigma$. This is further verified by exploiting various creep stress conditions to obtain the same prestrain level. It can also be applied to pursue towards achieving the full mechanical potential of the viscoelastic fibre prestressing, indicating further flexibilities for VPPMC technology.

(iii) Longer exposure of nylon 6,6 yarns to a higher strain level could increase the viscoelastic prestress-induced mechanical benefits. Compared to the ~3.4% prestrain level, an 18% increase in viscoelastic creep strain results in a ~34% increase in prestress benefits, and there is no deterioration in prestress benefits up to 0.5 years at real time.

(iv) The increase in impact energy is a function of creep time at a constant creep stress (590 MPa). There is an optimum prestrain level to maximise the prestress benefits. Increasing the creep stress (at constant creep time) or the creep time (at constant creep stress) have similar effects on the microstructures of the prestressed fibres; the prestress mechanisms are subsequently proposed.

These findings demonstrated that both materials and energy consumptions could be conserved for manufacturing of advanced composites, the viscoelastic fibre prestressing presents long-term effectiveness within a polymeric composite. Therefore, they promote further steps of VPPMC technology towards potential industrial applications, especially for impact protections.

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