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Viscoelastically prestressed polymeric matrix composites – Effects of delayed moulding on Charpy impact properties.

Yang Qin, Kevin S. Fancey*

GW Gray Centre for Advanced Materials,

School of Engineering & Computer Science

University of Hull, HU6 7RX, UK

Abstract

Viscoelastically prestressed polymeric matrix composites (VPPMCs) are produced by subjecting fibres to creep, then releasing the creep load before moulding. Previous work has demonstrated mechanical property improvements up to ~50% from nylon 6,6 fibre-polyester resin VPPMCs, compared with control (unstressed) counterparts. Since fibre stretching and moulding processes are decoupled, the time interval between releasing the fibre stretching load and moulding (delayed moulding) offers considerable production flexibility. This paper investigates delayed moulding over 0–1272 h, using fibres stored at 20 °C and -25.4 °C. Charpy impact tests demonstrated increased energy absorption from all VPPMC samples compared with control counterparts, this increase reducing with delayed moulding time. A 1272 h delay gave an increase of ~23% for fibre storage at 20 °C, and ~40% at -25.4 °C, the latter demonstrating "decelerated" ageing. For all samples, the magnitude of fibre-matrix debonding (the principal energy absorption mechanism) increased linearly with impact energy data.

Keywords: A. Polymer-matrix composites (PMCs); B. Debonding; B. Impact behaviour; Viscoelasticity.

* Corresponding author. Tel.: + 44 1482 465071.
E-mail address: k.s.fancey@hull.ac.uk (K.S. Fancey).

1. Introduction

Residual stress within polymeric matrix composites (PMCs) is usually considered to be an unwanted consequence of differential shrinkage from the processing route [1]. Nevertheless, there have been a number of investigations into exploiting intentionally induced stress during PMC production, the principal motivation being to enhance mechanical properties without the need to increase mass or section thickness within a composite structure.

Elastically prestressed PMCs (EPPMCs) can be produced, based on the principles utilised for prestressed concrete, in that fibres (e.g. glass) are stretched to a fixed elastic strain during matrix curing. Following curing, the fibre tensile load is released, so that compressive stresses are created within the solidified matrix, these being balanced by residual fibre tension. Early EPPMC studies were based on laminates, to decrease fibre distortion and improve laminate stiffness [2] or to reduce the effects of (unwanted) thermally induced residual stresses [3-5]. Later investigations with unidirectional glass fibre EPPMCs demonstrated increases in tensile strength and elastic modulus of ~25% and 50% respectively, compared with unstressed counterparts [6]. Impact toughness, flexural stiffness and strength were also found to increase by up to 33% [7, 8]. Within the last decade, EPPMC studies have included EPPMCs based on glass fibre, as possible dental materials [9]; also, other prestressing reinforcements have been investigated, including carbon fibre [10] and natural fibre (flax) [11]. The exploitation of EPPMCs for shape-adaptive (morphing) composite structures has also been of interest, either as prestressed laminates [12] or unidirectional fibre prestressed structural elements [13]. Most recently, EPPMCs have been reported to show significant improvements in fatigue life [14, 15].

Despite these studies demonstrating the benefits of elastic prestressing within a PMC, there are two potential drawbacks. First, the need to apply fibre tension during matrix curing can restrict fibre length, orientation and spatial distribution, which compromises mould geometry [16]. Moreover, it has been reported that stretching rig design with appropriate fibre clamping can be technically challenging [12, 17]. The second drawback originates from the matrix being a polymeric material: elastically generated prestress can be expected to promote localised matrix creep at fibre-matrix interface regions, which could cause the prestress to deteriorate progressively with time [16]. Evidence of this effect has been recently reported [15].

As an alternative approach to the EPPMC route, the mechanical properties of a PMC can be improved by exploiting the viscoelastic characteristics of certain fibre reinforcements. Viscoelastically prestressed polymeric matrix composites (VPPMCs) have demonstrated such improvements, in comparison with their unstressed counterparts, without the need to increase section size or weight [18]. Published results have shown 20–50% increases in Charpy impact strength and flexural stiffness [19-24] and up to 15% improvement in tensile strength [25] for nylon 6,6 fibre-based VPPMCs. Similarly, for ultra-high molecular weight polyethylene (UHMWPE) fibre-based VPPMCs, 20–40% increases in flexural and Charpy impact properties have been observed [26, 27]. In addition, VPPMCs with bamboo slivers [28] and cellulose fibres [29] have demonstrated flexural and tensile property improvements of 20% or more. To produce a VPPMC, the fibre reinforcement is first subjected to a tensile creep load for a designated time period; after removing the load, the loose fibres are moulded into a resin matrix. Following resin solidification, the viscoelastically recovering fibres generate compressive stresses within the matrix, which are counterbalanced by residual tension within the fibres [18].

In contrast with EPPMCs, long-term viscoelastic recovery processes in VPPMCs are expected to counteract localised creep at the fibre-matrix interfaces [16]. Thus a longevity study using accelerated ageing has shown no degradation in Charpy impact performance over a period equivalent to 25 years at a constant 50 °C with nylon 6,6 fibre-based VPPMCs [22]. Moreover, in contrast with EPPMC production, the fibre stretching and moulding processes are decoupled in VPPMC manufacturing, offering potentially significant benefits: (i) after releasing the stretching load, the fibre reinforcement can be chopped to any length and moulded into the resin matrix in any orientation; (ii) VPPMC production has no geometrical limitations; (iii) the stretching equipment can be relatively simple [18].

To date, all VPPMC samples in previous studies have been produced by moulding the prestrained fibres almost immediately after releasing the stretching load. This arises from investigations being limited to evaluating property improvements [16, 19-27, 29, 30-35] and the performance of bistable (morphing) structures [36, 37]; thus only relatively simple (effectively) one-dimensional VPPMC samples with unidirectional fibres have been required. In order to produce VPPMCs with more complex geometries (especially involving discontinuous fibres which would require a chopping process), the time interval between releasing the fibre stretching load and moulding could be longer. Moreover, during industrial manufacture, the fibre stretching and moulding processes may even be conducted at two different locations; i.e. prestrained fibres would need to be transported to another location for moulding, which could exacerbate the time interval. Therefore, the aim of this paper is to study the influence of delayed moulding, this being achieved by evaluating the Charpy impact properties of nylon fibre-based VPPMCs.

2. Background

2.1. Long-term viscoelastic recovery strain

Long-term viscoelastic properties can be evaluated by recovery strain measurement [16, 22, 23, 26, 32]. For example, Fig. 1 shows recently acquired recovery strain-time data from nylon 6,6 fibre, following 24 h creep at 330 MPa [23]. An equation based on the Weibull or Kohlrausch-Williams-Watts (KWW) function [38] is used to describe the time-dependent viscoelastic recovery strain $\varepsilon_{rvis}(t)$:

$$\boldsymbol{\varepsilon_{rvis}(t) = \varepsilon_r \left[\exp\left(-\left(\frac{t}{\eta_r} \right)^{\beta_r} \right) \right] + \varepsilon_f} \quad (1)$$

where η_r and β_r in the ε_r function represent the Weibull characteristic life and shape parameters respectively. Permanent strain resulting from viscous flow is represented by ε_f. The curve fitting parameters in Fig. 1 show that ε_f is less than 10^{-9} %; thus the strain from viscous flow is negligible.

2.2. The time-temperature superposition principle

For some materials with thermorheological properties, the results from experiments (creep, stress relaxation, etc.) performed at different temperatures can be assembled into a "master curve" over a wide range of timescales. Thus through the time-temperature superposition principle (TTSP), a change in temperature can be equivalent to a shift on the time scale, with a temperature shift factor, αT [39]. Published results from creep and stress relaxation experiments based on nylon 6,6 fibre, have demonstrated that a simple linear relationship exists between log αT and temperature over a wide temperature range [40, 41]. It has also been shown that αT can be applied to the viscoelastic recovery process [22, 32]. By applying the TTSP to increase viscoelastic recovery rate (accelerated ageing) at an elevated temperature of 70 °C for 2298 h (equivalent to 25 years at 50 ˚C), the longevity of VPPMCs was studied by Charpy impact testing. The results, as reported in Section 1, demonstrated no deterioration in improved impact energy absorption from fibre prestress [22].

Here in this paper, by subjecting prestrained nylon 6,6 fibres to a lower temperature, the viscoelastic recovery rate shown in Fig. 1 can be reduced in accordance with the TTSP. Thus, at a lower temperature, this "decelerated ageing" effect will enable the prestrained fibres to maintain their viscoelastic recovery strain over a longer timescale. Fig. 2 shows log α_T as a function of temperature (-65 ˚C to 35 ˚C) with a linear regression fit from stress relaxation experiments [40, 41]. This allows log α_T to be predicted at a low temperature with respect to a reference temperature, T_0, of 20 ˚C.

3. Experimental procedures

3.1. Sample production

In accordance with previous work [16, 20-23, 30-32, 34, 35], batches of VPPMCs were produced following the procedures summarised below. The fibre reinforcement was an untwisted continuous nylon 6,6 yarn consisting of 140 filaments (27.5 μm filament diameter) supplied by Ogden Fibres Ltd, UK. Two identical yarns, designated "test" and "control", were annealed simultaneously in a fan-assisted oven at 150 °C for 30 min. This removed any residual stresses induced during manufacturing, as required for the long-term recovery observed in Fig. 1 [18]. The test yarn was stretched under a 330 MPa creep stress for 24 h with a bespoke stretching rig [35], while the control yarn was positioned in close proximity to ensure exposure to the same environmental conditions (19–21 °C, 30–45% RH).

Five different time intervals were selected to study the delayed moulding effect, i.e. 0 h, 4 h, 48 h, 216 h and 1272 h. After releasing the creep load, both prestrained and unstrained yarns were stored loose in sealed polyethylene sample bags at room temperature (19–21 °C) for the respective time intervals. In addition, further batches of yarn were stored in a laboratory freezer (-25.4 ± 0.4 °C) for 1272 h to decelerate the viscoelastic recovery. According to the TTSP, the viscoelastic recovery of prestrained yarns at -25.4 °C for 1272 h was equivalent to ~10 min at 20 °C, based on Fig. 2. Following removal from the freezer, both test and control yarns were allowed to return to room temperature, by delaying the moulding procedure for 1 h. Both yarns were then individually folded, chopped into ~600 mm lengths and brushed into two flat ribbons ready for moulding.

The matrix material was a clear polyester casting resin (Reichhold Polylite 32032), mixed with 2% MEKP catalyst; this was supplied by MB Fibreglass, UK. Two identical aluminium moulds, each with a 10 mm wide, 3 mm deep and 450 mm long polished channel, were utilised to produce test (prestressed) and control (unstressed) composite strips with unidirectional continuous fibres. Both strips were then cut into five equal lengths for one batch of 5 test and 5 control samples after demoulding. Sample geometry was 80 × 10 × 3.2 mm, with a fibre volume fraction (V_f) of ~2 %. This low V_f minimised frictional energy losses during Charpy impact testing and facilitated visual inspection of the resulting debonded regions [35]; however, higher V_f values (3% – 53%) have also been investigated [19, 21, 25, 34, 36]. Subsequently, all samples were held under steel weights for 24 h to prevent possible distortion. Finally, all samples were stored at room temperature for ~336 h before impact testing.

3.2 Recovery strain measurement within the resin

To determine the onset of matrix solidification and resulting composite prestress generation, the nylon 6,6 fibre recovery strain within the polyester resin was measured as the matrix cured. Before the 24 h stretching process, two ink marks (150 mm apart) were applied to the nylon 6,6 yarn. After moulding the yarn into the resin (with minimal delay), another two marks (150 mm apart) were also applied within the resin immediately before solidification, as shown in Fig. 3. The change in strain during matrix curing for both nylon 6,6 yarn and the resin was measured at room temperature (19–21 °C) by a digital calliper with a measurement precision of ± 0.01 mm. In accordance with previous viscoelastic recovery strain studies [16, 23, 26, 29, 31, 32], results were recorded from single measurements below 1 h (due to high fibre strain rates) and from the mean of three readings at longer time intervals.

3.3 Charpy impact testing and debonded area assessment

Charpy impact tests were conducted with a Ceast Resil 25 Charpy machine utilising a 7.5 J hammer at 3.8 m/s, operating in accordance with BS EN ISO 179 [42]. Previous investigations have shown that for low V_f composites, nylon 6,6 fibres tend to sink towards the bottom of the mould before the resin cures, as demonstrated by photographic examples of composite sample cross-sections [19, 21, 33]. Thus all

impact tests were performed with the fibre-rich side facing away from the pendulum hammer, the configuration being previously reported [16, 30, 31]. All tests were performed at room temperature (19–21 °C) with a test span of 24 mm, in accordance with previous studies [16, 20-23, 27, 30-33, 35].

Fibre-matrix debonding is considered to be the principal impact energy absorption mechanism [21, 24]. Therefore, following Charpy impact testing, each sample was quantitatively studied by measuring the debonded area, this being facilitated by using *ImageJ* software for image enhancement. Here, the software was used to calculate area by manually outlining the debonded (brighter) region from a high contrast image.

4. Results and discussion

4.1. Recovery strain within the resin

Recovery strain data from the prestrained nylon 6,6 yarn within the resin (moulded immediately after releasing the load) are plotted against time in Fig. 4. For comparison, Fig. 4 also shows strain values from the resin. It is clear that during resin curing, the viscoelastic recovery process continues, while the resin appears to show little or no deformation. Although some matrix shrinkage may have been expected, Reichhold Polylite 32032 is a low-shrinkage resin. Moreover, the thin, flat strip geometry (a large surface area to volume ratio) would have restricted resin shrinkage through contact with the mould surfaces. Despite the residual stresses that can be expected from yarn contraction and resin solidification, previous work has demonstrated that fibre-matrix bonding is improved within VPPMCs [25, 34] and this improvement is discussed in Section 4.3. Fig. 4 indicates that the fibre contraction process (recovery) appears to cease at ~2 h, suggesting that the resin has sufficiently solidified to be capable of "gripping" the prestrained yarn; i.e. the fibre recovery strain becomes fixed. Thus, it can be assumed here that compressive stresses imparted to the surrounding matrix are initiated at ~2 h after moulding. Consequently, by adding this 2 h fibre recovery time to the time intervals reported in Section 3.1, the "true" delay time between releasing the fibre stretching load and compressive stress generation is obtained; i.e. 2, 6, 50, 218 and 1274 h under room temperature. For the low temperature delay condition, the 1 h interval for fibres to return to normal room temperature is added, giving 1275 h.

4.2. Charpy impact tests

Table 1 summarises the Charpy impact data. For each delay time at 20 °C, 9 batches (i.e. 45 test, 45 control) and at -25.4 °C, 13 batches (i.e. 65 test, 65 control) of composite samples were tested. The increase in energy absorption against the "true" delay time, t_{TD} is also plotted in Fig.5. In Table 1, it can be observed that all control samples show a consistent energy absorption of ~24 kJ/m^2, including those stored at -25.4 °C for 1272 h. This therefore indicates that exposure to the low temperature does not affect fibre properties (other than the deceleration in test fibre recovery rate) in composite production. The increase in energy absorption between VPPMCs and control samples shows a gradually decreasing trend with t_{TD}. Compared with the ~45% increase in value observed from VPPMCs with the immediate fibre moulding (i.e. a t_{TD} of 2 h), the 4 h and 1272 h delays (under room temperature) in fibre moulding still give ~40% and ~23% respectively.

More importantly, it is observed that VPPMCs with prestrained fibres stored for 1272 h under the low temperature condition (-25.4 °C) absorb ~40% more energy. This demonstrates successful retardation of viscoelastic recovery rate, enabling a significant fibre viscoelastic recovery strain to be maintained. As reported in Section 3.1, the t_{TD} was equivalent to ~10 min at 20 °C for the fibre refrigeration period (1272 h); subsequently, the fibres were stored for 1 h at 20 °C prior to moulding and fibres continued to recover during resin curing for a further 2 h (Section 4.1). Therefore, the total t_{TD} was ~3.2 h, suggesting that the increase in energy absorption for VPPMCs with previously refrigerated fibre reinforcements should be in

the range corresponding to the increase between 2 and 6 h delay times at 20 °C (41.86%–45.11%). Although the 40.68% result is slightly below this range, the difference is within the limits of measurement variations. This is verified by one-tailed hypothesis testing at 5% significance level. Consequently, the results suggest that the impact performance of VPPMCs produced from refrigerated prestrained fibres will be equivalent to the same time-temperature shift factor (α_T) applied directly to fibre viscoelastic recovery.

Table 1

Charpy impact results for composite sample batches; 9 batches (i.e. 45 test, 45 control) for each delay time at 20 °C and 13 batches (i.e. 65 test, 65 control) for batches delayed at -25.4 °C. SE represents the standard error of the mean.

Fibre delay conditions	Delayed moulding time (h)	t_{TD} (h)	Mean impact energy (kJ/m^2)		Mean increase in energy (% ± SE)
			Test ± SE	Control ± SE	
20 °C	0	2	35.05 ± 0.66	24.23 ± 0.57	45.11 ± 3.48
	4	6	34.55 ± 0.63	24.44 ± 0.60	41.86 ± 3.46
	48	50	33.49 ± 1.38	24.34 ± 0.91	38.02 ± 4.38
	216	218	31.54 ± 1.45	24.03 ± 0.80	31.47 ± 5.34
	1272	1274	29.11 ± 0.69	23.66 ± 0.42	23.25 ± 3.30
-25.4 °C	1272	1275*	34.40 ± 0.88	24.64 ± 0.62	40.68 ± 4.99

*Equivalent to ~3.2 h at 20 °C

In Fig. 1, the recovery strain value for nylon 6,6 fibres can be predicted for any time value, by the curve fitting parameters. Thus in Fig. 6, the increase in energy absorption is plotted against the recovery strain predicted at each t_{TD}. Since there appears to be a linear relationship, the increase in impact energy between VPPMC and control samples can be readily predicted from the recovery strain data. Moreover, the y-axis intercept is close to zero (i.e. no increase in impact energy from unstrained fibres), which provides self-consistent support for the linear relationship in Fig. 6.

4.3 Debonded area assessment

Fig. 7 shows the typical debonding patterns from both test and control composite samples, following Charpy testing, over the range of delayed moulding intervals. A larger debonded area is observed on all test samples, in accordance with previous findings. This is said to result from the residual shear stresses at the fibre-matrix interfaces induced by the prestrained fibres, which promote debonding over transverse fracture during the impact process [20, 31]. It is also believed that this debonding is the major energy absorption mechanism in impact tests [21, 24]. Thus for VPPMC samples, the larger debonded areas observed here, concur with the greater energy absorption results in Table 1 and Fig. 5. Moreover, there appear to be no discernible differences in debonded area for all the control samples, which is in accordance with the similar energy absorption values for these samples in Table 1. For VPPMCs with prestrained fibres delayed at room temperature, the debonded area decreases with increasing delay time interval. Nevertheless, for VPPMCs with prestrained fibre reinforcements stored at -25.4 °C for 1272 h,

the debonded area is significantly larger than that of the equivalent delay at room temperature and this concurs with the result in Table 1 and Fig. 5.

By assuming the debonding mechanism is consistent through the sample thickness, the differences in debonded area between test and control samples can be compared quantitatively. An approximately linear relationship between increase in impact energy absorption and increase in debonded area is shown in Fig. 8. This finding agrees with impact testing results from a previous VPPMC study [21] and also compares well with studies on glass fibre-epoxy plates, carbon fibre reinforced laminates and graphite fibre reinforced epoxy [43-45]. It is also interesting to note the positive y-axis intercept; this implies that even without a larger debonded area (i.e. the increase in area being 0%), test samples absorb ~9% more energy than their control counterparts. A recent study based on the scanning electron microscope mirror effect, has provided evidence that VPPMC samples trap fewer negative electric charges than their control counterparts; this indicates that VPPMCs possess a higher bonding strength at the fibre-matrix interfaces [34]. Therefore, we suggest that this effect may explain the ~9% increase observed for the intercept in Fig. 8. Here, a higher bonding strength at the fibre-matrix interfaces will result in more energy absorption from the debonding mechanism for the same debonded area.

5. Conclusions

The effect of delayed moulding from 0 h to 1272 h in VPPMC production has been investigated by Charpy impact testing of nylon 6,6/polyester VPPMCs. The major findings are:

(i) Over the full range of time intervals for delayed moulding of prestrained fibres, the impact energy absorption by VPPMC (test) samples was greater than the unstressed control samples. In comparison with the ~45% increase for no delay (i.e. fibres being moulded as soon as possible after releasing the stretching load), the increase was still ~23% after a delay of 1272 h at room temperature.

(ii) Comparing VPPMCs with prestrained fibres delayed at room temperature for 1272 h, VPPMCs with fibres refrigerated at -25.4 °C for the same time period showed a much greater increase in energy absorption; i.e. ~40%, over their control counterparts. By employing the TTSP, 1272 h at -25.4 °C was calculated to give an equivalent t_{TD} of ~3.2 h at 20 °C. The results suggest that VPPMC impact performance with prestrained fibres stored at low temperature will be equivalent to the same time-temperature shift factor α_T, as applied directly to fibre viscoelastic recovery strain.

(iii) The increase in VPPMC sample impact energy absorption shows a linear relationship with fibre recovery strain determined from the t_{TD} values; therefore, the impact performance of VPPMCs with prestrained fibres delayed under various conditions can be predicted.

(iv) A larger debonded area was observed on all test samples and the increase in energy absorption showed a linear relationship with increasing debonded area between test and control samples. There is evidence to suggest that test samples would still absorb ~9% more energy, independent of any increase in debonded area. This may be a result of higher bonding strength at the fibre-matrix interfaces for VPPMCs.

Our work has quantitatively demonstrated the effect of delayed moulding in the production of VPPMCs on impact strength. Of major importance is that this study has verified the feasibility of storing viscoelastically prestrained fibres under refrigerated conditions (on their own or as prepreg material) for subsequent VPPMC manufacture. Clearly, this demonstrates the flexibility that VPPMC manufacture may provide. Thus, for example, fibre stretching and subsequent moulding operations could be performed on different sites.

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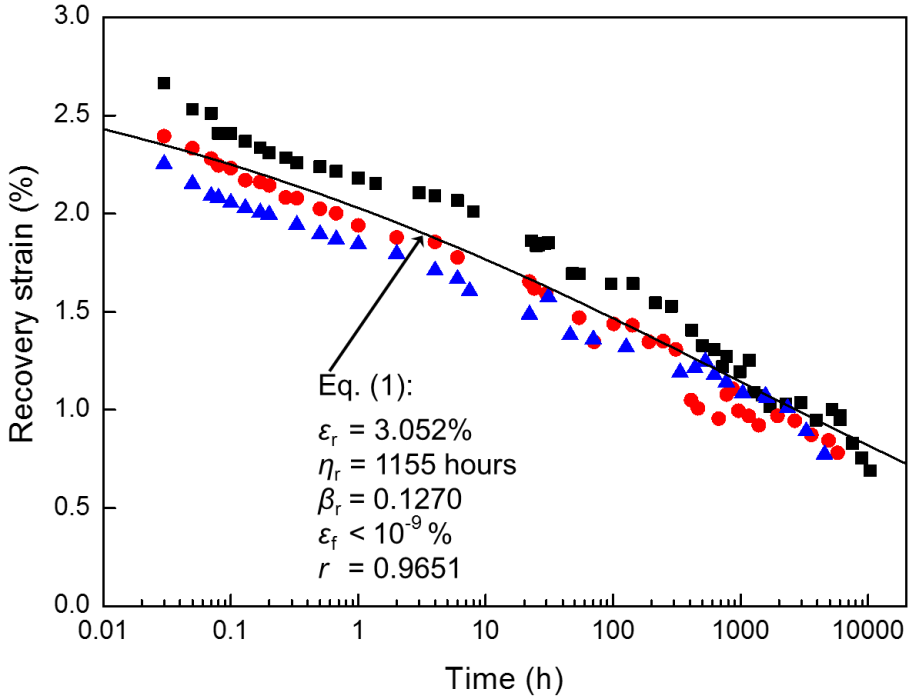

Fig. 1. Three sets (repeat runs) of recovery strain-time data from annealed (150 °C, 30 min) nylon 6,6 yarns recovering at room temperature (~20 °C) after 24 h creep at 330 MPa. Curve-fit parameters are from Eq. (1), r is the correlation coefficient; after Ref. [23].

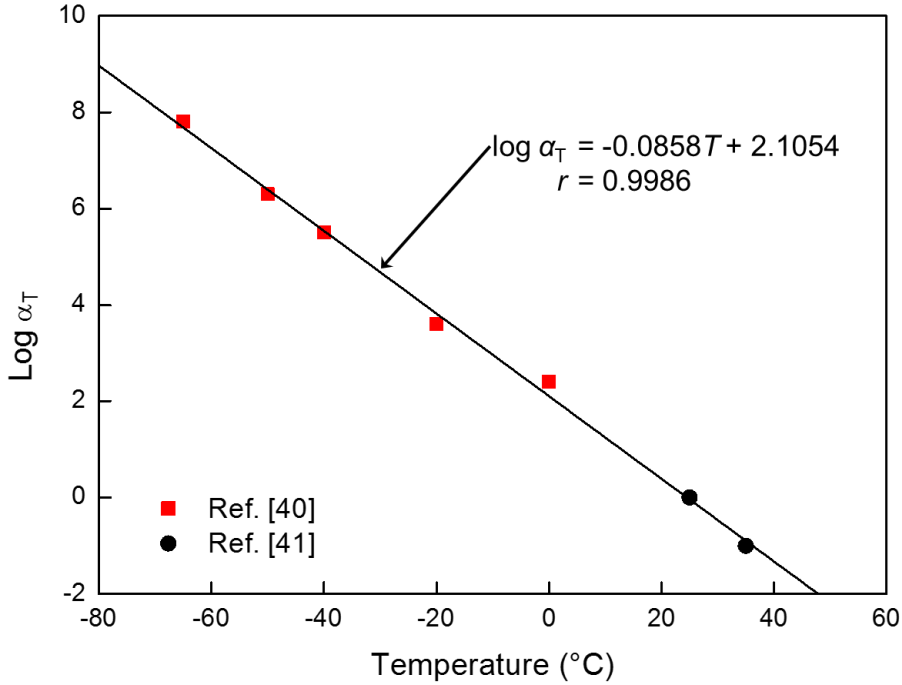

Fig. 2. Plot of the time temperature shift factor (αT) as a function of testing temperature (-65 ˚C to 35 ˚C) from published data for stress relaxation [40, 41] with nylon 6,6 fibre. Reference temperature (log α_T) was 25 ˚C. The trend and equation are from linear regression, r is the correlation coefficient.

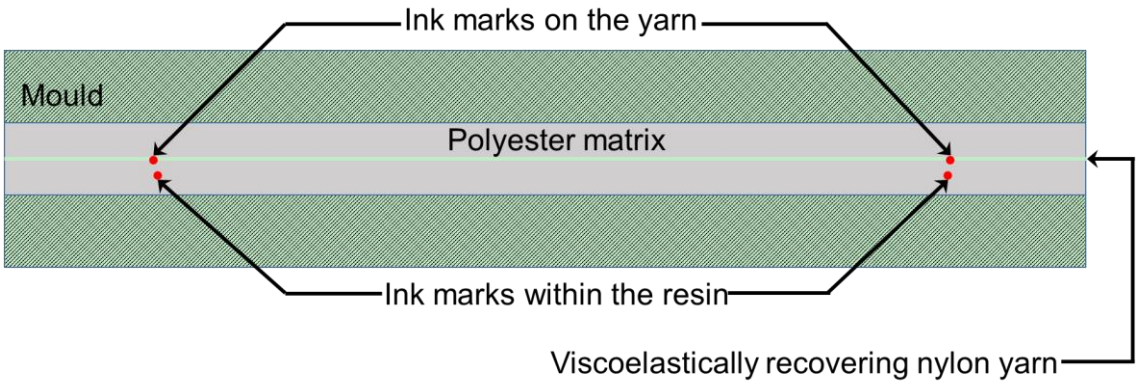

Fig. 3. Plan view of the fibre recovery strain measurement arrangement within the polyester resin.

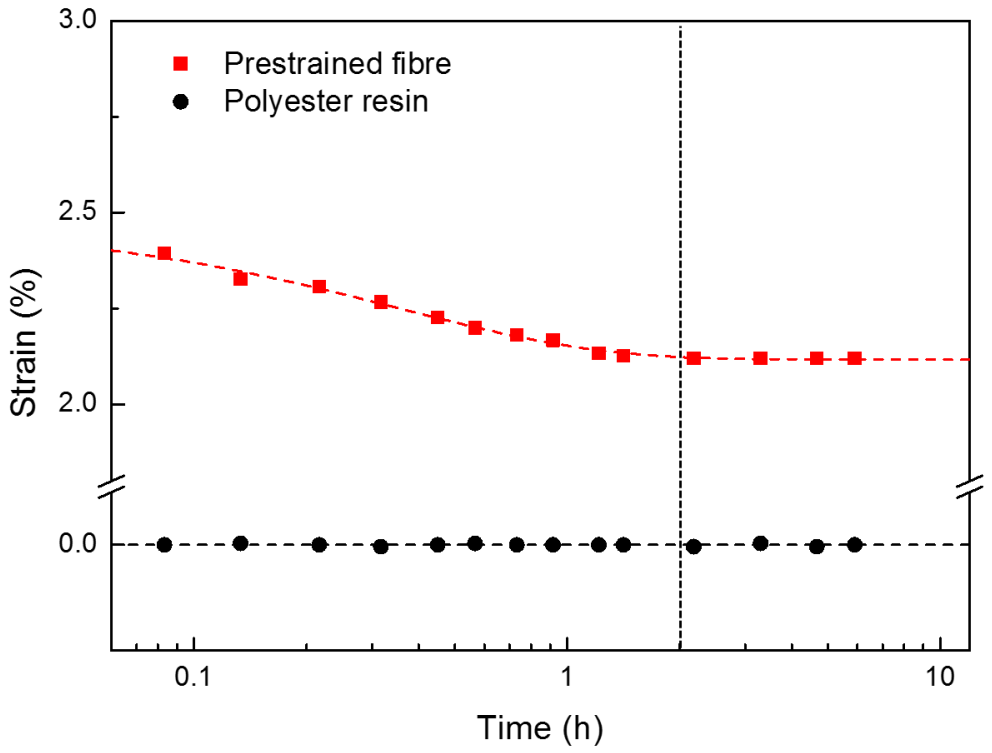

Fig. 4. Recovery strain-time data for the prestrained nylon 6,6 yarn within polyester resin (moulded with minimal delay); the strain for the resin during curing is shown for comparison.

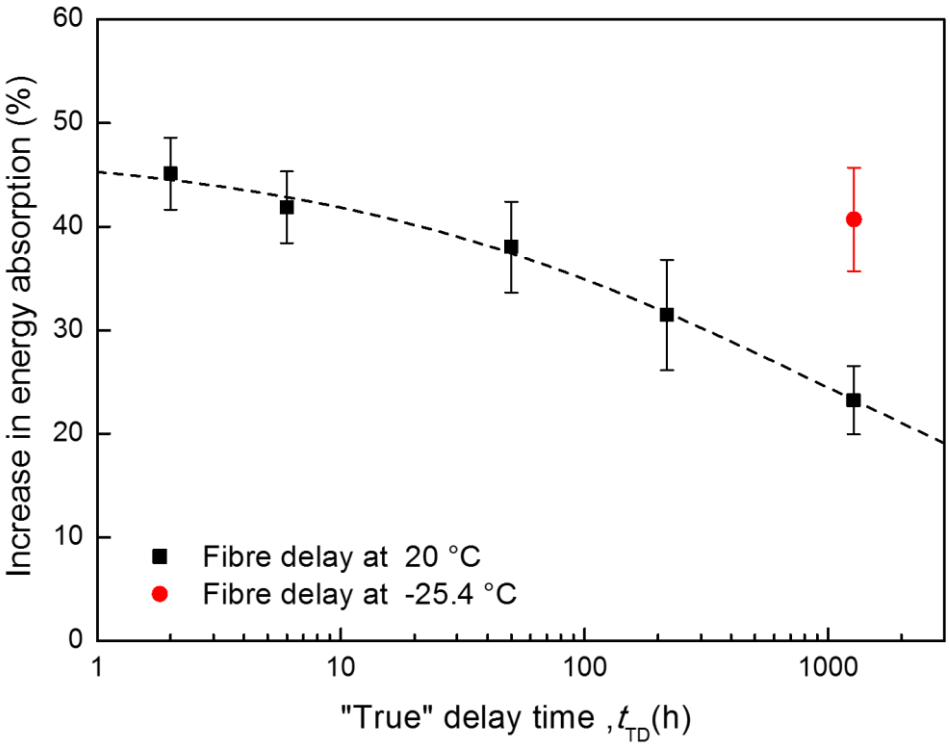

Fig. 5. Increase in impact energy absorption between test and control samples as a function of t_{TD} (data from Table 1); error bars indicate the standard error.

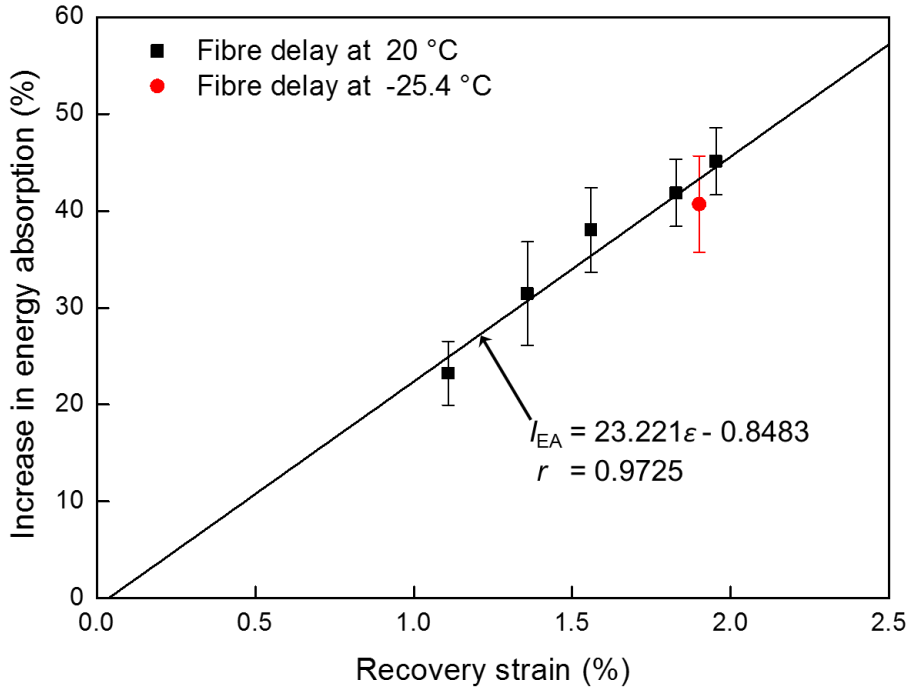

Fig. 6. Increases in VPPMC impact energy absorption versus test fibre recovery strain (predicted from Eq. (1) using Fig. 1 data at the t_{TD} values); error bars indicate the standard error. Line and equation are from linear regression. I_{EA} and ε represent the increase in energy absorption and the recovery strain respectively, r is the correlation coefficient.

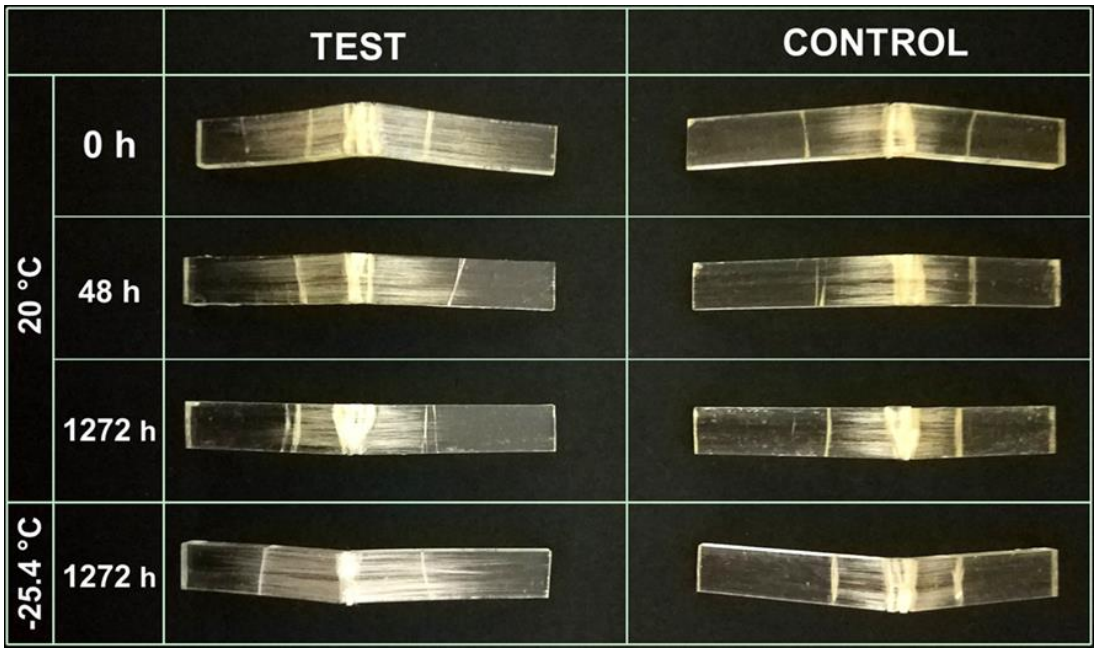

Fig. 7. Typical debonding characteristics for both test and control samples over the range of delayed moulding conditions studied. All samples are shown with the fractured central region deflected upwards, resulting from impact by the Charpy hammer on the opposite face.

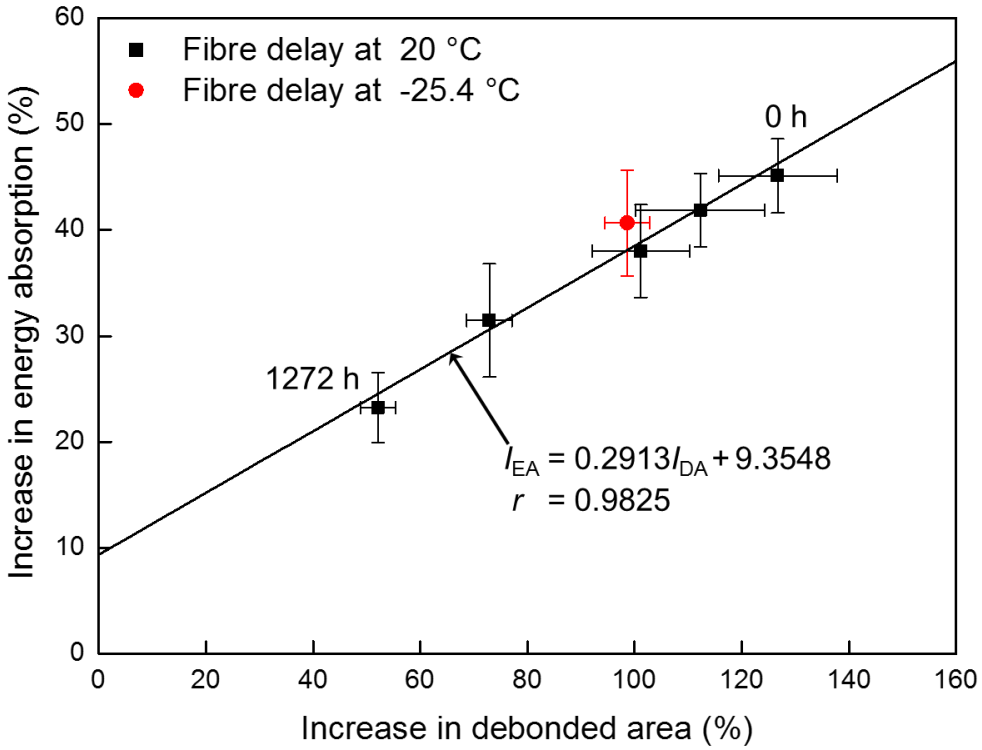

Fig. 8. Increases in VPPMC impact energy absorption versus increase in debonded area for samples under the various delayed moulding conditions. Error bars indicate the standard error. Line and equation are from linear regression. I_{EA} and I_{DA} represent the increases in energy absorption and debonded area respectively, r is the correlation coefficient.