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# Time-Temperature Superposition to Determine the Stress-Rupture of Aramid Fibres

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**Abstract** Conventional creep testing takes a long time to obtain stress-rupture data for aramid fibres at the low stress levels likely to be used in practical applications. However, the rate of creep of aramid can be accelerated by a thermally activated process to obtain the failure of fibres within a few hours. It is possible to obtain creep curves at different temperature levels which can be shifted along the time axis to generate a single curve know as a master curve, from which stress-rupture data can be obtained. This technique is known as the time-temperature superposition principle and will be applied to Kevlar 49 yarns. Important questions relating to the techniques needed to obtain smooth master curves will be discussed, as will the validity the resulting curves and the corresponding stress-rupture lifetime.

**Key words** Aramid fibres · accelerated testing · creep · stress-rupture

## 1. Introduction

Aramid fibres should have many structural applications, where their ability to carry significant loads with little creep can be exploited. However, uncertainty about the stress-rupture lifetime means that engineers are reluctant to use the fibres for tendons in prestressed concrete and stay cables in bridges, where any economy advantage relies on applying permanent high stresses.

Most of the stress-rupture models have been based on tests carried out at room temperature and at high stress levels, when creep failures can be expected in days, if not hours [1]. For permanent load applications, where lifetimes of 100 years may be specified, significant extrapolation is required. The degree of extrapolation, the short lifetimes reported in the literature, and the lack of test data all mean that engineers apply very large safety factors to cover the uncertainty.

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Creep of aramid is known to be sensitive to various environmental and testing conditions, such as humidity and temperature, as well as stress. The creep rate of fibres can be accelerated by changing these parameters [2]. In this study, creep will be accelerated by changing the temperature while other parameters are kept constant. This allows stress-rupture data to be obtained at low stress levels but raises questions about the validity of the results obtained.

The background to the Time Temperature Superposition Principle (TTSP) has been summarised by Markovitz [3]. He reports that the TTSP was probably first identified by Leaderman [4]. According to Leaderman's observations the creep compliance vs. *log* (time) curves for different temperatures for various materials are of the same shape, but increasing temperature has the effect of contracting the time scale. Tobolsky and Andrews [5] were the first to make use of Leaderman's observations to superpose the individual curves into a single reference curve.

Considerable subsequent use has been made of TTSP which is widely reported elsewhere [2, 3, 6–17]. The most relevant work related to the present study is that of Tamuzs et al. [18], who applied TTSP to SVM, an aramid fibre produced in Russia.

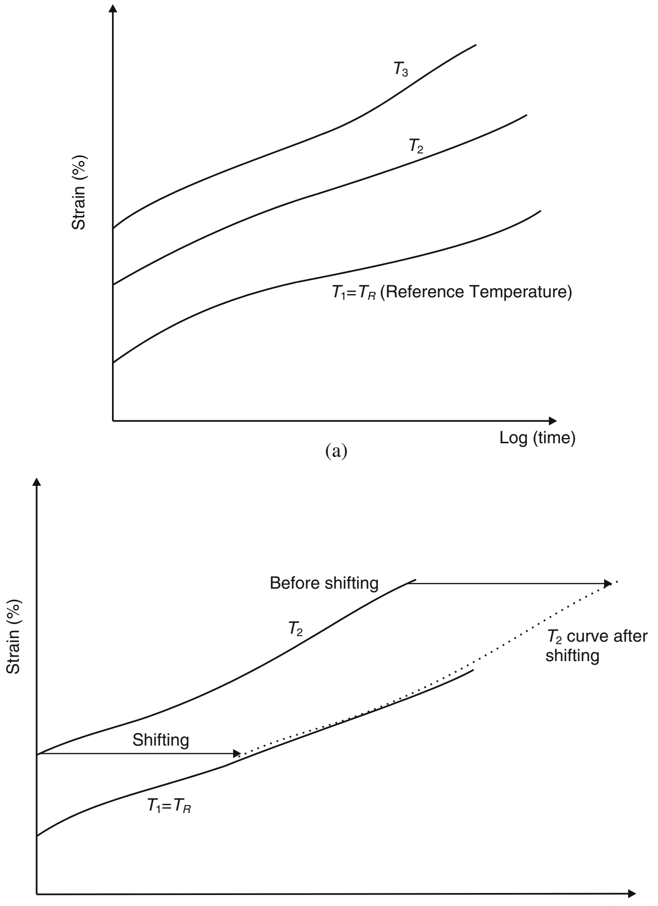
It should be pointed out here that this work does not address any mechanisms of fibre degradation, such as sensitivity to UV light or hydrolysis to which aramids are known to be susceptible. The results produced here are thus upper bounds to the predicted lifetime under sustained load in the absence of any other degrading influence.

## 2. Description of the TTSP

TTSP has been used to obtain the master curves for several properties such as creep, creep compliance and stress compliance against time (or *log* (time)) or dynamic modulus against frequency etc. [2]. When applied to the generation of a creep master curve the following steps are required.

- A material specimen is subjected to a constant load at a certain temperature as in conventional creep testing, and the variation of the creep of the specimen is observed against the *log* (time), shown diagrammatically in Figure 1(a).
- Similar experiments are performed for different specimens at different temperature levels and the relevant creep curves obtained.
- An arbitrary reference temperature is selected ($T_R$).
- All the individual creep curves corresponding to different temperature levels are shifted along the *log* (time) scale to superpose to a master curve.

To illustrate the shifting process more clearly two individual creep curves are considered in Figure 1(b); the same procedure is applicable to the other curves at different temperature levels. One of the important considerations is the smoothness at the overlap where two different individual curves meet. If the TTSP method is valid, the master curve represents the true behaviour of a long-term test at the reference temperature. That curve would be expected to be smooth, so it is a necessary condition that the master curve produced should also be smooth.



(a)

(b)

**Figure 1** (a) Idealised creep curves at different temperatures; (b) shift process

In the absence of reliable long-term data, it has been the practice of those working in this area to try to minimise the discrepancy between the individual curves when overlapped. This, in effect, turns the necessary condition into a sufficient condition to allow reliance on the TTSP method.

An additional factor, which is not tested in this paper but could be tested by these methods, is that the master curve should be independent of the TTSP test regime adopted. The same curve should be produced from tests carried out for different durations or at different temperatures.

It is generally difficult to obtain a smooth match at the overlapping region. However, partial matching of the two curves is possible. A small change at the overlap may lead to erroneous predictions of the rupture times. The error may be of the order of decades as the shifting is performed along the *log* (time) scale and the error may accumulate if multiple tests are used.

If it is possible to generate a smooth master curve by applying a horizontal shift along the *log* (time) axis, the material is classified as a thermorheologically simple material (TSM). However, for some materials a vertical shift factor may be needed to obtain a smooth master curve; they are classified as thermorheologically complex materials (TCM). The reasons for needing such factors are discussed below.

Although TTSP has been used for many decades for polymeric materials, no firm rules have been developed for obtaining the master curves. The accuracy of the master curve is dependent on the following factors:

- Variation of the shift factors with temperature.
- Existence of the same creep mechanism under different temperatures.
- The initial strain rate applied to the specimens in creep testing. Although the creep stress, $\sigma_0$ is assumed to be applied instantaneously, a finite time and a certain rate of strain are needed to reach the required stress level.
- The variation of humidity conditions.
- State of the polymeric material (glassy, rubbery, or at the transition range).
- Initial preparation of the specimens, the type of the clamping system and the type of the testing machine.
- Rate of application of heat to reach an isothermal temperature level.

### 2.1. Empirical Relationships for Horizontal Shift Factor

Several researchers have investigated the temperature dependent behaviour of the horizontal shift factor and proposed various empirical formulas. In particular, the WLF equation (Williams–Landel–Ferry) is often used [2], but it is restricted to materials above the glass transition temperature.

Since aramid fibres do not display a glass transition, but remain crystalline at all temperatures, the relationship between the temperature and the horizontal shift factor, *log* ($a_t$), is based on the Arrhenius equation. It is assumed that creep is a thermally activated process and will obey the kinetic rate theory. The time taken to failure is given by:

$$t_i = B.\exp^{\left[\frac{\Delta H}{RT}\right]} \tag{1}$$

where:

$\Delta H$ is the activation energy
$R$ is the universal gas constant
$T$ is the absolute temperature

This leads to the following relationship [19]:

$$log\,(a_t) = \frac{\Delta H}{2.30R}\left(\frac{1}{T} - \frac{1}{T_R}\right) \tag{2}$$

The superposition theory is only valid if the same mechanism is present at all the temperature levels; this can be checked by determining the activation energy of the process which should be independent of temperature.

### 2.2. Vertical Shift Factor

For thermorheologically complex materials the visco-elastic properties do not superpose completely if only horizontal displacements are performed. The need for an additional shift was identified by several researchers [2, 5, 7]. Vertical shift factors are needed to account for change in material density, hygrological effects, and thermal expansion and contraction. In the current tests the specimens were loaded after heating, so no such adjustment was needed.

## 3. Materials and Experimental Set-Up

In the sample tests described here, Kevlar 49 yarns were used with an average breaking strength (ABL), obtained from 12 short-term tests, of 445 N. The cross-sectional area of the yarn was $0.1685\times10^{-6}$ m$^2$.

The tensile tests were carried out in a conventional testing machine, using round bar clamps that have also been used for long-term dead-weight testing of yarns. The testing set-up is shown in Figure 2. The oven is set up within the test machine, with the two clamps mounted on extension pieces so that the complete test specimen lies inside the oven. The load was applied by moving the cross-head of the machine at a specific rate (5 mm/mm); the cross-head movement and the load level were recorded.

It is essential to know accurately the strain of the specimen just after the initial loading in order to compare the creep curves at different temperatures. A small error here results in displacing the creep curves on the creep strain axis which then makes it impossible to obtain valid, smooth master curves by only making time shifts. A detailed analysis has been carried out using the raw data (cross head movement of the machine and load level) to determine the absolute zero of the stress–strain curve of a yarn specimen, which then enables the accurate strain immediately after loading to be determined [20]. The technique is described briefly here for completeness.

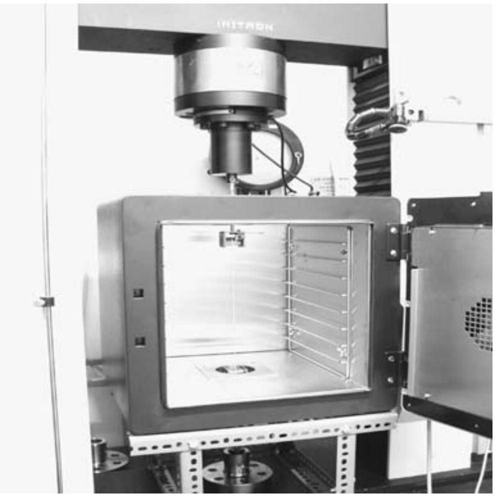


**Figure 2** Experimental set up for tensile and TTSP tests

An error is associated with the clamping system due to initial slack and lack of a well-defined point of load transfer around the jaws; this means the cross-head movement of the testing machine does not represent the accurate change of length of a yarn for a given load. It is also clear that the effective gauge length is not the same as the nominal gauge length (the centre to centre distance between clamps); this is known as the 'jaw effect.' The accurate effective length is therefore defined as the addition of the nominal gauge length and the jaw effect.

The secant modulus of the yarns varies with load level. It can be expressed in terms of measurable quantities and the unknown amount of slack and jaw effect:

$$E_j = \frac{stress}{strain} = \frac{p_{ij}\left(l_{gi} + l_{jawi}\right)}{A\left(e_{ij} - s_i\right)} \tag{3}$$

where:

$i$ is the specimen number
$j$ is the loading level
$E$ is the secant modulus
$A$ is the cross-sectional area of the yarn
$s$ is the initial slack
$e_{ij}$ is the cross-head movement
$l_{\mathrm{g}}$ is nominal gauge length
$l_{jaw}$ is the jaw effect

There will also be experimental variation in the amount of extension, which can be expressed by a term, $a_{ij}$ which varies from specimen to specimen. Re-arranging Equation (3):

$$e_{ij} = \frac{P_{ij}\left(l_{gi} + l_{jawi}\right)}{AE_j} + s_i + a_{ij} \tag{4}$$

There are three unknowns: The jaw effect and the amount of slack should be constant for any given test, while the secant modulus should be the same for all tests at the same load level. The error term has a different value for each reading. To determine the jaw effect, the amount of slack and the secant modulus, the error in Equation (4) was minimised using a built-in routine in Matlab.

It was found that the jaw effect does not vary with the loading level or with the nominal gauge length but is constant for a given machine and specimen combination. Once the jaw effect and initial slackness are determined, it is possible to determine the accurate stress vs. strain curve.

A similar procedure is carried out to determine the stress vs. strain curves for all the temperature levels used in the tests (Figure 3). This figure is used to determine the initial strains for a given stress level at different temperatures. For example, points at which the line AB crosses the stress–strain curves are the initial strain values at 70% ABL. The accurate values are listed in Table I.

## 3.1. Testing Procedure

A series of tests were carried out at different temperatures. A high load level was selected for the first set of tests (70% ABL) since the rupture point can be obtained with fewer temperature steps and there is a limited amount of conventionally

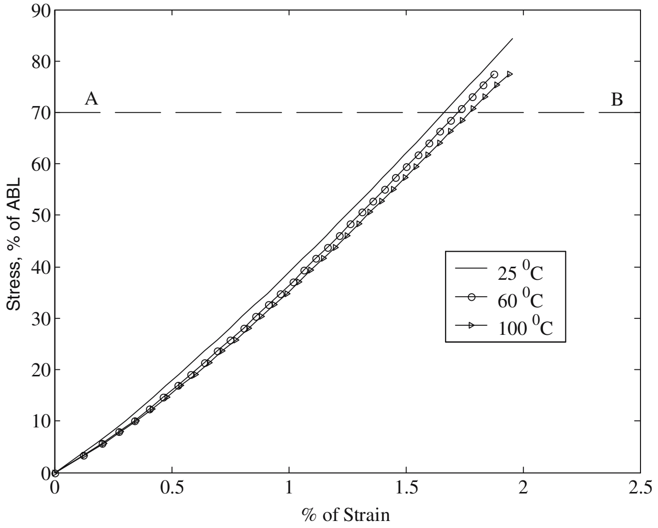


**Figure 3** Stress vs. strain curves at different temperatures

obtained data at this load level. It is then possible to check whether the TTSP could be successfully applied to aramid yarns. Later, a series of tests were carried out at a lower stress level (50% ABL) to obtain the long-term stress-rupture points. Each creep test was run for 16 h, until the yarn had reached its steady creep state. Tables II and III show the test sequences for 70% ABL and 50% ABL, respectively. In each test, the load was applied only after the temperature had reached the desired value. Thus, no vertical adjustment was needed and the curves were shifted only horizontally to obtain the master curve. Two separate specimens were tested at each temperature level to minimise the variability among the yarns and the results were averaged and referred to as a single test number. For example, tests at 70% ABL are denoted as TTSP70-01. '70' denotes the loading level and '01' denotes the test number. Specimens at the highest temperature level broke before the test ended and are marked as '*.'

**Table I** Initial creep values at 70% and 50% ABL.

| Temperature Level (°C) | Percent of Strain just after Loading | |
|---|---|---|
| | 50% ABL (222 N) | 70% ABL (311 N) |
| 25 | 1.2640 | 1.6721 |
| 40 | 1.2789 | 1.6925 |
| 60 | 1.2987 | 1.7196 |
| 80 | 1.3186 | 1.7467 |
| 100 | 1.3384 | 1.7738 |
| 120 | 1.3583 | – |
| 140 | 1.3782 | – |
| 160 | 1.3980 | – |

**Table II** Testing procedure for 70% ABL.

| Test No. | No. of Tests | Temperature (°C) | Time Duration (h) |
|---|---|---|---|
| TTSP70-01 | 2 | 25 | 16 |
| TTSP70-02 | 2 | 40 | 16 |
| TTSP70-03 | 2 | 60 | 16 |
| TTSP70-04 | 2 | 80 | 16 |
| TTSP70-05 | 2 | 100 | 8* |

## 4. Fitting a Curve to Creep Data

Various models have been used to describe creep data for aramid fibres, including linear variations, power low variations and hereditary type equations [21]. However, in this analysis a polynomial is used to describe the creep data of the master curve.

It was decided to select 25 °C as the reference temperature, since most of the long-term applications of this material would be at ambient temperature.

### 4.1. Shifting Procedure

To obtain a smooth master curve, the individual creep curve segments are shifted along the *log* (time) axis. The upper tail of the master curve represents the rupture time of the specimen if the last creep segment was tested to failure. Stress-rupture times are very sensitive to the shift factor values as the shifting is performed on the *log* (time) axis. A small change of the shifting values can lead to a noticeable change in the predicted stress-rupture lifetime.

### *4.1.1. Adjustment to Correct for Initial Strain*

Each specimen is tested once the temperature has been raised to a desired temperature level, which eliminates the need for a vertical shift. However, it is important to make an adjustment for initial strain before any shifting is carried out in the *log* time axis to obtain smooth master curves. The objective is to make a vertical adjustment so that the strain immediately after loading should be the same as the value from Table I. A similar procedure is carried out for all TTSP curves measured at different temperature levels and plotted in Figure 4 as horizontal lines.

**Table III** Testing procedure for 50% ABL.

| Test No. | No. of Tests | Temperature (°C) | Time Duration (h) |
|---|---|---|---|
| TTSP50-01 | 2 | 25 | 16 |
| TTSP50-02 | 2 | 40 | 16 |
| TTSP50-03 | 2 | 60 | 16 |
| TTSP50-04 | 2 | 80 | 16 |
| TTSP50-05 | 2 | 100 | 16 |
| TTSP50-06 | 2 | 120 | 16 |
| TTSP50-07 | 2 | 140 | 16 |
| TTSP50-08 | 2 | 160 | 10* |

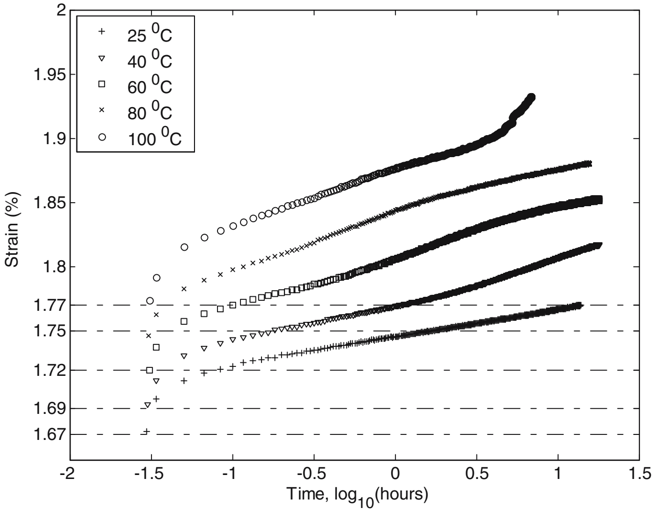


**Figure 4** Raw creep data at 70% ABL at different temperatures

Rapid changes of the creep gradient can be seen in the early stages, but after 1 h the curves seem to be straight, which indicates the steady state of the creep process. Jeon et al. [11] suggested that better matching of individual creep curves would be possible when the shifting was performed for the steady state regions of the creep curves, so the creep data up to 1 h were omitted when TTSP was applied.

Two specimens were tested at each temperature level and averaged to eliminate some of the yarn variability. All averaged curves were adjusted for the initial strain as described above.

### *4.1.2. TTSP Adjustment*

The yarns were loaded once the temperature had increased to the specified level, so vertical adjustments for thermal expansion/contraction were not needed. Smooth master curves were obtained only by horizontal translation of individual creep curves. This procedure was undertaken in stages. The first stage involved shifting the curves by eye until a smooth match was obtained; this was subsequently refined by a computer-based method in which fine adjustments were made to the shifts and the lack-of-fit minimized.

The master curve that results from this process is shown in Figure 5; it is not smooth at certain overlapping parts but it can be used as the starting point for iterative refinement techniques. Consider two creep curves at 40°C (OC) and 60°C ($A_1B_1$) (Figure 6). $A_1B_1$ is shifted along the logarithmic time axis by a shift factor, $log\ (a_t)$, to $A_2B_2$; $A_2C$ represents the overlap region of $A_2B_2$ and OC.

Hermida and Povolo [15] outlined a series of scaling properties that have to be satisfied to obtain the smooth master curves; they suggested that gradients of the two curves within the overlapping region should be similar to an acceptable accuracy. This procedure is difficult to implement when the curves are not straight within the overlapping region. In this study a different procedure was adopted.

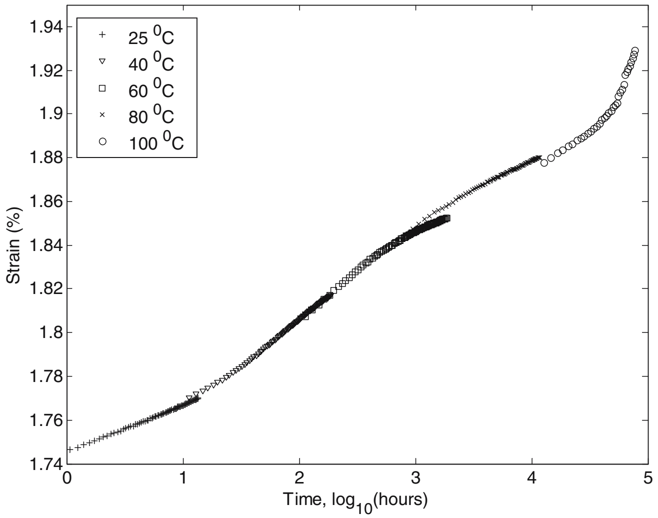


**Figure 5** Master curve at 70% ABL obtained graphically

An arbitrary point, $P_1$, on $A_1B_1$ is chosen at:

$$P_1 = [log\,(t_1), \varepsilon_1] \tag{5}$$

Point $P_1$ on $A_1B_1$ is accordingly moved to $P_2$ at:

$$P_2 = [(log\,(t_1) + log\,(a_t)), \varepsilon_1] \tag{6}$$

A slightly bigger region (2 h on each side of the overlap region, $A_2C$) was then considered. Data points on lines $A_2B_2$ and OC over that region were fitted to a third

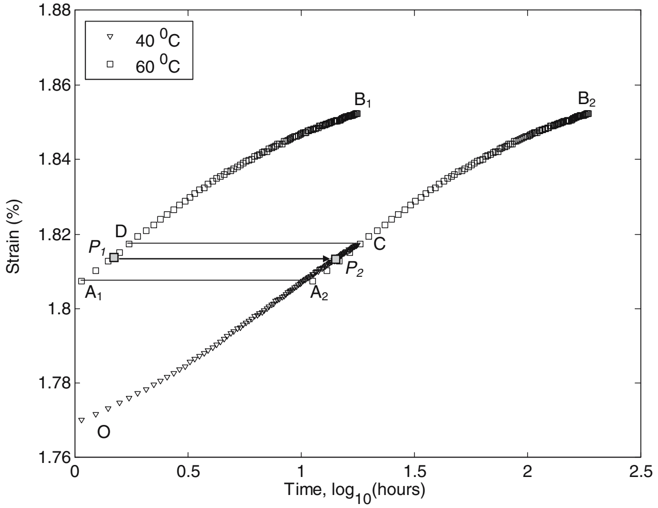


**Figure 6** Shifting procedure of two creep curves at 70% ABL

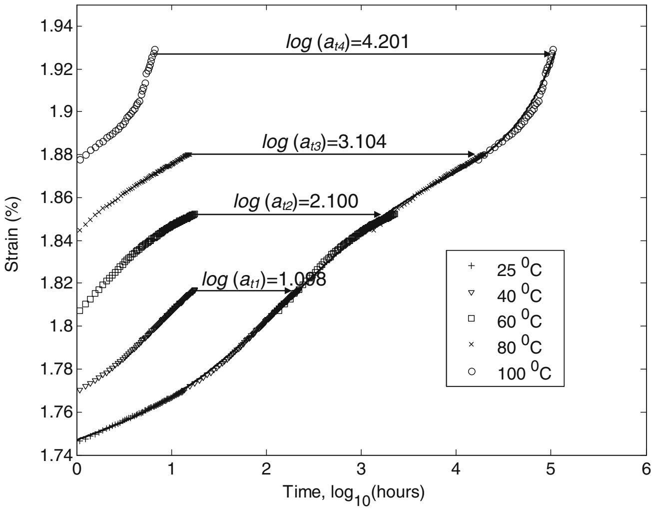


**Figure 7** Master curve at 70% ABL after numerical refinement

order polynomial, and the unknown value of the shift factor *log* ($a_t$) was altered numerically to minimize the sum of square error (SSE) between the data points and the third order polynomial. A similar procedure was adopted for subsequent pairs of curves. Figure 7 shows the master curve produced and the shift factors needed.

A higher order polynomial was then fitted to the master curve. The choice of the appropriate number of parameters for a model is open to debate and there is no firm rule. Figure 8 shows the Sum of the Squares of the Error vs. degree of polynomial for one set of data; as expected the error decreases as the order of polynomial increases. However, the change in error seems to be very small above a

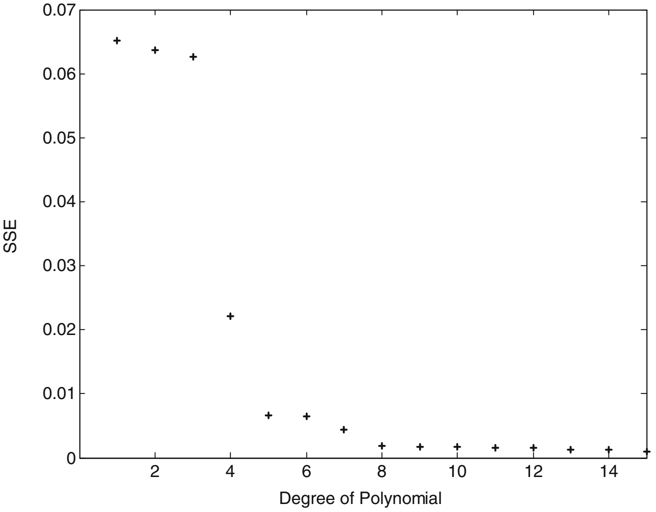


**Figure 8** Sum of square errors

sixth order polynomial and to avoid problems with over-fitting of data, this order of polynomial has been used subsequently.

An alternative was attempted in which all the curves were adjusted simultaneously. For every combination of shift factors data were fitted to a sixth order polynomial and the sum squares of error was calculated. Each of the shift factors was then altered numerically. The master curve that results from the optimum solution was not as smooth as that in Figure 7, despite the fact that the method could have reached the same result. This indicates problems with the numerical process since the discrepancy was much larger than the difference between the step sizes used in the search. The method was more time consuming since much iteration was involved and the order of the polynomial had to be assumed *a priori*. This method was not pursued further.

## 5. Discussion

A series of conventional creep tests have been performed to check the validity of this method which are reported elsewhere [20]. These tests have been carried out in a controlled temperature (25°C) and humidity (65% RH) environment, which were similar to the nominal condition of the master curve. Figure 9 shows the conventional creep curve plotted with the master curve for 70% ABL. The initial part of the conventional curve clearly follows the master curve, but it does not extend to the period between 1,000 and 10,000 h where the master curve shows double curvature; this is the subject of further study.

A similar procedure was used to analyse the 50% ABL data; Figure 10 shows that master curve plotted together with the conventional creep data. Once again the double curvature of the master curve is apparent between 1,000 and 10,000 h.

The double curvature of the master curve raises questions about whether a different mechanism is involved at different parts of the curve. It is expected that

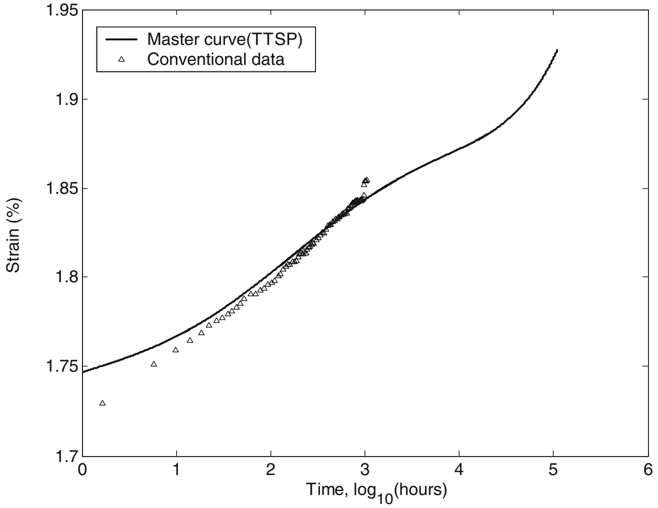


**Figure 9** Conventional creep curve plotted together with master curve at 70% ABL

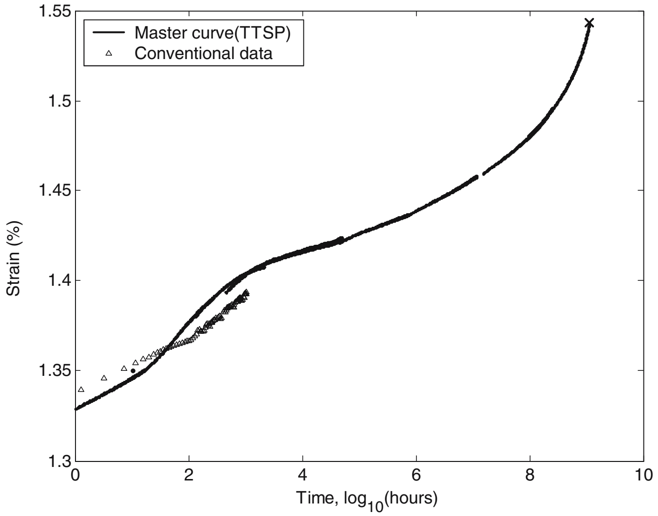


**Figure 10** Conventional creep curve plotted together with master curve at 50% ABL

the shift factors will be governed by Arrhenius' equation since aramids have no glass transition. This implies that a straight line should be obtained when *log* ($a_t$) is plotted against 1/*T*. Figure 11 shows that Arrhenius plots for both sets of tests give reasonable straight lines but with slightly different activation energies, as calculated using Equation (2) (116.3 kJ/mole for 70% ABL and 147.32 kJ/mol for 50% ABL). More tests are needed to determine whether the activation energy is stress dependent or if the results are simply experimental variation.

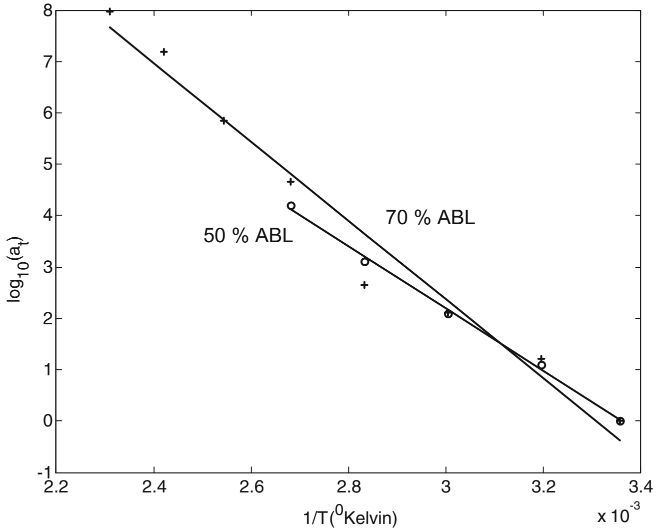


**Figure 11** Arrhenius curves plotted together

Tamuzs et al. [18] carried out a series of creep tests at different temperatures (20, 70, 100 and 150°C) on SVM fibres (another version of aramid fibres) and the master creep curve at 20°C was obtained using the TTSP technique. Using a similar approach to that reported here, the activation energy was found to be 103 kJ/mol.

Wu [22] performed a statistical analysis for the stress-rupture data of Kevlar-49 filaments which had been obtained by Wagner and Phoenix [23]. Wu described the scale parameter of the lifetime distribution of Kevlar filaments with the Arrhenius approach and the activation energies reported were 117 kJ/mol at 60% ABL, 109 kJ/mol at 65% ABL and 92.1 kJ/mol at 77% ABL. These values are of the same order as those obtained in the present analysis.

However, Ericksen [24] carried out creep tests on Kevlar-49 fibres at different temperatures. The creep curves were assumed to vary linearly on the logarithmic time scale. The creep rate parameter, $\alpha$ ($d\varepsilon/d\log(t)$), was determined for each curve and it was assumed that it would vary according to the Arrehenius equation. The plot of $log(\alpha)$ vs. $1/T$ was obtained and the activation energy was found to be 7 kJ/mol at 25% ABL which is significantly different from the values found here. In the present analysis the shift factors were assumed to vary according to the Arrhenius equation, whereas Ericksen assumed that the creep rate parameter varied according to the Arrhenius equation. A further investigation on activation energy is needed to resolve this matter.

## 6. Stress-Rupture Data Points

The final objective of the analysis is to obtain the stress-rupture data of the yarns. If the master curves produced are truly representative of the long-term creep behaviour, a fair prediction of the rupture times can be made. Since yarns were tested using TTSP until they failed, the upper limit of the master curves is a rupture point. The master curve at 70% ABL gave a rupture time around 12 years and at

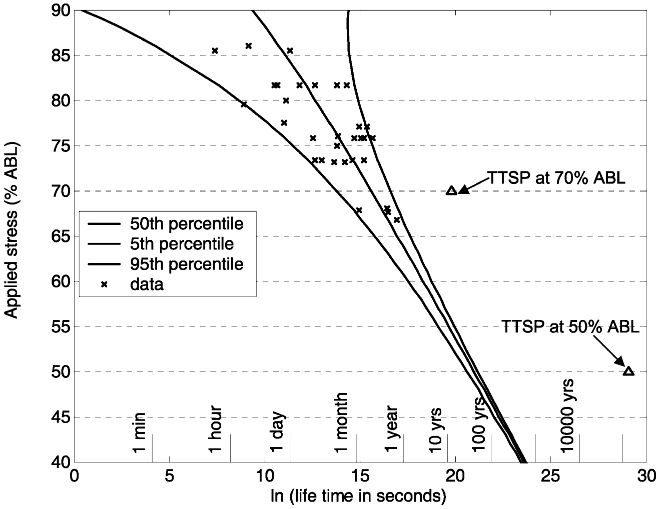


**Figure 12** Rupture times plotted together with the best model from [25]

50% ABL master curve gave around 130,000 years. For comparison, these rupture times are plotted together with the rupture model obtained by the authors from a statistical analysis of rope test data (Figure 12) [25]. It is apparent that the stress-rupture predictions do not match those predicted statistically but they are at load levels for which conventional stress-rupture testing is impractical because of the time scale involved.

## 7. Conclusion

It has been shown that TTSP can be used to produce master creep curves for aramid yarns from which stress-rupture data can be obtained. The necessary shift factors obey Arrhenius' Law, showing that creep is a thermally activated process. The stress-rupture lifetimes are much longer than predicted by extrapolation from short-term tests and clearly a more extensive testing programme needs to be undertaken that overlaps with the existing shorter-term data and also extends to other stress levels. It is also necessary to show that the same creep master curves can be obtained from different testing regimes.

The form of the creep master curve shows characteristics not before reported, in particular the reversed curvature, and this needs to be the subject of further investigation.

The authors have also undertaken a study of the Stepped Isothermal Method which is a development of the TTSP technique, in which a single yarn is tested at varying temperatures. This will be reported elsewhere [26].

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