# Accelerated and Real-Time Creep and Creep-Rupture Results for Aramid Fibers

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**ABSTRACT:** This article presents results from conventional creep tests (CCT) and two accelerated test methods (the stepped isothermal method (SIM) and the stepped isostress method (SSM)) to determine the creep and creep-rupture behavior of two different aramid fibers, Kevlar 49 and Technora. CCT are regarded as the true behavior of the yarn, but they are impractical for long-term use where failures are expected only after many years. All the tests were carried out on the same batches of yarns, and using the same clamping arrangements, so the tests should be directly comparable. For both materials, SIM testing gives good agreement with CCT and gave stress-rupture lifetimes that followed the same trend. However, there was significant variation for SSM testing, especially when testing Technora fibers. The results indicate that Kevlar has a creep strain

## **INTRODUCTION**

Uncertainty about creep rupture has held back the use of aramid fibers for structural engineering applications such as tendons in prestressed concrete, stay cables in bridges, and standing ropes in the marine industry. It has been proposed that a life span of about 120 years is possible if the tendons are subjected only to 50% of the short-term strength.<sup>1,2</sup> Similarly, Gerritse et al.<sup>3</sup> proposed limiting the initial stress in prestressing elements to 55% of the short-term strength. Even the manufacturers provide a large range of time-to-failure predictions when fibers are subjected to constant loads.<sup>4,5</sup>

Many creep-rupture models<sup>3,6–12</sup> have been proposed to predict the long-term creep-rupture behavior of aramid fibers. However, these models are based on data at high load levels (<70% breaking load), when creep failures can be expected in a short period of time. For lower stress levels extrapolation techniques have been suggested. The degree of extrapolation and the lack of test data introduce many uncertainties, and therefore for engineering

capacity that is almost independent of stress, whereas Technora shows a creep strain capacity that depends on stress. Its creep strain capacity is approximately two to three times that of Kevlar 49. The accelerated test methods give indirect estimates for the activation energy and the activation volume of the fibers. The activation energy for Technora is about 20% higher than that for Kevlar, meaning that it is less sensitive to the effects of increasing temperature. The activation volume for both materials was similar, and in both cases, stress dependent. © 2012 Wiley Periodicals, Inc. J Appl Polym Sci 125: 3856–3870, 2012

**Key words:** accelerated testing; creep; creep rupture; stepped isothermal method; stepped isostress method; Kevlar 49; Technora

design very large safety factors are applied. Therefore, engineers are currently forced to use low allowable stresses, resulting in significant economic disincentive. Less suitable materials are often used simply because there is more confidence about their long-term properties.

As an alternative, accelerated creep-rupture testing can be carried out at low stress levels, in such a way that the long-term creep and creep-rupture properties can be determined in reasonable times without having to extrapolate data. Creep can be accelerated in various ways. A materials' resistance to creep can be overcome by supplying energy; this is usually done by means of heat as in time temperature superposition testing (TTSP),<sup>13,14</sup> or the stepped isothermal method (SIM).<sup>15–17</sup> However, it can also be accelerated by stress, as in time stress superposition testing (TSSP),<sup>18–20</sup> or the stepped isostress method (SSM).<sup>21–23</sup>

A program of testing has been undertaken on two different aramid fibers (Kevlar 49 and Technora) using several different techniques, at a number of different stress levels, with a view to establishing with reasonably certainty their creep and creep-rupture behavior, and also to consider possible differences between them.

Conventional creep tests (CCT), with loads applied by dead weights and durations up to one year, can

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provide data on creep rates, and creep-rupture data at high loads. They can usefully validate accelerated testing, but inevitably have limitations on their duration.

The two accelerated methods used are the SIM, in which creep rates are accelerated by increasing the temperature, and the SSM in which creep rates are accelerated by increasing the stress. Detailed descriptions of both methods are given elsewhere,<sup>17,23</sup> although a very brief outline is given below. The purpose of this article is to present the results of three different testing regimes on the two different fibers, and to draw conclusions both about their long-term properties and about methods for accelerating creep.

Yarns from the same batches were used in all tests; they were handled by the same personnel and were gripped in the same clamps. The results of the tests should thus be directly comparable with each other, and it is believed that this is the first time that such a consistent comparative study has been carried out.

## DESCRIPTION OF THE TWO DIFFERENT ACCELERATING TESTING METHODS

The SIM can be considered as a special case of the TTSP. SIM testing involves loading a single specimen, under constant load, with the temperature increased in a series of steps to accelerate the creep. A full description of the method is given elsewhere<sup>16,17</sup> and will not be repeated here. Careful choice of the temperature step and step duration allow the test to be completed in about 24 h. At each temperature step a creep curve is obtained; these are then adjusted to compensate for the different temperature levels and a creep master curve at a reference temperature is produced. A creep-rupture point can then be determined as the very last point of each creep master curve. Four adjustments are required to produce the single master curve and are described briefly below.

- 1. The *initial vertical adjustment* allows for slight variations in the test setup between yarns.
- 2. The *vertical shifting* allows for the thermal contraction and creep that occurs during the temperature change.
- 3. The *rescaling* accounts for the thermal history that has occurred before each portion of the creep test.
- 4. The *horizontal shift* combines the individual creep curves to give a single master curve.

The fundamental premise of SIM testing is that viscoelastic processes are accelerated at elevated temperatures in a predictable manner. Both materials have high glass transition temperature, e.g. Kevlar 49 at 375°C and Technora at 318°C,<sup>24</sup> so the Williams–Landel–Ferry equation is not applicable<sup>24,25</sup> at the testing temperatures. Instead, Boltzmann's superposition principle and the Arrhenius equation provide justification for rescaling and horizontal shifting of the strain data obtained at each isothermal exposure to produce a creep master curve corresponding to the reference temperature.

It is possible to use different temperature step sequences to accelerate creep, and if the method is valid, the master curves produced by the SIM technique using these different time and temperature steps should overlap and must give consistent rupture times with acceptable accuracy.

The use of a single specimen minimizes concerns about specimen variability and handling effects; TTSP needs more specimens and more handling. SIM can be automated and takes less time than TTSP, so offers several advantages.

In SSM testing, a similar approach is adopted but the acceleration is obtained by increasing the stress in steps while keeping the temperature constant. Additional stress provides energy to the system in an analog of the effect of heat in SIM. As with SIM four different adjustments are required to produce the final master curve at a reference stress level at a constant temperature. More details about the method can be found elsewhere.<sup>21,23</sup>

In this method, the Eyring equation relates the reaction rate to stress; it follows from the transition state theory, and contrary to the empirical Arrhenius equation, the model is theoretical and based on statistical thermodynamics. Eyring and his colleagues assumed that the deformation of a polymer was an energetically activated rate process involving the motion of segments of chain molecules over potential barriers. The form of Eyring Equation resembles the Arrhenius equation, but the equivalent to the activation energy is a term with the units of volume, which results in an activation volume ( $V^*$ ). This can be envisaged as the volume of material that takes part in the process.

## MATERIALS AND EXPERIMENTAL SET-UP

Kevlar 49 and Technora yarns, available in reel forms, were used for all tests. In both cases, the tested yarns had been supplied by the manufacturers as flat yarns and had been twisted to 80 turns/m and rewound by a commercial supplier. They are typical of the yarns that would be used in the manufacture of ropes or larger components.

Kevlar 49 is an aramid fiber made by Du Pont<sup>4</sup> from a single monomer unit. Its chemical structure consists of aromatic polyamides containing chains of aromatic rings, linked together with -CO- and

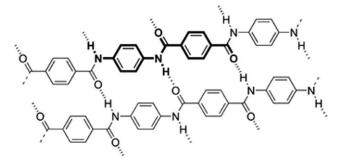


Figure 1 Chemical structure of Kevlar 49.

-NH- end groups, weakly linked with hydrogen bonds between adjacent long chain molecules. Figure 1 illustrates the molecular structure of Kevlar 49. The bold lines denote the repeating unit in a molecule and the dashed lines denote the hydrogen bonds. Technora is a copolymer, made by Teijin<sup>5</sup>; its chemical structure consists of two different monomer units, which are placed in a completely random sequence in the polymer chain. One of the monomer units is the same as Kevlar, while the other contains an extra benzene ring. Figure 2 illustrates the molecular structure of Technora. The symbols "*m*" and "*n*" denotes mol %, and always (*m* + *n*) = 100%.

The cross-sectional area (*A*) of the yarns, after removing moisture, is shown in Table I. The breaking loads were determined from 20 short-term tensile tests. From the dispersion of results, a mean value  $\mu_P$  (the average value) and a standard deviation  $\sigma_P$  (the measure of variability) were determined. The measured values shown in Table I are in agreement with the values given by the two manufacturers.<sup>4,5</sup> All subsequent stress levels will be expressed as a percentage of this ABL (average breaking load). Before testing the yarn reels were kept at constant room temperature (25°C) and humidity (50% relative humidity), placed in a black polythene bag inside a box to protect them from ultraviolet light.

Before creep testing, an extensive programme of testing was carried out using mechanical strain gauges to determine the jaw effect and to obtain accurate stress versus strain curves at different temperature levels (Fig. 3). These curves were used to determine the initial strains for a given stress level at different temperatures.

All yarn tests require a clamping device at each end, and because it was envisaged that a large num-

TABLE I Mean Value  $\mu_P$  and Standard Deviation  $\sigma_P$  of the Breaking Load

		0		
Material	Diameter of yarn (mm)	Cross-sectional area (mm <sup>2</sup> )	Mean value µ <sub>P</sub> (N)	Standard deviation $\sigma_P$ (N)
Kevlar 49 Technora	0.472 0.396	0.175 0.123	444.60 349.01	8.22 6.75

ber of long-duration tests were to be carried out, it was not feasible to use the standard horn grips used for short-term yarn testing. The clamps were also used for stress–relaxation tests, and retained– strength tests (neither of which are described here), but this requirement meant that it had to be possible to move the yarns from a dead-weight creep rig to a tension testing machine with as little disturbance to the yarns as possible.

The clamps thus had to be relatively cheap to fabricate, not to require an external power source, and also to ensure that failure should take place within the testing length and not within the clamp. The yarn is wrapped around a spindle and then fixed by a grip (Fig. 4). The main advantage of this arrangement is that the full load is not transferred to the grip, but it is spread over the perimeter of the spindle; this results in failure within the testing length.

The error associated with the clamping device due to initial slack and lack of a well-defined point of load transfer around the jaws, means that the cross-head movement of the testing machine does not represent the accurate change of length of a yarn for a given load. This "jaw effect" means that the effective gauge length is not the same as the nominal gauge length. Three different ways to determine the initial slack *s* and the jaw effect  $l_{jaw}$  values are available and described elsewhere.<sup>22,26</sup>

Conventional creep tests were carried out in a room where the temperature and humidity levels could be controlled; they were set at 25°C and 50% relative humidity (RH). Eighteen test stations were used; the top clamp was kept stationary and the lower clamp was free to move vertically between two metal rails, as shown in Figure 5. A constant load was applied by hanging dead-weights through a lever arm at the bottom clamp. Mechanical strain gauges of circular form were used to measure the elongation of the yarns, and an uninterruptible

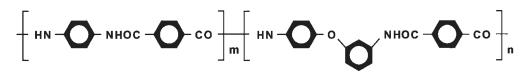


Figure 2 Chemical structure of Technora.

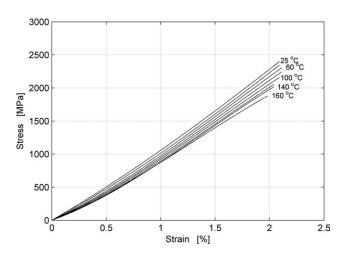


Figure 3 Stress versus strain curves at different temperatures.

power supply (UPS) was provided for the data logger and the computer.

Tensile, SIM, and SSM tests were conducted using the same experimental set-up in an Instron tension testing machine. Two clamps, as described earlier, were fixed to the machine by means of two Invar bars that projected through holes cut in the top and bottom of a Thermocenter-Salvis Lab oven, as shown in Figure 6. The holes in the oven were sealed by PTFE (polytetrafluoroethylene) blocks, so that the two clamps and the yarn were fully inside the oven. The Instron could be controlled in either load or displacement mode as necessary, and the temperature in the oven could be programmed The cross head movement, load cell reading and the temperature (as measured by thermo couples) were recorded by a data logger, at suitable intervals.



Figure 4 Clamping device used in this study.

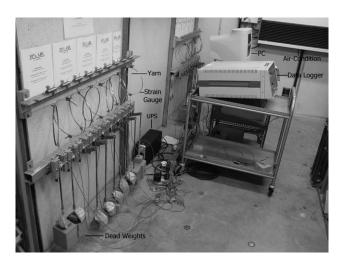


Figure 5 Experimental set-up for CCT tests.

## **TESTING PROCEDURE**

SIM tests and SSM tests for Kevlar 49 and Technora yarns at different load levels (50, 55, 60, 65, 70, 75, and 80% ABL) were carried out. Eight tests using SIM and four tests using SSM were conducted at each load level. Each step was chosen to last 5 h, except the final one which lasted until failure of the specimen. The steady state of creep is reached after about 1 h of testing, and therefore 5 h of testing at each step is satisfactory. Experiments were not conducted below 50% ABL, since Kevlar 49 and Technora show a nonlinear viscoelastic behavior below 40% ABL<sup>26,27</sup> so the superposition principle would



Figure 6 Experimental set-up for tensile and SIM tests. Journal of Applied Polymer Science DOI 10.1002/app

Test label	Temperature level (°C) 25	Time (h)		Rescaling factor <i>r</i> (h)		Shift factor log $(\alpha_{\sigma})$	
SIM-75-01-01; SIM-75-01-02		5	5	0.00	0.00	0.00	0.00
	40	5	5	4.60	4.58	1.00	1.00
	60	5	5	9.62	9.62	2.07	2.07
	80	3	2.6	14.50	14.50	3.14	3.16
SIM-75-02-01; SIM-75-02-02	25	5	5	0.00	0.00	0.00	0.00
	40	5	5	4.40	4.67	0.86	1.03
	60	25.1	17.1	9.70	9.98	2.06	2.48
SIM-75-03-01; SIM-75-03-02	25	5	5	0.00	0.00	0.00	0.00
	40	5	5	4.42	4.70	0.95	1.18
	60	5	5	9.64	9.90	2.03	2.51
	70	4.6	2.1	14.23	14.44	2.80	3.31
SIM-75-04-01; SIM-75-04-02	25	5	5	0.00	0.00	0.00	0.00
	40	5	5	4.42	4.57	0.95	1.04
	50	5	5	9.72	9.87	2.15	2.24
	70	1.7	1.8	14.63	14.49	3.20	3.20

TABLE IISIM Tests and Shifting Factors at 75% ABL for Kevlar 49

not have been applicable. Typical SIM and SSM test results at 75% ABL are shown for Kevlar 49 in Tables II and III, respectively. Each test is identified by a test label, e.g. SIM-75-02-01 or SIMT-75-02-01, where "SIM" denotes SIM tests for Kevlar 49 and "SIMT" denoted SIM tests for Technora, "75" denotes the starting reference load level, "02" denotes the test number, and "01" denotes the repetition of the test. The two repetitions of each test are shown in two adjacent columns, the second italicized.

Conventional creep tests (CCT) were also carried out, at different stress levels (10, 20, 30, 40, 50, 55, 60, 65, 70% of ABL), under constant temperature and humidity. Two specimens were tested at each load level. None of these was expected to fail by stress-rupture; the purpose was to obtain creep strain versus time curves for comparison with the master curves produced by SIM and SSM.<sup>23</sup>

A second set of creep tests were carried out at much higher loads until rupture of the specimens. The main purpose of these experiments was to measure the time to failure at different load levels and not to determine their creep curves so no strains were recorded and only a clock device was attached to the bottom clamp to record the time at which the specimen failed. To have failure of the specimens within a reasonable time period (maximum 6 months), tests were carried out at 77.5, 80, 82.5, 85, 87.5, 90, 92.5, and 95% ABL. At each load level four test repetitions were conducted.

## **CREEP TEST RESULTS**

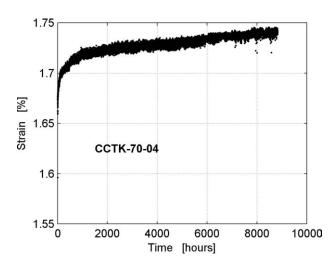
## **Conventional creep tests**

The strain versus time curves for each of the conventional creep tests (CCT) were plotted; a typical curve for test CCTK-70-04 is shown in Figure 7. The observed spread is due to the inherent noise of the measuring equipment (accuracy of strain gauges  $\pm 0.0003$ ). For calculation purposes, to diminish this noise, the value of strain at any time is the one corresponding to the center of the spread (mean value).

Some creep tests were discarded because slip events were observed. These were caused by slip between the mechanical strain gauge and the yarn or due to a sudden change of the testing room temperature (when visiting the room), which caused small jumps in the creep curves.

SSM Tests and Shifting Factors at 75% ABL for Kevlar 49							
Test label	Load sequence (% ABL) 75	Time (h)		Rescaling factor r (h)		Shift factor log $(\alpha_{\sigma})$	
SSM-75-01-01; SSM-75-01-02		5	5	0.00	0.00	0.00	0.00
	77.5	5	5	3.52	3.41	0.69	0.65
	80	5	5	8.63	8.68	1.53	1.48
	82.5	3.9	2.0	13.84	14.30	2.40	2.49
SSM-75-02-01; SSM-75-02-02	75	5	5	0.00	0.00	0.00	0.00
	77.5	5	5	3.50	3.52	0.71	0.79
	80	8.2	8.3	8.51	8.64	1.45	1.67

TABLE IIISSM Tests and Shifting Factors at 75% ABL for Kevlar 49



**Figure 7** A typical strain versus time curve.

The temperature and humidity variation with time in the testing room was monitored; it was verified that they were kept practically constant throughout the testing period. The shape of all strain versus time curves is similar, showing a primary creep region that levels out and a secondary creep region that starts at about 1000 h and is almost linear with a slope. No tertiary region is present since all creep tests were stopped at either 100 or 365 days, and the tertiary region at 70% ABL is expected to start at about 5 years.<sup>17</sup>

All creep curves (strain vs.  $\log_{10}(t)$ ) for Kevlar 49 (set 1–4) are plotted in Figure 8. It is observed that using a logarithmic time scale the creep curves are practically straight, which confirms the conclusions of other researchers.<sup>2,28–30</sup> The creep curves obtained in this study will be used to validate the two accelerated techniques.

#### SIM tests

Detailed results are presented below for one test (at 75% ABL on Kevlar 49) to show how the method works, followed by a summary of all the results for Kevlar 49 and Technora.

The test readings monitored throughout each SIM tests areas follows: specimen elongation ( $\Delta l$ ) versus

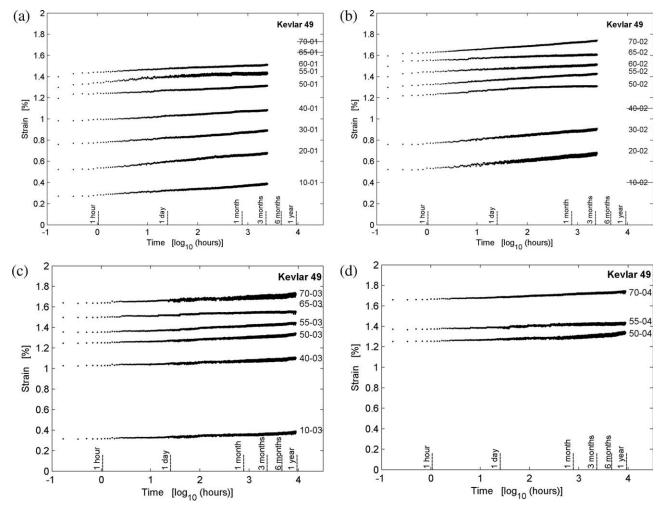


Figure 8 Creep curves for Kevlar 49 (Sets 1–4).

**Figure 9** Elongation versus time curve.

10

Time [hours]

40 °C

time (t) (Fig. 9), applied load (P) versus time, and temperature (T) versus time (Fig. 10).

The elongation versus time curve, for a given constant applied load, was then converted to a strain versus time curve. This was done by using the following relationships:

$$\sigma = P/A \tag{1}$$

15

80 °C

60 °C

rupture

20

$$\varepsilon = (\Delta l - s)/l_{\text{eff}}$$
 where  $l_{\text{eff}} = l_{\text{nom}} + l_{\text{jaw}}$  (2)

where  $\sigma$  is stress in MPa, *P* is the applied tensile load in *N*, *A* is the cross-sectional area of the yarn in mm (values are given in Table I),  $\varepsilon$  is the strain in %,  $\Delta l$  is the elongation in mm, *s* is the initial slack in mm,  $l_{\text{eff}}$  is the effective length in mm,  $l_{\text{nom}}$  is the nominal gauge length in mm, and  $l_{\text{jaw}}$  is the jaw effect in mm.

The initial slack *s* and the jaw effect  $l_{jaw}$  have been determined by the method described elsewhere<sup>26</sup> and were found to be 0.42 mm and 140.0 mm

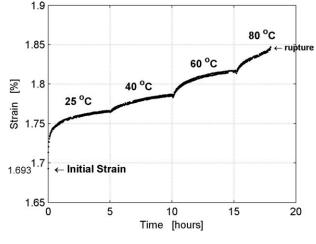


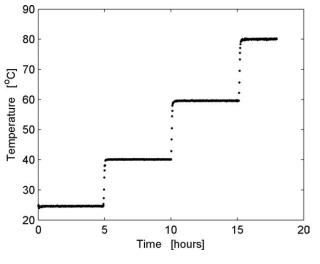
Figure 11 Strain versus time curve

respectively for Kevlar 49, and 0.20 mm and 128.0 mm respectively for Technora.

The resulting strain versus time curve at an applied stress level is adjusted up and down to give an initial strain at zero time which is the same as that from the stress versus strain curves of Figure 3 obtained by the mechanical strain gauge; the result is given in Figure 11.

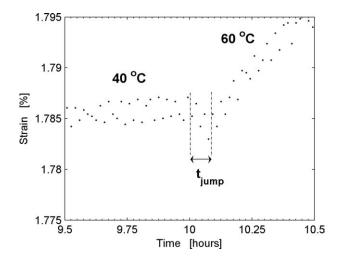
A local drop of strain is observed at each temperature change caused by the negative coefficient of axial thermal expansion of aramids, as seen in Figure 12, which shows an enlarged portion of the curve around the second temperature jump (40– 60°C). The as-measured strain versus time curve given in Figure 12 is adjusted vertically to remove this effect: both curves are shown in Figure 13.

Each part of the curve of Figure 13, corresponding to a different temperature level, has to be rescaled by horizontal shifting to take into account the thermal history of the specimen and to form a creep master curve. To obtain a smooth master curve, a





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**Figure 12** Strain versus time curve at the temperature jump 40–60°C.

9.6

9.4

9.2

9

8.8

8.6

0

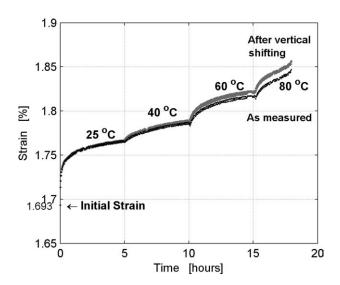
8.732

25 °C

← Initial Elongation

5

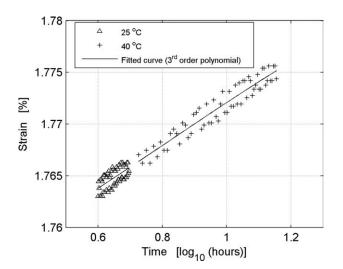
Elongation [mm]



**Figure 13** Strain versus time curve as measured and after vertical shifting.

third-order polynomial was fitted to the curves just before and after each temperature jump (Fig. 14); the result is the final smooth master curve (Fig. 15). The very last point of this curve corresponds to the creep-rupture point of the specimen. Details of all applied adjustments mentioned earlier are given elsewhere.<sup>17,27</sup>

The aforementioned procedure is followed at all load levels: 50, 55, 60, 65, 70, 75, and 80% ABL. All SIM master curves from all tests are shown together in Figures 16 and 17 for Kevlar 49 and Technora, respectively. Examining the SIM master curves (gray lines) at each load level, which resulted from eight tests with different temperature histories, shows that they match both in form and position with some experimental scatter. Less good agreement is observed at lower load levels for Technora.



**Figure 14** Smooth match of strain versus time curve at the temperature jump  $25-40^{\circ}$ C.

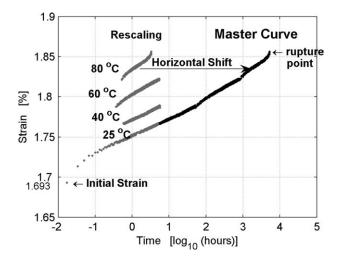


Figure 15 Individual creep curve after rescaling and master curve after horizontal shifting.

By plotting the shifting factors, obtained from the horizontal shifting, with the inverse of temperature (*K*), a linear variation is observed with a small experimental scatter (Fig. 18). This indicates that creep can be regarded as an Arrhenius process. Figure 19 shows the combined results for all load levels; the overlapping curves imply that the activation energy *E* of the reaction is constant and therefore the same creep mechanism is operative at each temperature level and at each load level. For Kevlar and Technora, the mean activation energies were found to be 119 and 133.7 kJ mol<sup>-1</sup>, respectively.

## SSM tests

As with SIM, throughout each SSM test three readings are monitored and the following plots are

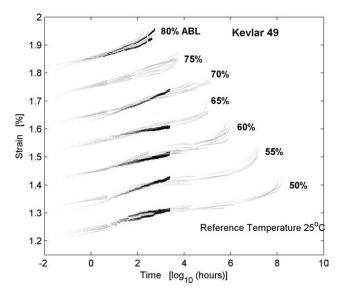
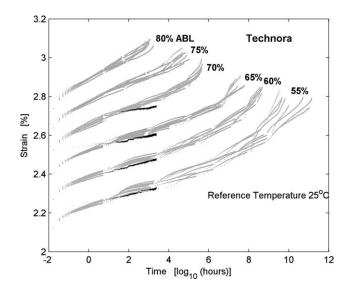


Figure 16 SIM master curves (gray) and conv. creep curves (black) for Kevlar 49 (No CCT test at 75% ABL).

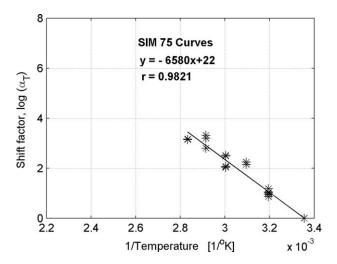


**Figure 17** SIM master curves (gray) and conv. creep curves (black) for Technora (No CCT tests at 75% and 80% ABL).

produced: specimen elongation versus time (Fig. 20), applied load versus time (Fig. 21), and temperature versus time.

The elongation versus time curve is then converted to a creep strain versus time curve and the resulting curve is further adjusted to give the accurate initial strain at zero time, as with SIM. The resulting strain versus time curve of SSM-75-01-01 after the initial vertical adjustment is given in Figure 22.

An increase of strain is observed in each stress jump in Figure 22 caused by the elastic extension. This is shown more clearly in Figure 23 where an enlarged portion of the curve is given around the second stress jump (77.5–80% ABL). The as-measured strain versus time curve of SSM-75-01-01 given in Figure 22 is adjusted vertically, to remove the elastic portion.



**Figure 18** Arrhenius plot of SIM curves at 75% ABL for Kevlar 49.

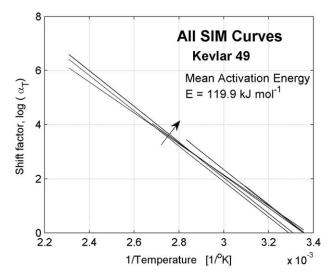


Figure 19 Arrhenius plot of SIM master curves for Kevlar 49.

The next step is to apply the rescaling and horizontal shifting procedures to each part of the curve of Figure 24 corresponding to a different stress level. At each stress jump a third-order polynomial was fitted to the parts just before and after the jump, so that a smooth match between the two parts was achieved. The rescaling factor and shift factor that resulted for the load sequence of SSM-75-01-01 are given in Table III. Using the rescaling factors the individual creep curves are produced, each of which would have been obtained from TSSP tests. Then, using the horizontal shifting factors the final master curve is produced (Fig. 25). The very last point of this curve corresponds to the creep-rupture point of the specimen.

The aforementioned procedure is followed at all starting reference loads: 50, 55, 60, 65, 70, and 75% ABL. All SSM master curves (black lines) from all tests for Kevlar 49 and Technora are shown in Figures 26 and 27, respectively.

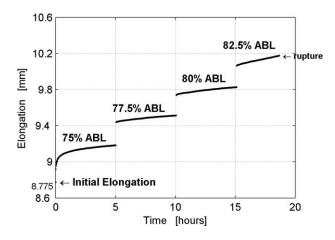


Figure 20 Elongation versus time curve.

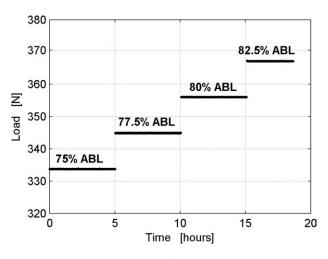


Figure 21 Load versus time.

From the plots of shift factor (log ( $\alpha_{\sigma}$ )) versus accelerating stress (% ABL), a linear variation is observed, with a small experimental scatter, and which shows that the same creep mechanism is operative for each load sequence. A typical plot for an initial reference stress of 75% ABL is shown in Figure 28. This validates the use of the SSM procedure for Kevlar 49 and Technora, i.e. the use of the superposition theory in adding creep curves with different load sequences to the creep master curve.

The slope of the lines is equal to  $V^*/(2.30kT)$ , where  $V^*$  is the activation volume and k is Boltzmann's constant (=  $1.38 \times 10^{-23}$  J K<sup>-1</sup>) and T is the temperature. The resulting values of  $V^*$  at different starting reference loads, given in Tables IV and V, show an increase with the increase of the applied load. The variation of activation volume with stress for both fibers is shown in Figure 29. This implies a relationship between activation volume and stress, which might be expected since activation volume is actually a stress coefficient. According to transition state theory, activation volume  $V^*$  is interpreted as the difference between the partial molar volumes of

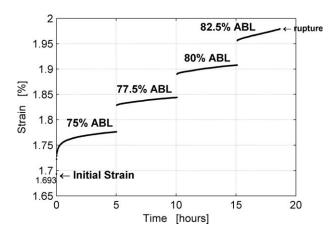


Figure 22 Strain versus time curve.

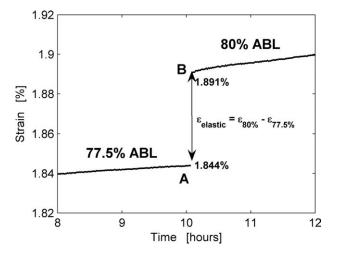


Figure 23 Strain versus time curve at the stress jump 77.5–80% ABL.

the transition state and the sums of the partial volumes of the reactants at the same temperature and pressure. This implies that for higher stress levels, higher activation volumes are expected. However, as discussed elsewhere,<sup>23</sup> there is a conundrum because if  $V^*$  varies with stress, plots such as Figure 29 should not give straight lines. The experiments do show, however, that both Kevlar 49 and Technora have similar activation volumes that show similar stress dependency.

#### DISCUSSION OF TEST RESULTS

#### Comparison of SIM and CCT

Conventional creep tests have been carried out at 10–80% ABL, as explained in the previous sections. These tests can be used to validate the two accelerated testing methods, by comparing the conventional

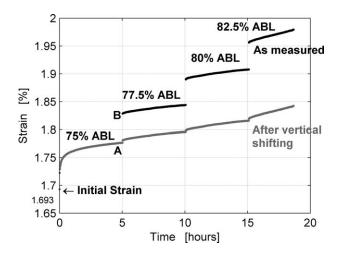


Figure 24 Strain versus time curve as measured and after vertical shifting.

8 1.8

Strain

1.9

1.85

1.75

1.7

1.65

2

1.9

1.8

1.7

1.4

1.2

1.1<sup>L</sup> -2

0

Strain [%] 9.1 [%] -2

**Figure 25** Individual creep curves after rescaling and master curve after horizontal shifting.

1

2

Time [log<sub>10</sub>(hours)]

3

Kevlar 49

Temperature 25°C

8

10

Rescaling

82.5%

80%

77.5%

Initial Strain

0

-1

75% ABL

**Master Curve** 

Horizontal Shift

← rupture

point

4

5

creep curves with the corresponding SIM master curves. All CCT curves (black lines) are plotted with SIM master curves (gray lines) at the reference temperature (Figs. 16 and 17 for Kevlar 49 and Technora, respectively). Excellent agreement is observed for Kevlar 49 up to the 1 year duration of the testing. The agreement for Technora is less good, with clear differences between the creep rates at 65% and 70% of the ABL.

#### Comparison between SIM and SSM testing

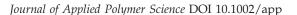
Comparisons between SIM and SSM testing are made in Figures 26 and 27 for the two materials. For Kevlar 49 there is very good agreement between the two sets of results for stresses above 60%, but less good agreement at 50% and 55%. There is less good agreement for Technora fibers, especially at low load

Figure 26 All SIM (gray) and SSM (black) master curves for Kevlar 49.

4

Time [log<sub>10</sub> (hours)]

6



2

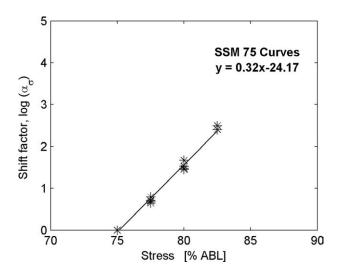


Figure 28 Eyring plot of SSM curves at starting reference

load 75% ABL for Kevlar 49.

Technora 3 60% 2.8 [%] strain 2.6 2.4 Temperature 25°C 2.2 -2 2 10 12 6 8 4 time [log10 (hours)]

3.2

Figure 27 All SIM (gray) and SSM (black) master curves for Technora.

levels. Technora fibers exhibit higher activation energy than Kevlar 49, which implies that high temperatures are needed to get failures within 25 h, especially at low stress levels.

A typical SIM master curve (SIM-50-01-01) plotted into a linear time scale is given in Figure 30. It can be observed that the shape of this curve is in general agreement with those found from conventional creep tests on parallel-lay aramid ropes, exhibiting primary, secondary, and tertiary creep phases. The initial very fast creep rate reduces during the primary phase and is constant during the secondary phase. During the tertiary phase the rate again increases leading to failure. All SIM curves in this study show these three distinct creep regions.

TABLE IV Activation Volume at Different Reference Loads for Kevlar 49					
	Poly	ynomial			
Reference load (% ABL)	Slope a	Constant b	Activation volume V* (nm <sup>3</sup> )		
50	0.22	-10.91	0.082		
55	0.25	-13.27	0.091		
60	0.24	-14.59	0.091		
65	0.27	-17.61	0.100		
70	0.32	-22.45	0.120		
75	0.32	-24.17	0.120		

## Creep strain capacity

Chambers<sup>9</sup> and Guimaraes<sup>2</sup> suggested that Kevlar had a limiting creep capacity. According to Guimaraes' study, the creep capacity  $\varepsilon_{cp}$  is defined as:

$$\varepsilon_{\rm cp} = \varepsilon_2 - \varepsilon_0$$
 (3)

where  $\varepsilon_0$  is the strain just after the initial strain. Chambers considered  $\varepsilon_0$  to be the strain 1 min after the initial strain.

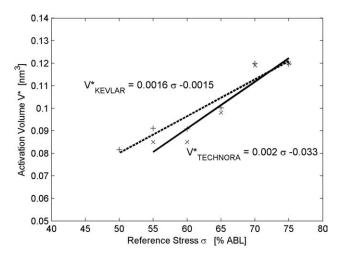
 $\varepsilon_2$  is the strain at the beginning of the tertiary creep region. Chambers defined  $\varepsilon_2$  as the intersection point of the extension of the secondary line with the rupture time.

Both researchers suggested that aramid fibers might fail when a certain amount of creep capacity had been consumed, and suggested that this effect could be used to provide experimental predictions of the creep-rupture lifetime if the creep rates could be established.

Following Chambers' approach, the creep strain capacity values were determined from all SIM and SSM master curves at all load levels. The results indicate that creep strain capacity decreases as the applied stress increases. The fitted lines to the experimental results are shown in Figure 31 for both methods and materials. Good agreement is observed for the SIM and SSM fitted lines for Kevlar 49, while for Technora there is a significant difference at high stress levels and the creep capacity is much higher.

TABLE V Activation Volume at Different Reference Loads for Technora

lechnora				
	Poly	ynomial		
Reference load (% ABL)	Slope a	Constant b	Activation volume $V$ (nm <sup>3</sup> )	
55	0.26	-14.10	0.085	
60	0.26	-15.33	0.085	
65	0.30	-19.01	0.098	
70	0.36	-25.02	0.119	
75	0.36	-27.08	0.120	



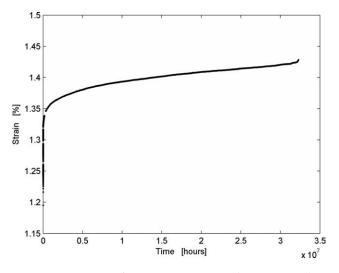
**Figure 29** Variation of activation volume with reference stress for Kevlar and Technora.

This difference can be explained from the slightly different chemical and physical composition of the two materials, which is further discussed elsewhere.<sup>22</sup>

#### **Creep-rupture times**

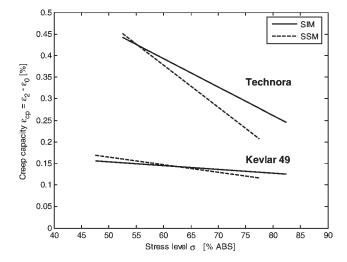
All the accelerated creep tests and some of the conventional creep tests were carried out until failure of the specimen. The last point on the master curve corresponds to the rupture time of the specimen at the reference load and constant temperature 25°C. The rupture times from all creep tests at various load levels are summarized in Table VI for Kevlar 49 and Technora yarns.

The creep-rupture predictions for Kevlar 49 and Technora are shown in Figures 32 and 33, respectively. The results from the CCT, SIM, and SSM tests



**Figure 30** A typical SIM master curve (SIM-50-01-01) in linear time scale.

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**Figure 31** Creep strain capacity values for Kevlar 49 and Technora from SIM and SSM master curves.

are shown separately, together with the best fit lines for both the SIM and SSM tests. It is significant that the SIM test results align with the CCT results for both materials, whereas the SSM results lie below the SIM results, indicating that stress-rupture would be predicted to occur more rapidly.

The alignment of the SIM and CCT results in these two figures, and the uniformity of the predictions for the activation energy discussed earlier, lead to the conclusion that the SIM results are a more reliable predictor of the stress-rupture lifetimes than the SSM tests. This is probably associated with the variation with stress of the activation volume of the two aramids, as shown in Figure 29.

There does appear to be a clear difference between the two materials; the difference between the SIM and the SSM predictions for Technora is much larger than that for Kevlar 49. No explanation is suggested for this observation, but it is clearly something of interest for future study.

It is possible to determine a best-fit line to the data from all three sets of test for each material. This will be conservative if the SSM tests should be ignored, since it will have the effect of giving a lower value for the predicted lifetime. The fitted lines are given by:

$$\log (t_r) = 15.86 - 0.17 \text{ P}$$
 for Kevlar 49 (4)

$$\log(t_r) = 19.52 - 0.21 \text{ p}$$
 for Technora (5)

where  $t_r$  is rupture time in hours and *P* is the load expressed as a percentage of ABL.

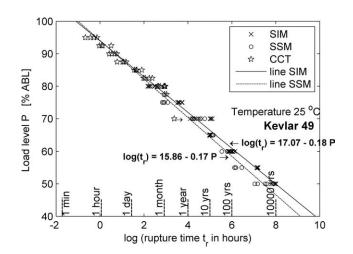
The variation of the test data at all load levels about the two fitted regression lines is small (r = 0.9905 and r = 0.9828 for Kevlar 49 and Technora, respectively).<sup>31</sup>

	Rupture time (years)			
Test number	Kevlar 49	Technora		
SIM-50-01-01	11661.8	_		
-02	10798.5	-		
SIM-50-02-01	10294.1	-		
-02	8970.2	-		
SIM-50-03-01	7206.2	-		
-02	7404.8	-		
SIM-55-01-01	1537.3	5,759,536		
-02	1614.4	12,490,275		
SIM-55-02-01	1755.8	514,918		
-02	1702.0	380,093		
SIM-55-03-01	1786.6	750 <i>,</i> 337		
-02	1671.5	517,005		
SIM-55-04-01	1786.6	1,115,324		
-02	1671.5	15,593,377		
SIM-60-01-01	111.1	29,608		
-02	77.0	26,982		
SIM-60-02-01	122.0	51,777		
-02	155.9	41,944		
SIM-60-03-01	95.2	54,386		
-02	84.3	49,702		
SIM-60-04-01	88.6	49,459		
-02	113.9	50,914		
SIM-65-01-01	10.8	3,421		
-02	11.9	1,880		
SIM-65-02-01	11.2	3,082		
-02	12.4	4,358		
SIM-65-03-01	11.3	6,058		
-02	11.6	7,210		
SIM-65-03-01	11.3	4,393		
-02	11.6	2,669		
SIM-70-01-01	14.72	46.50		
-02	12.07	49.96		
SIM-70-02-01	1.87	48.82		
-02	3.41	48.39		
SIM-70-03-01	1.47	54.13		
-02	4.01	49.14		
SIM-70-04-01	11.59	48.93		
-02	13.14	50.20		
SIM-70-05-01	2.72	_		
-02	2.27	_		
SIM-75-01-01	0.60	7.38		
-02	0.63	9.01		
SIM-75-02-01	0.46	4.12		
-02	0.59	5.16		
SIM-75-03-01	0.38	6.65		
-02	0.63	12.43		
SIM-75-04-01	0.42	5.78		
-02	0.45	5.52		
SIM-80-01-01	0.43	0.09		
-02	0.04	0.05		
SIM-80-02-01	0.03	0.13		
-02	0.02	0.13		
SIM-80-03-01	0.02	0.12		
-02	0.03	0.19		
-02 SIM-80-04-01	0.04	0.10		
-02	0.02	0.08		

 TABLE VI

 Rupture Times for Kevlar 49 and Technora Yarns

These predictions have all been made for tests carried out at 25°C. It is possible to use the values for activation energy determined above to



**Figure 32** Stress-rupture predictions for Kevlar 49 using CCT, SIM, and SSM.

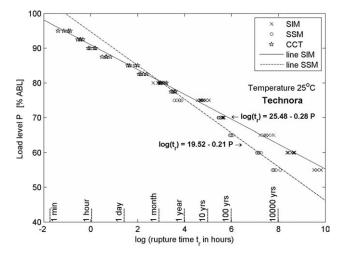
predict the behavior at different temperatures, giving:

$$\log (t_r) = -4.54 + \frac{6270}{T} - 0.18 \text{ P} \text{ for Kevlar 49}$$
(6)

log 
$$(t_r) = +0.51 + \frac{7248}{T} - 0.26$$
 P for Technora (7)

where T is the temperature in Kelvin.

Applying the aforementioned relationships at different reference temperatures, it is shown that increasing the temperature decreases the rupture times. More details on how the variability and different activation energies of the two materials effect the stress-rupture behavior is given elsewhere,<sup>17</sup> and the implications for the stress limits that should be used by structural designers are given in Giannopoulos et al.<sup>31</sup>



**Figure 33** Stress-rupture predictions for Technora using CCT, SIM, and SSM.

#### CONCLUSIONS

The tests have shown that accelerated testing of high modulus aramid fibers is possible and gives results that match well with the results of conventional creep tests.

The results show significant differences between the behavior of Kevlar 49 and Technora. Technora has a better creep-rupture performance than Kevlar 49 at long durations. It is also shown the Technora has a higher activation energy, and therefore is less affected by changes of temperature than Kevlar 49.

The tests have shown good agreement between SIM and SSM testing for Kevlar 49 above 60% ABL, and also when predicting creep rates for Technora, there is a significant difference in the creep-rupture lifetime predicted for Technora, especially at low stress levels.

#### References

- 1. Burgoyne, C. J. In Conctruction Materials Reference Book; Doran, D. K., Ed.; Oxford: Butterworths, 1992.
- Guimaraes, G. B. Parallel-lay aramid ropes for use in structural engineering. University of London, PhD, 1988.
- Gerritse, A.; Taerwe, L. Proceeding of the 4th Int Symposium on Fiber Reinforced Polymer for Reinforced Concrete Structures (FRPRCS-4), 1999; p 829.
- 4. DuPont. Data manual for fibre optics and other cables. E. I. D. P. d. N. A. C. (Inc.), Ed., 1991.
- 5. Teijin, L.; High Tenacity Aramid Fibre. T. Bulletin, Ed., 1986.
- Chiao, T. T.; Wells, J. E.; Moore, R. L.; Hamstad, M. A. Composites Materials: Tesing and Design (3rd Conference), 1974; p 209.
- Phoenix, S. L.; Wu, E. M. In Mechanics of Composites Materials; Hashinand, Z.; Herakovich, C. T., Eds.; New York: Pergamon Press, 1983; p 135.
- Glacer, R. E.; Moore, R. L.; Chiao, T. T. Compos Technol Rev 1984, 6, 26.
- 9. Chambers, J. J.; Parallel-lay aramid ropes for use as tendons in prestressing concrete. University of London, PhD, 1986.
- 10. Guimaraes, G. B.; Burgoyne, C. J. J Mater Sci 1992, 27, 247.
- Yamaguchi, T.; Kato, Y.; Nishimura, T.; Uomoto, T. Proceeding of the 3rd Int. Symposium on Non-metallic Reinforcement for Concrete Structures (FRPRCS-3), 1997; p 179.
- Ando, N.; Matsukawa, T.; Hattori, M.; Mashima, M. 3rd International Symposium on Non-metallic Reinforcement for Concrete Structures (FRPRCS-3), 1997; p 203.
- 13. Markovitz, H. J Polym Sci: Polym Symp 1975, 50, 431.
- 14. Alwis, K. G. N. C.; Burgoyne, C. J. J Appl Compos Mater 2006, 13, 249.
- 15. Thornton, J. S.; Paulson, J. N.; Sandri, D. 6th International Conference on Geosynthetics, Atlanta, USA, 1998.
- 16. Burgoyne, C. J.; Alwis, K. G. N. C. J Mater Sci 2008, 43, 4789.
- Giannopoulos, I. P.; Burgoyne, C. J. 5th Conference on Advanced Composite Materials in Bridges and Structures (ACMBS-V), Winniped, Canada, 2008.
- 18. Lai, J.; Bakker, A. Polymer 1995, 36, 93.
- 19. Hadid, M.; Rechak, S.; Tati, A. Mater Sci Eng 2004, 385, 54.
- 20. Jazouli, S.; Luo, W.; Bremand, F.; Vu-Khanha, T. J Mater Sci 2006, 41, 531.
- Giannopoulos, I. P.; Burgoyne, C. J. 9th International Conference on Fibre Reinforced Polymers for Reinforced Concrete Structures (FRPRCS-9), Sydney, Australia, 2009.
- 22. Giannopoulos, I. P.; Creep and Creep-Rupture Behaviour of Aramid Fibers. University of Cambridge, PhD, 2009.

- 23. Giannopoulos, I. P.; Burgoyne, C. J. J Mater Sci 2011, 46, 7660.
- 24. Yang, H. H. Aromatic High-Strength Fibers; Wiley, 1989.
- 25. Ward, I. M.; Sweeney, J. An Introduction to the Mechanical Properties of Solid Polymers; Wiley, 2004.
- 26. Giannopoulos, I. P.; Burgoyne, C. J. 16th Hellenic Conference in Concrete Structures, Paphos, Cyprus, 2009.
- 27. Burgoyne, C. J.; Alwis, K. G. N. C. J Mater Sci 2008, 43, 7091.
- 28. Howard, A. TECQ Proceedings, University of Surrey, 1983.
- 29. Ericksen, R. H. Polymer 1985, 26, 733.
- Alwis, K. G. N. C. Accelerated testing for long-term stressrupture behaviour of aramid fibres. University of Cambridge, PhD, 2003.
- 31. Giannopoulos, I. P.; Burgoyne, C. J. Structures and Buildings 2009, 162, 221.