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Material Properties

Tensile and creep properties of ultra high molecular weight PE fibres

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Abstract

In order to design multimaterial structures made of ultra high molecular weight PE fibres, their main mechanical properties were characterised from tensile and creep tests performed on single filaments or bundles, with various conditions of temperature and loading speed. After having described the experimental procedures, comparisons and deviations between the reported measures and data given by the manufacturer or published elsewhere, have led to interpretations and modelling. Thus, these tests showed the small dependence of the Young's modulus and tensile strength on the strain rate, and gave rise to the description of the drop in mechanical properties near the melting temperature. Also, a constitutive law related to creep behaviour was determined through tensile tests at constant loading for various temperatures.

Keywords: Fibres (A); Polymers (A); Thermomechanical properties (B); Creep (B)

1. Introduction

Since the 1970s, studies have been devoted to the development of new materials for reinforcement, based on the potential properties of polymers. Indeed, the mechanical performance of this class of materials is far from that which theoretically could be obtainable in the direction of the molecular chain. Thus, it has been expected to process high performance fibres by aligning polymeric molecular structures according to a fibrous morphology. For instance, manufacturing polyethylene (PE) fibres by gel spinning has given rise to stiffness and strength far superior to those related to non-oriented PE [1–5].

However, the use of PE fibres in composites has been impeded by the strong temperature dependence of their properties and the difficulty of obtaining sufficient chemical interaction with polymeric matrices [6–8]. In order to overcome these drawbacks, applications have been limited to low temperatures, and surface treatments have been performed successfully to ensure suitable conditions of load transfer at fibre/matrix interfaces [9].

Consequently, the increasing interest for these fibres is not surprising in the broadening domain of multimaterials which can be designed for satisfying the multifunctional requirements of new applications [10]. Also, it means that quantitative mechanical characteristics must be available for the design department while, presently, the performance of these fibres remains quite uncertain and corresponds to great differences between the values given by the manufacturer and those published elsewhere [11].

As an example, the use of PE fibres as the reinforcement of a container wall whose major function is the

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release (deconfinement) of the contained energetic matter at moderate temperature (120 °C), requires knowledge of various characteristics such as: the fibre rigidity and strength in a rather large domain of temperature, the significance of the viscoelastic component of the fibre behaviour and their creep characteristics in the vicinity of the deconfinement temperature. As a matter of fact, dimensioning such a multimaterial structure and above all simulating the mechanism of deconfinement cannot be performed without measuring precisely the concerned characteristics [12].

Thus, the present contribution has aimed at determining the main mechanical characteristics of ultra high molecular weight polyethylene (UHMW-PE Dyneema SK 75) fibres from tensile and creep tests performed on single filaments and bundles, with different loading speeds and at various temperatures ranging between room temperature and 150 °C.

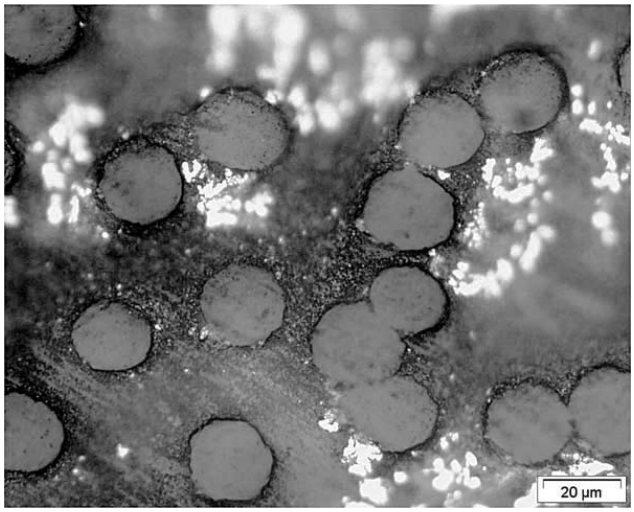

Fig. 1. Micrography of UHMW-PE single filaments cross-section in a bundle.

2. Experimental procedure

2.1. UHMW-PE single filaments and bundles

The single filaments used for the tensile and creep tests were extracted from high modulus Dyneema SK 75 fibres bundles. The first step in the determination of the mechanical properties of the PE fibres was the measurement of single filament diameter and the determination of the number of filaments in bundles. Diameter measurement was not easy because, unlike most of the classically used fibres, the PE fibres used are not cylindrical. Indeed, the fibrous structure, resulting from the manufacturing process, induces changes in the cross-section along filaments. Thus, to be representative of the fibres, the diameter measurement had to be carried out on a great number of single filaments.

As the single filament diameter is of the order of 20 µm and their section is not rigorously circular, the use of a laser diffraction method for this type of polymeric fibres would not give the expected accuracy. Thus, a micrographic method was chosen to obtain an average value of the diameter by direct measurement from images, as illustrated in Fig. 1.

The diameter measurement by image analysis on more than 100 elementary filaments belonging to several bundles, led to an average diameter of $20.5 \pm 1.3\mu m$.

The number of filaments in bundles was derived from bundle weights for various lengths ranging between 25 mm and 5 m from the average filament diameter and from the known fibre (UHMW-PE Dyneema SK 75) density (0.97). This evaluation led to an average bundle section of 0.186 ± 0.005 and $550 \pm 65 mm^2$ filaments in a bundle.

2.2. Testing conditions

2.2.1. Specimen loading

Given their small diameter, each elementary filament was bonded with epoxy resin on a stiff frame in order to facilitate their manipulation (Fig. 2(a)). The tensile tests were carried out on a 5 kN tensile testing machine, equipped with a LVDT load sensor having a maximum range of 10 N (Fig. 2).

Specimen loading was applied through the control of the machine cross head displacement at speeds ranging between 0.04 and 160 mm/min. Concerning the creep

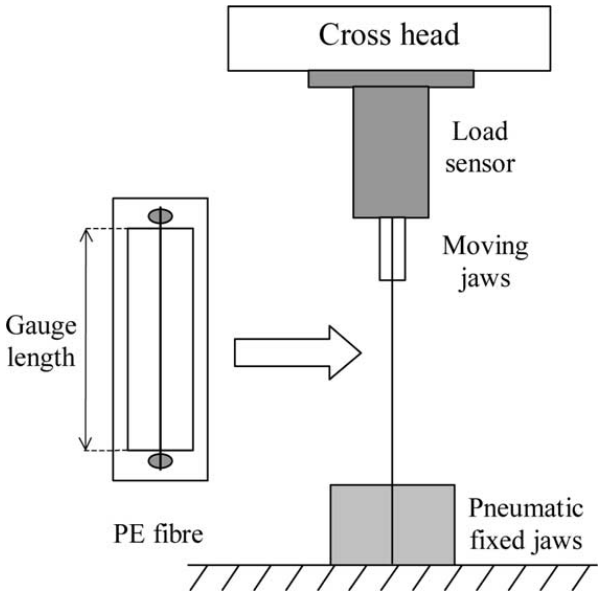

Fig. 2. Schematic representation of the tensile tests procedure.

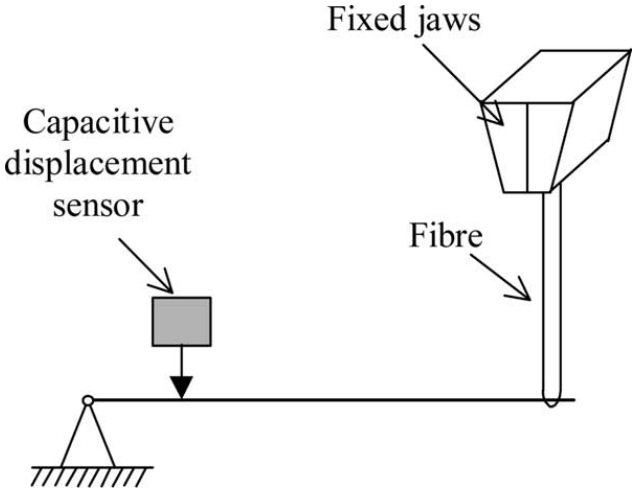

Fig. 3. Creep tests equipment.

tests, a constant load was applied to specimens through a small metallic lever as schematically illustrated in Fig. 3.

2.2.2. Displacement and compliance measurement

As the use of an extensometer is difficult on such thin specimens, the elongation of the single filaments or fibre bundles was determined through the displacement of the testing machine cross head. However, it is theoretically necessary to operate a correction since the recorded displacements combine the extensions of both the specimen and the whole testing device. So, determining the fibres' true strain requires the measurement of the whole testing device compliance. The related procedure consists of testing specimens with different gauge lengths and deriving from the corresponding load–displacement curves the total compliance, which is a linear function of gauge length. Extrapolating this function as illustrated in Fig. 4 to zero gauge length, leads to the testing device compliance which is, in the present case, very small (0.236 mm/N) compared to those of the different specimens.

Then, subtracting for each recorded load, the corresponding testing device elongation from the machine cross head displacement, enables the determination of the true specimen compliance.

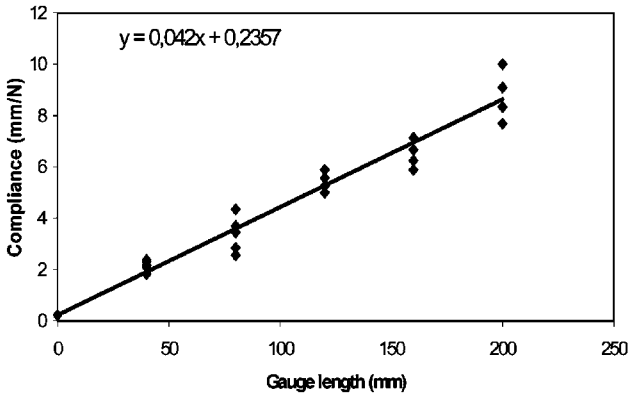

Fig. 4. Determination of the testing device compliance.

Concerning the creep tests, the displacements were measured with the help of a capacitive sensor positioned above the lever, allowing an accurate measurement of large displacements (Fig. 3).

2.2.3. Testing temperatures

Since the drop in mechanical properties when the temperature increases and the rather low melting temperature of the UHMW-PE fibres (about 150 °C) impede their use in the composites industry, the maximum service temperatures have been limited to 100 °C in classical applications. However, some new applications such as those previously mentioned, propose to take advantage of this temperature induced drop in performance. For example, using the fibre melting as a security device replacing a valve, needs the fibre behaviour to be described near the melting temperature.

In order to depict the UHMW-PE fibre properties in a domain of temperature ranging from room temperature up to temperatures close to their melting point, tensile tests on elementary filaments were performed in a narrow electric furnace.

The temperatures chosen for the tests were 70, 100, 125 and 140 °C. For each test, the tensile strength was noted and the Young's modulus was calculated using the previously described procedure.

3. Experimental results and discussion

3.1. Influence of the strain rate

Since on the one hand, PE often exhibits at room temperature viscoelastic behaviour and on the other hand the UHMW-PE fibre crystallinity is presumably very significant due to their processing mode, it is necessary to assess the contribution of the viscoelasticity in the UHMW-PE fibre behaviour through the influence of the strain rate on their mechanical properties. Thus, tests were conducted with strain rates ranging between 0.1 and 100%/min (0.1; 1; 4 and 100%/min). For each series of tests, four gauge lengths from 40 to 200 mm were used, with at least 15 tests for each one, to enable the compliance correction as previously defined. Since the load–displacement curves are generally non-linear as illustrated in Fig. 5, the Young's moduli were calculated from the slope at the beginning of the curve. The whole results related to more than 150 tests on single filaments are shown in Table 1 and in Fig. 6, using a logarithmic scale for the strain rates.

These results confirm that the orientation in the fibre direction of the molecular chain of PE gives the fibre microstructure a high degree of crystallinity. It leads not only to high mechanical performance but also reduces

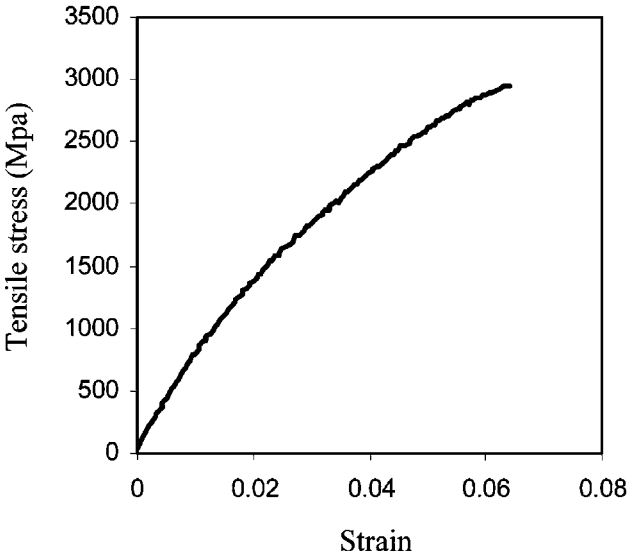

Fig. 5. Tensile stress versus strain curve related to a UHMW-PE single filament.

Table 1
UHMW-PE fibre characteristics according to various sources

	Young's modulus (GPa)	Tensile strength (GPa)	Elongation at rupture (%)
1%/min	71	2.4	3.95
4%/min	71	2.4	4
100%/min	62	2.4	3.76
0.1%/min	56	2.4	5.88
Manufacturer [12]	89	2.7	3.5
Ref. [11]	68	2.25	4.89

drastically the viscoelastic contribution of the fibre behaviour, since there is no dependency of the mechanical properties on strain rate. Indeed, only the elongation at rupture seems to exhibit some sensitivity to strain rate, although the related deviation is quite small with regard to the investigated range of strain rates.

It is also interesting to compare these results with the values given by the manufacturer [13] or published elsewhere. Indeed, Table 1 shows some deviations which can be correlated with experimental uncertainties but which might be related to other explanations.

Considering there are always doubts concerning the similitude of the conditions of testing, tensile tests were also performed on fibre bundles in order to find other explanations for variation of values than difficulties in measuring single filament diameter accurately. However, before performing such tests on bundles, a statistical approach to the results obtained on single filaments was also attempted in order to estimate the coherence of the related results.

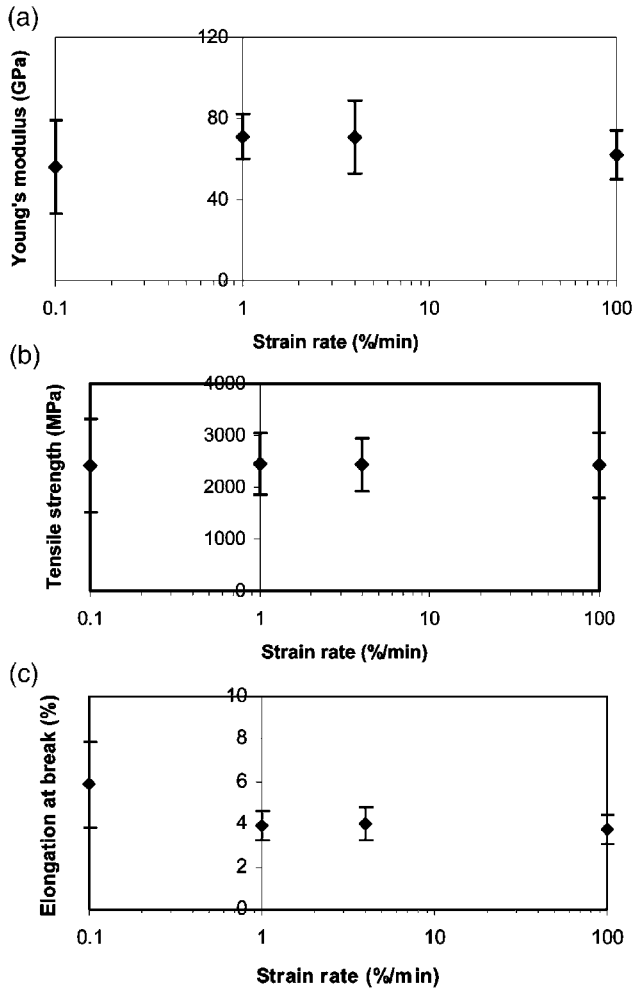

Fig. 6. Influence of the strain rate on (a) the rigidity, (b) the strength, (c) the ductility.

3.2. Statistical approach of tensile strength

Although the tensile behaviour of the UHMW-PE filaments is not typically brittle elastic, but exhibits a not negligible non-linear contribution, it was assumed that their strength might be controlled by the presence of defects such as, for instance, diameter deviations and reductions along filaments.

Thus, the tensile strengths obtained on filaments for various gauge lengths have been investigated according to the Weibull approach in terms of survival probability versus rupture strength as illustrated in Fig. 7 for a 80 mm gauge length [14].

Although a greater number of tensile tests would have been preferable, the whole results reported in Table 2 and more particularly the Weibull modulus, *m*, related to different gauge lengths, give some indications concerning features of the results.

The significant increase of the Weibull modulus as the gauge length is raised may be related to a systematic presence of a defect type for gauge lengths higher than

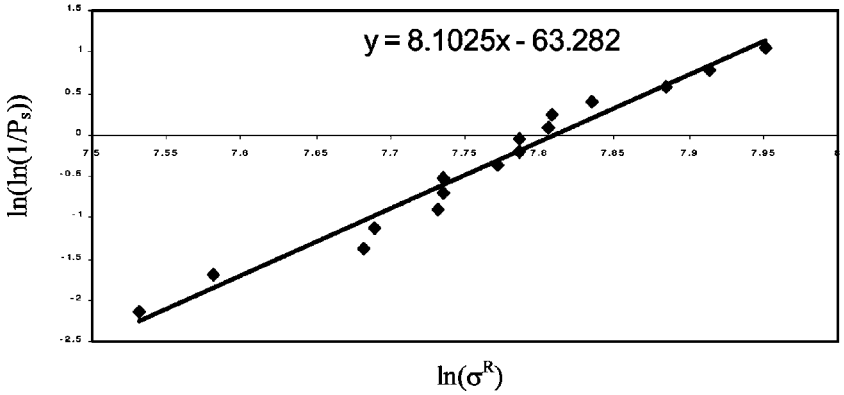

Fig. 7. Weibull chart for 80 mm gauge length filaments.

Table 2
Weibull modulus and tensile stress corresponding to a 0.5 survival probability for various gauge lengths

Gauge length (mm)	Weibull modulus	$\sigma_{0.5}$ (MPa)
40	4.77	2377
80	8.10	2356
120	15.97	2271
200	8.95	2486

80 mm. However, the very small decrease of the stress related to a survival probability of 0.5 when the gauge length is raised and, even more, the significant increase of this strength for the highest gauge length leads to consider that the exclusion of the weakest filaments due to friction between filaments during their extraction from bundles might increase the Weibull modulus and prevent any decrease of filament strength as gauge lengths are raised. In fact, the longer the gauge length, the stronger the friction forces are and the more effective the filament selection. This explanation tends to invalidate the tensile tests performed on filaments of large gauge lengths (120–200 mm) because of the selection effect and to invalidate those related to small gauge lengths because the volume tested is not sufficiently representative of the diameter fluctuations.

The previous considerations and discussion related to difficulties of fibre mechanical evaluation through tensile tests on filaments justify an attempt to evaluate performance through tensile tests on bundles. Nevertheless, an average strength of 2400 MPa can be presently considered as the performance derived from tests on single filaments.

3.3. Influence of coupling between single filaments gathered in bundles

Fibre bundle tests are generally used to decrease the total number of tests in comparison with filament tests [15]. However, deriving intrinsic characteristics of single filaments from bundle tests requires taking precautions. For instance, obtaining the single filament strength from that of bundles needs to know, or to assume, an appropriate strength distribution law, such as the most usually chosen Weibull distribution [16–19]. More precisely, it is noteworthy that the smaller the Weibull modulus, the lower the bundle strength compared to those of single filaments.

The tests on bundles were performed using five different gauge lengths to apply the correction on the displacement measurement. The obtained experimental curves are illustrated in Fig. 8.

As illustrated in Fig. 9, the rupture strengths obtained on filaments are rather close to those obtained on bundles, which is indicative of small dispersions in single filament strengths corresponding to high Weibull moduli. This means that tensile tests on bundles with gauge length higher than 100 mm are able to give directly the rupture strength of UHMW-PE fibres. Also, these results indicate that the Weibull approach is not particularly suitable for this type of fibre.

Comparison between rigidities obtained on single

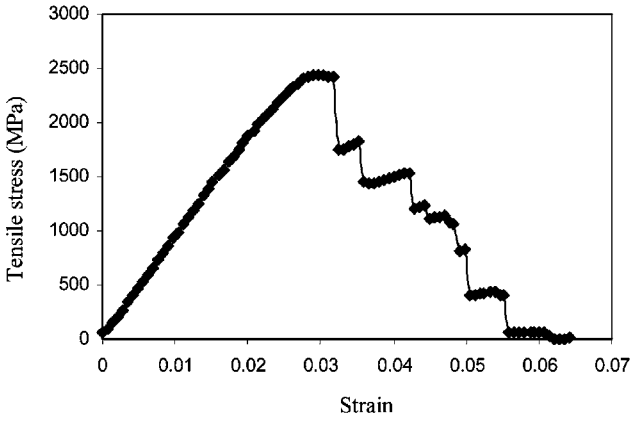

Fig. 8. Example of experimental tensile test curve related to UHMW-PE fibre bundle.

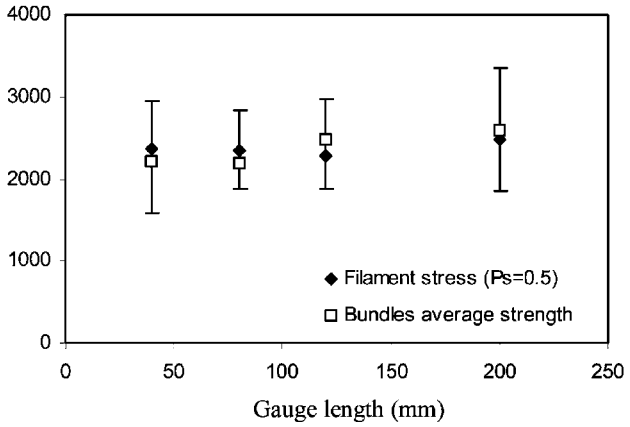

Fig. 9. Comparison between filament and bundle tensile tests.

filaments and from bundles, points out a noticeable difference which cannot be merely and only explained by uncertainties affecting the filament diameter measurement, which is also involved in rupture strength determination (Table 3).

In order to find an explanation to the deviation between the rigidities obtained either by single filament tests or through bundle tests, the effects of the links observed between filaments were studied from a schematic representation of two opposite cases of filaments coupling in bundles, as illustrated in Fig. 10.

Applying tensile loads to bundles which could be represented by model (a) leads to a global stiffness, $k_{\#}$, which is quite different to $k_{//}$ related to model (b). Computing the difference $k_{\#}-k_{//}$ gives rise to a term proportional to $(k_2k_3-k_1k_4)^2$ which is always positive. This justifies the proposal that the filament linkage within bundles significantly increases bundle stiffness.

Also, concerning bundle failure, local links between filaments are able to redistribute loading when a weaker filament is about to fail, thanks to the non-linear feature of the filament tensile curves in the vicinity of rupture. This load redistribution is able to delay the failure of bundles compared to those of single filaments.

Thus, the deviation between UHMW-PE fibre stiffness related to different sources can be explained by links observed between single filaments within bundles or/and by twisting induced friction between single filaments which would lead to similar effect.

Table 3
Comparison between bundle tests and single filament tests

	Young's modulus (GPa)	Tensile strength (GPa)
Single filaments	71	2.4
Fibre bundles	104	2.4

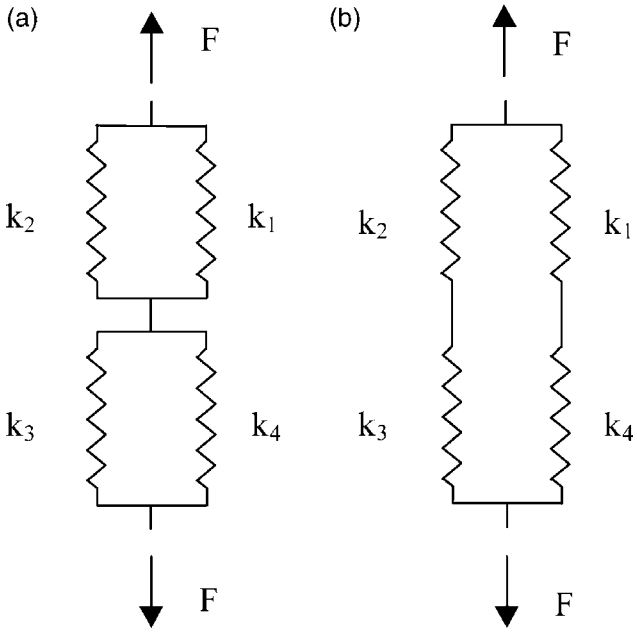

Fig. 10. Models of filaments coupling in UHMW-PE fibre bundles (a) with interfacial links between filaments (stiffness $k_{\#}$) and (b) without any interfacial link (stiffness $k_{//}$), k_1, k_2, k_3, k_4 represent the stiffness related to various filament zones in which the PE molecular chain orientations are different or correspond to various degrees of crystallinity or rate of polymerisation.

3.4. Influence of the temperature

The load–displacement curves reported in Fig. 11 related to single filament tensile tests performed at various temperatures, show the deviations in the UHMW-PE fibres' behaviour when raising temperature. An increase in temperature broadens significantly the domain of fibre

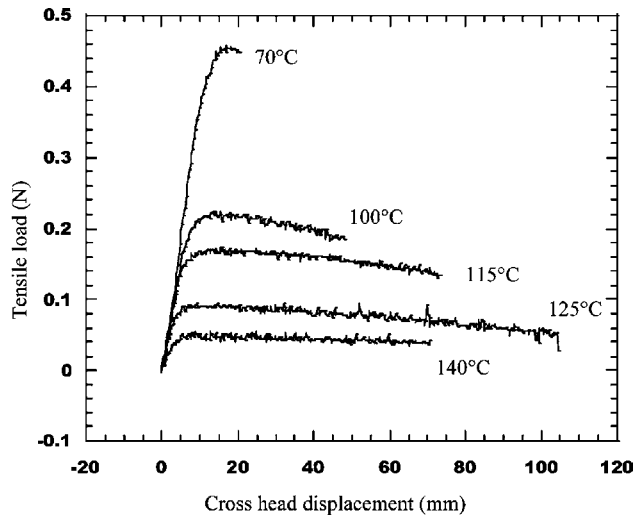

Fig. 11. Tensile load–displacement curves related to UHMW-PE filaments tested at various temperatures.

viscous flow under quasi-constant stress which can be considered as the ultimate tensile strength. This deviation shows that despite the high degree of polymerisation of this type of PE, the very long molecular chains are able to slide as soon as the temperature increases. Such deviation could be enhanced by structural change which will be considered later.

From the load–displacement curves, the influence of temperature on the UHMW-PE fibre rigidity and strength could be pointed out as illustrated in Fig. 12(a) and (b) which exhibits some difference in features.

While on one hand, the rigidity drops significantly between 20 and 70 °C to about half the nominal value obtained at room temperature and decreases more progressively from 70 up to 150 °C, on the other hand, the decrease of tensile strength is rather moderate up to 70 °C and becomes drastic between 70 and 120 °C. Such a difference between rigidity and strength deviations versus temperature might be important for the design of structures whose deconfinement mechanism is based on a temperature threshold.

3.5. Creep behaviour

The numerical simulation of the previous deconfinement mechanism requires more than mechanical performance at various temperatures, data related to deformation kinetics are also needed. More precisely, creep laws have to be determined for the temperatures considered in the previous investigation. As already established elsewhere, a power law should describe the variation of the stress relaxation modulus as a function of time and a decreasing exponential should be a suitable function for representing the temperature dependence [20,21]. Thus, the law which was chosen and had to be verified can be expressed as follows:

$$\varepsilon^{c} = C_0 \sigma^{C_1} t^{C_2} e^{(-C_T/T)},$$

where ϵ^{c} is the creep strain, σ the applied stress and C_i the constants of the model.

The aim of the determination of this creep law is only to provide the user with an expression of the creep strain, without considering whether it is recoverable or not, contrary to the previous studies.

For verifying this creep law, tests were performed at five temperatures and with two different loads in order to determine the constants C_i. It is noteworthy that the deviations in strain as a function of time are linear up to failure, because of the absence of tertiary creep. Consequently C_2 could be equalled to 1.

Then, representing the strain rates as a function of temperature for two different tensile loads, as illustrated in Fig. 13, allows C_0, C_1 and C_T to be derived by fitting the experimental results with the previously proposed creep law.

The resolution of this problem gives the following Bailey–Norton creep law for the PE fibre:

$$\varepsilon = 1.72\sigma^{3.82} t e^{(-13450/T)}.$$

In addition, these tests give confirmation concerning the creep behaviour of the fibre that was described by Dessain et al. [11]. Indeed, this previous study has shown that a solid phase transformation in the fibre, from an orthorhombic structure toward an hexagonal structure, takes place at about 5 °C, and induces strain rates far superior to those observed at lower temperatures. How-

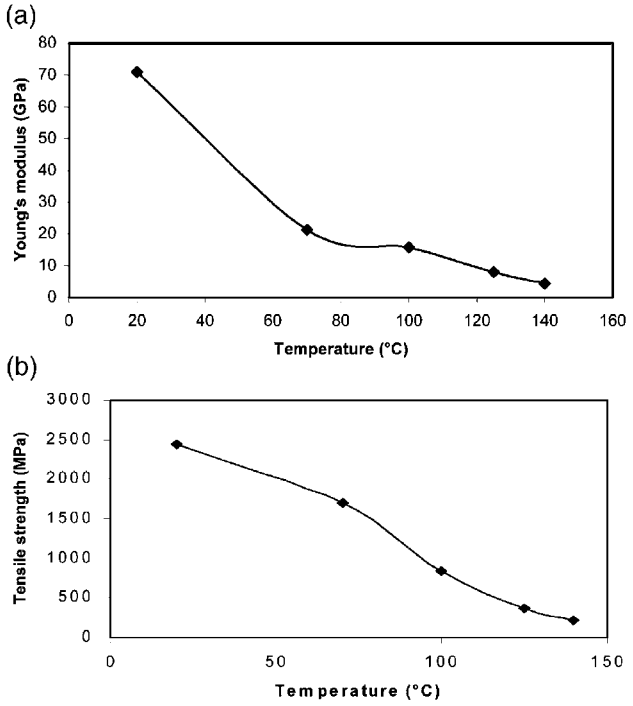

Fig. 12. Influence of the temperature on (a) the Young's modulus and (b) the tensile strength.

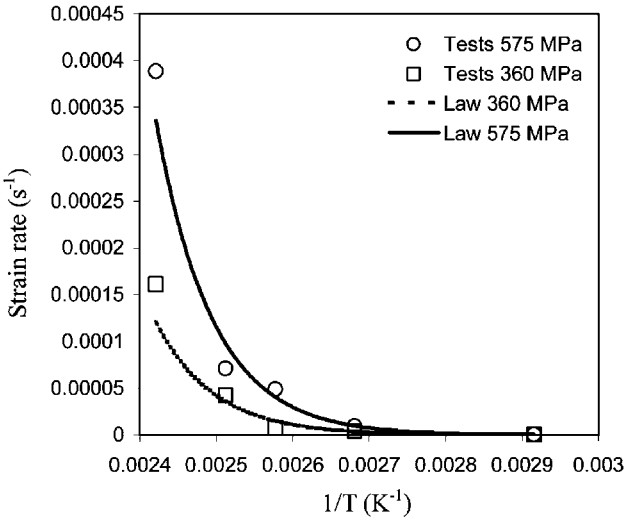

Fig. 13. Deviation in strain rate versus temperature.

ever, this study was limited to a temperature domain below 100 °C, whereas the present tests have shown that strain rates keep increasing exponentially up to the melting temperature.

4. Conclusions

The tensile and creep properties of PE fibre have been studied. The tests have shown the importance of the molecular chains' alignment which is characteristic of the fibre microstructure. Indeed, the mechanical performance and more particularly the fibre stiffness has been found to be about 20 times higher than that of non-oriented PE, and barely depend on the strain rate.

Furthermore, the way in which the tensile properties drop near the melting temperature has been defined precisely, allowing the corresponding characteristics of the PE Dyneema SK 75 fibre to be used for the numerical simulation of a thermally activated failure related to PE fibre reinforced structures. The identification of an exponential law through creep tests has enlarged the validity domain of the constitutive representation established in previous papers.

Acknowledgements

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