DUAL VISCOSE AND PAN-BASED CARBON FILAMENTS: MECHANICAL AND POROUS PROPERTIES

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Introduction

Carbon fibres of high sorption capacity may be obtained from various precursors including rayon, isotropic pitch and thermosetting polymers and their potential use in various types of application has been reviewed recently (1). Fabrics made of adsorbent carbon fibre, *i.e.* activated carbon fibre, are of interest for the confection of protective clothes. However, the mechanical properties of activated carbon fibre are usually rather weak and hardly meet those required in current textile use. On one hand, high strength carbon fibres cannot be easily activated and on the other hand, the strength of microporous carbon filaments is very low. A way to combine strength and porosity in a same filament would be to assemble two types of carbon fibres in a concentric way : a core bearing strength and an external layer developing microporosity. This study explores the possibility of obtaining dual carbon filaments of sufficient mechanical strength and sorption capacity for protective clothes.

Experimental

The core of the dual filament consists of about 50 discontinuous PAN-based carbon fibres (mean diameter equal to 7 μm, average length equal to 48 mm) which are hold together by a continuous filament of polyvinylic alcohol (PVA filament). An external layer made of discontinuous viscose fibres (mean diameter equal to 12 μm, average length equal to 150 mm) is thereafter wrapped in a concentric way around the core using a friction spinning process (DREF 3) designed for monitoring the amount of deposited filaments.

The dual filament was then carbonized in inert atmosphere using a linear heating rate equal to 10°C/min up to 900°C and kept at this temperature for 15 min. During carbonization, the PVA filament is completely pyrolyzed and both types of viscose undergo a weight loss equal to 85%. The proportion of core and external layer in the dual carbon filament is equal to 40 and 60% by weight respectively. Activation of carbon filaments was carried out in CO_2 at 900°C and at atmospheric pressure using a linear gas flow rate equal to 36 cm/min.

Ultimate tensile load of filament was measured using a dynamometer on samples with a gauge length equal to 20 mm (crosshead rate equal to 4 mm/s). 60 samples were tested for each type of filaments. Micropore volume of fibres, V, was determined by adsorption of N_2 at -196°C according to the Dubinin-Radushkevich equation.

Results and Discussion

Two types of viscose, Va and Vb have been used in dual filaments denoted C-Va and C-Vb respectively. For comparison the properties of core PAN-based filaments without an external viscose layer have also been examined (sample C).

Tensile load of dual carbon filaments

A statistical analysis of the ultimate load data was done using the following Weibull equation :

$$P(f) = 1 - \exp\{-A\,[(f-fu)/fo]^m\}\ [1]$$

where $P(f)$ is the cumulative probability of failure at load f, A is a constant, m is the Weibull parameter which is related to flaw distribution, fu is the lowest load at which filament failure is observed and fo is a scaling parameter. Figure 1 represents the cumulative probability of failure as a function of applied load for filaments C and C-Va. The solid line corresponds to the values calculated using Eq [1]. The experimental values can be satisfactorily fitted by the Weibull equation.

The mean value of ultimate tensile load of carbon filaments, f^*, and the standard deviation, s, of the distribution are shown in Table 1. For all filaments, the elongation to failure is close to 2%.

Table 1. Tensile load of dual carbon filaments.

Filament	f^* (N)	s	f_h (N)	m
C	8.9	1.9	8.1	3.8
C-Va	13.0	3.2	11.7	2.2
C-Vb	11.9	2.6	11.1	3.0

The load corresponding to a survival probability of filaments equal to 0.5, f_h, and the value of m are also listed in Table 1. Average load values, f^*, are in fairly good agreement with those of f_h. There is a small increase in ultimate load of C-VA or C-Vb filaments as compared to the one of single core. This increase is due to the carbonized viscose and may originate from the shrinkage

(about 30%) of viscose fibres around the core during pyrolysis. However, as expected, the largest part of the load is supported by the core. The value of the Weibull parameter, *m*, decreases after setting the external layer of viscose-based carbon which indicates that the distribution of flaws inducing the filament failure has been broaden.

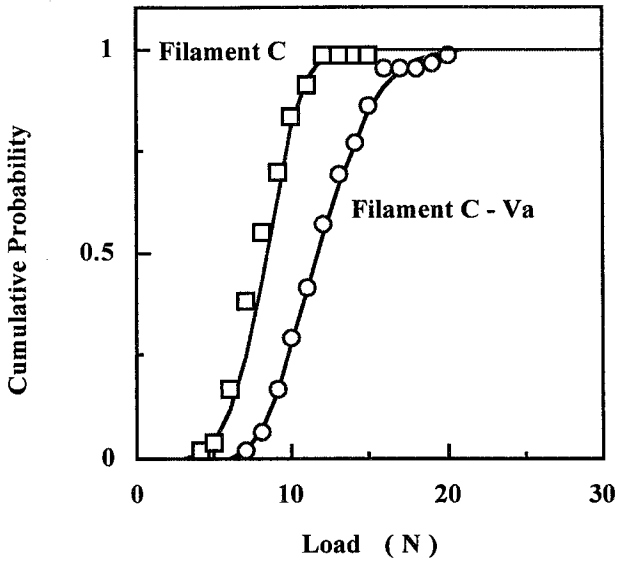

Figure 1. Tensile load distribution of carbon filaments

Activated dual carbon filaments

As expected, the rate of weight loss of PAN-based fibres during activation is quite small (~0.03 %/min) as compared to Va (~2 %/min) and Vb (~0.6 %/min) carbon filaments. The difference in gasification rate of Va and Vb fibres is attributed to the presence of sodium (0.7 % by weight of metal) in the former one.

Table 2. Activation of carbon filaments

Filament	Weight loss (%)			*V*
	Total	Ext. layer	Core	(cm^3/g)
C-Va-10	17	28	< 1	0.18
C-Va-15	32	53	1	0.28
C-Vb-90	34	55	3	0.22
C-120	4		4	-

Physical gas adsorption measurements on activated viscose-based filaments indicate that a significant micropore volume is obtained in the case of Va, since after 30 % weight loss the micropore volume, *V*, is equal to 0.34 cm^3/g. In the case of viscose Vb the micropore volume developed at the same level of burn-off is only equal to 0.25 cm^3/g. Consequently, the activation time of dual filaments was set in order to submit the viscose-based carbon to a burn-off which develops a sufficient micropore volume. Filament tretament time (in min) is indicated in sample designation in Table 2.The respective weight losses of core and external layer after activation are shown in Table 2. There is a significant difference in activation time between the two dual filaments for obtaining 50% burn-off of the viscose-based carbon. Micropore volume, *V*, expressed per unit mass of dual carbon filament is also shown in Table 2. The values obtained are similar to those found for activated carbon clothes. However carbon filaments based on viscose Va develop a larger micropore volume than with viscose Vb.

The mechanical characteristics of the activated filaments differ also for similar levels of burn-off of the external layer (Table 3). The ultimate load of C-Va filaments is not significantly affected by the activation treatment even after 55% of burn-off (sample C-Va-15). For a similar extent of activation of viscose Vb, an important decrease in ultimate tensile load is observed (sample C-Vb-90). The decrease in filament strength originates from the weakening of the core PAN-based fibres due to a longer tretatment time which induces a slight gasification. The distribution of flaws becomes also larger after gasification of PAN-based fibres as indicated by a smaller value of *m* (2.6 as compared to 3.8). It is therefore important to keep the activation time of the carbon fibres of the external layer as short as possible.

Table 3. Characteristics of activated carbon filaments

Filament	f^* (N)	*s*	f_h (N)	*m*
C-Va-10	12.9	3.0	13.2	2.4
C-Va-15	13.5	2.3	12.5	2.9
C-Vb-90	3.4	1.2	4.5	2.6
C-120	4.6	1.6	5.3	2.6

Conclusion

Carbon filaments made of a core of PAN-based fibres and an external layer consisting of viscose-based fibres exhibit after activation in carbon dioxide a microporous character associated to sufficient mechanical strength for their use as adsorbent cloth. For high enough an activation rate of viscose-based carbon fibres, the mechanical strength of PAN-based core fibres is preserved. Hence the properties of the starting viscose fibre will be a critical factor for the optimization of the mechanical and the porous properties of dual carbon filaments.

Reference

1. Suzuki, M., *Carbon*, 1994, 32, 577.