HIGH STRENGTH CARBON FIBERS FROM MESOPHASE PITCH

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Introduction

It has been demonstrated in the past that carbon fibers from mesophase pitch are capable of reaching a tensile strength of 3.5-4.0 GPa[1,2]. So far, such high strengths have been obtained only in selected, thin monofilaments spun from a mesophase pitch that was prepared from a carefully processed commercial precursor material. The purpose of the present study was to confirm the initial results and to analyze the data statistically as a means of estimating the intrinsic strength of these carbon fibers.

Experimental

Additional monofilaments have been spun from a mesophase pitch prepared from a processed precursor. After thermosetting and carbonizing, the filaments had round cross-sections with diameters between 7.3 and 8.5 µm. Most filament in one sample were free of voids that could be seen at 1000X magnification, but the filaments in another sample had a large number of voids. These two samples were compared to determine the effect of voids on tensile strength.

Filaments were mounted on perforated cardboard tabs and broken in tension routinely at gauge lengths of 1.4 to 40 mm on an Instron testing machine. A perfect vertical alignment of very short filaments was difficult to achieve, particularly at the gauge length of 1.4 mm.

Results and Discussion

The average tensile strength and Young's modulus, along with the frequency of break histogram, at each gauge length, are shown in Figures 1 and 2 for weak and strong filaments, respectively. Clearly, most weak filaments broke under a stress of 1.0-2.0 GPa. This level of strength, attributed to the presence of holes, occurs in the strong filaments with a relatively low frequency which decreases with decreasing gauge length and practically disappears at and below 3.2 mm.

The probability of failure and the fiber strength can be correlated analytically by means of the Weibull function[3]. Figure 3 shows the tensile strength of the strong filaments at 3.2 mm length plotted on the Weibull probability chart. The Weibull plot allows one to identify and eliminate "stray" values that do not fall on the straight line whose slope is the flaw dispersion parameter m, a characterisitc constant for the material. Table I shows the values of m at various gauge lengths calculated from the two-parameter form of the Weibull equation. Calculations of the mean strength $\bar{\sigma}$ from the Weibull data was performed in the manner described by Street and Ferte[4].

Table I

Weibull Dispersion Parameter m at Different Gauge Lengths

Gauge length (mm)	Weibull Slope m: Strong filaments		Weibull Slope m: Weak filaments	
1.4	8.2		3.1	
3.2	5.2	Avg.=5.0	3.2	Avg.=3.7
10	4.1		3.7	
20	5.6		4.2	
40	2.8		2.4	

The relationship between $\bar{\sigma}$ and the gauge length, L, for a particular m can be represented as $\ln \bar{\sigma} = -(1/m) \ln L + \text{constant}$. Figure 4 shows the double logarithm plots of $\bar{\sigma}$ vs. L for weak and strong filaments. The average tensile strength derived from simple sample statistics (see histograms) are also included in the plot. With one exception, the results are almost identical. The solid lines were drawn through the points, using -1/m as the slope where m is the average value shown in Table I. The points at L = 1.4 mm are excluded because of the poor reliability of measurements at low gauge lengths[4,5]. The values of m at L = 40 mm are excluded because the Weibull distribution plots indicated failure due to a secondary flaw population, probably caused by surface damage.

A tensile strength of 7 GPa is obtained for the strong filaments by extrapolation to a length just below 0.1 mm. Thorne estimates from the loop test at an effective length of 0.1 mm that the strength is greater than 6 GPa for PAN-based carbon fibers[6]. Others have made estimates of strength for PAN-based carbon fibers at even smaller lengths. Diefendorf and Tokarsky report a strength of 8 GPa at 0.02 mm length[7]. Watt cites a strength value of 7 GPa by extrapolation to near zero length[8]. Obviously, carbon fibers made from mesophase pitch are capable of reaching at least equivalent levels of strength.

In terms of the potential performance of fibers in composites, it is more practical to consider only fiber lengths larger than the load transfer length as defined by Kelly[9]. This length is estimated to be about 0.2 mm for the fibers in this study. Morita et al. reported good agreement between strand results and single filament data extrapolated to a length of 0.6 mm, using Weibull statistical analysis[10]. At this gauge length, our strong fibers have a tensile strength of 4.7 GPa, while the fibers with voids are 30% weaker.

Acknowledgments: This work was supported in part by the Air Force Materials Laboratory, Wright-Patterson Air Force Base, Ohio. The authors are grateful to J. D. Grigsby for fiber test measurements and to M. B. Carter for assistance with the Weibull calculations.

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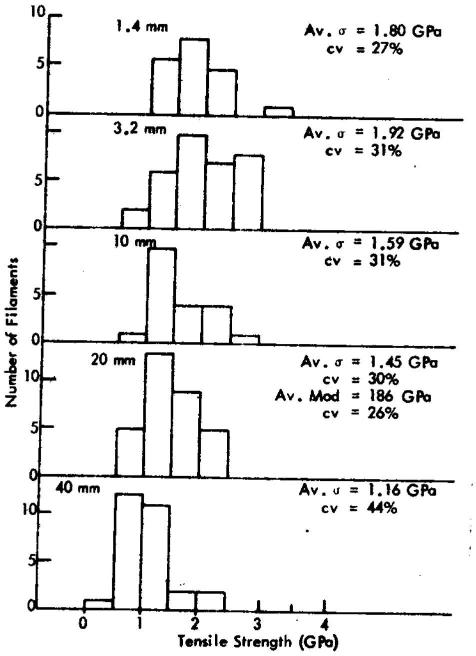

Figure 1. Tensile Strength Distribution for Various Gauge Lengths of Weak Filaments.

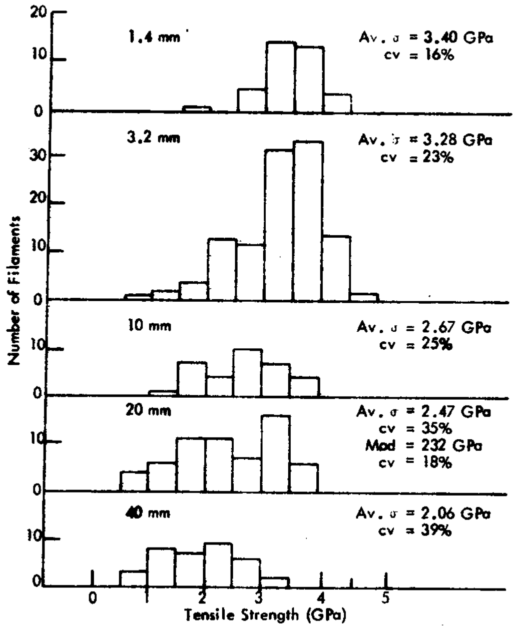

Figure 2. Tensile Strength Distribution for Various Gauge Lengths of Strong Filaments.

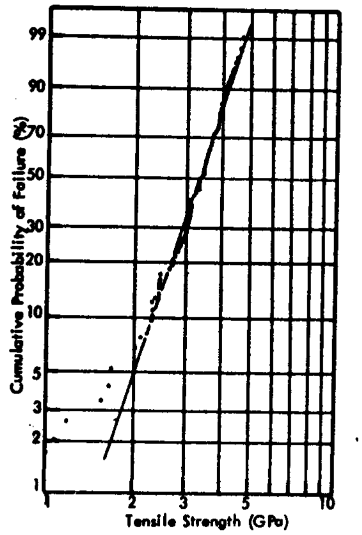

Figure 3. Weibull Strength Distribution of Strong Filaments at 3.2 mm Gauge Length.

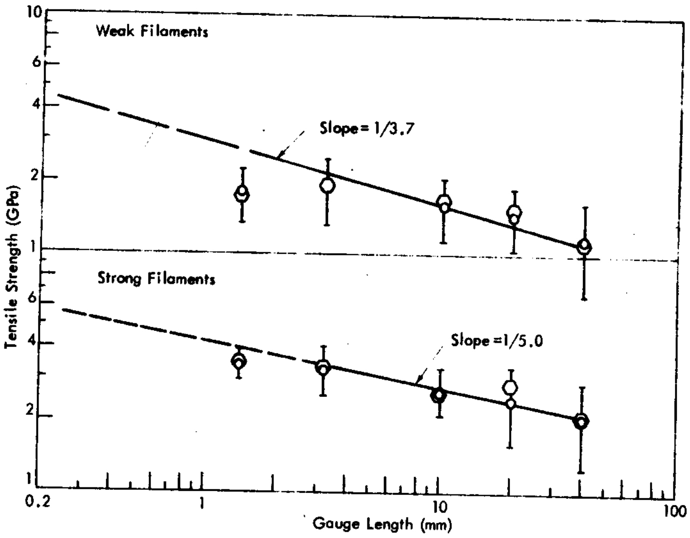

Figure 4. Tensile Strength as a Function of Gauge Length ○Av. Strength ◯Calc. Strength $\overline{\sigma}$.