SIZE EFFECTS ON STRENGTHS OF GLASSY CARBON

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Introduction

Weibull predictions of strength ratios for brittle materials of different volumes that are evaluated under two different test conditions have the general form [1]

$$\frac{\sigma_1}{\sigma_2} = \left(\frac{V_2}{V_1}\right)^{1/m} \text{ X stress-distribution term(STD)}, \quad (1)$$

where

STD = $[2(m+1)^2]^{1/m}$ for the ratio of three-point bend strength to tensile strength,

= $[4(m+1)^2/(m+2)]^{1/m}$ for the ratio of four-point bend strength (quadrant loading) to tensile strenth,

= $[m/2+1]^{1/m}$ for the ratio of three-point bend strength to four-point bend strength,

with the Weibull modulus m providing a measure of the scatter found within experimental strength data.

These predictions have agreed reasonably well with experiment when graphite specimens of the same volume have been tested under different stress distributions [2], but the agreement has not usually been good when different volumes of graphite have been tested under the same conditions [3,4]. The theoretical volume term has generally been found to considerably overestimate the actual size effect for large-grained graphites [2-6], but it has been suggested that the agreement here would improve if test samples were made large in relation to their grain size [7,8]. Therefore, it is of interest to examine size effects on the strength of a glassy carbon that has an x-ray crystallite size of less than 20Å [9], as opposed to values for graphite that are often 10^6 times as large.

Experimental Details

The material tested was a commercial glassy carbon manufactured by Beckwith Carbon Corporation. This disc-shaped material was designated as 1800 grade, indicating heat treatment to 1800°C; it was 125 mils thick and had a bulk density of 1.39 g/cm^3. When thin strips taken from the surface were polished to a mirror finish and viewed in the microscope under the proper angle of incident light, a great many spherical pores were observed. While most of these pores were only 1 to 5μm in diameter, several were 10 to 15μm, and the largest observed was about 30μm. Pores larger than 1μm virtually disappear at depths greater than 8 mils into this particular material, however, as previously observed [10]. Therefore, a 10 mil thickness was removed from all outer surfaces in order to leave only the remaining bulk material; this is justified because it is possible to produce glassy carbon in the laboratory that does not have a porous surface layer [11]. Discs 4 mils thicker than the desired thickness of the bend specimens were longitudinally sliced from this bulk material with a mechanically controlled diamond section wheel; these thin sections were sealed between glass slides and then cut into strips 40 mils wide with the sectioning wheel. Finally, these strips were attached to an arbor and sanded down uniformly by 2 mils on each side to remove the small edge chips (<20μm) that almost inevitably result during cutting of this brittle material, particularly on the exit side of the cut. The final polishing compound was 3μm diamond powder, which produced test specimens with mirror-like surfaces.

The finished specimens were examined under a microscope to ensure that they were free from serious chips or scratches, and the surface that appeared best was selected to be the tension face in the three-point bend test. To ensure uniformity before testing, the width of each specimen was measured in a toolmaker's microscope to an accuracy of ±0.04 mils at three points, which corresponded roughly to the load points during subsequent testing, and the thickness was measured at about the same points to an accuracy of ±0.05 mils with a sharp-point micrometer. Then, after testing, dimensions of each fragment were measured adjacent to the fracture and averaged to get values for the failed section. Specimens were tested over a span width of 0.2 in. on a three-point bend fixture, which had been designed for use on pyrocarbon strips removed from graphite discs that were coated along with nuclear fuel particles [12]. Specimens were loaded with the use of a table-model Instron machine at a crosshead speed of 0.01 in./min, and the load and deflection were simultaneously recorded. Fracture stresses were calculated from expressions [13] that take into account large deflections and frictional effects at the knife edges [14]; these corrections were significant only for the thinnest specimens tested (3 mils), where the calculated fracture stresses exceeded those calculated from simple-beam theory by as much as 10%.

Results and Discussion

The test plan for this study eventually calls for three sample thicknesses to be tested under three-point bending at two different span widths to investigate size effects, with two of these specimen types also being tested under four-point bending to investigate stress-distribution effects. However, only interim results can be reported at this time. Three-point bend testing for a single span width has been completed to date for specimens with thicknesses of 12 and 6 mils, and partial results are available for 3-mil specimens. The experimental strength data are plotted in Fig. 1, and results of a Weibull analysis on these data are summarized in Table 1. Strength ratios for the three different test volumes are compared to Weibull predictions in Table 2. It is observed from Fig. 1 that the strength distributions for the 6-mil and 12-mil specimens have essentially the same shape, as verified by the closeness of their Weibull moduli (Table 1), but that the entire distribution for the thinner specimens is shifted upward in strength by an average amount that agrees well with the 4-ksi difference in mean values for the two distributions. The Weibull parameters which best characterize both of these curves are given by: m=6.0 and σ_0=2.9 ksi·in.$^{3/m}$, where these values have been determined to best fit (in the sense of maximum likelihood estimates) the data of Fig. 1 with the three-point-bend survival probability $S=\exp[-V(\sigma/\sigma_0)^m/2(m+1)^2]$.

The partial data for the 3-mil specimens (Fig. 1) were not sufficient for use in these distribution determinations, but once such parameters were determined then the still higher mean strength (Table 1) for these thinnest specimens **was** useful for comparison with Weibull predictions of the various strength ratios (Table 2). It is observed from these comparisons that the Weibull volume term gives good agreement with experiment for glassy carbon, as was previously found to be the case for structural composites reinforced with carbon fibers having small crystallite sizes [15]. Finally, based on results of this study, Weibull theory was applied to calculate the tensile strength expected for a 1cm gauge length of 25μm glassy carbon fiber having the same modulus ($E=4\times10^6$psi) as the bulk material investigated, and the comparison with experiment [16] is very good (Fig. 2).

References

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Table 1. Three-Point Bend Data for Glassy Carbon

	Specimen Thickness		
	12 mil	6 mil	3 mil
Number Specimens	24	24	3
Mean Strength (ksi)	27.86	31.86	36.12
1 Stdard Deviation (ksi)	5.38	6.28	6.54
Test Volume (10^{-5} in.^{3})	9.90	4.58	2.40
Weibull Modulus (m)	6.00	5.90	6.46
σ_0 Parameter (ksi. in.$^{3/m}$)	2.98	2.85	3.60

Table 2. Weibull Predictions Versus Measurement

Strength Ratios	Theory	Experiment
σ(3 mil)/σ(6 mil)	1.11	1.13
σ(6 mil)/σ(12 mil)	1.14	1.14
σ(3 mil)/σ(12 mil)	1.27	1.30

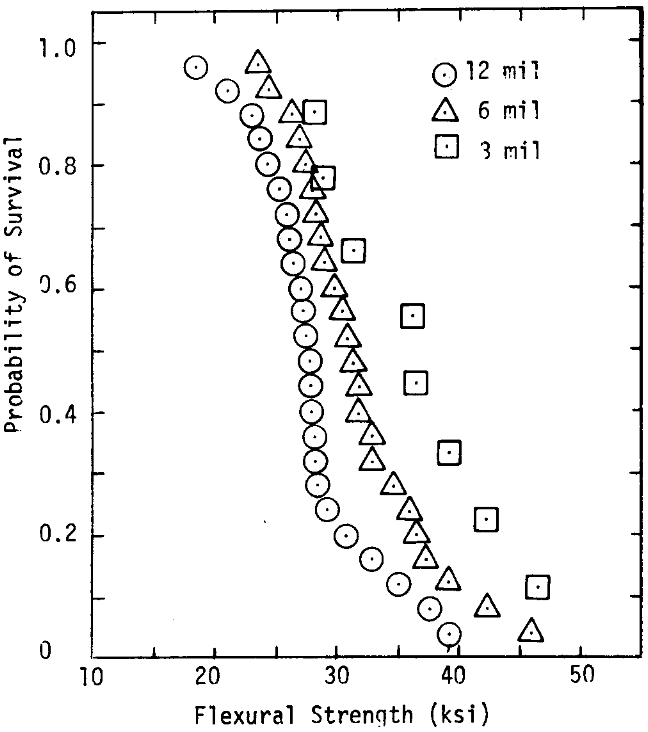

Fig. 1. Strength distributions for glassy carbon.

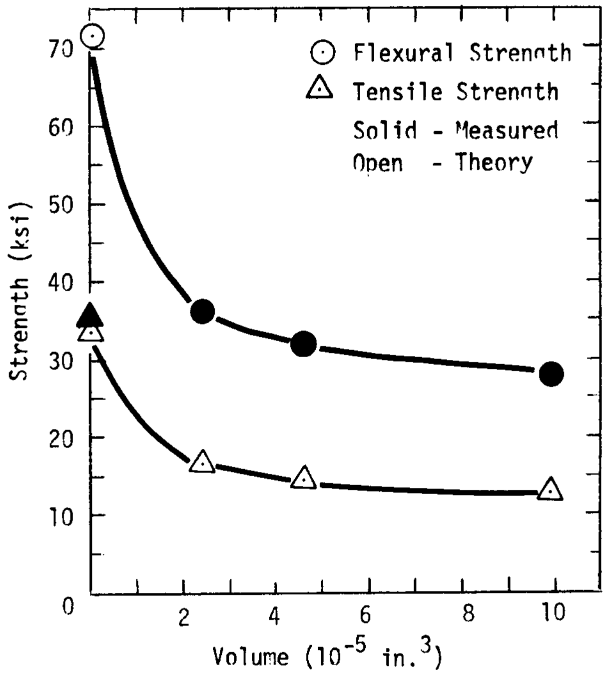

Fig. 2. Size effects on strengths of glassy carbon.