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New Perspectives on the Fracture of Nicalon Fibers

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NEW PERSPECTIVES ON THE FRACTURE OF NICALON FIBERS

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ABSTRACT: Experimental studies have been conducted to examine the strength and fracture behavior of monofilament Nicalon³ SiC fibers ranging in diameter from 8 to 22 μm . The effects of varying fiber diameter, flaw location and flaw population on the tensile behavior of individual fibers were investigated using fractography. Results indicate that variations in fiber diameter influence the apparent fiber fracture toughness (K_{Ic}), with higher K_{Ic} values observed for decreasing fiber diameters. Observations also suggest that the location of the critical flaw may play a role in the fracture of Nicalon fibers. In addition to surface flaws, three distinct internal flaw populations are seen to cause fracture in Nicalon fibers.

KEYWORDS: Nicalon, SiC fibers, fracture, fracture toughness, fractography

Nicalon SiC fibers represent attractive reinforcement material for elevated-temperature structural components due to their high strength, high Young's modulus, excellent resistance to oxidation, and broad commercial availability. As such, there exists a critical need to examine and understand the mechanical behavior of these fibers in order to develop improved high-temperature composites. Determining the strength and fracture behavior of Nicalon fibers at ambient conditions is particularly important, as these

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findings provide a fundamental understanding of the various parameters and processes that contribute to fiber fracture.

Ceramic fibers derived from polymeric precursors and processed via spinning techniques typically show a broad range of fiber diameters within a given tow of fibers. Previous studies have documented this behavior for Nicalon [1-8] as well as other ceramic fibers [9]. Given the broad distribution of fiber diameters which may exist in a fiber tow, the possible effects of varying fiber diameter on fracture behavior demand serious consideration. To date, however, no such studies have been performed with these goals in mind.

This work has been conducted in an attempt to identify the effects of varying fiber diameter on fracture of Nicalon fibers at ambient conditions. Extensive fractographic analysis is used to correlate specific fracture parameters, such as the fracture mirror size and the critical flaw size, with tensile strength data for individual fibers obtained from the same tow, but varying significantly in diameter. These data are used to characterize the apparent fiber fracture toughness with respect to fiber diameter.

The effects of critical flaw location and individual flaw populations on strength and fracture properties of the fibers are also addressed.

Experimental Procedure

Individual ceramic-grade Nicalon SiC fibers with PVA sizing were glued at the ends onto sandpaper mounts. Fibers were selected from varying regions of the tow to maximize sampling volume. Mounted fibers were mechanically secured into an alignment jig which was then attached to the grips of a micro-tensile test machine. Fibers were loaded in tension to fracture at a constant displacement rate of 0.3 mm/min. Load-displacement curves were recorded, and the peak load and measured cross-sectional area were used to calculate fiber strength.

Fibers were strength-tested at three different gauge lengths (10, 25 and 50 mm) in mineral oil, with data sets for each gauge length consisting of approximately 50 individual fiber tests. Tests conducted in mineral oil permit consistent recovery of the fractured fiber ends for fractographic analysis on a Scanning Electron Microscope (SEM). Tested fibers were recovered as two distinct fractured ends and mounted onto aluminum stubs in matched pairs with the fracture surfaces facing up. Fibers breaking outside of the gauge length, or at the extreme ends of the fiber, were discarded. A field-emission SEM operated at 2kV accelerating voltage was used to measure fiber diameter and to examine the fracture surface characteristics of each fiber. When possible, the fracture mirror size, critical flaw size and normalized flaw location (defined as the radial distance of the critical flaw from the fiber center, divided by the fiber radius)

FIG. 1--Distribution of fiber diameters for total sample population

were determined for each fiber.

Results

Fig. 1 shows the distribution of fiber diameters for all samples tested in this study. Individual fiber diameters vary significantly within the tow, ranging from approximately 8 to 22 μm .

FIG. 2--Characteristic fracture features for a) surface and b) interior flaws

Figs. 2a and 2b show typical fracture surface features for fibers failing due to surface and interior flaws, respectively. The characteristic mirror, mist and hackle features are clearly evident in these micrographs. Fractographic analysis of the fractured fibers reveals that interior flaws can be grouped according to three distinct flaw populations: individual pores, pore clusters, and granular defects. Representative SEM micrographs of each flaw type are presented in Fig. 3. Pore clusters (Fig. 3b) represent collections of small, individual pores. Granular defects (Fig. 3c) can be classified as aggregates of small granular particles, which have previously been shown to be richer in carbon than the bulk fiber [6].

FIG 3--Critical flaws in the form of a) individual pore, b) pore cluster, and c) granular defect

A dependence of strength on fracture mirror size is common in many glass and polycrystalline ceramic materials, and is characterized by the empirical relation

$$\sigma_f \sqrt{r_m} = A_m \quad (1)$$

where σ_f is the tensile stress at fracture, r_m is the mirror radius as measured on the fracture surface, and A_m is the mirror constant which is related to the fracture toughness of the material. Strength is plotted as a function of the reciprocal square root of the mirror

FIG. 4--Strength dependence on the reciprocal square root of the mirror radius.

radius ($r_m^{-1/2}$) in Fig. 4 for all fibers tested in mineral oil. The slope of this plot yields a mirror constant value of $2.51 \text{ MPa m}^{1/2}$, which is consistent with A_m values of $2.51 \text{ MPa m}^{1/2}$ [10], $2.0 \text{ MPa m}^{1/2}$ [6], and $1.81 \text{ MPa m}^{1/2}$ [4] reported previously for Nicalon fibers.

As Fig. 5 illustrates, a linear relationship exists between the critical flaw size and the mirror radius for SiC fibers. The flaw-to-mirror size ratio obtained here (0.19) is very similar to that reported previously (0.22) for Nicalon fibers [11]. Empirical evidence

relating mirror size and flaw size is critical to the study of fracture processes in fibrous materials, as mirror zone size measurements can ultimately lend insight to the critical flaw dimensions of brittle fibers when the flaws cannot be directly identified.

The fracture mirror constant (A_m) and fracture toughness (K_{Ic}) can ultimately be related through the Griffith-Orowan-Irwin relation

$$\sigma_f \sqrt{a_c} Y = (E \gamma_c)^{1/2} \quad (2)$$

where a_c is the critical flaw radius, Y is a geometry factor which is dependent on the flaw size and shape and the specimen geometry, E is the Young's modulus, and γ_c is the critical fracture energy. The geometry factor Y is presumed to be a function of both the flaw-to-fiber-size ratio (a_c/r) and the flaw location (λ) within the fiber. Substituting $0.19r_m$ for a_c , and taking

$$(E \gamma_c)^{1/2} = K_{Ic} \quad (3)$$

the modified equation now yields

$$\sigma_f \sqrt{r_m} Y^* = K_{Ic} \quad (4)$$

where $Y^* = \sqrt{0.19} Y$, and Y^* is now a function of r_m/r . Invoking Eq. 1, we see that

$$A_m Y^* = K_{Ic} \quad (5)$$

and the relationship between mirror constant and fracture toughness is established. In practice, Y should be independent of the flaw size if $a_c/r \ll 1$, so that a dependence of Y^* on r_m/r is not expected. Because no direct measurements of Y^* or K_{Ic} have been made for

FIG. 5--Linear relationship between critical flaw radius and fracture mirror radius.

glass or ceramic fibers of such small dimensions ($d \approx 15 \mu\text{m}$), the significance of Eq. 5 relates to its ability to predict these factors from other experimental data.

Fig. 6a is a plot of the measured mirror constant versus fiber diameter. Despite appreciable scatter in the data, the mirror constant appears to increase with decreasing fiber diameter. A t test was conducted on the data in Fig. 6a to better evaluate the apparent trend, and the data is shown to have a non-zero slope at a confidence level of 99%. The calculated slope is negative, suggesting that the mirror constant does, in fact, increase with decreasing fiber diameter. Fig. 6b shows the relationship between the mirror-to-fiber-size ratio (r_m/r) and the fiber diameter, demonstrating that this ratio does not vary with diameter. The critical flaw location is plotted as a function of fiber diameter in Fig. 6c. Flaw location is best characterized as a normalized parameter, λ , which is defined as the distance from the critical flaw center to the fiber center, divided by the fiber radius. (Using this convention, the fiber center is defined as $\lambda=0$, while the fiber surface is represented by $\lambda=1$). Although an obvious trend is

FIG. 6--a) mirror constant, b) mirror-to-fiber size ratio, and c) critical flaw location versus fiber diameter

not apparent in the plot, the data suggests that λ either increases slightly with decreasing diameter, or remains constant. Specifically, λ does not decrease with decreasing diameter. If λ is constant with diameter, then the trends observed in Fig. 6 would suggest that the apparent fiber K_{Ic} increases with decreasing fiber diameter. If λ increases slightly with decreasing diameter, and we invoke the relationship established by Kobayashi *et. al* [12] stating that Y^* increases with increasing λ , then Y^* must increase with decreasing diameter, and consequently K_{Ic} should increase with decreasing diameter with a stronger trend than that observed for A_m in Fig. 6a.

Fig. 7 examines the effects of critical flaw location on fiber fracture. Fracture strength is plotted as a function of flaw location in Fig. 7a. Fibers that fracture due to flaws located near the center of the fiber show lower average strengths than those failing due to outlying flaws, with mean strength increasing as normalized distance from fiber center increases. It is also suggested that centrally-located flaws yield less scatter in

strength than flaws existing closer to the surface. These apparent trends are in marked contrast to that observed between mirror constant and flaw location as shown in Fig. 7b. Here, the mirror constant is shown to remain constant with varying flaw location, suggesting that the ratio of K_{Ic} to Y^* is unaffected by changes in flaw position.

FIG. 7--a) strength and b) mirror constant as a function of normalized (critical) flaw location.

Discussion

Experimental results obtained in this study suggest that variations in both fiber diameter and flaw location may affect the fracture behavior of Nicalon fibers. We have concluded from Fig. 6 that the apparent K_{Ic} increases with decreasing diameter for Nicalon fibers. One possible explanation may be that smaller fibers possess intrinsically higher densities, greater purities and thus more homogeneous microstructures, and may therefore have higher K_{Ic} values. Based upon findings by Yajima and co-workers [13], which showed a trend for increasing Young's modulus with decreasing fiber diameter, Andersson and Warren [1] suggested that fibers of higher purity SiC can be achieved at smaller diameters, possibly as a result of a more effective pyrolysis.

The scatter observed in Fig. 6a may also be related to the presence of different flaw populations and to variations in critical flaw location, both of which can contribute to data scatter and thus affect mirror constant values. Similarly, the moderate scatter observed in Fig. 6b can be attributed to several sources. The determination of Y^* for fracture in a particular sample depends not only on the geometry and size of the specimen, but also on the size and location of the flaw. For bulk specimens of standard geometry and pre-determined flaw shape, calculation of Y^* is a fairly straight-forward procedure as the flaw is assumed to be in a fixed location and to possess a known geometry. These assumptions, however, do not apply to the fracture of Nicalon fibers. As fractography of the fibers demonstrates, flaws do not occupy a fixed location, nor do they conform to a fixed geometry. Furthermore, deviations from circular cross-sections in Nicalon fibers result in a variety of different effective specimen geometries which further contribute to the scatter in Y^* observed in Fig. 6.

Contrary to results reported previously [6] for fracture of Nicalon fibers, none of the *interior* flaw populations examined in this study correlate to specific strength ranges, nor do they exhibit specific strength-flaw size relationships. Rather, as Fig. 8 suggests, fiber strength appears to be more dependent on flaw size than on flaw type, as all of the

FIG. 8--Strength versus critical flaw size for all fibers tested. Fiber strength shows a greater dependence on flaw size than on flaw type.

flaw populations contribute to fracture over a broad spectrum of strength values. In Fig. 9a, surface flaws are shown to be prevalent at small fiber diameters due to the high surface-to-volume ratio of the fiber. Fig. 9b reveals that surface flaws are also preferentially sampled over interior flaws at longer gauge lengths, where statistically weaker flaws dominate failure. Additionally, fractography shows that the three interior flaw populations tend to occupy preferred locations in the fibers. This behavior is illustrated in Figs. 10, 11, and 12, where statistical

calculations are used to obtain the accumulative flaw distribution (F), the associated flaw distribution (f), and the critical flaw density (p) as a function of critical flaw location (λ) for individual pores, pore clusters and granular defects, respectively.

FIG. 9--Percentage of fibers failing due to surface flaws as a function of a) fiber diameter, and b) fiber gauge length.

The statistical distribution of flaw location for each of the three populations can be inferred from Figs. 12-14. For each flaw population, individual flaws are ranked in order of increasing λ . The accumulative flaw distribution function is obtained by plotting the normalized flaw locations in a given population, and then fitting the data to a power law of the form

$$F(\lambda) = A\lambda^n \quad (6)$$

where A and n are empirical constants. These curves are shown in part (a) of Figs. 12-14. These figures demonstrate that very few critical flaws are located close to the fiber center, as would be expected given the small volume that a flaw could potentially occupy if positioned in close proximity to the fiber center.

FIG. 10

FIG. 11

FIG. 12

FIGS. 10-12 -- The a) accumulative flaw distribution and b) the associated flaw distribution (solid line) and flaw density (dashed line) plotted against the normalized flaw location for individual pores (FIG. 10), pore clusters (FIG. 11) and granular defects (FIG. 12).

The associated flaw distribution is determined from the first derivative of $F(\lambda)$:

$$f(\lambda) = nA\lambda^{n-1} \quad (7)$$

Physically, $f(\lambda)$ describes the distribution of flaws contained within an element ($d\lambda$) of the fiber volume, as illustrated in Fig. 13. Dividing $f(\lambda)$ by $2\pi\lambda$ yields the critical flaw density

$$p(\lambda) = \frac{nA\lambda^{n-2}}{2\pi} \quad (8)$$

which represents the critical flaw distribution normalized to a specific volume of the fiber (see Fig. 13). This function, when integrated over the entire locus of normalized flaw location, yields $F(\lambda)$:

$$\int_0^\lambda p(\lambda) 2\pi\lambda d\lambda = F(\lambda) \quad (9)$$

Both $f(\lambda)$ and the critical flaw density $p(\lambda)$ are plotted as a function of critical flaw location in part (b) of Figs. 10-12. These plots yield some intriguing results regarding the dependence of different flaw populations on critical flaw location.

Individual pores can be found throughout most of the fiber domain, and are primarily responsible for fracture

occurring due to flaws located near the center of the fiber. The solid line in Fig. 10b indicates that individual pores which serve as critical flaws remain equally distributed over the entire range of normalized fiber distance. The density of these critical flaws (dashed line) is highest at the fiber center ($\lambda=0$) and is seen to decrease with increasing λ . Critical flaws in the form of pore clusters and granular defects are found predominantly at larger values of λ . The critical flaw densities (dashed lines) are shown to increase with increasing λ for both pore clusters and granular defects.

The demonstrated propensity for individual interior flaw populations to occupy select regions of the fiber may partially explain the trends observed in Fig. 7a, which shows the dependence of fracture strength on critical flaw location. Average strength values, as well as the overall scatter in strength, are seen to increase as the critical flaw location moves further away from the fiber center ($\lambda=0$). Although the variation in strength with flaw location could possibly be explained by changes in critical flaw size and fiber gauge length, another explanation is offered by the dependence of individual flaw populations on critical flaw location. Close to the fiber center, only one type of critical flaw is likely to exist -- the individual pore -- and at low λ values, this population is shown to have very little variation in strength values. However, moving further away

FIG. 13--Schematic view of fiber cross-section depicting physical parameters used to characterize flaw population.

from the fiber center, all three interior flaw populations could potentially serve as fracture initiators, which may explain why the scatter in strength becomes so large with increasing λ .

Conclusions

The strength and fracture behavior of Nicalon fibers exhibiting a broad distribution of diameters have been characterized using extensive fractographic analysis. Results suggest that variations in both fiber diameter and flaw location can influence the mechanical response of fibers fractured under tensile loading. As fiber diameter decreases, the apparent fiber fracture toughness (K_{Ic}) appears to moderately increase. Tensile strength is shown to vary with flaw location, with higher strengths accompanying flaws located further away from the fiber center. Larger scatter in strength values is also observed with increasing distance from fiber center. This scatter has been attributed primarily to variations in critical flaw population with varying flaw location. Individual pores are seen to be distributed throughout the fiber domain, and are primarily responsible for fracture occurring due to flaws located closest to the fiber center. Pore clusters and granular defects are located predominantly in the outer regions of the fiber. The individual flaw populations are not observed to correlate to specific fiber strength values, suggesting that flaw size, and not flaw type, plays a more prominent role in determining fiber strength values.

This work is expected to have relevant implications to other types of ceramic fibers derived from polymeric precursors which are likely to possess inherently broad distributions of fiber diameters.

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