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Time/Temperature Dependent Tensile Strength of SiC and Al_2O_3-Based Fibers

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Abstract

In order to understand and model the thermomechanical behavior of fiber-reinforced composites, stress-rupture, fast-fracture, and warm-up rupture studies were conducted on various advanced SiC and Al_2O_3-based fibers in the temperature range from 20 to 1400 °C in air as well as in inert environments. The measured stress-rupture, fast fracture, and warm-up rupture strengths were correlated into a single master time/temperature-dependent strength plot for each fiber type using thermal activation and slow crack growth theories. It is shown that these plots are useful for comparing and selecting fibers for CMC and MMC reinforcement and that, in comparison to stress rupture tests, the fast-fracture and warm-up tests can be used for rapid generation of these plots.

Introduction

Substantial interest in continuous fibers based on silicon carbide and aluminum oxide has developed because of their potential reinforcement of high temperature composites for structural components in advanced gas turbine engines, such as Si-based ceramic matrix composites (CMC) for combustor and turbine components and Ti-based metal matrix composites (MMC) for fan and compressor components. Based on performance requirements, small diameter multifilament fibers are preferred for CMC, and large diameter monofilaments for MMC [1].

Currently, for CMC reinforcement, small diameter SiC fibers derived by polymer pyrolysis possess high stiffness, high room temperature strength, and high thermal stability. The recently developed Dow Corning fiber (nearly stoichiometric beta-SiC) and the Hi-Nicalon fiber (beta-SiC with excess carbon and trace oxygen) are leading candidates because they have been observed to display creep and rupture behavior superior to that of the first-generation high-oxygen containing Nicalon SiC fiber [1-3]. In a similar manner for Al_2O_3-based fibers, the new Nextel 720 fiber is more creep and rupture resistant than the older Nextel 610 fiber. These results were obtained using as-produced single fibers removed from multifilament tow [4,5]. Likewise for MMC use, newly developed large diameter chemically vapor deposited (CVD) monofilaments, like the developmental SCS-X or Ultra-SCS fibers [6], possess higher strength, and higher creep and rupture strength than the earlier version, SCS-6 monofilament. The single crystal alumina-based monofilament, like Saphikon, may also have MMC potential.

Since the structural performance of high temperature composites relies mainly upon the fiber thermomechanical performance, understanding the time and temperature dependent strength behavior of the fibers is essential. Therefore, the objectives of this study were (1) to measure the time/temperature-dependent fracture behavior of SiC and Al_2O_3-based fibers of current technical interest, (2) to develop simple tests, theories, and methodologies to calculate and describe the measured properties, and (3) to compare the fracture strength behavior of the various fibers.

Experimental Procedure

Two types of SiC multifilament and two types of SiC monofilament fibers were studied: as-produced Hi-Nicalon and Nicalon (Ceramic Grade) fibers from Nippon Carbon; and as-produced Ultra-SCS and SCS-6 (both contain similar C-rich double layer coatings) from Textron Specialty Materials. For the Al_2O_3-based fibers, multifilament fibers and one type of monofilament were studied: as-produced Nextel 610 and Nextel 85-15 (early version of Nextel 720) fibers from 3M, and single crystal C-axis alumina monofilament from Saphikon. As reported elsewhere [4], the fiber stress-rupture strength behavior was measured by recording creep deformation versus time at a constant deadweight load using a ~25mm and 100mm hot zone length for two air furnaces and a 115mm hot zone for an argon furnace. The warm-up rupture test [3] is a similar test to the stress-rupture test, but it is conducted by heating a pre-loaded fiber at a constant heating rate using the air or argon furnace chamber and then measuring the rupture temperature. The third test on the fibers was a conventional fast fracture tensile test in a 25mm hot zone furnace in air using a constant displacement rate of 1.25 mm/min.

Result and Discussions

STRESS-RUPTURE STRENGTH:

Fig. 1-a shows typical creep strain vs. time curves for the Hi-Nicalon and other SiC fibers at 1400 °C in air. Stress-rupture curves for the Hi-Nicalon fibers are shown in Fig.1-b at 900 C to 1400 °C in air. From 1200 to 1400 °C, rupture strength vs. time curves were nearly independent of hot zone lengths from 25 to 100 mm, suggesting a high Weibull modulus for fiber fracture and that the controlling mechanism was related with creep-induced cavitation. At low temperatures, like 900 °C, the strength degradation rate was so small that the stress-rupture exponent N was ~ 25 in the rupture time relation: $t \sim \sigma^{-N}$. This high number is similar to those N values typically reported to be related with slow crack growth in monolithic ceramics [8,9]. With increasing testing temperature, the strength degradation rate increased, so that the N value at 1400 °C was ~ 3.

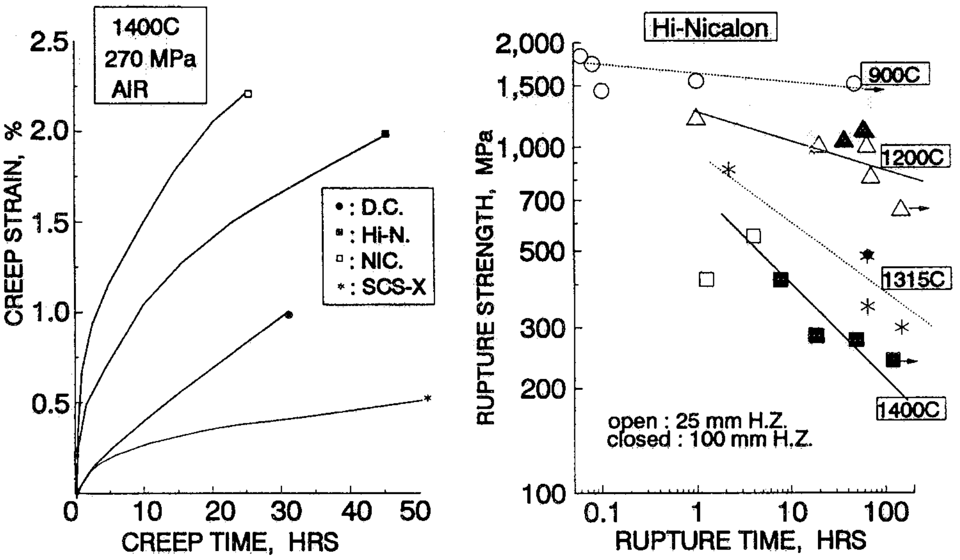


Fig. 1 Representative (a) creep (b) rupture curves for as-produced SiC fibers in air.

As discussed elsewhere [7], the decrease in N value as a function of temperature indicates that the fiber rupture is a thermal activated process, thereby allowing the use of general thermal activation theory. For example, in a stress rupture test under constant stress (σ), fiber rupture time (t) and temperature (T) are assumed to be related by $\Theta(\sigma) = t \exp[-Q(\sigma)/RT]$, where Θ is the activation time parameter and Q is the effective rupture activation energy. Thus average fiber rupture strength $S(=\sigma)$ can be described by

$$S = S(t,T) = S(\Theta,Q). \tag{1}$$

For the rupture of SiC and Al_2O_3-based fibers, within the data scatter the parameter Θ was observed to be constant (Θ_0) and independent of stress conditions, so that average rupture strength can be described as a function only of q, i.e.

$$S = S(q) \tag{2a}$$

where

$$q = Q/(2.3R) = T(\log t + D) \tag{2b}$$

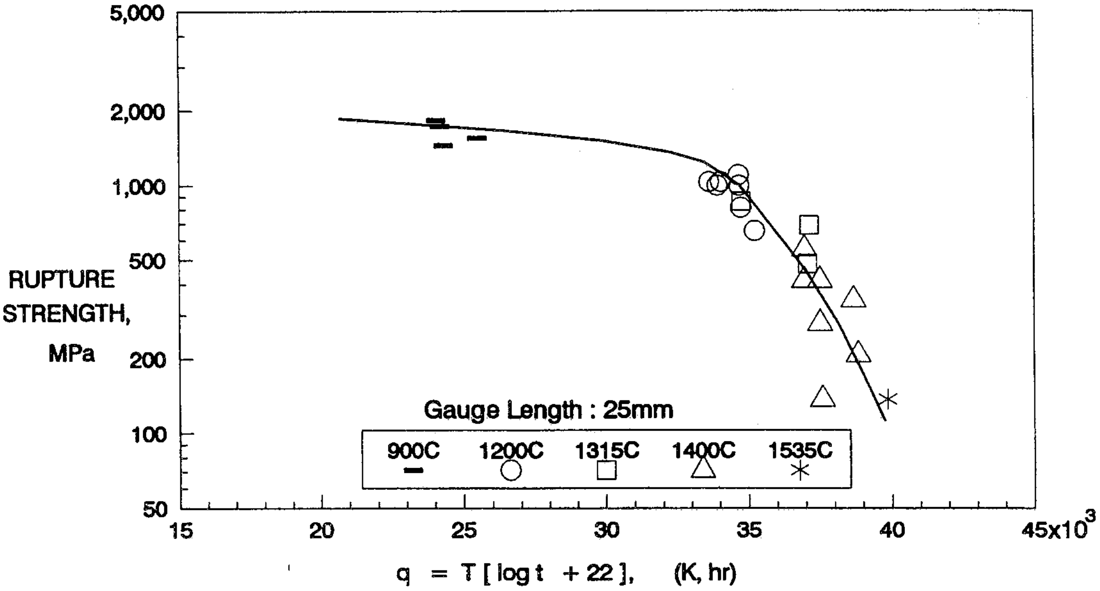


Fig. 2 Thermal activation plot for Hi-Nicalon rupture strength in air.

Here $D = \log(\Theta_0) = 22$, a value which best fits the SiC and Al_2O_3-based fiber results in the high temperature range. Fig. 2 shows how the application of thermal activation theory allows condensation of all the stress rupture data for the Hi-Nicalon fiber into a single master curve for average fiber strength. For Fig. 2, fibers are in their as-produced condition; the environment is air; and the hot zone length is 25 mm. This plot is typical of monolithic fracture where fracture below a given q value (~32000 for Hi-Nicalon) is generally accepted as due to slow crack growth of as-produced flaws in the fiber and fracture above this q value is generally attributed to the growth of new flaws due to creep-induced cavitation.

FAST FRACTURE STRENGTH:
To expand the Fig. 2 thermal activation plot to low q values, fiber fast-fracture strengths were determined vs. temperature. The equivalent stress rupture time t* in the T-A plot for the fast fracture strength results can be estimated assuming that the fast fracture occurs by slow crack growth. Based on slow crack growth theory,

$$t^* = t_{FF} / (1+N) \qquad (3)$$

where t_{FF} is fast fracture time and (1/(N+1)) is the slope of log (average strength) vs. log (stressing rate). For typical fiber fast fracture tests, t_{FF} ~30 sec for a 1.25mm/min displacement rate and N ~30. Thus it is assumed that the average fiber fast fracture strength data can be plotted at the static fatigue or rupture time t* ~1 sec. or q = 18.3 T. The Hi-Nicalon T-A plot including the fast fracture strength values (solid points) is shown in Fig. 3 for the fibers of this study. The fast fracture strength values from room temperature to 1200 °C are on the same line as the stress-rupture strength values measured at 900 °C. Also shown in Fig. 3 are the fast-fracture results of Bodet et. al. [10] for a stronger Hi-Nicalon fiber. The results of Fig. 3 suggest that the slow crack growth of as-produced flaws is the probable strength degradation mechanism below 900 °C for stress-rupture and up to ~1200 °C for fast fracture.

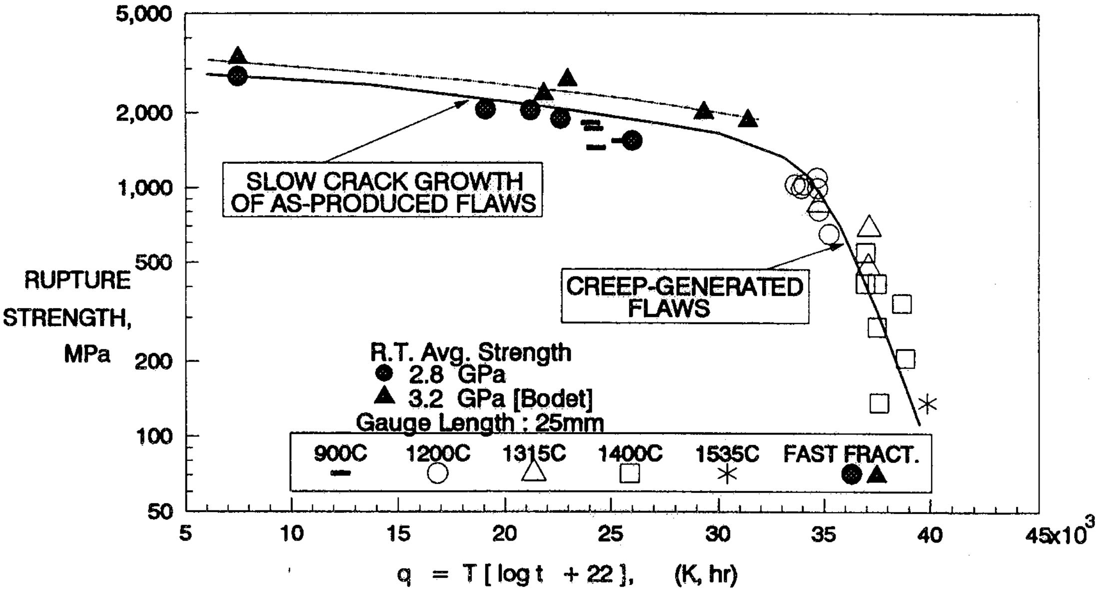


Fig.3 Thermal activation plot for Hi-Nicalon fast-fracture and rupture strength in air.

WARM-UP RUPTURE STRENGTH:
The warm-up rupture strengths as a function of temperature are shown in Fig. 4 for the Hi-Nicalon fibers in air and argon. These data cover the stress range from ~50 to 1600 MPa and temperatures from 1000 to 1700 °C. Approximately 1600 MPa is a proof stress for the Hi-Nicalon fibers at room temperature with a grip-to-grip length of ~250mm. Below ~1600 MPa, the rupture is related with temperature dependent slow crack growth or creep cavitation during the warm-up experiment. In air, the rupture temperatures for the 25 mm (open symbol) hot zone or the 100 mm (closed symbol) hot zone were higher than those in argon (~115mm hot zone). The higher strength in air was similarly observed in the stress-rupture strength [4].

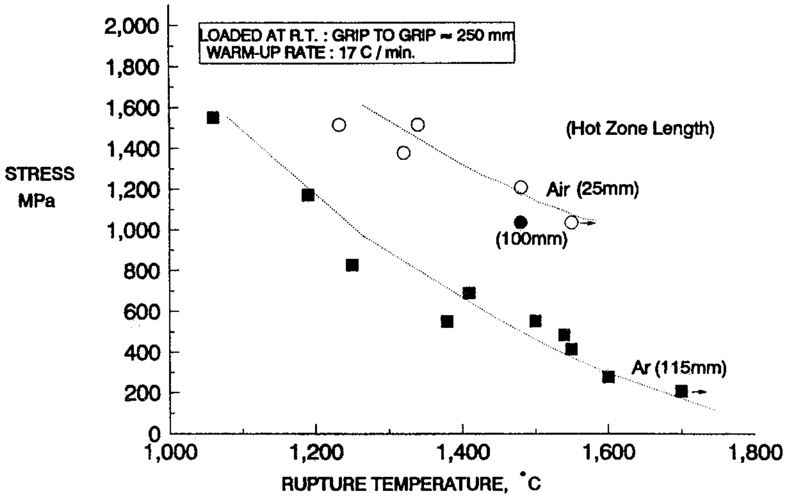


Fig. 4 Warm-up rupture strength for Hi-Nicalon fiber in air and argon.

Like fast fracture, the warm-up tests are easy and rapid in comparison to the stress-rupture tests. To correlate the warm-up strength results with the rupture data, it was assumed that if at a given warm-up rate (α), fiber fracture occurs at a temperature (T) for a deadweight load (P), then the average rupture time t** for a conventional stress-rupture tests at same load (P) and constant temperature (T) will be given by $t^{**} = A / \alpha$. The constant (A) can be determined by fitting the temperature-dependent warm-up strength data at a given (α) to existing T-A plots. This approach yields t** ~ 1min for a 17 °C/min warm-up rate; i.e. $q_{WR} = 20.2$ T. The combined T-A plot for the three types of tests is shown in Fig. 5 for the Hi-Nicalon fiber in air and argon. Clearly there is excellent strength agreement between these tests, suggesting that one or more of the tests can be used to generate T-A plots for a particular fiber type. Fig. 5, as well as Fig. 4, shows also that the Hi-Nicalon behavior is better in air plausibly due to the formation of a SiO_2 overlayer which slows down decomposition.

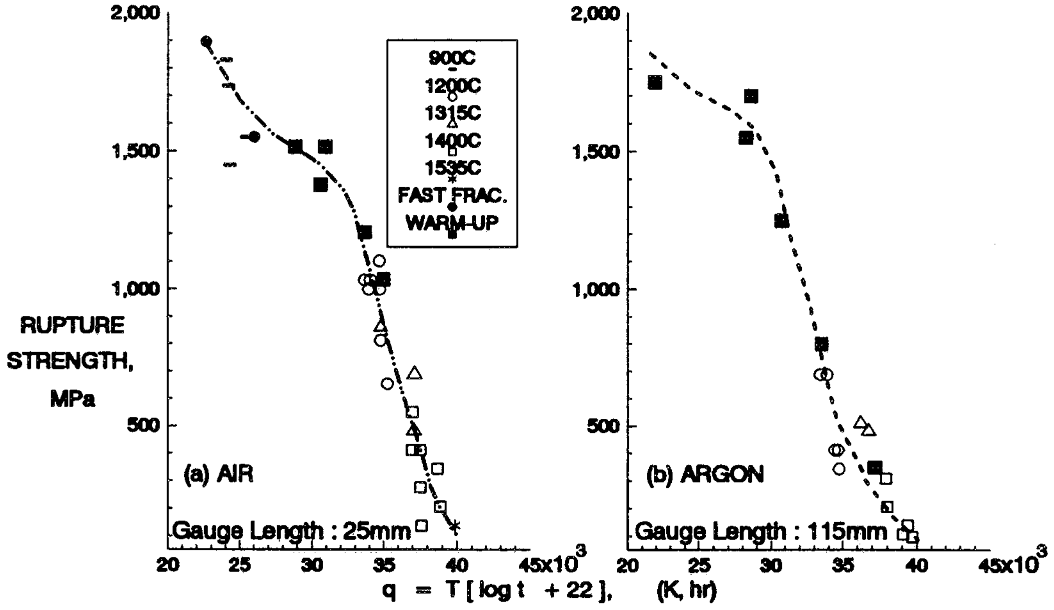
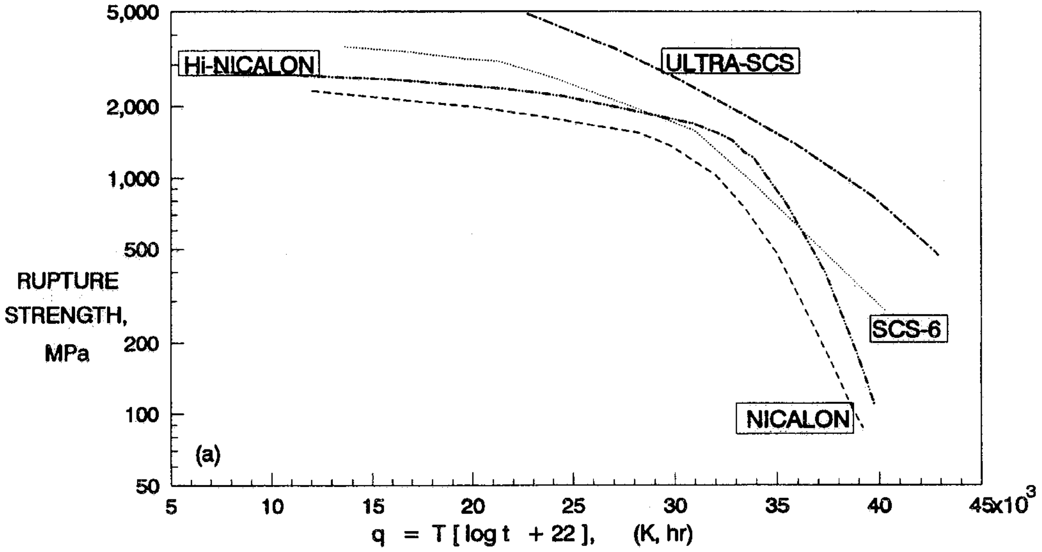


Fig. 5 Thermal activation plot for Hi-Nicalon fiber strength in (a) air and (b) argon.

COMPARISON OF T-A PLOTS FOR SiC FIBERS:

As with Hi-Nicalon, T-A plots for other SiC and Al_2O_3 fibers (ceramic grade Nicalon, SCS-6, Ultra-SCS, Nextel 610, Nextel 85-15, and Saphikon) were generated using D = 22 for the three types of strength tests. The best fit master curves for the various SiC fibers are shown in Fig. 6-(a), with the Nicalon and Hi-Nicalon tested in air and the SCS-6 and Ultra-SCS in inert environments, and for the Al_2O_3-based fibers in Fig. 6-(b), with the Nextel 610 and 85-15 tested in air, and the Saphikon fiber in inert environment. At q ~ 30000, the vertical line separates the SiC curves into two regions; one related with slow crack growth, and the other with cavity growth. The boundary between two regions for the polycrystalline Al_2O_3 was as low as q ~ 20000. Most long-time high-temperature applications are basically in the region of cavity growth. At a q value of 37000, ~1000 hours at 1200 °C, the CVD-processed Ultra-SCS and SCS-6 have better strength capability than the polymer-derived Hi-Nicalon and Nicalon SiC fibers. However at q ~32000, ~1000 hours at 1000 °C, the Hi-Nicalon fibers have better strength capability than the CVD SCS-6. For the Al_2O_3 fibers, the Nextel 85-15 possessed higher strength and temperature capability than the Nextel 610, and the Saphikon indicated a superiority at higher temperatures.



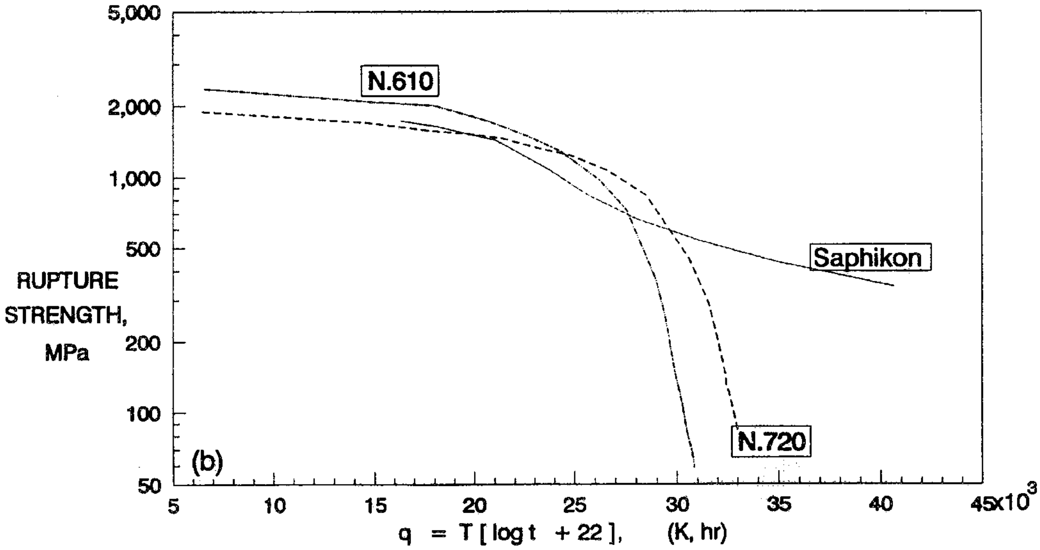


Fig. 6 Thermal activation plots for average strength of as-produced (a) SiC fibers and (b) Al_2O_3-based fibers at a gauge length of ~25 mm.

Summary and Conclusions

The time/temperature-dependent strength behavior of various SiC and Al_2O_3-based multifilament and monofilament fibers were measured using stress-rupture, fast fracture, and warm-up rupture tests. The strength results were correlated into master thermal activation plots using thermal activation and slow crack growth theories. These plots allow fiber comparison across a wide time/temperature range; for example, the Ultra-SCS and Hi-Nicalon fibers have superior strength capability than the SCS-6 and Nicalon fibers, respectively. Beside allowing fiber comparison, the development of thermal activation plots for each fiber provides a simple method to predict strength behavior outside of the data set, to understand generic and specific mechanisms controlling flaw growth, and to predict the behavior of high temperature composites [11]. The good agreement between the strength data provided by the long-term stress rupture test and the data provided by the short-term fast fracture and warm-up rupture tests suggests that these latter two tests can be used for rapid generation of master strength plots for newly-developed fibers.

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