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High Temperature Structural Fibers—Status and Needs

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HIGH TEMPERATURE STRUCTURAL FIBERS—STATUS AND NEEDS*

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ABSTRACT

The key to high temperature structural composites is the selection and incorporation of continuous fiber reinforcement with optimum mechanical, physical, and chemical properties. Critical fiber property needs are high strength, high stiffness, and retention of these properties during composite fabrication and use. However, unlike polymeric composites where all three requirements are easily achieved with a variety of commercially available carbon-based fibers, structural fibers with sufficient stiffness and strength retention for high temperature metal and ceramic composites are not available. The objective of this paper is to discuss in a general manner this thermomechanical stability problem for current high performance fibers which are based on silicon and alumina compositions. This is accomplished by presenting relevant fiber property data with a brief discussion of potential underlying mechanisms. From this general overview, some possible materials engineering approaches are suggested which may lead to minimization and/or elimination of this critical stability problem for current high temperature fibers.

INTRODUCTION

With the performance advantages demonstrated for fiber-reinforced polymeric composites at ambient and near ambient temperatures, there is currently significant activity in government and industry aimed at developing metallic and ceramic composites which can extend these advantages to much higher temperatures. This is especially the case in the aerospace industry where there is a strong need for structural composites which cannot only provide specific strength properties better than conventional aluminum, titanium, and nickel-based alloys, but also operate at higher use temperatures. Prime aerospace applications expected to significantly benefit from these new materials are hypersonic airframes and advanced propulsion systems, such as gas turbine engines where composite materials are envisioned for essentially all of the hot section components. With their higher specific strength and operating temperatures, these metal matrix composites (MMC) and ceramic matrix composites (CMC) have the potential for not only reducing system operating costs, but also for improving system performance in terms of thrust, range, and mission life.

As in the case for low temperature composites, the key to high temperature structural composites is the selection and incorporation of continuous fiber reinforcement with the optimum mechanical, physical, and chemical properties. Although some of these properties such as fiber diameter and interfacial bonding depend on whether the matrix is metallic or ceramic (ref. 1), in general, as with polymeric composites, the critical fiber property needs are high strength, high stiffness, and retention of these properties during composite fabrication and use. However, unlike polymeric composites where all three requirements are easily achieved with a variety of

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commercially available carbon-based fibers, structural fibers with sufficient overall performance for high temperature composites are not available. That is, because of the poor oxidation resistance of carbon-based fibers above 400 °C (ref. 2), high temperature composite engineers have had to look to other fiber compositions. Although commercially available, these noncarbon fibers have been found to be lacking to various degrees in the key need of structural property retention, i.e., thermomechanical stability. Clearly, if fiber producers are to improve their products and high temperature structural composites are to achieve their potential, the underlying mechanisms for these instabilities must be understood. With this in mind, the objective of this paper is to discuss in a general manner the status for the thermomechanical instability problem in current high temperature structural fibers and, based on this discussion, to suggest some approaches that might be attempted to address this critical issue.

COMPOSITIONS FOR HIGH TEMPERATURE STRUCTURAL FIBERS

To put the current situation for high temperature fibers into perspective, it is important first to discuss three important factors that strongly influence the composite engineer's choice for fiber chemical composition. These factors, which are related to maintaining fiber strength within the composite environment, are (1) thermal stability, (2) service gas durability, and (3) matrix compatibility. That is, the engineer typically selects fiber compositions which during composite processing and use (1) have the potential for intrinsic physical and chemical stability, (2) do not significantly react with any expected environmental gas, and (3) are chemically and physically compatible with the anticipated matrix composition.

Because high temperature composites are generally fabricated at temperatures well above their anticipated use temperatures and because oxygen is the most aggressive service gas for most high temperature applications of practical interest, composite engineers and fiber producers have satisfied the first two needs by considering only fiber compositions that remain solid to above 2000 °C and are oxidation-resistant at composite service temperatures. Generally, oxidation-resistance implies compositions which react little with oxygen or in interaction with oxygen form a protective diffusion barrier oxide. Based on these criteria, compositional fiber choices can be currently narrowed down to two generic classes of ceramic fibers: silicon-based nonoxides and alumina-based oxides. In the presence of air environments, the Si-based compositions, such as SiC, form protective SiO_2 coatings which significantly inhibit oxygen diffusion to the fiber surface (ref. 2). Alumina, on the other hand, is not only stable in oxygen but also displays one of the best thermomechanical stabilities of all well-studied oxide compositions (refs. 3 and 4). Other property advantages for both compositions are low densities and high elastic moduli.

Based on the needs for thermal stability and oxidation resistance, essentially all current commercial fibers developed for high temperature application possess either Si-based or Al_2O_3-based compositions. These commercial fibers are summarized in table I together with some of their important properties. For convenience, the fibers are divided into six generic types based on primary composition and method of fabrication since these two factors typically result in fibers with similar mechanical, physical, and chemical properties. Also indicated in table I are approximate current cost per kilogram and typical trade names.

Matrix compatibility, the third factor influencing fiber composition selection, has both physical and chemical aspects. On the physical side, it is critical that the fiber composition have a coefficient of thermal expansion (CTE) very nearly equal to that of the matrix. Without this condition, internal residual stresses can develop in composites which, during thermal cycling, can severely degrade the fiber, matrix, or fiber/matrix interface. To avoid this problem for CMC, the fiber and matrix are often chosen to possess the same composition, e.g., SiC/SiC. For MMC, however, achieving an expansion match with the table I fibers is generally not possible because most metal matrices of high technical interest have CTE's greater than 10×10^{-6} °C^{-1}. This MMC problem has led to research for the identification and development of new fiber compositions with high CTE and to the implementation of such concepts as compliant fiber coatings which, by virtue of their plasticity, can serve to reduce high internal stresses at fiber-matrix interfaces (ref. 5).

Another important aspect of the matrix compatibility factor is the attainment of proper interfacial interaction between fiber and matrix so that sufficient mechanical load transfer is achieved. For MMC in which strong interfacial interaction is desirable (ref. 1), fiber and matrix compositions are often chosen which allow some chemical reaction between fiber and matrix at the temperatures needed for matrix consolidation. Care must be taken, however, that at the lower temperatures associated with composite use, this chemical reaction proceeds at a rate which does not severely degrade fiber strength. For CMC in which weak interfacial bonding is desirable (ref. 1), fiber and matrix compositions are often selected based on the avoidance of chemical reaction during composite processing or use. Weak bonding can then be achieved by mechanical interlocking due to fiber surface roughness or by friction between fiber and matrix. For those MMC and CMC cases where the above interaction approaches are not effective, fiber coatings can sometimes be used. It should be realized, however, that many of the same issues associated with thermal stability, oxidation resistance, thermal expansion compatibility, and chemical compatibility will also apply to the coating composition.

MECHANISMS AFFECTING FIBER STRENGTH RETENTION

For high temperature application, perhaps the most important properties to be examined for the table I fibers are as-produced tensile strength and thermomechanical stability as revealed by their ability to retain strength under composite processing and use conditions. Since the fibers are brittle ceramics, as-produced tensile strengths are controlled by surface and volume flaws introduced during fiber processing; whereas strength retention is affected by the creation of larger flaws either during fiber exposure or during fiber loading at high temperature.

For the six fiber types of table I, the above mechanisms are revealed in the tensile strength versus temperature results shown in figures 1 and 2. For figure 1, the strength results were obtained at room temperature after typically aging the fiber for short time periods (1 to 10 hr) in inert environments for the nonoxide fibers and in air for the oxide fibers; whereas for figure 2, the strength results were typically obtained at the test temperature after a short (~10 min) soak time in air. Common practice is to measure tensile strength on individual fibers of 25 mm gauge length at a strain rate of approximately 5×10^{-5} sec^{-1}. Because of the statistical nature of ceramic fiber failure, it should be expected that in their as-produced condition and after high temperature treatment, the fiber types of table I will display a large scatter in strength data. For this reason, it is convenient to group the figures 1 and 2 results into approximate bands of observed behavior. More detailed information on as-produced strength and strength retention behavior can be found in the table I references and in the literature (ref. 24).

Examining first the Si-based fiber data of figure 1, it can be seen that the large diameter SiC monofilament fibers produced by chemical vapor deposition (CVD) show significant strength loss after short time thermal treatments between 1200 and 1500 °C. Current theory for the strength degradation mechanism centers on flaw growth by reaction of SiC with metallic substrates (e.g., tungsten) (ref. 6) or by reaction of free silicon metal in the CVD SiC with carbon-rich substrates and surface coatings (ref. 7). For the small diameter Si-based fibers produced by polymer pyrolysis, process-generated oxide phases within the fiber can react with base nonoxide phases at temperatures as low as 1000 °C, resulting in such large internal flaws that these fibers have effectively no strength above 1500 °C (ref. 8). For the Al_2O_3-based fibers of figure 1, the large diameter single crystal monofilament grown by the edge-defined film growth (EDFG) method (ref. 9) appears to be best for retaining strength to above 1400 °C. The mechanism for the slow strength loss with temperature is not certain but may be related to thermal effects on dislocation densities. Finally, for the small diameter polycrystalline Al_2O_3-based fibers produced by sol gel (SG) or slurry spinning (SS), the strength degradation beginning near 1200 °C appears to be related to the growth of strength-limiting grains (ref. 10).

From a practical point of view, the figure 1 results can be summarized by the conclusion that no current commercial fiber can maintain its as-produced strength for short time exposures above 1200 °C. This suggests that if these materials are to be used as reinforcement, MMC and CMC consolidation temperatures should be kept below 1200 °C in order to avoid strength degradation due to internal fiber mechanisms. Obviously, the situation can become even worse if fiber-matrix chemical reaction also occurred during composite fabrication.

To understand the upper use temperature for these MMC and CMC as structural materials, one can examine the data of figure 2 which plots strength at temperature for the six fiber types. It can be seen that under tensile loads, strength degradation begins as low as 800 °C for all types. Indeed, these data were obtained in fast fracture tests, suggesting that under long term structural use, the upper use temperature needed to maintain the as-produced fiber strength would be less than 800 °C. For the polycrystalline fibers, it would appear that the underlying mechanisms for the figure 2 data are creep related. That is, all fiber types begin to show creep at the threshold temperatures for strength degradation, which suggests creep-induced flaw growth as the controlling mechanism. Creep in the polycrystalline fibers occurs by grain boundary sliding which is enhanced by small grain sizes and low viscosity grain boundary phases, such as, free silicon in the SiC (CVD) fibers and SiO_2 in the Si-based (PP) fibers (ref. 11). For the single crystal Al_2O_3 (EDFG) fiber, the dislocation mobility increases significantly above 800 °C, suggesting that the figure 2 data is caused by a drop in yield strength of this material with increasing temperatures.

An additional undesirable aspect of fiber creep is that it results in internal and external dimensional changes in a structural composite which can seriously limit use temperatures and service life. In figure 3, estimates are presented on the upper use temperatures for the most creep-resistant SiC-based and Al_2O_3-based fibers currently produced commercially (refs. 11 and 3). For these estimates, an upper creep strain limit of 1 percent was assumed for a service stress of 100 MPa and service life of 300 hr. For the current polycrystalline fibers, the SiC-based and Al_2O_3-based fibers can reach ~1200 and 1000 °C, respectively; whereas for the current single crystal fibers, the limits are increased to ~1900 and 1600 °C, respectively. It should be noted that today no continuous single crystal SiC based fiber exists so that the figure 3 estimate is based on bulk material data. It should also be realized that the upper creep-limited temperature estimates can decrease significantly for smaller creep strain limits, higher service stresses, or longer service lives.

APPROACHES FOR STABILITY IMPROVEMENT

Based on the previous discussion, it should be clear that current Si-based and Al_2O_3-based fibers, which can withstand oxidation environments to temperatures well above 1000 °C, cannot be used at this temperature for any length of time in structural composites. The primary limiting mechanisms are impurity-induced internal reactions and creep-induced flaw growth. To make improvements in these fibers, obviously the fiber producers must eliminate or minimize the limiting mechanisms for each type. For example, removing metallic substrates and free silicon in the SiC (CVD) fibers is required. Eliminating oxide phases and metallic inclusions for the Si-based (PP) fibers and free SiO_2 in the Al_2O_3-based (SG, SS) fibers is also needed. But perhaps, more importantly, there is a need to minimize creep as much as possible.

Since grain boundary sliding is the primary creep mechanism in polycrystalline fibers, one approach to reduce fiber creep is to increase grain size (ref. 11). However, grain size should be kept below ~ 1 μm if fiber strengths greater than ~ 1 GPa are desired. For current Si-based fibers, grain sizes are typically below 0.1 μm, so that opportunities appear to exist here for creep improvement. A second approach which may be more effective is the elimination of low viscosity grain boundary phases, such as, free Si and SiO_2, and the insertion of stable grain boundary pinners, such as, carbon for Si-based fibers. These grain boundary pinners can also aid in inhibiting grain growth. A third approach might be to utilize large aspect ratio grains with the small dimension transverse to the fiber axis for good tensile strength and the large dimension along the fiber axis for good high temperature stability (ref. 12).

For single crystal fibers, creep is caused by dislocation motion. One approach for creep reduction would then be to add stable dislocation pinning phases to the ceramic fiber as is often done in precipitate and solution-strengthened metal alloys. Another approach might be to select a fiber crystal structure in which dislocation mobility is intrinsically inhibited. A third approach for oxide fibers might be to develop a fine-sized diameter fiber or a fine-sized single crystal eutectic structure aligned along the fiber axis so that dislocation mobility would be hindered by the phase boundaries.

The microstructural approaches discussed above for stability improvement of current fibers are summarized in table II. Using bulk ceramic behavior as an example of the effectiveness of these approaches, one can make crude estimates of the improvements in creep resistance to be expected. These are displayed as upper use temperatures in figure 3 for the bars labeled optimized polycrystalline and single crystal. From these estimates, one might conclude that the Si-based and Al_2O_3-based fiber producers can possibly increase temperature capability by as much as 400 and 200 °C, respectively, over current fibers. Figure 3 also suggests that if strong continuous single crystal fibers can be fabricated, they would offer the highest use temperatures. For the Si-based compositions, although SiC and Si_3N_4 whiskers are commercially available, no known source exists for continuous single crystals. The reasons for this probably center on the need for controlled atmosphere growth facilities and, as with current continuous single crystal Al_2O_3 fiber, the unfavorable economics associated with significantly slow growth kinetics.

CONCLUDING REMARKS

The objective of this paper was to indicate that in the area of high temperature structural fibers, there exists a significant deficiency in the thermomechanical performance of current commercial fibers. The opportunity thus exists for fiber producers to develop new fibers which can seriously impact the potent area of high temperature composites. By understanding the intrinsic microstructural mechanisms underlying the instability problem for each fiber type, approaches such as those suggested here might be attempted to develop improved behavior. Although processing methods were not discussed, one hopes that current techniques and facilities could be used so as to reduce costs and to achieve new fibers as soon as possible. However, if this were not the case, it would seem that the potentially high technical and economic payoff predicted for high temperature structural fibers and composites would be sufficient to spur research and development.

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TABLE I.—CURRENT CERAMIC FIBERS FOR HIGH TEMPERATURE STRUCTURAL USE

(a) Si-based

Primary composition	Typical processing[a]	Crystal morphology[b]	Density, gm/cc	Approximate diameter,[c] μm	Approximate modulus, GPa	Approximate cost, $/kg	Average CTE, 10^{-6} °C^{-1}	Typical tradenames	References
SiC	CVD	POLY	3	80 to 100	330 to 420	5 000	4.5	SCS-6, Sigma	6,7,13
Si-C-O	PP	POLY	2.5	15	190	1 000	4	Nicalon, Tyrrano	8,14,15, 16,17
Si-N-C-O	PP	AMOR	2.5	10	220	1 000	3	HPZ, Tonen	18

(b) Al_2O_3-based.

Primary composition	Typical processing[a]	Crystal morphology[b]	Density, gm/cc	Approximate diameter,[c] μm	Approximate modulus, GPa	Approximate cost, $/kg	Average CTE, 10^{-6} °C^{-1}	Typical tradenames	References
Al_2O_3	EDFG	SINGLE	4	80 to 250	480	50 000	9	Saphikon	9,19
Al_2O_3	SS	POLY	4	20	380	1 000	9	FP, PRD166	10,20
Al_2O_3-SiO_2	SG	POLY	3	12	200	1 000	4.5	Nextel, Sumitomo	10,21, 22,23

[a] CVD = Chemical Vapor Deposition; PP = Polymer Pyrolysis; EDFG = Edge Defined Film Growth; Ss = Slurry Spinning; SG = Sol Gel.

[b] POLY = Polycrystalline; AMOR = Amorphous; SINGLE = Single Crystal.

[c] DIAM. > 50 μm; MONOFILAMENT FORM; DIAM. < 20 μm; TOW FORM - 100 to 1000 FIBERS/TOW.

TABLE II.—OPPORTUNITIES FOR NEW FIBERS WITH IMPROVED THERMOMECHANICAL STABILITY

Polycrystalline fibers

- * Eliminate reactive phases in Si-based fibers
- * Reduce grain boundary mobility
  - Tailor grain size
  - Eliminate low viscosity phases
  - Add stable pinning phases
  - Align high aspect ratio grains

Single crystal fibers

- * Develop cost-effective processing for continuous Si-based fibers
- * Reduce dislocation mobility
  - Add stable pinning phases
  - Select low mobility crystal structure
  - Develop fine-sized diameter
  - Develop fine-sized aligned eutectic

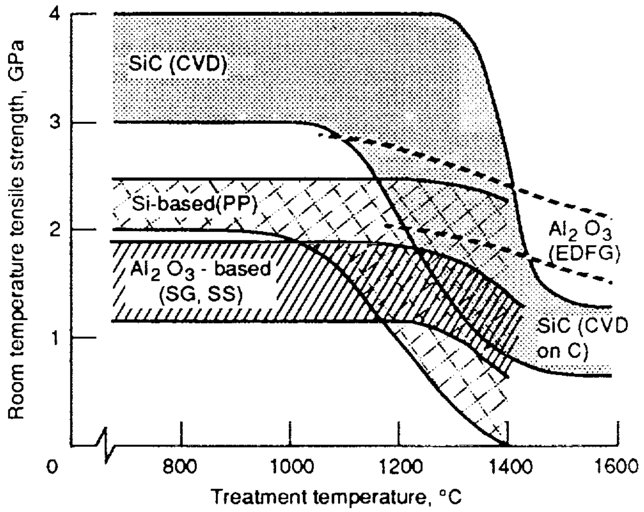

Figure 1.—Strength retention of current high temperature structural fibers. Typical thermal treatment times range from one to ten hours.

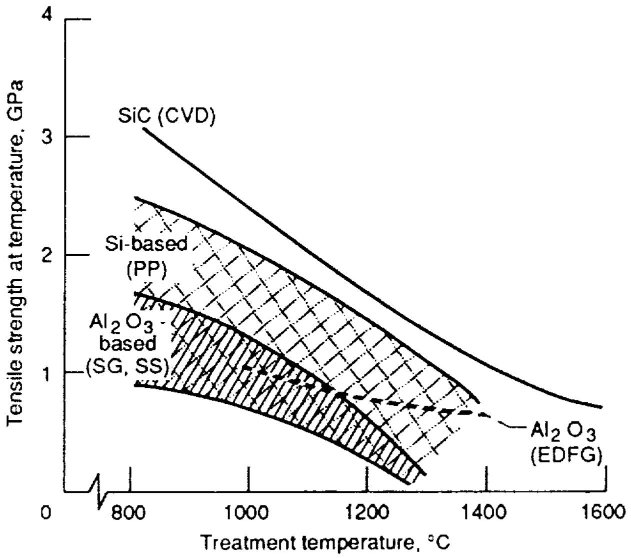

Figure 2.—Temperature–dependent strenght of current high temperature structural fibers. times at the test temperature are typically less than 15 min.

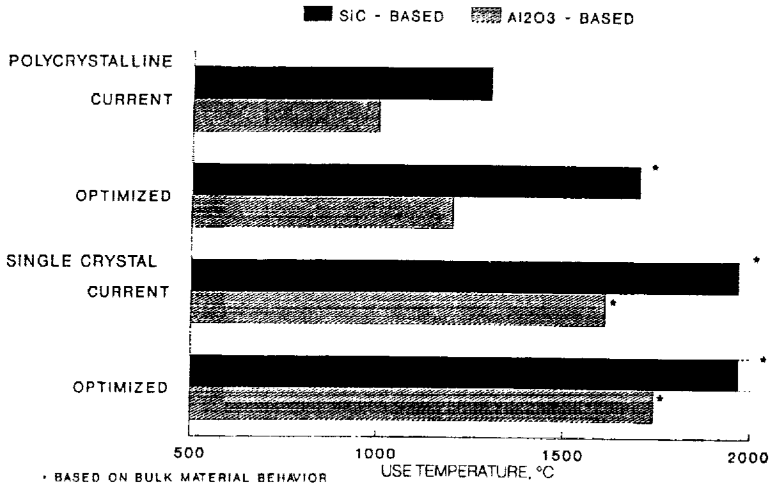

Figure 3.—Estimated creep–limited use temperatures of current fibers and new fibers with optimized microstructures. Estimates assume 1% creep stain in 30 hours at 0.1 GPa stress.

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