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FRACTURE BEHAVIOR OF ADVANCED CERAMIC HOT-GAS FILTERS*

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DISCLAIMER

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ABSTRACT

Microstructural, mechanical, and thermal-shock behavior of hot-gas candle filters obtained from different manufacturers have been evaluated. These filters include both monolithic ceramic and composite materials. Based on the results obtained so far, composite filters perform better than monolithic ceramic filters in a thermal-shock environment. During thermal-shock testing, the monolithic ceramic filters failed in a brittle (catastrophic) mode, while composite filters showed a non-catastrophic mode of failure and very little degradation in ultimate strength. Fractographic evaluations were performed to identify and characterize critical flaws in Nextel fibers for the determination of in-situ fiber strength in Nextel/SiC filters. Average in-situ fiber strength was determined to be 1.7 GPa.

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INTRODUCTION

Hot dirty gas exiting from a gasifier or an advanced combustor contains sufficient particulates to warrant it undesirable for direct utilization in gas turbines and heat engines. Today's hot dirty gas cleanup systems, such as cyclones, can remove only the fraction containing the larger particulates. Smaller particulates can be removed only by cooling and filtering the gas. The resulting enthalpy loss causes a significant decrease in overall system efficiency. Because of the enthalpy loss and decrease in overall efficiency, there is a critical need for cleaning hot dirty gas with little or no cooling. The U.S. Department of Energy and others are currently supporting the development of ceramic/composite filter technology for combined-cycle power generation with coal gasification. Ceramic filters should essentially remove all of the fines from a hot dirty gas stream and be stable in hot dirty gas environments.

This project supports the development of candle ceramic/composite filters for cleanup of hot dirty gases. Effort has been directed toward developing materials qualification technology in order to ensure satisfactory performance of filters in a hot dirty gas stream. To predict long-term performance of such filters, it is important to understand and evaluate the fracture behavior of filters in service environments. Mechanical properties should be evaluated to establish baseline data. Thermal-shock resistance should be measured to predict filter performance in a service environment.[1] Failure modes must be identified and failure mechanisms must be established.

In this paper, results are presented on the evaluation of strength and thermal-shock resistance for the first set of filters obtained from 3M, Du Pont Lanxide, and Industrial Filter and Pump (IFP).

SPECIMENS FOR FRACTURE STUDIES

Initial effort concentrated on filters obtained from the three industrial sources: Nextel/SiC composite filters from 3M, PRD-66 filters from Du Pont Lanxide, and monolithic SiC filters from IFP. The 3M filters consist of layered composite structures, with a tubular filter element sandwiched between two Nextel/SiC composite tubes. Bonding between the Nextel/SiC composite tubes and the filter layer is achieved by chemical vapor infiltration (CVI) of SiC. The inner and outer diameters of these filters are ≈2.54 and ≈2.86 cm, respectively. The PRD-66 is an all–oxide ceramic consisting of a layered microstructure of alumina, mullite, cordierite, and some amorphous material. Inner and outer diameters of the filters are ≈2.26 and ≈2.98 cm, respectively. The monolithic SiC filters consist primarily of SiC grains; inner and outer diameters are ≈2.05 and ≈2.96 cm, respectively.

EVALUATION OF STRENGTH AND THERMAL–SHOCK RESISTANCE

Strength of the candle filters was evaluated on 1–in.–long O-ring specimens machined from each of the three filters. These specimens were loaded to failure in a diametrical compression mode at a crosshead speed of 0.13 cm/min. Maximum stress is at the inner diameter across the load points, which simulates the thermal-shock stresses developed during pulse–cleaning cycles. The fracture stress, σ_f, is given by the following equation:[2]

$$\sigma_f = PK/\pi bl, \qquad (1)$$

where P is the fracture load, K is a function of the ratio of inner and outer diameters,[2] and l is the length of the ring. As shown in Fig. 1, load-deflection plots for the filter specimens in as-fabricated condition indicated a nonbrittle mode of failure for the Nextel/SiC composites and PRD-66 filters, while the IFP monolithic SiC filters showed brittle failure.

Thermal-shock testing of these filters was performed on 1-in.–long ring specimens machined from the filters and insulated on the outer surfaces to simulate heat transfer conditions in service. The specimens were heated to preselected temperatures (25-1100°C) in an electric furnace, as shown in Fig. 2. Subsequently, they were quenched in silicone oil at room temperature (≈25°C). Thermal-shock damage was estimated by measuring the strength of each ring specimen before and after thermal quenching.

Results of the thermal-shock experiments are given in Fig. 3, which shows the retained strength of specimens when subjected to varying degrees of thermal quenching (ΔT). Vertical bars represent standard deviation when three to four specimens were tested. Other data points represent values for single specimens.

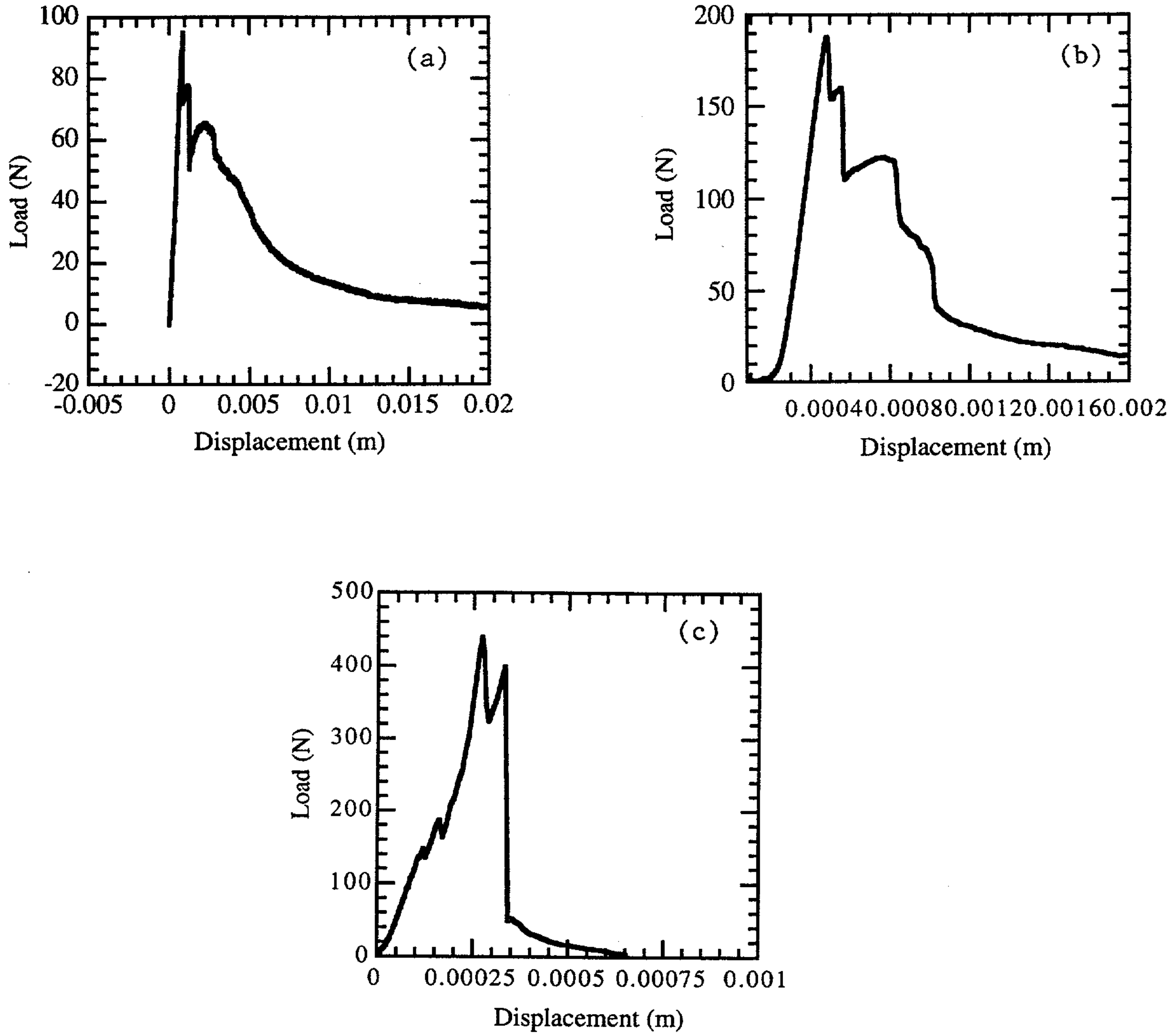


Fig. 1. Load-displacement plots for (a) Nextel/SiC, (b) PRD-66, and (c) IFP SiC filters, showing nonbrittle and brittle fractures.

Limited results obtained so far indicate that the 3M Nextel/SiC composite filters and Du Pont Lanxide PRD-66 filters show little or no strength degradation when quenched from temperatures up to ≈1000°C. On the other hand, the monolithic SiC filters from IFP showed strength degradation when quenched from temperatures >800°C. Further experiments are currently in progress to confirm the statistical reliability of these results. Microstructural evaluations are also underway to evaluate failure modes and mechanisms for improved prediction of filter performance.

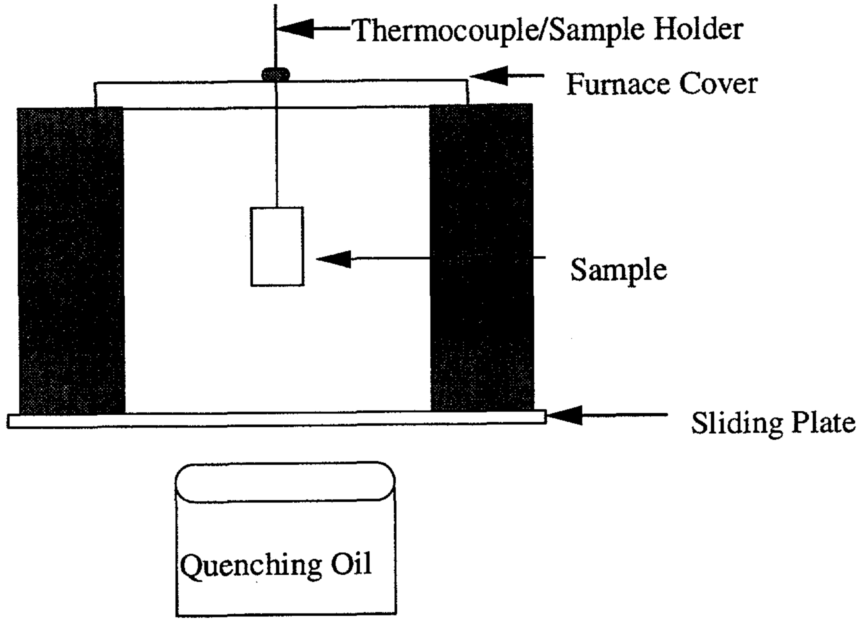


Fig. 2. Schematic diagram of thermal-quench test apparatus.

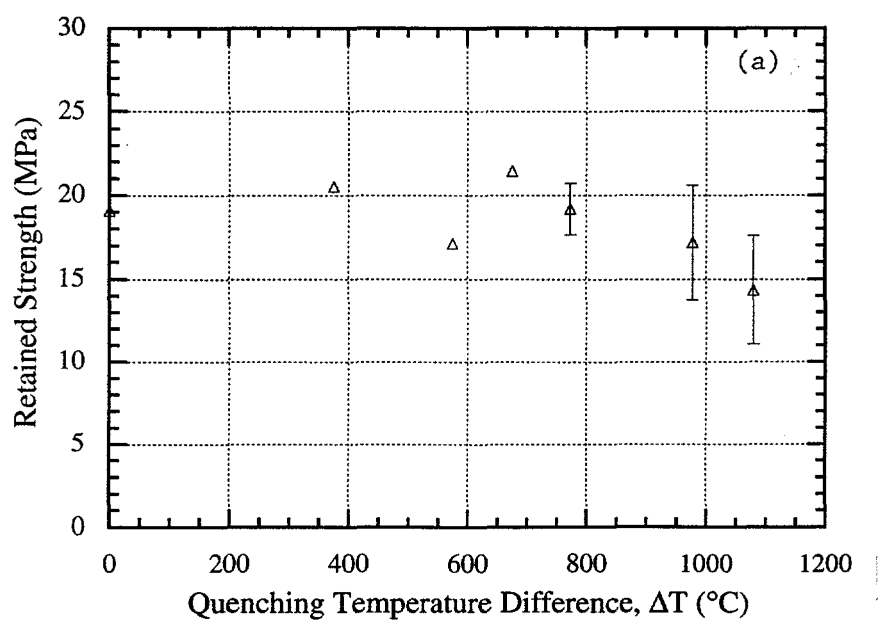
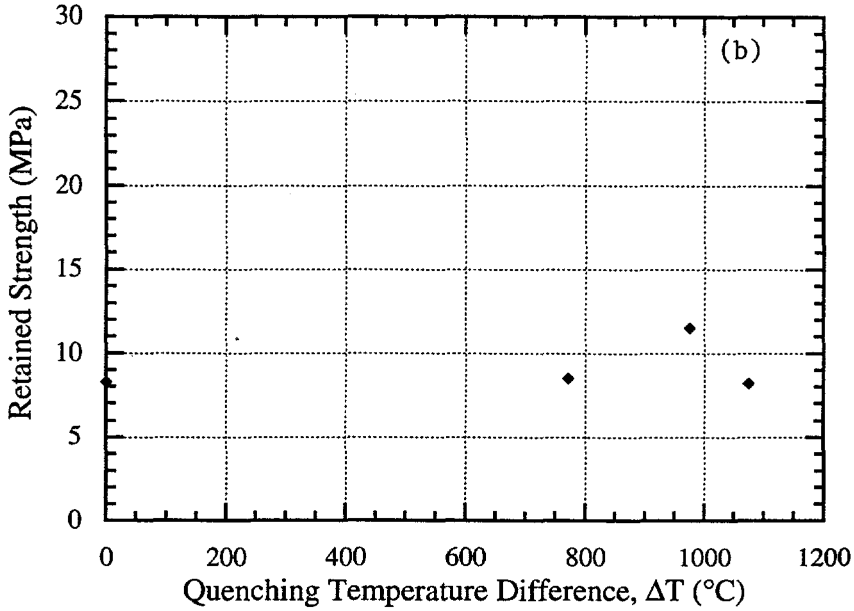
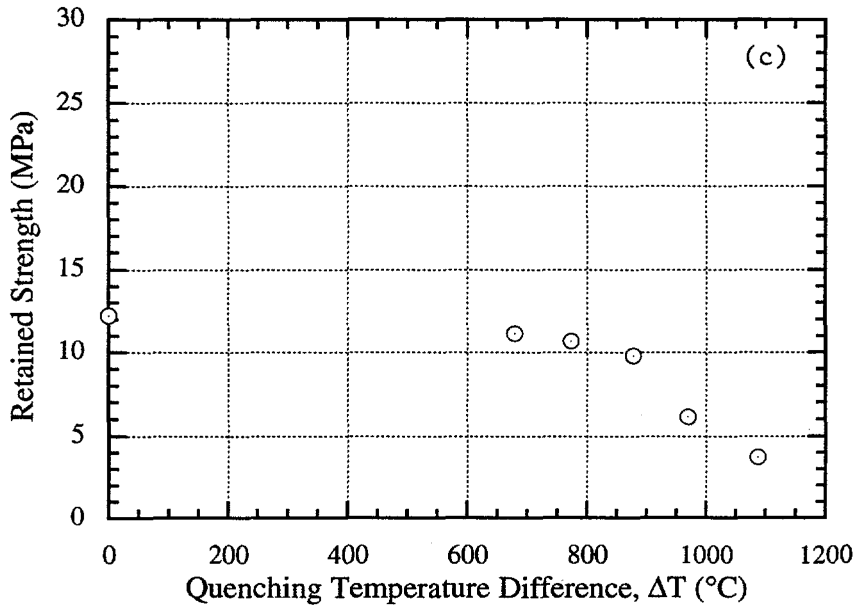


Fig. 3. Dependence of retained strength of ring specimens on quenching temperature difference (ΔT): (a) Nextel/SiC composite filters, (b) PRD-66 filters, and (c) monolithic IFP SiC filters.



(b)
Retained Strength (MPa)
30
25
20
15
10
5
0
0
200
400
600
800
1000
1200
Quenching Temperature Difference, ΔT (°C)



(c)
Retained Strength (MPa)
30
25
20
15
10
5
0
0
200
400
600
800
1000
1200
Quenching Temperature Difference, ΔT (°C)

Fig. 3. (Contd.)

As noted in other studies,[3,4] a substantial degradation of in-situ strength of Nicalon fibers in Nicalon–fiber-reinforced SiC matrix composites was observed during composite processing and in elevated–temperature service environments. Therefore, a study has been initiated to evaluate the nature of flaw generation and resulting strength degradation of Nextel fibers in the Nextel/SiC composite filters during filter processing and in service. Strength of in-situ Nextel fibers in the Nextel/SiC composites after filter processing has been determined by evaluating critical flaw sizes using fractographic techniques, as described below.

IN–SITU FIBER STRENGTH BEHAVIOR

In-situ strength of fibers in the Nextel/SiC composite filters tested at room temperature was evaluated from characteristic fracture features of the fibers. Strength of fractured fibers was determined from the measured values of fracture mirror radii, as discussed in regard to use of the empirical relationship proposed by Kirchner and Gruver[5] and as given in

$$\sigma_f \sqrt{r_m} = A_m, \tag{2}$$

where σ_f is the fiber fracture strength, r_m is the measured fiber mirror radius, and A_m is the mirror constant and is taken to be 3.5 MPa$\sqrt{m}$.

The measured values of in-situ fiber strengths were described by the Weibull strength distribution function, as shown in

$$F(\sigma) = 1 - \exp\left[-\left(\frac{\sigma}{\sigma_o}\right)^m\right], \tag{3}$$

where $F(\sigma)$ is the cumulative failure probability at an applied stress σ, σ_o is the scale parameter signifying a characteristic strength of the distribution, and m is the Weibull modulus that characterizes the flaw distribution in the material.

Figure 4 shows the distribution of in-situ Nextel fibers in Nextel/SiC composite filters. Average fiber strength is approximately 1.7 GPa. An effort was also made to evaluate the strength of as-fabricated fibers by fiber bundle testing.[6] Average strength of as-fabricated Nextel fibers, obtained from preliminary fiber bundle tests, was 2.1 GPa. This suggests that there is an approximately 20% degradation in the strength of as-fabricated Nextel fibers during filter processing. Further experiments are being conducted to validate the results obtained from bundle tests.

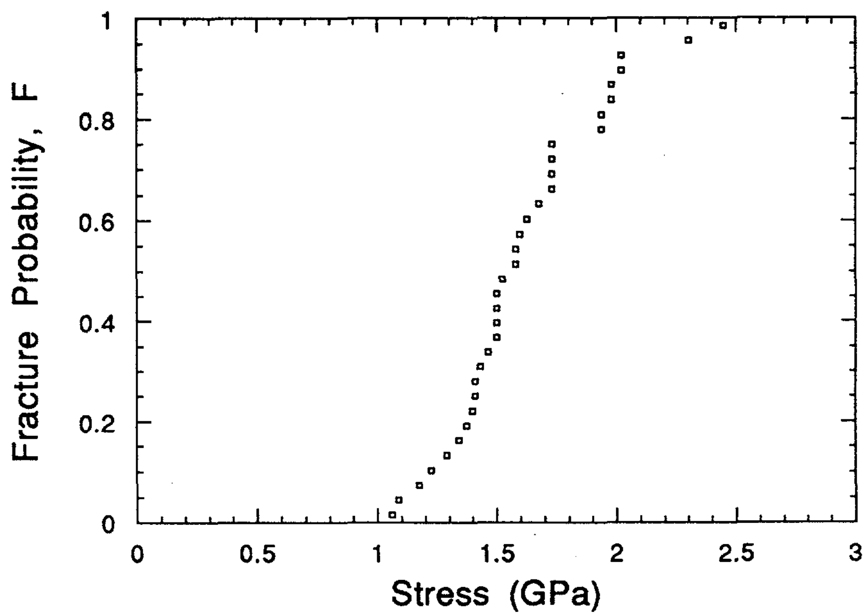


Fig. 4. Weibull strength distribution of Nextel fibers in Nextel/SiC composite filters.

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