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HOT-GAS FILTERS\***

J. P. Singh, M. Sutaria, and W. Bielke  
Energy Technology Division  
Argonne National Laboratory  
Argonne, Illinois 60439

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## THERMAL-SHOCK BEHAVIOR OF ADVANCED CERAMIC/COMPOSITE HOT-GAS FILTERS

J. P. Singh, M. Sutaria, and W. Bielke  
Energy Technology Division, Argonne National Laboratory  
Argonne, Illinois 60439

### ABSTRACT

The thermal shock/fatigue behavior of monolithic and composite hot-gas candle filters obtained from various manufacturers was evaluated. The composite filters were made of both oxide and nonoxide materials; the monolithic filters were made only of nonoxide materials.

During single-cycle thermal shock tests, composite filters show little or no strength degradation when quenched from temperatures between 900 and 1000°C. At higher quenching temperatures, slow strength degradation was observed. Regular monolithic SiC filters showed no strength degradation when quenched from temperatures up to  $\approx$ 700-900°C, whereas at higher quenching temperatures, the strength decreased at a relatively sharper rate. On the other hand, recrystallized monolithic SiC filters showed higher initial strength and retained this strength to higher quenching temperatures when compared with regular SiC filters. This finding may be related to the difference in the strength of grain boundary phases in the two filters. For thermal cycles between room temperature and 800-1000°C, composite filters show little (18-24%) strength degradation up to three cycles, beyond which the strength remains unchanged. Similar behavior, with an initial strength drop of 15-28%, was observed for monolithic filter specimens that were thermally cycled between room temperature and 800°C.

### INTRODUCTION

Hot and 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 that contains larger-size 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. Thus, there is a critical need for a way to clean 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.

To predict long-term performance of the ceramic/composite hot-gas filters, it is important to understand and evaluate fracture behavior of these filters in service environments. Mechanical properties should be evaluated to establish baseline data. Thermal-shock resistance should be measured to predict filter performance in the service environment [1]. Failure modes must be identified and failure mechanisms must be established.

In this paper, we will present the results of the evaluation of strength and thermal-shock resistance of filters obtained from Minnesota Mining and Minerals (3M), Du Pont Lanxide, Industrial Filter & Pump Mfg. Co. (IFPM), Pall Corp., and Babcock & Wilcox.

#### SPECIMENS FOR FRACTURE STUDY

Filters studied were obtained from 3M (Nextel™/SiC composite filters), Du Pont Lanxide (PRD-66 filters), Babcock & Wilcox (oxide composites), IFPM (monolithic SiC and recrystallized SiC filters) and Pall Corp. (monolithic SiC). The 3M filters consist of layered composite structures. A tubular filter element is sandwiched between the two Nextel™/SiC composite tubes. The bonding between the Nextel™/SiC composite tubes and the filter layer is achieved by the chemical vapor infiltration of SiC. The inner and outer diameters of these filters are ≈5.08 cm and ≈5.72 cm, respectively. The PRD-66 is an all-oxide ceramic consisting of a layered microstructure of alumina, mullite, cordierite, and some amorphous material. The inner and outer diameters of the filters are ≈4.52 cm and ≈5.96 cm, respectively. The Babcock & Wilcox filter is an oxide composite with chopped and continuous Nextel fibers made by a sol-gel technique. The inner and outer diameters of the filters are ≈5.00 cm and ≈5.96 cm, respectively. Both monolithic SiC filters primarily consist of SiC grains; the inner and outer diameters of the filters are ≈4.52 cm and ≈5.96 cm, respectively.

## EVALUATION OF STRENGTH AND THERMAL-SHOCK RESISTANCE

The strength of the candle filters was evaluated by both diametral compression testing of O-ring specimens and by burst testing of short filter segments with rubber plugs. Both of these tests induce maximum tensile stresses on the inside surface of the filter, simulating service conditions.

### O-ring Compression Test

One-inch-wide O-ring specimens were cut with a diamond saw from each of the filters. Subsequently, these specimens were ground with a 45  $\mu\text{m}$  diamond wheel to smooth the cut surfaces. The specimens were then loaded to failure in a diametral compression mode at a crosshead speed of 0.13 cm/min. The maximum stress develops at the inner surface across the load points, which simulates the thermal-shock stresses developed during a pulse cleaning cycle. The fracture stress  $\sigma_f$  is given by Equation 1 [2]:

$$\sigma_f = \frac{PK}{\pi b l} \quad (1)$$

where,  $P$  is the fracture load,  $K$  is a function of the ratio of inner and outer diameters [2],  $b$  is the outer radius of the specimen, and  $l$  is the length of the tube. This equation is applicable for a homogeneous cylinder; for layered composite cylinders, it provides only the relative load-carrying capability. The load-displacement plots for the filter specimens in as-fabricated condition indicated a nonbrittle mode of failure for all composites, and brittle failure for the monolithic filters.

Failure of 3M composite filters was initiated by debonding of the interface between the inner composite tube and the filter layer under the load point, followed by subsequent growth of the debonded zone along the interface, as shown in Fig. 1.

### Burst Testing

Burst testing was performed on 1-in.-wide O-ring specimens machined from a Nextel<sup>TM</sup>/SiC composite filter in as-fabricated condition. As shown in Fig. 2, uniform radial loading was applied to the O rings through a rubber plug (slightly smaller in diameter than the O ring), which was compressed between two aluminum

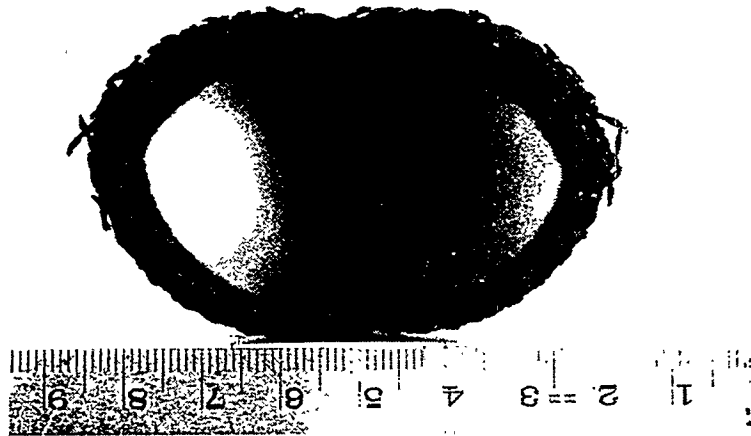


Fig. 1. Filter failure due to debonding.

$$\sigma_t = p^L \left( \frac{r_o^2 + r_i^2}{r_o^2 - r_i^2} \right) \quad (2)$$

$$p^L = \frac{\nu (P_f - P_o)}{(1 - \nu) \pi r_i^2} \quad (3)$$

plates. Figure 3 shows a typical load-displacement plot for a Nextel™/SiC composite filter specimen in as-fabricated condition. The maximum hoop stress  $\sigma_t$  occurs at the inner wall and is given by Eqs. 2 and 3 [3,4], where,  $p^L$  is the radial pressure on the inner wall of the filter,  $r_o$  and  $r_i$  are the outer and inner radii of the filter, respectively,  $P_f$  is the maximum applied load at fracture,  $P_o$  is the load at which the rubber plug makes radial contact with the filter, and  $\nu$  is the Poisson's ratio (value taken to be 0.5). The fracture load  $P_f$  and contact load  $P_o$  were obtained from the load-displacement plot. A limited number of specimens was evaluated by burst testing to compare the strength data with data obtained by O-ring compression testing. The burst strength of the Nextel™/SiC composite filter specimens was found to be  $6.4 \pm 0.22$  MPa. This value is smaller than that obtained by O-ring tests. The difference is believed to be partly due to the larger specimen volume subjected to high stresses during burst testing than in O-ring compression testing.

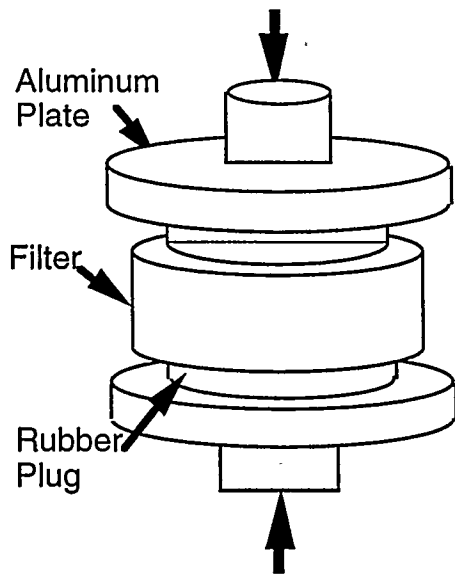


Fig. 2. Schematic diagram of burst testing setup.

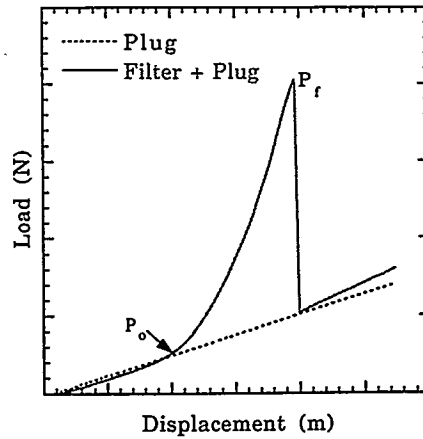
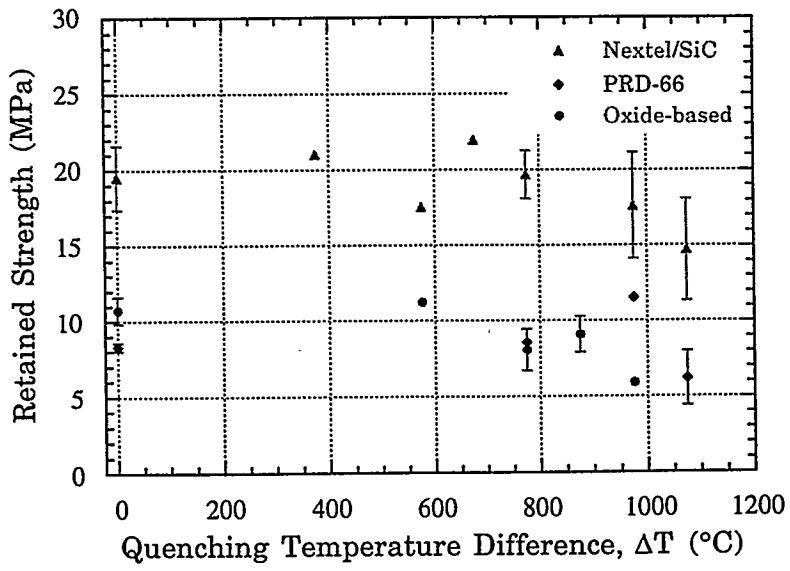


Fig. 3. Typical load-displacement plot for Nextel™/SiC filter specimen.

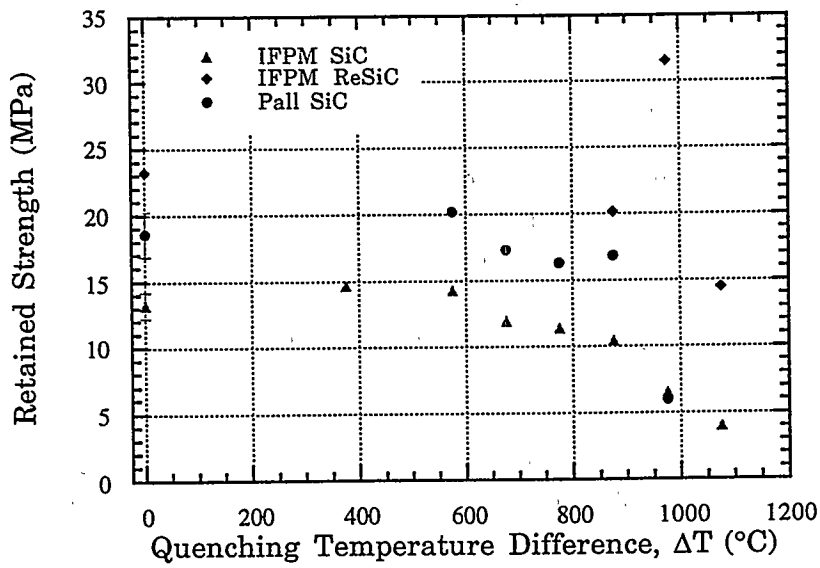
### Thermal-Shock Testing

Thermal-shock testing was performed on 1-in.-wide ring specimens machined from the filters. The ring specimens, insulated with a high-temperature alumina insulating tape on the outer surface to simulate in-service heat transfer conditions, were heated to preselected temperatures (25-1100°C) in an electric furnace. Subsequently, the specimens were quenched in silicone oil at room temperature ( $\approx 25^\circ\text{C}$ ). Thermal-shock damage was estimated by measuring the strength of the ring specimens by the O-ring compression test before and after thermal quench. The results of the thermal-shock experiments are shown in Fig. 4, which shows the retained strength of specimens subjected to varying degrees of thermal quench ( $\Delta T$ ).

Results obtained indicate that composite filters show little or no strength degradation when quenched from temperatures up to  $\approx 900$ - $1000^\circ\text{C}$ . At higher quenching temperatures, slow strength degradation was observed. Although no significant strength degradation was observed, the area under the load-displacement plots decreased significantly in the specimens quenched from temperatures above  $600^\circ\text{C}$ , indicating an increase in brittleness of composites quenched from these temperatures.



(a)



(b)

Fig. 4. Dependence of retained strength of ring specimens on quenching temperature difference ( $\Delta T$ ): (a) Nextel<sup>TM</sup>/SiC, PRD-66, and Babcock & Wilcox composite filters, (b) IFPM monolithic SiC, IFPM monolithic recrystallized SiC, and Pall monolith SiC filters. Vertical bars represent standard deviation where three to four specimens were tested. Other data points represent values for a single specimen.

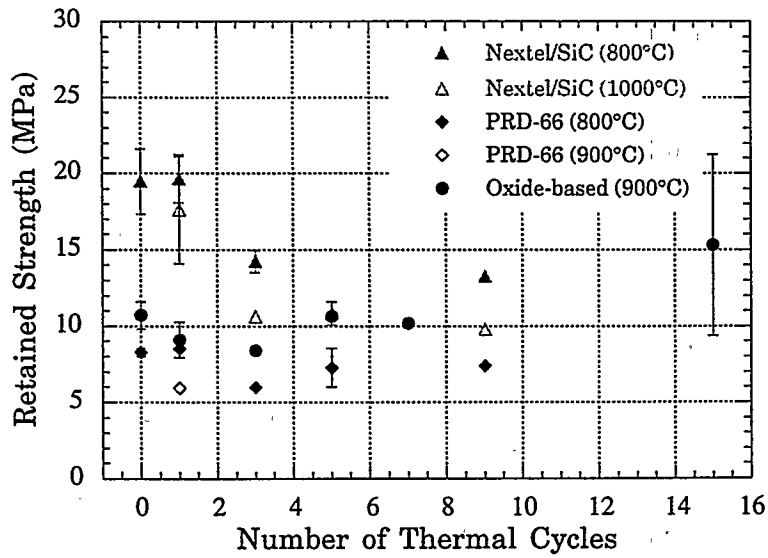
Regular monolithic SiC filters showed no strength degradation when quenched from temperatures up to  $\approx 700-900^{\circ}\text{C}$ . At higher temperatures, the strength decreases at a relatively sharper rate. On the other hand, as seen in Fig. 4b, recrystallized monolithic SiC filter specimens showed higher initial strength and retained this strength to higher quenching temperatures when compared with regular SiC filter specimens. This may be related to the difference in the strength of grain boundary phases in the two cases.

#### Evaluation of Thermal-Fatigue Behavior

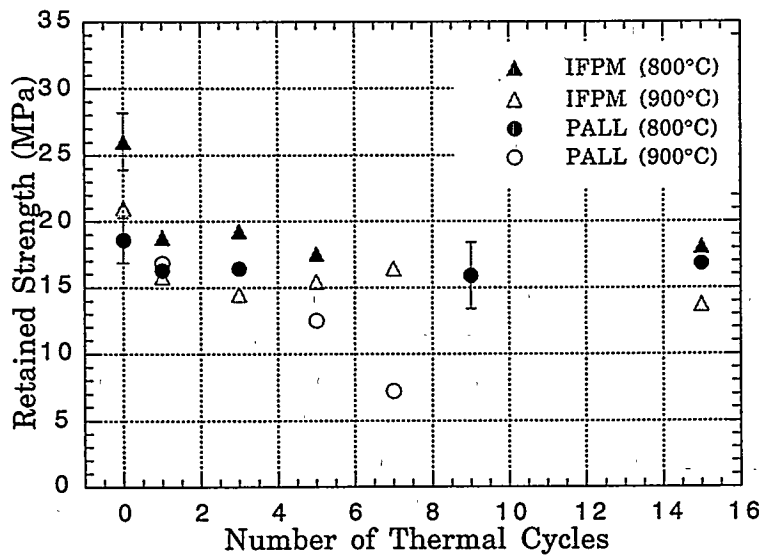
Thermal-fatigue testing of both monolithic and composite filters was performed on 1-in.-wide ring specimens machined from the candle filters. These specimens were thermally cycled between room temperature and preselected elevated temperatures. The elevated temperatures ( $800-1000^{\circ}\text{C}$ ) were selected for thermal cycling close to the critical quenching temperature observed in single-cycle thermal quench experiments. The ring specimens were insulated on the outer surface, as before, to simulate in-service heat transfer conditions. Thermal-fatigue damage was estimated by measuring strength (by O-ring compression tests) of the ring specimens before and after thermal cycling. The results of the thermal-fatigue experiments are shown in Fig. 5a and 5b, which shows the retained strength of filter specimens as a function of thermal cycles.

For thermal cycling between room temperature and  $800-1000^{\circ}\text{C}$ , Nextel<sup>TM</sup>/SiC, PRD-66 and Babcock & Wilcox composite filters show a small strength degradation (18-24%) up to 3 cycles, beyond which strength remained unchanged. The strength decrease for the Nextel<sup>TM</sup>/SiC composites is accompanied by a large decrease in the area under the load-displacement plots, indicating an increase in the brittleness of the composites with increasing thermal cycles. As discussed previously, this is believed to be related to fiber/matrix interface degradation during thermal cycling.

Similar strength degradation was observed in the monolithic filters that were thermal cycled between room temperature and  $800^{\circ}\text{C}$ . Initially, strength decreased by 15-28% up to approximately three cycles, beyond which the strength remained constant. On the other hand, for thermal cycling between room temperature and  $900^{\circ}\text{C}$ , a large drop in strength was observed for the monolithic filter obtained from Pall Corp. This behavior is currently under investigation to determine if the drop is



(a)



(b)

Fig. 5. Dependence of retained strength on number of thermal cycles: (a) Nextel<sup>TM</sup>/SiC, PRD-66, and Babcock & Wilcox composite filters, (b) IFPM monolithic SiC and Pall monolithic SiC filters. Vertical bars represent standard deviation, where three to four specimens were tested. Other data points represent values for a single specimen.

just a scatter in data. Only one specimen was tested per condition because of the lack of availability of filter specimens. As expected, both monolithic and composite filters showed a slight decrease in strength when they were thermally cycled at higher temperatures.

#### ACKNOWLEDGMENTS

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