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**Session 8a**

*Filter Technical Issues*

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**8a.1 Tidd PFBC Hot Gas Filter Operating Experience:  
July 1993 - April 1994**

**CONTRACT INFORMATION**

**Contract Number** DE-FC21-89MC26042

**Contractor** Ohio Power Company  
c/o American Electric Power Service Corporation  
1 Riverside Plaza  
Columbus, Ohio 43215  
(614)223-1585

**Contractor Project Manager** Michael J. Mudd

**Principal Investigator** John D. Hoffman

**METC Project Manager** Richard A. Dennis

**Period of Performance** August 2, 1989 to June 30, 1995

**Schedule and Milestones**

**Program Schedule**

	FY1990		FY1991		FY1992		FY1993		FY1994		FY1995	
	1	2 3 4	1	2 3 4	1	2 3 4	1	2 3 4	1	2 3 4	1	2 3 4
Design Specification/ APF Procurement	_____											
Detailed Design	_____											
Test Plan	_____											
Hardware Procurement	_____											
Testing	_____											
Data Analysis	_____											

## OBJECTIVES

The objective of this program is to evaluate the design and obtain operating experience for up to two Advanced Particle Filter (APF) systems through long-term testing on a slipstream at Ohio Power Company's Tidd Pressurized Fluidized Bed Combustion (PFBC) Demonstration Plant. Performance and reliability of commercial-scale filter modules will be monitored to aid in an assessment of the readiness and economic viability of this technology for commercial PFBC applications.

design to install a slipstream on the PFBC exhaust gases between the fluidized bed and the gas turbine to test an APF system. In November 1988, AEP submitted a proposal to the DOE for the HGCU Program, and in August 1989, a cooperative agreement was signed. In July 1990, AEP awarded a contract to Westinghouse Science and Technology Center to provide a candle-based APF. Installation of the slipstream began in December 1991, and the filter was commissioned in October 1992. Since then, over 3,000 hours of operating time have been logged on the filter.

## BACKGROUND INFORMATION

The 70 MWe Tidd PFBC Demonstration Plant in Brilliant, Ohio was completed in late 1990, and is currently in a four-year test program as part of the Department of Energy's (DOE) Clean Coal Technology Program. Provisions were included as part of the original

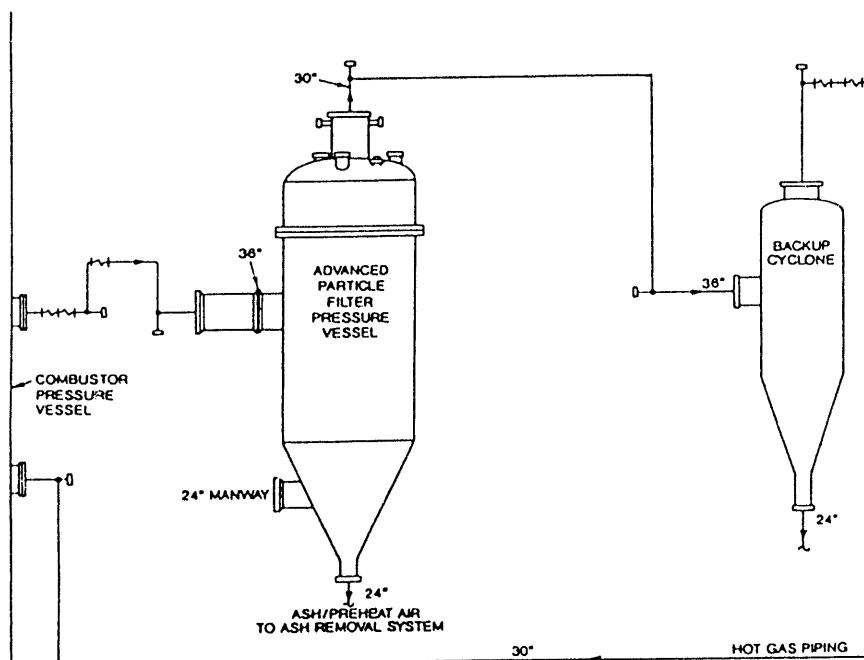


Figure 1. HGCU System Schematic

## PROJECT DESCRIPTION

### System Design

In the original design, the Tidd PFBC Demonstration Plant utilized seven strings of primary and secondary cyclones to remove 98% of the particulate matter from the gases between the fluidized bed and the gas turbine. The HGCU slipstream replaces one of the seven secondary cyclones by taking the discharge gas of one of the primary cyclones to outside of the combustor vessel and into the APF. After passing through the APF the gas flows through a backup cyclone, and then returns to the combustor vessel, where the slipstream flow rejoins the combustor gas at the discharge of the other six cyclone strings.

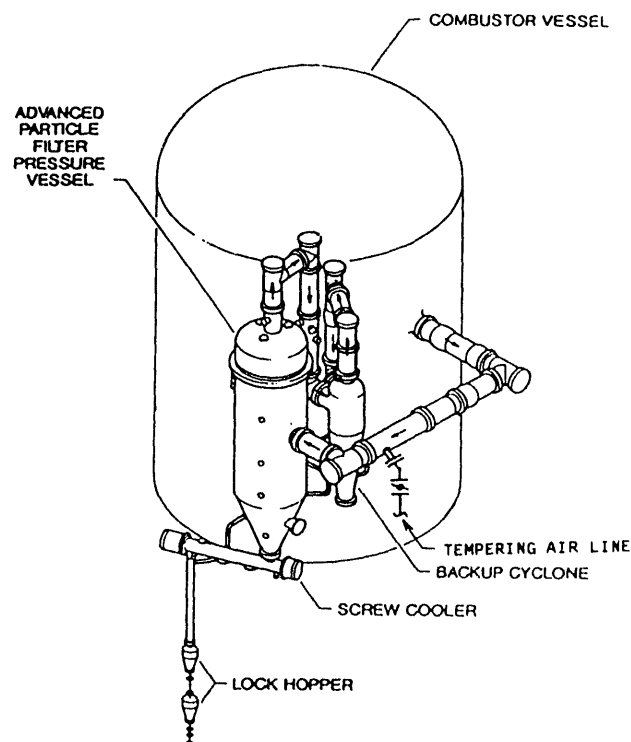
Figure 1 provides a simplified schematic of the APF system, and Figure 2 shows an isometric view of the system.

At maximum load conditions, gas at approximately 150 psig, 1550F flows into the filter at 7600 acfm with a dust loading of approximately 600 ppmw. (In January, 1994, the dust loading to the filter was increased from approximately 600 ppmw to 3400 ppmw by detuning the primary cyclone upstream of the APF.) Ash collected in the APF is discharged to a screw cooler and into lockhoppers which feed a vacuum pneumatic ash transport system. A backup cyclone downstream of the filter is installed to clean the gas in case of a filter malfunction, and to balance the pressure drop of the slipstream with the other six cyclone strings.

Table I provides the design basis of the APF system.

**Table I**  
**APF Design Basis**

Maximum Temperature	1670F
Operating Temperature	1550F
Operating Pressure	164 psia
Gas Flow Rate	100,700 lb/hr
Inlet Dust Loading	500 - 5,000 ppmw
Outlet Dust Loading	<15 ppm
Average Particle Size	1.5 microns
Temperature Drop	5F
Pressure Drop	3 psi
Face Velocity	7.1 ft/min



**Figure 2. Isometric View of HGCU System**

## Filter Vessel

The filter vessel is 10 ft. in diameter and 44 ft. long. It is internally insulated with alumina-silica ceramic insulation, with an internal 316 stainless steel liner to protect the insulation from erosion. The hot gas enters the side of the vessel radially, flows through the candle elements, through the tubesheet, and exits from the top of the vessel head. The exterior of the APF vessel is not insulated, and is coated with temperature sensitive paint to indicate hot spots.

## Filter Internals

The filter, shown in Figure 3, contains 384 candle filter elements, arranged in three clusters, spaced 120 degrees apart. Each cluster holds three plenums, each arranged vertically, with 38 candles in each of the upper and middle plenums, and 52 candles in each of the lower plenums. The candles are attached to the tubesheets in each plenum by bolted collars and high temperature gaskets.

The candles are Schumacher Dia-Schumalith F40 candles consisting of a clay-bonded sintered silicon carbide support matrix that is coated by an aluminosilicate fibrous membrane. Each candle is 2.36 in. OD and 4.92 ft. long.

The 2-in. thick tubesheet is made of RA-333 alloy, and is supported from an inverted "V" expansion cone.

## Backpulse System

The backpulse system receives high pressure backpulse air from a reciprocating compressor rated at 282 scfm and 1500 psig. The air discharges from the compressor to an air dryer, and to a primary air accumulator. The air is then directed to a backpulse skid installed near

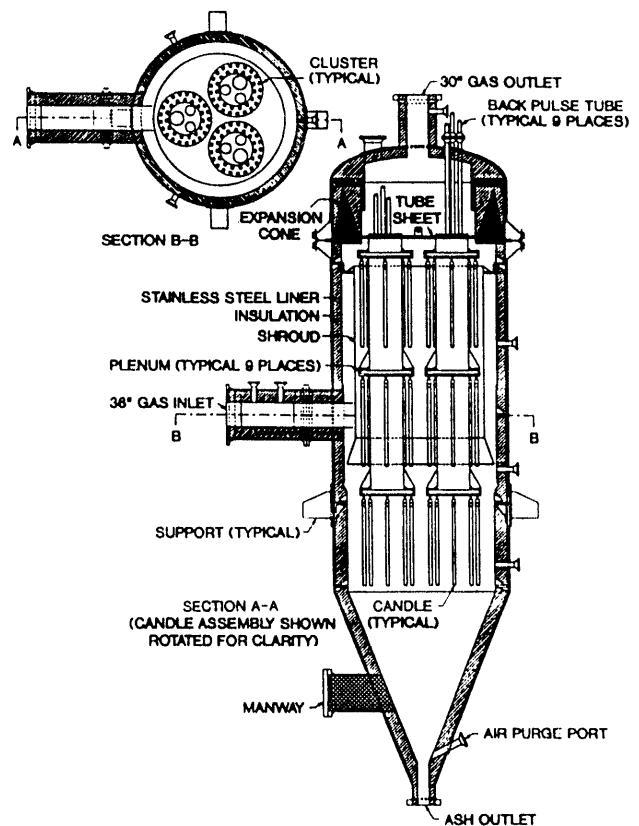


Figure 3. APF Candle Arrangement

the APF, which is comprised of secondary accumulators and the backpulse valves. The backpulse valves are fast acting (200 to 700 msec stroke time) 2-inch pilot-operated Atkomatic solenoid valves. There are three strings of backpulse valves, with redundant valves on each string.

## RESULTS

The Tidd Hot Gas Filter was commissioned on coal fire on October 28, 1992. Table II presents operating statistics through April, 1994. There have been three distinct test periods since initial operation, as summarized in Table II. The results of Test Period I have been



documented in Reference 1, and will not be discussed in this paper. The results of Test Periods II and III are discussed below and summarized in Table III.

**Table II  
Tidd HGCU Test Periods**

Test Period	I	II	III
Date	10/92- 12/92	6/93- 9/93	1/94- 4/94
No. of Runs	4	7	7
Test Period Total Hours	464	1295	1279
Longest Run, Hours	286	597	444

**Test Period II: 6/93 - 9/93**

Test Runs 5 and 6 - 6/30 to 7/5/93

The unit was operated for 60 hours on coal fire during Run 5, and 17 hours during Run 6. While burning Pittsburgh No. 8 coal and Plum Run Greenfield dolomite, the unit achieved a maximum load during Run 5 of 53 MW and 116-inch bed level resulting in an APF gas temperature of approximately 1400F. The APF differential pressures (DPs) remained stable during these runs and reached a maximum of 90 inches at 1400F gas temperature. (NOTE: All DP's are expressed in inches of water.)

Test Run 7 - 7/18 to 8/5/93

The unit was operated for 426 hours on coal fire burning Pittsburgh No. 8 coal and Plum Run Greenfield dolomite. PFBC performance tests were conducted at 95-inch and 80-inch bed

levels and at 85%, 90%, and 95% sulfur retention. Due to problems with gas turbine vibration, the maximum unit load obtained was 46 MW at 110-inch bed level resulting in 1350F APF gas temperature.

Most of the operating time during this run was at 80-inch bed level and 1150F gas temperature. During these operating conditions, the APF DPs were stable at approximately 60-inches trigger and 50-inches baseline. The maximum DP obtained during this run was 105 inches during an excursion at 100-inch bed level and 1250F gas temperature.

The unit was shut down on 8/5 due to ash buildup in the APF hopper that was approaching levels up to the candles. Following shutdown, an inspection revealed three to four inches of ash buildup in areas on the hopper walls. Additionally, a large amount of ash fell off the hopper walls during unit shutdown. Prior to this test series, a pneumatic vibrator was installed in the APF vessel manway and linked to the hopper liner in an effort to keep ash from accumulating on the wall of the hopper. The vibrator, however, did not prove to be effective. Therefore, it was replaced with a larger single impactor-type pneumatic vibrator following this run.

Post-test inspection revealed a small amount of ash buildup on the candles; however, there was no splotching. In general, the candles appeared to be in good shape.

Test Runs 8 and 9 - 8/9 to 8/14/93

During most of the run the unit was operated at 115-inch bed level resulting in 1400 and 1450F APF gas temperatures. The APF DPs were unstable at this load. The trigger DP increased from 90 inches on the afternoon of 8/11 to 150 inches on the morning of 8/14. On 8/12, the backpulse pressure was increased

**Table III**  
**Summary of HGCU Operation - Test Periods II and III**

	Operating Hours	Cumulative Hours	Operating Temp.	Pressure Drop	Inspection Observations
<b>Test Period II</b>					
Run 5 & 6	75	541	<1400°F	Stable	● No filter issues
Run 7	426	967	1150°-1350°F	Stable	● No filter issues
Run 8 & 9	80	1047	1450°F	Increasing $\Delta p$ , Pulse pressure increased from 800 to 1000 to 1200 psig	● Patchy cleaning ● No ash bridging
Run 10	116	1163	1450°F	Slowly increasing $\Delta p$ , pulse pressure at 1200 psig	● Patchy cleaning ● No ash bridging
Run 11	597	1759	1450°F 1200°F	Increasing $\Delta p$ $\Delta p = 240$ in. wg Stable $\Delta p$	● Outlet dust first detected (after 300 hours) ● Significant ash bridging, failed candles
<b>Test Period III</b>					
Run 12	19	1778	1300°-1400°F	Stable	—
Run 13	333	2111	1300°-1400°F	Stable	● Uniform residual ash cake ● No failures ● Ash accumulated on dust sheds
Run 14	23	2134	1200°-1300°F	Stable	—
Run 15	151	2285	1250°-1370°F	Stable	—
Run 16	145	2430	1350°-1400°F	Stable	—
Run 17	164	2594	1300°-1425°F	Gas flow rate decreased during run	● Ash cake about 1/4" thick ● No failures ● Ash accumulation on sheds
Run 18	444	3038	1350°-1450°F	Baseline $\Delta p$ increased $\approx 20$ in. wg	● 28 broken candles ● Inner rows of top and middle plenums heavily bridged

from 800 to 1000 psig. On 8/13, the tank pressure was increased to 1200 psig and the backpulsing time interval was increased from 30 minutes to 60 minutes. Prior to the unit trip on 8/14, the APF DP reached 150 inches trigger and 115 inches baseline.

#### Test Run 10 - 8/19 to 8/24/93

The unit was operated for 116 hours burning Pittsburgh No. 8 coal. The unit was started up using Plum Run Greenfield dolomite, and on 8/24 sorbent feed was switched to Delaware limestone. The unit was shut down 13 hours later due to unstable bed and evaporator conditions experienced while burning limestone. Sulfur retention was maintained at 95% from start-up through the morning of 8/22 and then at 90% for the remainder of the run. The unit was operated at 115-inch bed level and 1450F APF gas temperature for most of the run. The maximum unit load was 52 MW at the conditions stated above.

During operation at 115-inch bed level and 1450F gas temperature, the APF DPs were somewhat unstable; however, not as severe as in Run 9. The trigger DP increased from 95 inches on 8/21 to 115-inches on 8/24, while backpulsing at 1200 psig. When sorbent feed was switched over to Delaware limestone, the baseline DP increased from 90 to 100 inches while the trigger DP remained fairly constant.

Post-test inspection revealed splotchy areas of ash buildup on the candles. The candles were backpulsed during unit shutdown with little success in removing the ash buildup. The APF hopper walls also had areas of ash buildup.

#### Test Run 11 - 8/29 to 9/23/93

The unit was operated for 597 hours burning Pittsburgh No. 8 coal. The maximum unit load

achieved during this run was 55 MW at 125-inch bed level, resulting in an APF gas temperature of 1500F. During start up while increasing load to 115-inch bed level and 1450F gas temperature, sorbent fines (Plum Run Greenfield dolomite, 100% less than 75 microns) were added to the coal water paste while reducing the pneumatic sorbent feed rate by approximately 50%. During this time period the APF DPs started to increase at a rate greater than had been experienced during previous load increases. While at temperatures over 1400F, the APF trigger DP increased from 100 to 168 inches over a period of two days. The baseline DP increased from 84 to 154 inches during this same time period. It is unknown whether this increase in DPs was due to the sorbent fines or whether it was a more aggressive deterioration of APF performance (due to some other phenomenon) than what had been experienced previously at temperatures above 1400F.

On 9/9, the APF trigger DP reached 250 inches, at which time it was decided to reduce unit load to 50-inch bed level, 1000F gas temperature to backpulse the APF. The APF DP was reduced to 145 inches by load reduction and dropped to 36 inches after minimum load backpulsing. However after unit load was brought back to 115-inch bed level, 1450F gas temperature, the APF trigger DP reached a level of 250 inches in less than two days. Load was reduced to 80-inch bed level, 1200F gas temperature to maintain DPs within acceptable limits.

On 9/12 and again on 9/13, indications of candle breaks were noticed by a sudden decrease in APF DP (by approximately 10 inches) and a sudden increase in individual plenum gas flow which occurred simultaneously while backpulsing. Later on 9/13, fragments of broken candles were recovered from the ash removal system. The total fragments recovered could not positively account for more than one

candle. Figure 4 shows a graph of filter pressure drop and temperature for the entire run. The unit was removed from service on 9/23 due to a leak in the sorbent transport pipe caused by erosion.

**Post-Test Inspection and Modifications**

The APF internals were inspected through instrument nozzles and the manway nozzle on 9/27 and 9/28. During this inspection approximately 24 candles were observed to be broken. The ash hopper was filled with ash and broken candles to a level approximately 6 to 12 inches above the manway nozzle.

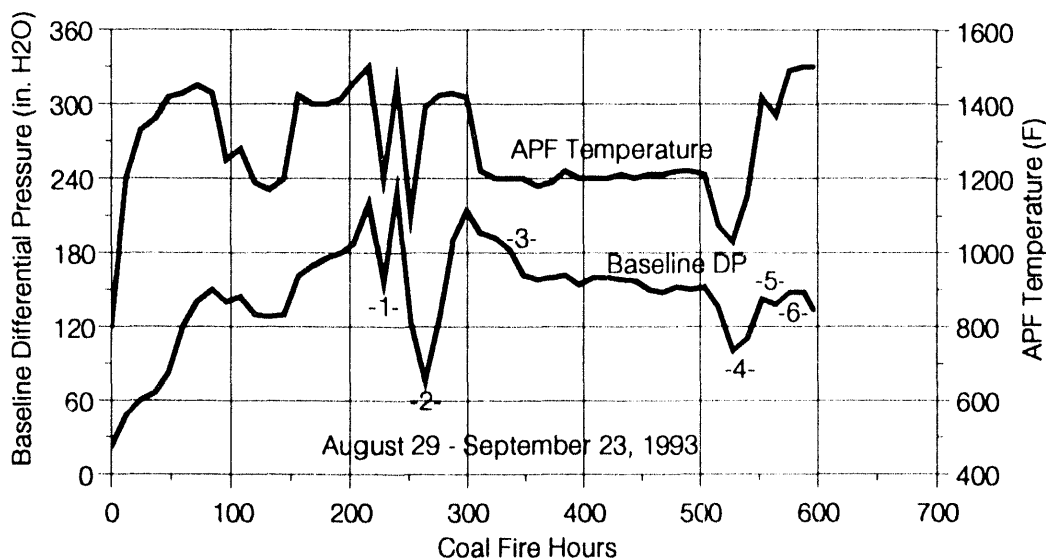
On 9/30, the APF internals were removed from the APF. Upon inspection, 62 candles were found to be broken. Approximately three candles were broken during removal from the APF vessel. The following table shows a summary of the location of the broken candles.

**Location of Broken Candles  
September 1993**

	Plenum A	Plenum B	Plenum C
Top	3	1	2
Middle	6	24	5
Bottom	0	1	20

Very heavy ash bridging was apparent between candles and between candles and support pipes. The deposits were very hard and difficult to break into smaller pieces. Many candles were also found to be bowed. During subsequent cleaning of the ash deposits from the candles, approximately 40 more candles broke.

Two surveillance candles were recovered by Westinghouse and tested for remaining hot strength. One candle from the original set, which had been exposed to operating conditions for 1,759 hours, exhibited a 37 percent loss of



- 1. Reduced load.
- 2. Reduced load.
- 3. Candle breaks.
- 4. Reduced load.
- 5. Spoiling air on.
- 6. Candle breaks.

**Figure 4. Filter DP and Temperature - Test Run 11**

its original strength. The second candle, which had been installed in December 1992 and had experienced 1,295 hours of operation, had lost 51 percent of its strength.

All filter candles were replaced with new silicon carbide candles between Test Series II and III, except for nine surveillance candles which were reused.

The backpulse solenoid valves exhibited random failures to actuate during these tests. In all cases, the backup valve functioned properly. Following Test Period II, the backpulse solenoid valves were inspected and found to be galled on both the piston and cylinder walls. The valve pistons were Stellite coated and the valve body bores were nickel-boron plated to improve resistance to galling. Following these modifications, the valves successfully underwent accelerated cycle testing to verify the design. Operation of the valves during Test Period III was much improved, and subsequent inspections revealed no galling.

At the end of Test Period II, one backpulse tube was cut up and examined for degradation. Microcracking was evident on the inside surface of the tube, with cracks as deep as 0.020". Based on this observation it was decided to replace the Incoloy 800HT material with Haynes 230 alloy, which has better resistance to thermal fatigue. The new backpulse tubes were fabricated and installed prior to the start of Test Period III.

Between Test Series II and III, nine additional air purge nozzles were installed on the APF hopper to facilitate the removal of ash accumulation in the hopper. The hopper vibrator was moved from inside the APF nozzle to outside the nozzle because it proved to be unreliable inside the nozzle. It was mechanically linked to the hopper liner.

In an effort to improve the filter

performance, it was decided to install a system to detune the primary cyclone upstream of the filter. It was believed that the resulting coarser ash would be easier to remove from the filter candles and less likely to accumulate on the hopper walls. An air line was connected to the dip leg of the cyclone and a valve was installed external to the combustor to allow the detuning air to be controlled. Air injected into the dip leg created an upward velocity and thereby detuned the cyclone. All testing during Test Period III was conducted with the cyclone detuned.

It was noted in previous runs that the filter DP sometimes became unstable at gas temperatures over 1400F. To prevent limiting the load on the unit because of the 1400F limit, a tempering air line was installed upstream of the filter which allowed 580F process air at a slightly higher pressure to be mixed with the gas and thereby reduce the gas temperature by as much as 150F.

#### **Test Period III: 1/94 - 4/94**

##### Test Run 12 - 1/10 to 1/11/94

This test run was terminated after 19 hours of coal fire when several primary cyclones became plugged. Due to the brevity of this run and lack of steady state operation near design conditions, it will not be discussed further.

##### Test Run 13 - 1/15 to 1/29/94

The unit was operated for 333 hours on coal fire burning Pittsburgh No. 8 coal and Plum Run Greenfield dolomite. The primary objective of this run was to assess the performance of the APF while operating with the primary cyclone ahead of the filter detuned to produce a coarser ash and higher ash

loading. The tempering air system was commissioned during this run and functioned without any problems.

During the run ash sampling was performed upstream and downstream of the APF using specially designed sampling probes. The results showed that by detuning the primary cyclone, the ash loading to the filter increased from a design value of 600 ppmw to about 3400 ppmw, and the mean particle size of the ash increased from about 3 to 7 microns. The higher ash concentration resulted in significantly higher ash loading. Despite the higher ash loading, the filter performed very well.

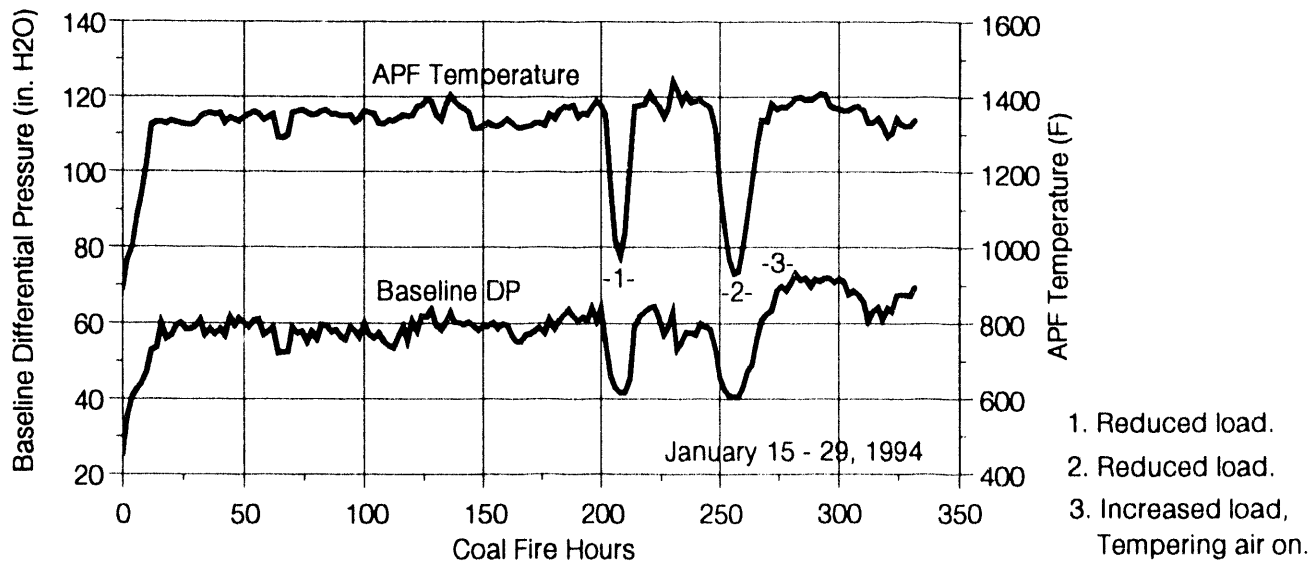
Figure 5 shows a graph of filter pressure drop and temperature for Run 13. The unit load was reduced at about 200 and 250 hours into the run due to bed sintering problems, which accounts for the dips in temperature and DP during these periods. During the remaining periods of this run, the filter pressure drop

remained stable and exhibited the usual trend of following gas temperature. During the last 50 to 75 hours of the run, the unit bed height was increased to the maximum (142 inches), and the filter DP increased somewhat due to higher gas flow and dust loading.

Following shutdown of the unit, the filter was inspected via nine 3-in. nozzles on the side of the vessel. In general the candles looked in good condition with very little residual ash accumulation. However, some ash bridging was seen between the bottoms of the inner rows of candles and the dust sheds on the plenum support pipes.

Test Run 14 - 2/17 to 2/18/94

This test run was terminated after 23 hours of coal fire when a 1" nipple on the HGCU piping developed a leak that could not be repaired in service.



**Figure 5. Filter DP and Temperature - Test Run 13**

Test Run 15 - 2/19 to 2/25/94

This run was a hot restart of the previous run. The APF performed without any major problems during this run. Some minor problems, however, arose. Shortly after start-up, the APF dome exhibited elevated temperatures (up to 730F) near the gas outlet nozzle. The gas temperature was held to the 1250F range in order to keep the hot areas below 750F. On 2/21 an on-line repair was successfully made which reduced the hot areas to below 300F in some areas and to below 400F in others. The repair involved drilling and tapping three 1/4" holes in the outlet nozzle near the head and pumping in 45 gallons of pumpable insulation. Following this repair, the unit was brought up to full bed height (142 inches). Tempering air was again used to limit the gas temperature in the APF to 1400F. Figure 6 shows a graph of APF DP and temperature versus time for this run. The unit load was reduced about 110 hours into the run when a coal paste pump stopped working.

After the bed again stabilized, the unit was returned to full load. The HGCU system functioned without further problems from this point until the unit tripped due to loss of the sorbent booster compressor.

Test Run 16 - 3/3 to 3/9/94

Most of this test run was conducted at 142 and 150-inch bed levels. As in the previous run, tempering air was used to limit the filter gas temperature to 1400F. This run was the smoothest so far the HGCU system. The system operated for over 145 hours without any major problems. Figure 7 shows a plot of APF DP and temperature during Run 16. Figure 8 shows DP and flowrate. The APF DP exhibited a gradual decline throughout this run. The reason for this is unknown. The test conditions throughout Run 16 were the most steady of all the runs. Therefore, data from this run should be considered a good baseline reference for APF operation at 1400F. This run was termin-

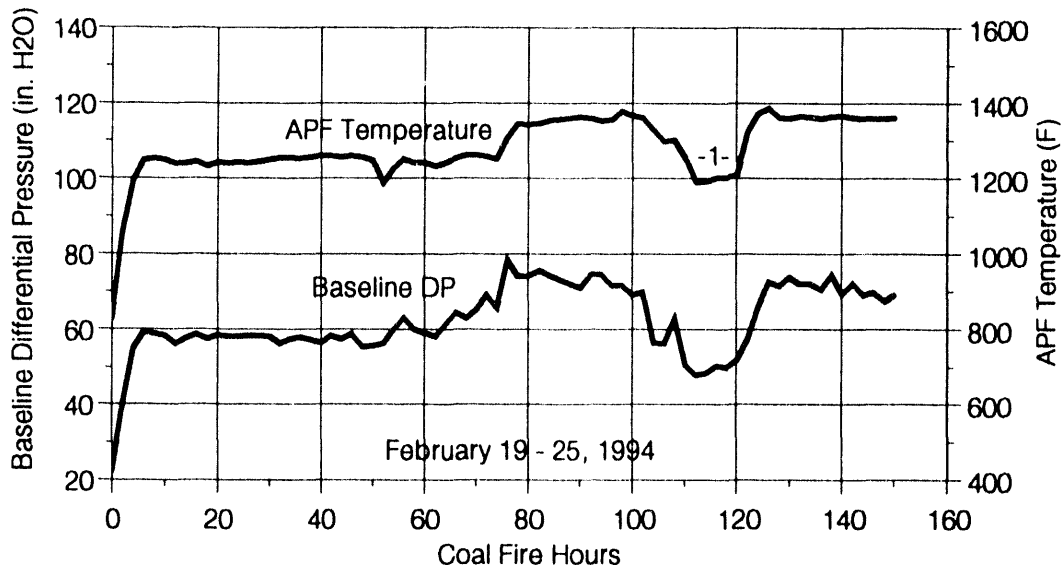
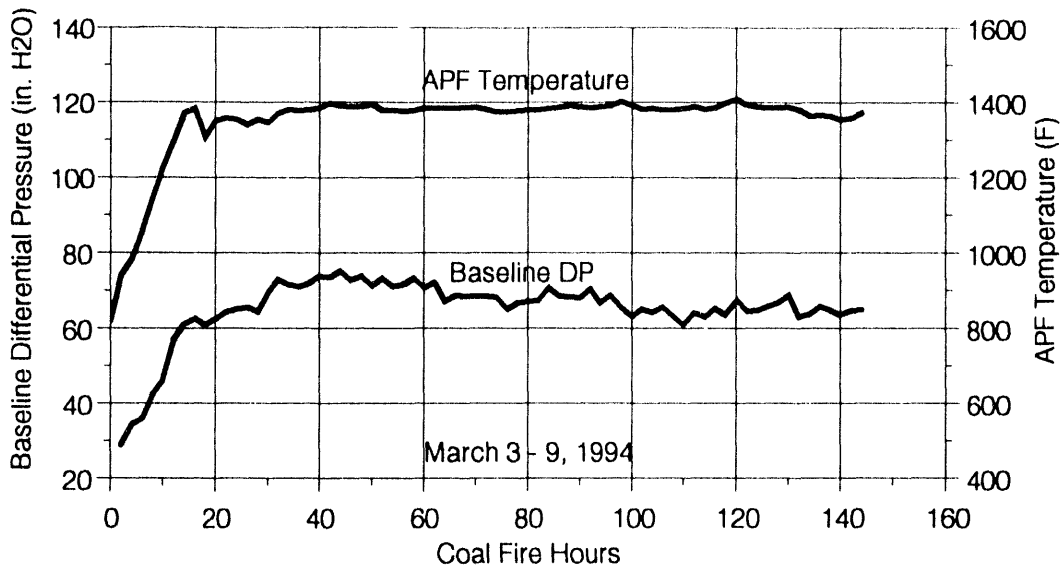
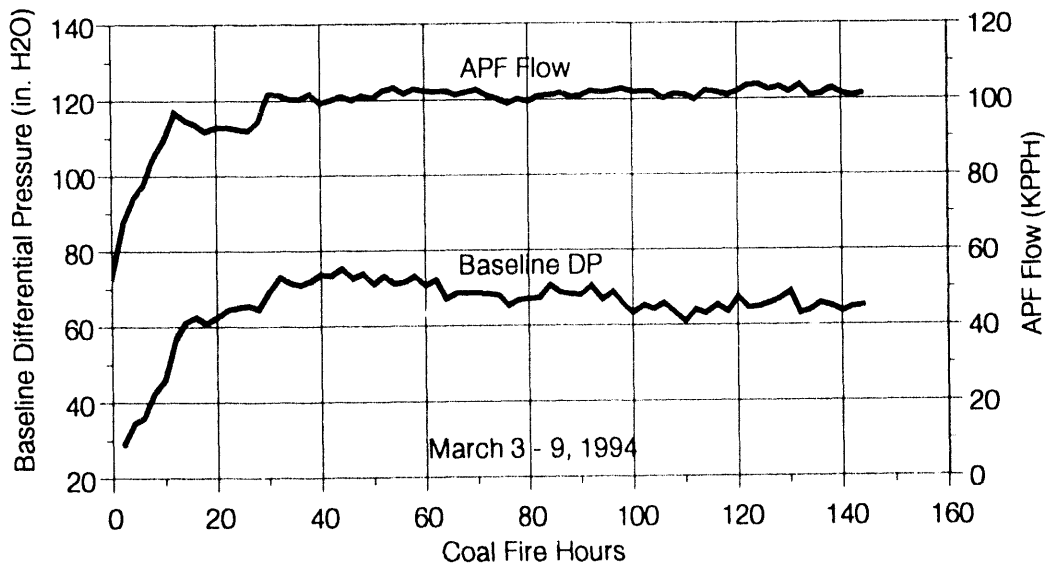


Figure 6. Filter DP and Temperature - Test Run 15



**Figure 7. Filter DP and Temperature - Test Run 16**



**Figure 8. Filter DP and Flow - Test Run 16**



ated when the unit tripped due to the loss of two coal paste pumps.

#### Test Run 17 - 3/16 to 3/23/94

This was another relatively uneventful run on the HGCU system. The unit operated for approximately 164 hours on coal fire. Most of the run was conducted at 128-inch bed level, with portions at 142-inch and 115-inch. Due to bed sintering problems, the bed level could not be maintained at 142 inches. The tempering air was turned off during this run which allowed the APF to operate up to 1450F. The filter pressure differential was relatively stable throughout the run as shown in Figures 9 and 10. However, the gas flowrate through the filter decreased throughout the run. During this run additional insulation was pumped into the APF head to lower the surface temperature in one area from 550F to 400F. The unit tripped due to a low oil pressure indication on the gas turbine.

The APF internals were inspected through the nine instrument nozzles following Test Run 17. No broken candles were observed. The residual ash layer on the candles was somewhat thicker than seen following Test Run 13 and appeared to be about 1/8" to 1/4" thick. The ash coating was uniform from candle to candle but also very rough looking on all the candles. The ash bridging previously seen between the dust sheds and the inner rows of candles was still evident, but not obviously worse than before. The amount of ash accumulation on the dust sheds varied considerably (1/2" to 4") among the six dust sheds. No candle-to-candle bridging was seen.

#### Test Run 18 - 3/31 to 4/18/94

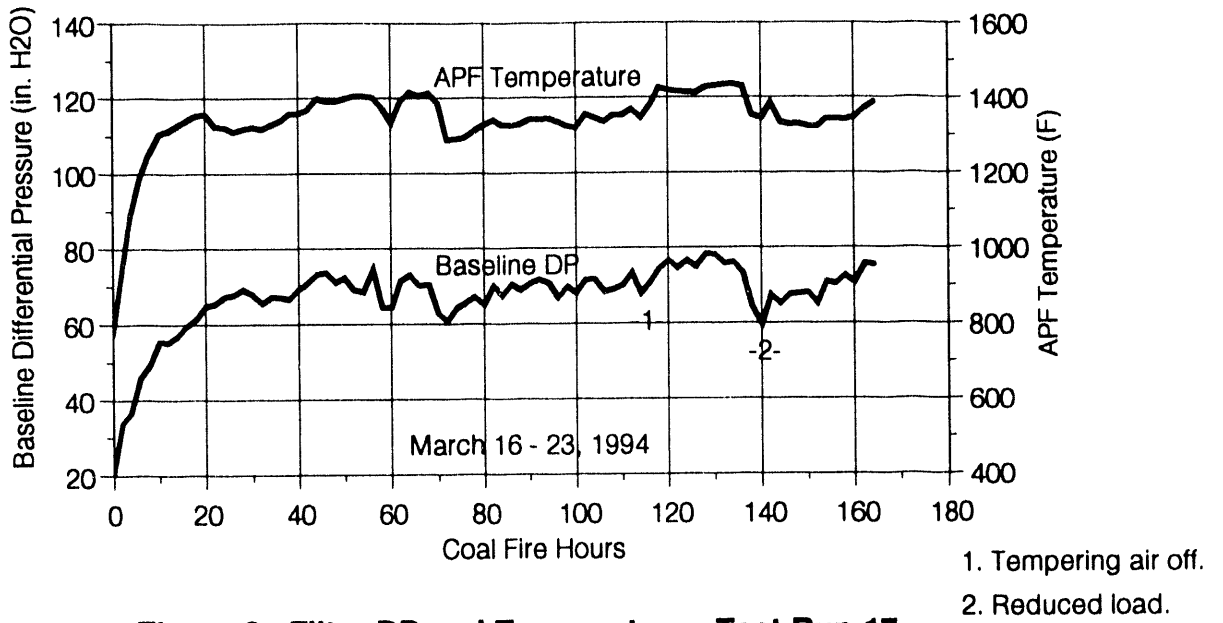
This was the longest run of this test period with almost 444 hours of coal fire. The bed

height during most of the run was 115 inches and the APF temperature was generally in the 1350 to 1400F range. The temperature was approximately 1450F for about 30 hours of the run. Due to a corroded instrument tube, flowrate data were not obtained during this run. Hazardous air pollutant sampling was conducted during this run. No major problems arose with the APF during this test. The baseline pressure differential increased during this run from about 70 to 90 inches as shown in Figure 11. This was the first run of this test series in which the DP increased noticeably from the beginning to the end of the run.

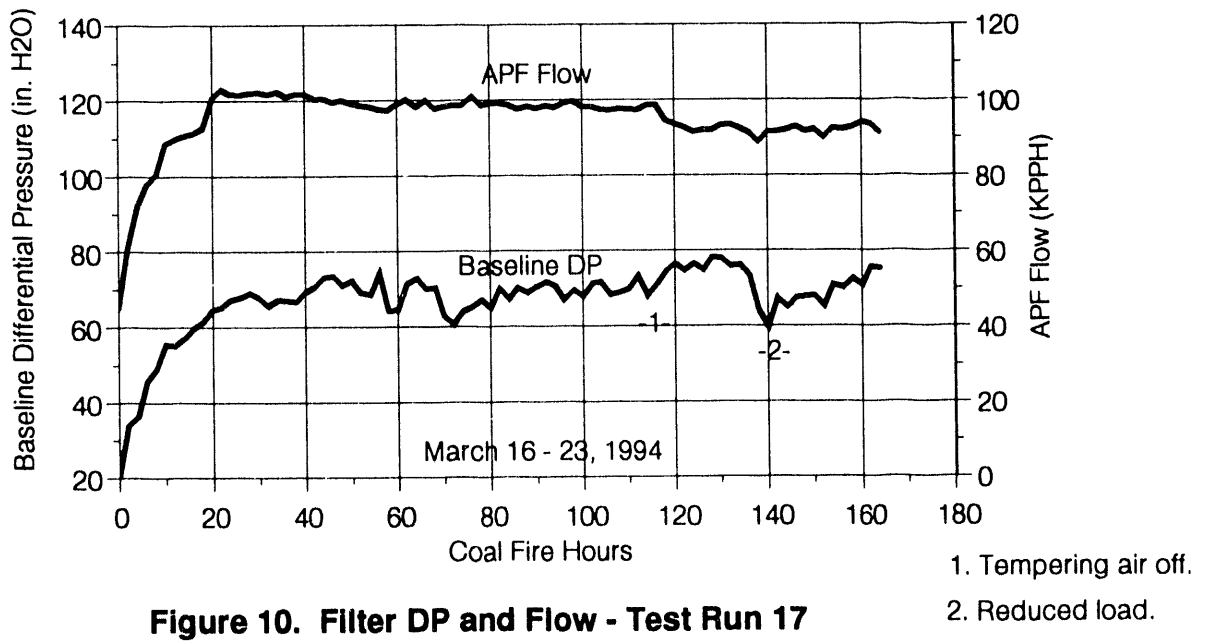
As part of Hazardous Air Pollutant testing, SO<sub>2</sub> data were obtained upstream and downstream of the APF. Preliminary results indicated that the SO<sub>2</sub> level in the gas was reduced approximately 45% by the APF. Post-test analysis of ash samples retrieved from the APF and precipitator hoppers confirmed this observation. The percent sulfation (calculated as moles of SO<sub>3</sub>/moles of CaO) of the APF hopper ash was 78.6% as compared to 50.0% for ash that did not pass through to APF sampled from the precipitator hopper. Additional data will be obtained in future tests to confirm these results.

#### Post-Test Inspection and Modifications

The APF internals were inspected via the instrument ports after this run. The filter candles had a thin layer (about 1/8") of residual ash, however broken candles were observed during this inspection. The filter internals were removed from the APF vessel on 5/5/94 and a more detailed inspection was performed. A total of 28 broken candles were found at the following locations:



**Figure 9. Filter DP and Temperature - Test Run 17**



**Figure 10. Filter DP and Flow - Test Run 17**

**Location of Broken Candles**  
**May 1994**

	Plenum A	Plenum B	Plenum C
Top	7	7	2
Middle	0	2	8
Bottom	0	2	0

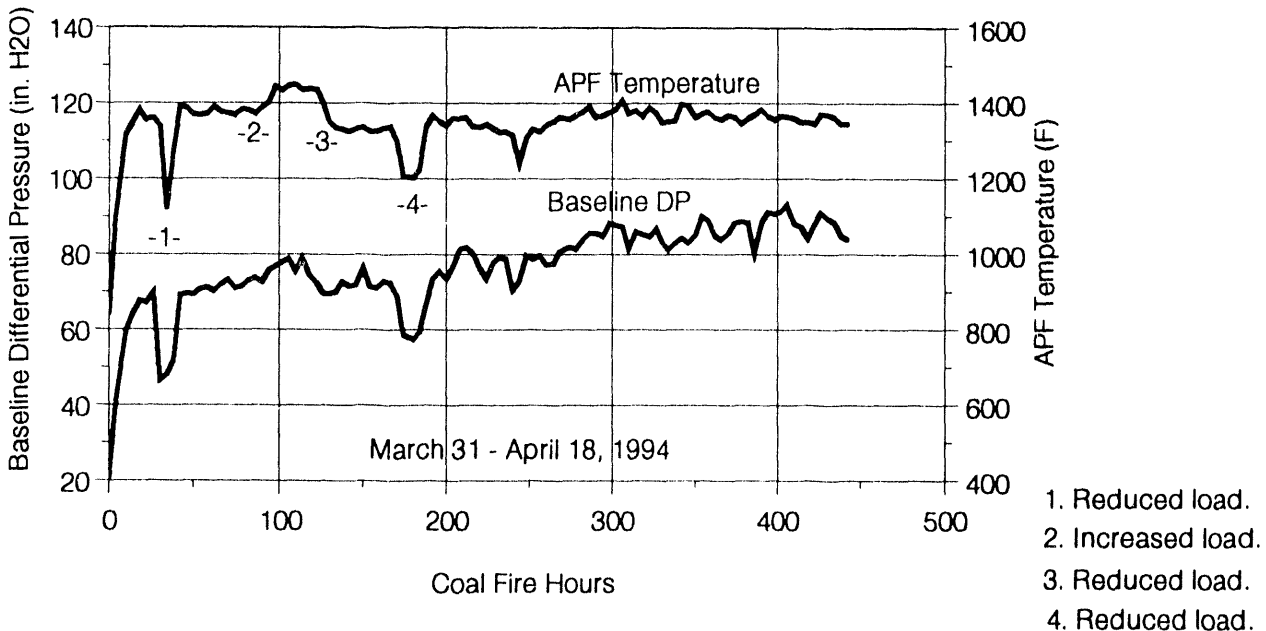
The two breaks in the bottom plenum are believed to have occurred during removal of the filter internals from the filter housing. Very heavy ash bridging was observed between the inner rows of candles and the support pipe on the top and middle plenums. The ash build-up extended over the entire length of the inner candles. In some cases the ash bridging extended into the outer rows of candles. Many of the inner candles were bowed outward and in some cases almost contacted the candles in the outer row. All of the candle breaks except one appeared to be fresh breaks judging from

the clean fracture surfaces. All of the breaks occurred at the top of the candle just below the candle holder. The breaks appeared to result from bending forces from ash bridging from the inner candles to the outer candles. Only one break was in an inner row; the remainder were in outer rows.

The outlet side of the filter internals were very clean with virtually no ash deposits. This is further indication that the failures occurred during or after plant shutdown, and that the filter was not leaking ash to the clean side during operation.

**FUTURE WORK**

At the end of April the system was out of service and plans were being formulated for modifications and future testing. It is planned to remove the inner rows of candles from the



**Figure 11. Filter DP and Temperature - Test Run 18**

top and middle plenums since the ash bridging phenomenon appeared to begin with ash building up on the dust sheds and bridging across to the bottoms of the inner candles. It is hoped that the removal of the inner rows of candles will eliminate this problem. In addition, plans are being implemented to modify the primary cyclone ahead of the filter so it can be rendered totally inactive. This will increase the ash loading and mean particle size to the filter and thereby improve the flowability of the ash. During this shutdown some alternate filter candle materials will be installed to expose them to the Tidd PFBC service conditions.

Assuming that these modifications are successful in overcoming the ash bridging problem, the system will be operated until the fall of 1994 with the current silicon carbide candles and selected alternate candles. The condition of the alternate materials will be assessed when the filter is re-opened in the fall of 1994. At that time, a decision will be made as to what filter materials will be installed for the final phase of testing which will run until February, 1995.

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3. Dennis, R. A., Lippert, T. E., Bruck, G. J., Alvin, M.A., and Mudd, M. J., 1993. Initial Test Results from the Department of Energy's Pressurized Bed Combustion Hot Gas Clean Up Program, Presented at the Tenth Particulate Control Symposium and Fifth International Conference on Electrostatic Precipitation, Washington D.C., April 5-8, 1993, Sponsored by EPRI and the International Society for Electrostatic Precipitation.
4. Mudd, M. J. and Hoffman, J. D., 1992. Initial Operation of the Tidd PFBC HGCU Test Facility, 27-35, Proceedings of the Ninth Annual Coal-Fueled Heat Engines, Advanced Pressurized Bed Combustion (PFBC) and Gas Stream Clean Up Systems Contractors Review Meeting, Morgantown, WV, October 27-29, 1992, DOE/METC-93/6129 (NTIS/DE 93000232).
5. Tidd PFBC Demonstration Project Public Design Report, 1992, DOE/MC/24132-3195 (NTIS/DE93000234).

8a.2

**Karhula Hot Gas Cleanup Test Results**

**CONTRACT INFORMATION**

**Contract Number** DE-FC21-89MC26042

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**METC Project Manager** Richard A. Dennis

**Period of Performance** October 1992 thru September 1994

**Schedule and Milestones**

**Schedule and Milestones FY94**

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	S	O	N	D	J	F	M	A	M	J	J	A
Filter Supply	Completed											
Test Segment 1	Completed											
Test Segment 2	_____											
Test Segment 3	_____											
Test Reports					Δ			Δ			Δ	

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**OBJECTIVES**

The objective of this work is to develop a practical hot gas filter design that meets the performance and operational requirements of pressurized fluidized bed combustion - bubbling bed, circulating bed and second generation - applications. The Westinghouse hot gas candle filter system is currently installed in the Ahlstrom Pyropower 10 MW (thermal) pressurized circulating fluidized bed combustor (PCFB) test facility located in Karhula, Finland. The overall

objective of the testing is to evaluate the filter design and operating reliability for selection and implementation into the Midwest Power DMEC-1 PCFB 150 MW<sub>e</sub> repowering project (Clean Coal III Selection).

**BACKGROUND INFORMATION**

High temperature particulate filters are a key component in the advanced, coal based gas turbine cycles (IGCC and PFBC) that are

currently under development by DOE/METC for clean coal demonstration and future commercialization. In these applications the hot gas particulate filter protects the downstream heat exchanger and gas turbine components from particle fouling and erosion effects and cleans the gas sufficient to meet environmental particulate emission requirements (Newby et al., 1992). Both PFBC and IGCC plants benefit because of lower cost downstream components, improved energy efficiency, lower maintenance and the elimination of additional and expensive flue gas treatment systems.

Ceramic barrier filter devices such as candles and cross flow filters are under development for hot gas filter application. These devices have been shown to be basically absolute filters on ash material, can be operated at relatively high gas throughput with acceptable pressure drop and cleanable by simple reverse pulse jet methods. Clay bonded silicon carbide (SiC) candle filters are commercially available. The structure of these elements is mainly a coarse-grained SiC bonded by a clay-based glass binder. Each element is provided with a fine grained SiC and aluminosilicate fiber outer skin that serves as the filtration surface. Alternate, oxide based ceramic materials are also being developed for ceramic barrier filter application. Both first generation, full scale cross flow and candle filter elements have been constructed using a homogeneous sintered structure that is an alumina/mullite (A/M) matrix containing a small percentage of amorphous (glass) phase. Laboratory and field evaluation of these and other materials are being conducted to identify, characterize and compare their respective chemical and thermal stability for IGCC and PFBC applications (Alvin et al., 1992).

Westinghouse is developing system designs to effectively package and operate these barrier filter elements and exploit their beneficial operating features (Lippert et al., 1993). An important aspect of the filter system development

includes the selection and qualification of the ceramic filter element and the implementation and integrated operation of these filters in pilot plant IGCC and PFBC facilities. Hot gas filters have been implemented and operated in four different test facilities: Subpilot scale entrained gasifier, located at the Texaco Montebello Research facilities in California, Foster Wheeler Advanced Pressurized Fluidized Bed Combustion pilot plant facilities, located in Livingston, New Jersey, Slipstream of the American Electric Power (AEP) 70 MW (electric) Tidd-PFBC, located in Brilliant, Ohio, and in the Ahlstrom 10 MW (thermal) Circulating PFBC facility, located in Karhula, Finland (Lippert et al., 1993). Table 1 identifies and summarizes the key operating characteristics of these facilities and the type and scale of filter unit tested. This paper updates the results of the pilot plant testing currently being conducted at the Ahlstrom Pyropower PCFB facility.

## PROJECT DESCRIPTION

Ahlstrom Pyropower has designed and built a pilot-scale 34 mm Btu/hr Pressurized Circulating Fluidized Bed (PCFB) Combustor - Ceramic Barrier Filter Testing Facility in Karhula, Finland. This facility is a key tool for verifying the design and scaleup parameters of the first commercial scale PCFB, a repowering demonstration planned by Midwest Power Co. of Des Moines, awarded under Round 3 of the Department of Energy Clean Coal Technologies Program. A key aspect of the PCFB testing is the evaluation of the ceramic barrier filter technology.

In 1989, Electric Power Research Institute (EPRI) and Pyropower Corporation (PPC) signed an agreement (RP3161-1) for EPRI's support in the testing of Asahi Ceramic Tube Filter (ACTF) on the Ahlstrom's 10 MW<sub>th</sub> PCFB pilot plant. The testing of ACTF was completed in June 1992.

**Table 1. Characteristics of Coal Based Test Facilities Used in Hot Gas Filter Testing**

	Entrained Gasifier (Texaco)	Advanced PFBC (Foster Wheeler)		PFBC (AEP-Tidd)	PCFB (Ahlstrom)
		Carbonizer	Combustor		
Facility Size	<2 MW <sub>t</sub>	2 MW <sub>t</sub>	1.2 MW <sub>t</sub>	10 MW <sub>e</sub>	10 MW <sub>t</sub>
Coal Type	Pgh #8	Varied	Varied	Pgh #8	Varied
Coal Feed Method	Slurry	Dry	Dry	Paste	Paste
Oxidant	Air/O <sub>2</sub>	Air	Air	Air	Air
Process Gas	Reducing	Reducing	Oxidizing	Oxidizing	Oxidizing
Method of Sulfur Control	External	In Bed	In Bed	In Bed	In Bed
Precleaning	None	None	None	Cyclone	None
Filter Unit(s)	Cross Flow	Cross Flow	Candle (Cross Flow)	Candle	Candle
Filter Arrangement	Full Flow With Bypass	Full Flow No Bypass	Full Flow No Bypass	Full Flow No Bypass	Full Flow No Bypass
Status	Completed	Ongoing	Ongoing	Ongoing	Ongoing

( ) - Planned

PPC, in cooperation with American Electric Power Service Corporation (AEP), the U.S. Department of Energy (DOE), Electric Power Research Institute (EPRI), and Westinghouse Electric Corporation (WEC), have developed a program for testing of the Westinghouse Candle Filter (WCF). The Westinghouse Candle Filter is being tested in tandem with the PCFB combustor. Testing was initiated in the Fall of 1992. The agreement between EPRI and PPC was modified in the summer of 1992 to include continued support of EPRI for the WCF testing. EPRI's participation has included supply of coal and technical consultation. The design and supply of the Westinghouse candle filter unit was conducted in support of the cooperative agreement (DE-FC21-89MC26042) between the American Electric Power Service Corporation and U.S. Department of Energy. Westinghouse is cost sharing operation of the test facility.

The prime objectives of the WCF test program are:

- I. To demonstrate a commercial scale HTHP filter to operate at the downstream of a PCFB.
- II. To study the performance characteristics of the filter with specific reference to:
  - pressure drop across the candle elements as a function of gas velocity, dust loading, pressure, temperature, and time of operation
  - pulse cleaning frequency, air pressure and quantity required.
- III. To establish the reliability of the system by monitoring:
  - the effect of cleaning under different operating conditions

- the collection efficiencies
- the mechanical capability to withstand normal and abnormal operation transients such as startup, part load, load change, loss of load reflected by sudden drop in pressure or emergency shutdown, and normal shutdown
- the aging of the filter material and metal specimens
- the degradation in physical and chemical properties, if any, due to chemical effects and mechanical disturbances
- the limits of the material to withstand chemical and mechanical extremes
- the downstream dust loading.

The PCFB facility, schematically shown in Figure 1, consists of five major components: compressor, circulating bed combustor, barrier filter, flue gas cooler and pressure reduction station. High pressure air is used to burn coal in the circulating bed combustor unit. The high temperature, high pressure product gases, containing significant particulate (ash/sorbent), pass through the high temperature barrier filter, are then cooled prior to pressure reduction and discharged through the plant stack. The barrier filter removes substantially all of the particulates entrained in the hot flue gas stream.

The primary design parameters for the test facility are given in Table 2. The PCFB test facility is started by first burning natural gas. Air is provided by an electrically driven, radial four-stage compressor. As the plant pressure is increased, the gas burner is switched off and heavy fuel oil is fed into the combustor. When adequate bed temperature and plant load are achieved, coal is fed into the furnace as a paste. The paste is a mixture of crushed coal, sorbent and water. Bed material (either sieved natural sand or limestone) is injected through a separate

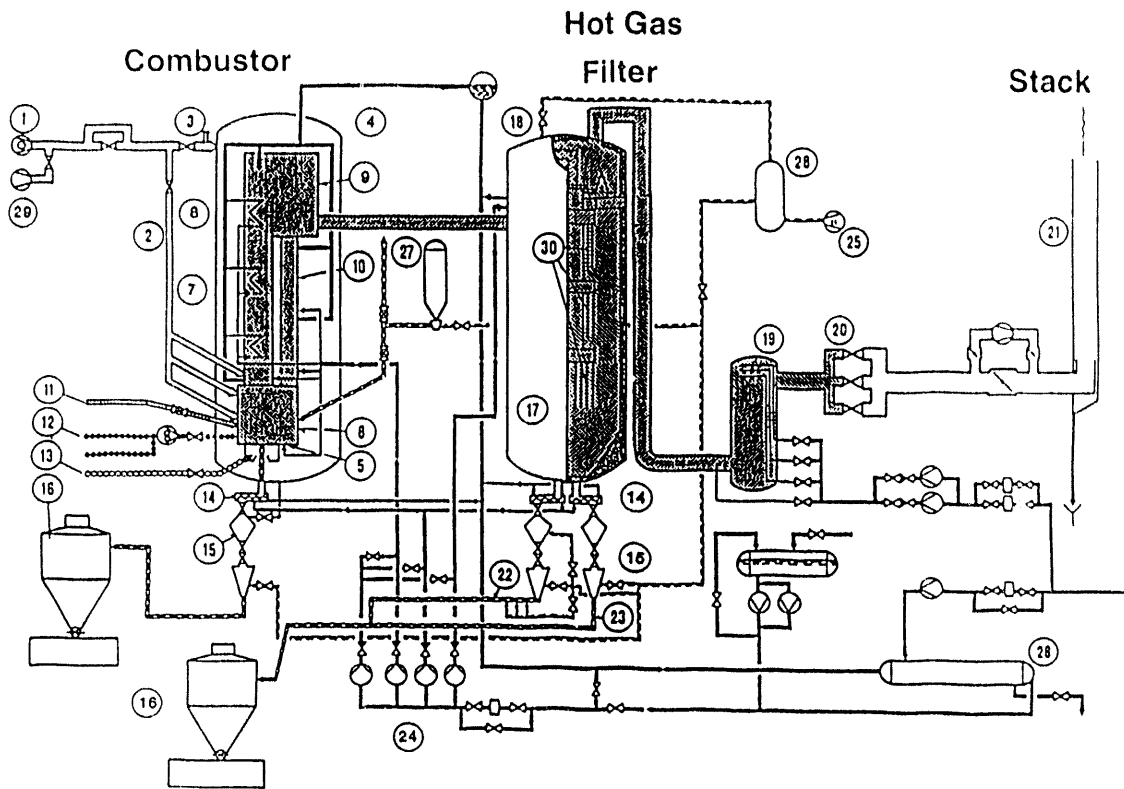
pneumatic line into the combustor. Solids recycle is achieved with a cyclone unit housed within the combustor pressure vessel.

**Table 2. PCFB Test Facility Design Parameters**

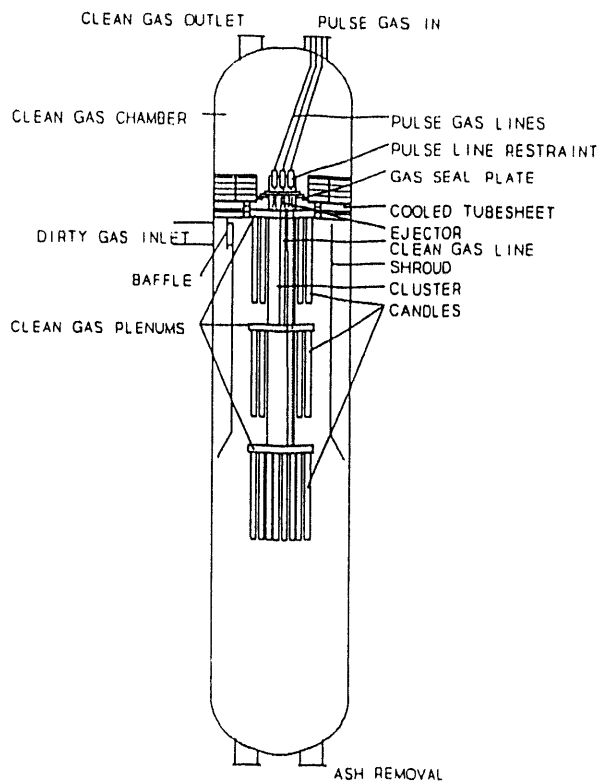
Thermal Rating	34 mm Btu/hr	10 MW <sub>th</sub>
Fuel Feed (Max)	15870 lb/hr	3 kg/s
Air Flow (Max)	43650 lb/hr	5.5 kg/s
Operating Temperature	1615 °F	880 °C
Operating Pressure (Max)	232 psia	16 bar

The Westinghouse candle filter unit, schematically shown in Figure 2, installed in the Ahlstrom facility, was backfitted to the pressure vessel previously housing the Asahi ceramic tube filter unit. The Westinghouse unit consists of a single filter cluster containing 128 candle elements that are arrayed on three plenums. The top and middle plenums each contain 38 candles. The bottom plenum contains 52 candle elements. The total filter surface area is approximately 384 ft<sup>2</sup> (35.7 m<sup>2</sup>). The three plenum sections connect to a common support pipe and hot seal plate arrangement that hangs from a water cooled tubesheet. The water cooled tubesheet is integral with the pressure vessel. The tubesheet and seal plate arrangement form the separation between the dirty and clean gas chambers inside the pressure vessel. The filters are cleaned on-line by high-pressure air pulses that are controlled by fast acting valves and directed through pulse pipes that are connected through the pressure vessel and terminate at the outlet side of the filter seal plate. Three pulse pipes are provided, corresponding to the three plenum sections. Air for the pulse cleaning is provided by a booster compressor (from the main plant air source) that discharges to a high pressure reservoir.





**Figure 1. Ahlstrom/Karhula PCFB - HGF Test Facility**



**Figure 2. Schematic of PCFB Hot Gas Candle Filter**

The test facility and filter unit are instrumented to monitor the key process and filter performance parameters. Measurements include gas flow, system pressure, filter pressure drop, gas temperatures, metal temperatures, and reservoir pressure.

The Westinghouse filter was installed in September 1992 and operated in accordance with schedule given in Figure 3. The testing is divided into three segments with the goal of achieving between 500 to 1000 hours operation in each segment. To date a total of 1741 hours of coal operation has been achieved, Table 3. Test segments 1 and 2 have been completed while testing in segment 3 is ongoing. Results from the first test segment have been reported (Lippert, et al., 1993).

Following Test Segment 1 operation several facility and filter modifications were implemented to address expected root cause events that had resulted in candle element damage during this testing. These modifications addressed the occurrence of process upsets and thermal transient events and include:

- improved main air compressor surge control
- increased the range of control to the fuel feed pump
- improved pulse valve operation
- improved distribution of the gas flow entering the filter unit by modifying the inlet baffle and shroud.

Year	1992				1993												1994													
Month	9	10	11	12	1	2	3	4	5	6	7	8	9	10	11	12	1	2	3	4	5	6	7	8	9	10	11	12		
Filter Modification	█																													
Test Segment I - Illinois #6 Coal - Iowa Rawhide Coal - Newland Coal - Kentucky Coal		█																												
Test Segment II - Illinois #6 Coal - Black Thunder Coal														█																
Test Segment III - Black Thunder Coal																			█			█								

Figure 3. Westinghouse Filter Testing - Karhula PCFB Schedule

**Table 3. Summary of Westinghouse Hot Gas Filter Operation at the Ahlstrom PCFB Facility - Karhula**

<u>Coal</u>	<u>Segment 1 (Hours)</u>	<u>Segment 2 (Hours)</u>	<u>Segment 3 (Hours)</u>	<u>Total</u>
Illinois No. 6	85	221	-	306
Iowa Rawhide	61	-	-	61
Newland	300	-	-	300
Kentucky	270	-	-	270
Black Thunder	<u>-</u>	<u>291</u>	<u>513</u>	<u>804</u>
Total Coal	716	512	513	1741

## RESULTS

This section summarizes Test Segment 2 results and current status of Test Segment 3. During 1026 hours of operation represented by Test Segment 2 and current testing in Test Segment 3, the filter unit and test facility has performed very well and operated without major equipment failures. The filter has demonstrated stable pressure drop and has operated without candle failure. Tables 4 and 5 summarize the filter operating parameters during the Test Segments 2 and 3, respectively. In Test Segments 2 and 3, commercially available clay bonded silicon carbide candle filters (Vitropore<sup>®</sup>) have been utilized, and filter operating conditions maintained below approximately 1560° F (850° C) to better accommodate these candles.

### Test Segment 2

Test Segment 2 test runs, Table 4, were initiated on November 6, 1993 and continued until December 17, 1993. Operation was initiated

utilizing high sulfur Illinois No. 6 coal and an Iowa industrial No. 1 limestone sorbent. Operation on the Illinois coal consisted of a relatively short run (1 to 2 days) due primarily with coal feeding problems associated with frozen coal. Nine separate test runs were made on the Illinois coal with plant load ranging from about 65 to 75%, and operating pressures between 138 to 152 psi (9.5 to 10.5 bar). Stable filter operation was observed through each test segment as determined by the filter baseline pressure drop measurements. In this testing with high sulfur Illinois coal, changes of the coal paste Ca/S (calcium to sulfur) molar ratio and load changes resulted in corresponding changes in the filter inlet dust loading ranging from 7200 to 12,300 ppm. Under these widely varying conditions, the filter operated without issue.

The longest continuous test run on the Illinois coal was approximately 53 hours. Thus, during this period of 221 coal operating hours, the filter was cycled through nine startups and, in addition, experienced over 170 hours of additional operation on oil firing.

**Table 4. Summary of Representative Filter Operation During Karhula Test Segment 2**

<u>Filter Parameter</u>	<u>Test Segment 2 (511 Hrs) 11/6/93 through 12/17/93</u>		
	Illinois #6 Iowa	Black Thunder Iowa	Black Thunder None
Coal Sorbent			
Coal Operation, hrs	221	116	174
Fuel Input, MW	6.9 - 8.2	4.9 - 8.2	7.6 - 8.6
Operating Temperature, °F (°C)	1470 - 1522 (800 - 828)	1333 - 1488 (723 - 809)	1471 - 1509 (800 - 821)
Filter Face Velocity, ft/min (cm/s)	5.5 - 6.5 (2.8 - 3.3)	3.4 - 6.5 (1.7 - 3.3)	6.2 - 6.4 (3.1 - 3.2)
Baseline Pressure Drop, in wg (mbar)	18 - 30 (46 - 75)	7 - 26 (18 - 66)	26 - 29 (65 - 72)
Cleaning Cycle, min	45 - 55	30	20
Dust Loading, ppmw	7200 - 12300	2000 - 6900	6400

**Table 5. Summary of Representative Filter Operation During Karhula Test Segment 3**

<u>Filter Parameter</u>	<u>Test Segment 3* (513 Hrs) 2/7/94 through 3/20/94</u>	
	Black Thunder None	Black Thunder Iowa Ind #1
Coal Sorbent		
Coal Operation, hrs	258	256
Fuel Input, MW	7.8 - 10	4.6 - 5.1
Operating Temperature, °F (°C)	1490 - 1560 (810 - 850)	1273 - 1327 (690 - 720)
Filter Face Velocity, ft/min (cm/s)	5.9 - 7.9 (3.0 - 4.0)	3.0 - 4.2 (1.5 - 2.1)
Baseline Pressure Drop, in wg (mbar)	26 - 48 (65 - 120)	10 - 14.1 (25 - 35)
Cleaning Cycle, min	40 - 55	40 - 55
Dust Loading, ppmw	4000 - 7000	3000 - 6000

\* Additional Testing Planned - May/June 1994

Operation with the low sulfur Black Thunder coal included testing both with and without sorbent addition and under plant load conditions between 50 to 75 percent and system pressure ranging from 152 to 167 psi (10.5 to 11.5 bar). The Black Thunder coal testing occurred in two operating segments of 53 and 238 hours. Stable filter operation was again achieved with the Black Thunder coal. The filter was operated without issue over a range of inlet loadings that depended on plant load and use of sorbent.

Following the 238 hour continuous operation on the Black Thunder coal, the filter testing was terminated by a scheduled shutdown and the unit inspected. Visual inspection showed all the candles to be intact with no ash bridging evident between candles. Ten of the bottom plenum candles were removed (and replaced) for subsequent destructive testing. Ash samples were also collected for analysis. Routine facility maintenance was performed prior to restarting of the unit, initiating Test Segment 3.

### **Test Segment 3**

Test Segment 3 testing, Table 5, was initiated early February 1994, with continuing operation utilizing Black Thunder coal, and operating both with and without sorbent addition. The initial operation in this test segment was at full load conditions, but cold weather again resulted in frozen coal and subsequent coal feeding problems causing frequent interruptions resulting in shutdown or hot standby on oil firing. After approximately 258 hours of coal operation under this mode, plant operation was reduced and maintained to about 50% load conditions. This circumvented the issues caused by the cold weather condition and provided opportunity to obtain important filter performance and operating data at the low load conditions. With this data, the ability to extrapolate filter operating

characteristics and performance over a range of conditions is improved. Later testing in this test segment will focus on full load operation.

During this testing, filter operation was stable with no pressure drop issues. Testing was terminated late March 1994 for scheduled facility maintenance. Preliminary inspection of the filter again showed the filter to be in good operating condition. No dust was present on the clean side of the filter confirming filter performance. Following the initial inspection several candle filters were removed from the bottom plenum and inspected. This inspection showed several of the candle elements to be slightly elongated and some with hairline cracks near the flange neck. Subsequent follow-up showed the manufacturing (firing conditions) of these candles had been modified relative to standard processing. The filter elements from these manufacturing lots (approximately 45 candles) have been identified and removed and replaced with candles manufactured using standard processing. Test Segment 3 candle testing has now been resumed.

**Summary.** Over 1741 hours of operation have now been achieved with the Westinghouse candle filter unit operating in the Ahlstrom PCFB facility. Extended, trouble-free, operating periods have been achieved in which the candle filter unit has demonstrated excellent particle collection efficiency and stable filter pressure drop while operating over a wide range of coal/sorbent types and gas temperature to 1650° F (900° C).

### **FUTURE WORK**

Operation of the Westinghouse candle filter unit will be continued through June 1994. Following this testing the unit will again be inspected and decisions for continued testing made.

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8a.3

Durability of Ceramic Filters

CONTRACT INFORMATION

**Contract Number** DE-AC21-89MC25034  
 (DE-FC21-89MC26042)  
 (DE-AC21-86MC23252)

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**METC Project Manager** Theodore J. McMahon

**Period of Performance** September 30, 1988 - February 28, 1995

**Schedule and Milestones**

Schedule and Milestones FY94

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	S	O	N	D	J	F	M	A	M	J	J	A
Experimental Testing	<hr/>											
Field Analysis	<hr/>											
Model Development	<hr/>											

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OBJECTIVES

The objectives of this program are to identify the potential long-term thermal/chemical effects that advanced coal-based power generating systems have on the stability of porous ceramic filter materials, as well as to assess the influence of these effects on filter operating performance and life.

BACKGROUND INFORMATION

Westinghouse Advanced Particulate Filtration (W-APF) systems have typically operated between temperatures of 540° C and 900° C in both oxidizing and reducing advanced coal-fired process applications (Table 1). First generation Coors alumina/mullite and clay bonded silicon carbide Pall Vitropore 442T and

**Table 1. Westinghouse Advanced Particulate Filtration Systems**

Process	Temp., °C	Conditions	Operating Hours	Candle Filter Type	Facility
AEP PFBC	540-810	Oxidizing	3038	SiC	Demonstration
Karhula CFBC	850-900 690-850	Oxidizing Oxidizing	716 1341	Alumina/Mullite SiC	Pilot Plant Pilot Plant
FW PFBC	700-870 700-870	Oxidizing Oxidizing	58 210	Alumina/Mullite SiC	Pilot Plant Pilot Plant
FW Carbonizer	<760	Reducing	63	Alumina/Mullite	Pilot Plant
Texaco Gasfler	700	Reducing	400	SiC and Alumina/Mullite	Pilot Plant

Schumacher Dia Schumalith F40 candle filters have been utilized in these installations, and to date have operated for test periods as long as 3,038 hours.

The porous alumina/mullite filter matrix consists principally of mullite which co-exists with an amorphous phase that contains anorthite and corundum. In contrast both the Vitropore 442T and Dia Schumalith F40 candle filters consist of silicon carbide grains that are bonded together via a silica-rich binder phase. Both clay bonded silicon carbide candle filters have outer membrane coatings (i.e., a finer grain silicon carbide layer along the Vitropore 442T candles; an aluminosilicate fibrous mat along the Dia Schumalith F40 candles), while the alumina/mullite candle filter is currently manufactured without an external membrane or coating.

When the alumina/mullite, Vitropore 442T or Dia Schumalith F40 filter materials are subjected to 870 °C process temperatures in a

static air environment, the bulk material strength of these porous ceramic filter matrices is generally retained during the first 3,000 hours of exposure (Table 2). The as-fabricated morphology of the oxide and nonoxide-based materials is also retained, with negligible observable changes occurring in the ceramic microstructure (i.e., crystallization, etc.) during the extended static air exposure period. These results are expected in view of the substantially higher manufacturing temperatures that are used to produce the various candle filter elements.

Recent field experience in the W-APF systems has indicated that porous ceramic candle filter elements are subjected to damage and degradation resulting from ash bridging within the various candle filter cluster arrays, thermal process transient events, and pulse gas cleaning cycles which are required for dust cake removal. An assessment of the alumina/mullite and clay bonded silicon carbide candles which experienced 712 and 1,341 hours of operation, respectively, in



**Table 2. High Temperature Static Air Exposure of As-Fabricated Porous Ceramic Candle Filters**

Candle ID	Temp., °C	Time, Hrs	Room Temperature Strength		Hot Strength	
			C-Ring Compression, psi	C-Ring Tension, psi	C-Ring Compression, psi	C-Ring Tension, psi
<b>Alumina/Mullite</b>						
AC-0	—	—	3287 ± 213	4001 ± 477	2841 ± 300 (a)	2996 ± 304 (a)
AC-0	870	1824	NA	NA	2474 ± 265 (b)	2441 ± 172 (b)
AC-0	870	3000	NA	NA	2635 ± 262 (b)	2926 ± 245 (b)
<b>Dia Schumalith F40</b>						
S/482-315C	—	—	1790 ± 112	2308 ± 275	1569 ± 90 (a)	3158 ± 271 (a)
S/482-315C	870	1824	NA	NA	1468 ± 58 (b)	1836 ± 144 (b)
S/482-315C	870	3000	NA	NA	1327 ± 203 (b)	1745 ± 108 (b)
<b>Vitropore 442T</b>						
R4-143	—	—	2585 ± 202	2235 ± 174	2406 ± 247 (b)	2536 ± 360 (b)
R4-148	—	—	2510 ± 158	2654 ± 256	2497 ± 227 (b)	2730 ± 238 (b)
R10-151	870	8	2576 ± 314	3027 ± 114	2646 ± 137 (b)	2683 ± 178 (b)
R3-148	870	1815	2593 ± 156	2734 ± 407	2489 ± 116 (b)	2765 ± 326 (b)
R3-148	870	3000	2335 ± 225	2464 ± 206	2403 ± 87 (b)	2705 ± 165 (b)

(a) Hot Strength Tested at 900°C

(b) Hot Strength Tested at 870°C

NA: Not Available

in the circulating fluidized-bed combustion (CFBC) test facility in Karhula, Finland, and 3,038 hours of operation of the clay bonded silicon carbide candles in the pressurized fluidized-bed combustion (PFBC) test facility at the American Electric Power (AEP) demonstration plant in Brilliant, Ohio, indicated that the porous ceramic filter materials typically lose bulk strength during hot gas filtration. In addition both the oxide and nonoxide-based filter materials undergo various phase changes within the bulk matrix and/or binder phases which reflect operating temperature and process flue gas chemistry. Microcrack formations have been observed within the field-tested alumina/mullite filter matrix, and are also expected to occur within the binder phase, and possibly through the silicon carbide grains in the clay bonded filter materials.

Thermal fatigue has been identified, particularly along the ID wall of the alumina/mullite candle filters as a result of contact with

cold pulse cleaning gas. Similarly a decrease in the fracture toughness of the alumina/mullite matrix resulted during high temperature, pressurized process operation. Alternately creep crack growth has been observed in the Vitropore 442T clay bonded silicon carbide candle filters after 500 hours of service at process operating temperatures of 800-850 °C. Bowing and tilting of the clay bonded silicon carbide candles have frequently been encountered when ash bridges form within the candle arrays, as well as during creep crack growth. Debonding of the outer candle filter membrane, and binder coating that encapsulates the silicon carbide grains resulted when the porous ceramic filters were contacted with a mixed sodium-potassium sulfate phase in the Foster Wheeler PFBC test facility. The resulting phase and microstructural changes, microcrack formations, and thermal conditioning effects that occur during hot gas filtration will be discussed in depth throughout the remainder of

this paper for both the alumina/mullite and clay bonded silicon carbide filter materials.

## PROJECT DESCRIPTION

Westinghouse has undertaken a two phase program to determine the possible long-term, high temperature influence that advanced coal-based power systems have on the stability of porous ceramic filter elements. Accomplishments during the past year have included:

- Developing an understanding of the fundamental changes or "conditioning" mechanisms that have occurred in the alumina/mullite and clay bonded silicon carbide candle filters during test operation in the AEP PFBC and Ahlstrom CFBC test facilities, and identifying how these changes impact bulk filter material strength, and possibly filter operating life.
- Conducting high temperature, thermal fatigue tests in which as-manufactured alumina/mullite, Vitropore 442T clay bonded silicon carbide, and Dia Schumalith F40 clay bonded silicon carbide filters, as well as alumina/mullite candle filters which had been exposed to CFBC operations for periods of up to 716 hours, were subjected to 10,000 pulse cleaning cycles under simulated PFBC process operating conditions. All candles retained their physical integrity after experiencing the extended accelerated thermal fatigue tests.
- Establishing the threshold stress intensity for slow crack growth in the alumina/mullite and clay bonded silicon carbide filter materials at temperature of 800 and 900 °C via interrupted static and dynamic fatigue testing.

A discussion of the results in each of these areas follows.

## RESULTS

### Field Testing

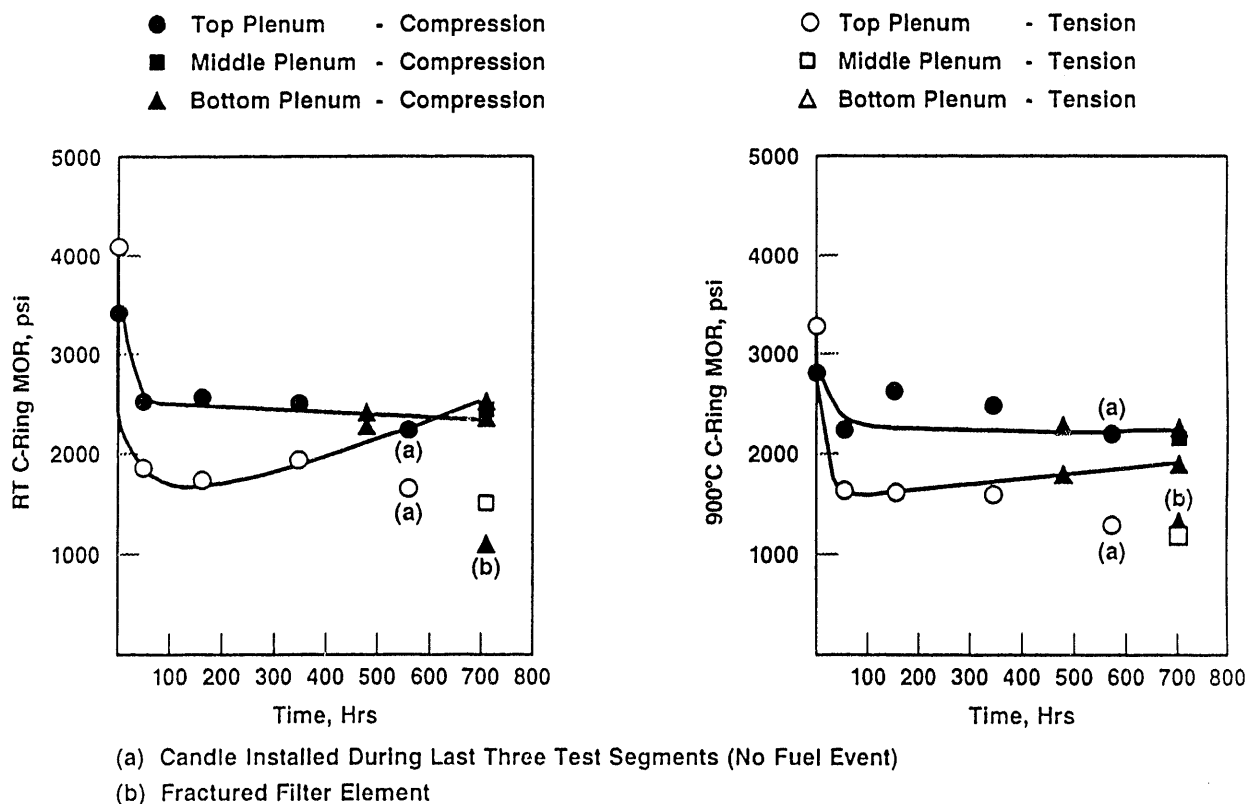
**Characterization of the Alumina/Mullite Candle Filters after Exposure in the W-APF System at the Ahlstrom CFBC Test Facility in Karhula, Finland (Test Segment 1).** One hundred twenty-eight (128), 1.5 m full length alumina/mullite candle filters were installed in the W-APF system in the Ahlstrom circulating fluidized-bed combustion (CFBC) pilot plant in Karhula, Finland prior to October 1992. As shown in Table 3, the W-APF operated for a period of 716 hours at nominal temperatures of 900 °C. Post-test characterization of the CFBC-exposed alumina/mullite candle filters typically included room temperature and process temperature (i.e., 900 °C) C-ring compressive and tensile strength testing, burst strength and fracture toughness analyses, x-ray diffraction (XRD) analyses, and scanning electron microscopy/energy dispersive x-ray analyses (SEM/EDAX).

Figure 1 illustrates the residual bulk strength of the CFBC-exposed alumina/mullite filter matrix as a function of process operating time. An initial loss of the bulk strength of the alumina/mullite filter matrix is experienced within the first 50 to 100 hours of operation in the CFBC process gas environment. The bulk strength of the matrix then appears to stabilize, remaining relatively constant throughout the remainder of the 616 to 666 hours of test operation.

Typically a greater loss of bulk material strength is experienced along the ID surface of the CFBC-exposed alumina/mullite filter matrix (i.e., tensile testing: 46-49% residual ID wall strength vs compressive testing: 71-83% residual OD wall strength; Table 4). Strength reduction is

**Table 3. W-APF Testing at Karhula Using Alumina/Mullite Candle Filters**

Test Segment 1 Run No.	Coal Fire Date	Coal Fire Time, Hrs	Comment	Coal/Sorbent
1	Oct/Nov 92	98	2 Broken Candles	Illinois #6/Iowa #6
2	Nov/Dec 92	45	Fuel Gas Ignition	Rawhide
3	Jan/Feb 93	202	1 Broken Candle In Middle Plenum	Illinois #6/Iowa #6 Newland/Fujiwara
4	March 93	144	Ash Bridging 23 Broken Candles	Newland/Fujiwara Kentucky
5	May/June 93	227	Shroud Shift 10 Broken Candles	Kentucky/Wilbur Newland/Tsukumi



**Figure 1. Residual Bulk Material Strength of the Alumina/Mullite Filter Matrix After Exposure to CFBC Process Gases in the W-APF System**

**Table 4. Percent Strength Retention in CFBC-Exposed P-100A-1 Alumina/Mullite Candle Filters**

Candle ID	Temp., °C	Time, Hrs	Room Temperature C-Ring		Hot Strength, 900°C C-Ring	
			Compression	Tension	Compression	Tension
AC-106RA (T14)	900	45	76	45	80	49
AC-170 (T35)	900	143	76	41	93	51
AC-129RA (T5)	900	345	73	47	88	50
AC-160 (B1)	900	488	69	55	84	55
AC-128RA (T3)	900	572 (a)	65	37	76	40
AC-274 (B22)	900	716	70	61	81	59
AC-204 (M2)	900	716	71	36	78	37

(a) AC-128RA Was Installed and Operated During the Last Three Test Segments and Did Not Experience the Initial Fuel Ignition/Thermal Shock Event

principally attributed to thermal fatigue of the alumina/mullite filter matrix as a result of the ID surface being contacted with the cold pulse cleaning gas.

During the 716 hours of CFBC test operation in Karhula, Finland, the W-APF vessel experienced a fuel ignition event, isolated ash bridging, and a shift in the vessel shroud position -- all of which were considered to contribute to failure of thirty-six (36) candle filter elements throughout the entire test period. As shown in Figure 1 and Table 5, the fractured candles and/or ash hopper candle sections exhibited a lower residual bulk strength, as well as reduced fracture toughness and/or elastic modulus in comparison to candles that remained intact throughout the course CFBC testing.

Optical microscopy analyses were conducted on several of the CFBC field-tested alumina/mullite candle filters in order to determine

whether microcracks were present in the porous ceramic matrix. "Tight" microcrack formations were infrequently identified to extend into the 10 mm alumina/mullite candle filter wall from the ID surface. The position and number of microcracks randomly varied along the length of the candle filter. Microcracks infrequently extended completely across the candle wall thickness. Characterization of as-manufactured alumina/mullite candle filters which had not undergone field testing also indicated the presence of microcracks within the porous ceramic filter body.

Characterization of the alumina/mullite filter matrix utilizing SEM/EDAX analytical techniques indicated that during the initial several hundred hours of test operation, surface crystallization of the amorphous phase occurs in the CFBC-exposed filter matrix. Mullitization (i.e., rod-like grain growth) is evident along the ID pore cavity walls. Nucleation or grain growth within the amorphous phase of the matrix leading

**Table 5. Fracture Toughness and Elastic Modulus of Alumina/Mullite Candle Filters Tested at Karhula**

Candle	Location	CFBC Process Test Runs	Time Hrs	Density, g/cc	Strength, MPa	Fracture Toughness, MPa√m	Elastic Modulus, GPa
<u>1992 Production Lot</u>							
C-35	---	---	---	1.69±0.05	---	0.62±0.03	21.6±1.4
AC-0	---	---	---	1.72±0.01	23.9±1.2	0.71±0.003	25.5±1.8
AC-Section (a)		1-2	<143	1.67±0.02	---	0.43±0.01	12.9±2.2
AC-79	T19	1-2	143	1.73±0.06	18.8±1.3	0.51±0.20	17.7±2.4
AC-121RB	T22			1.69±0.04	15.2±0.9	0.50±0.01	15.3±2.7
AC-150RA	B38	1-4	488	1.69±0.02	13.6±1.8	0.41±0.01	---
AC-128RA	T3	3-5 (b)	573	1.70±0.01	18.4±2.3	0.61±0.01	22.3±7.1
AC-204	M2	1-5	716	1.73±0.11	13.0±3.3	0.61±0.01	19.2±4.5
AC-274	B22	1-5	716	1.71±0.02	19.8±0.9	0.60±0.01	26.8±3.9
AC-80 (c)	B19	1-5	716	1.69±0.01	18.4±1.6	0.70±0.01	27.5±6.5
<u>1993 Production Lot</u>							
BC-337	---	---	---	1.69±0.02	21.7±1.0	0.70±0.004	24.5±0.9

(a) Fractured Section Of Candle Removed From The Ash Hopper.

(b) AC-128RA Was Installed And Operated During The Last Three Test Run Segments.

Note This Candle Did Not Experience The Initial Fuel Event As All Of The Other Candle Filters.

(c) Fractured Candle.

to the formation of mullite, appears to be enhanced as a result of repetitive exposure to cold pulse gas cycling conditions.

After 716 hours of CFBC-exposure, the alumina/mullite filter matrix appears to be nearly fully crystallized. Grain growth is evident throughout the entire ligament structure of the filter body. Extensive mullitization is also evident, particularly along the pulse cycled surface (i.e., 2-4 μm from the ID wall).

As shown in Table 6, the as-fabricated amorphous phase in the alumina/mullite matrix is converted initially into alumina and cristobalite. Further reactions include depletion of the anorthite phase, as the concentrations of mullite (i.e., the principal phase), and cristobalite (i.e., a secondary phase) increase. Note that after 488 hours of CFBC-exposure, the glass phase in the alumina/mullite matrix appears to have been completely converted into a crystalline phase.

After 716 hours of CFBC-exposure, tridymite which is a mineralized form of cristobalite, is not detected within the alumina/mullite filter matrix. This implies that virtually little or no gas phase sodium was sorbed via the alumina/mullite filter matrix during the 900° C CFBC-test operation. Cristobalite or tridymite are expected to crystallize from the silica glass phase that is rejected during production of the anorthite in the alumina/mullite matrix. The formation of cristobalite can lead to displacive phase transformations and large volume changes in the 150-200° C temperature range. Microcrack formations can be expected to result during cooling through this temperature range, unless the grain size and volume fractions are very small.

During the last 227 hours of test operation with the alumina/mullite candle filters, fourteen (14) Vitropore 442T and seven (7) Dia Schumalith F40 candle filters were placed in various positions throughout the top, middle, and

**Table 6. Phase Composition of Alumina/Mullite Candle Filters Tested at Karhula**

Candle ID	Time, Hr (Karhula)	Phase, Wt%						
		Mullite	Anorthite	Alumina	Glass	Cristobalite	Tridymite	Cordierite
P-100A-1 As-Manufactured	—	67.8	29.5	2.7	5*	—	—	vm
Fracture Section Retrieved From Hopper	45	60.9	31.0	2.1	5*	t	—	—
AC-170	143	M	s	m**	5*	-5+	—	—
AC-129RA	345	M	s	m**	<5*	-10	—	—
AC-160	488	M	s	m**	5*	-5	—	—
AC-128RA	572 (a)	M	m	t	—	m	—	—
AC-204	716	M	m	t	—	m	—	—

\* May Be As High As 10%  
 \*\* >2.1%  
 M: Major  
 s: Significant  
 m: Minor  
 t: Trace  
 (a) AC-128RA Was Installed and Operated During the Last Three Test Runs at Karhula. This Candle Did Not Experience the Initial Full Event as All of the Other Candle Filters

**Table 7. W-APF Testing at Karhula Using Alumina/Mullite and Clay Bonded Silicon Carbide Candle Filters**

Candle Filters	Alumina/Mullite* Test Segment 1	Vitropore Test Segment 2			Vitropore Test Segment 3	
Coal Operating Hours	716	221	116	174	258	256
Operating Temperature, °C	850-900	800-828	723-809	800-821	810-850	690-720
Coal	Illinois #6 Rawhide Newland Kentucky	Illinois #6	Black Thunder	Black Thunder	Black Thunder	Black Thunder
Sorbent	Iowa #6 Fujiwara Wilbur Tsukumi	Iowa	Iowa	None	None	Iowa

\* Last 227 Hours of Operation Included 14 Vitropore 442T and 7 Dia Schumalith F40 Candle Filters

bottom plenums in the W-APF vessel. As shown in Table 7, the operating temperature of the W-APF vessel was nominally 900 °C during this phase of testing.

Post-test inspection of several Vitropore 442T filters matrix which had been operated for 227 hours in the 900 °C CFBC process gas environment indicated that bowing and/or tilting of the filter elements had occurred. Westinghouse as-manufactured candle filter dimensional tolerance specifications permit a 3 mm maximum candle bow, and a perpendicularity tolerance of 0.5° over the 1,500 mm candle length (i.e., 12.7 mm from the center line at the bottom of the candle filter end cap). After 227 hours of operation in the 900 °C CFBC gas environment, one candle was shown to have a bow of 5.3 mm, and was 50.8 mm out-from-perpendicular. Bowing and/or tilting of the Dia Schumalith F40 candles was not observed.

Characterization of the residual strength of the 227 hour, 900 °C, CFBC exposed Vitropore 442T candle filter material indicated a loss of bulk material strength, primarily along the ID surface of the candle (Table 8). As shown in Figure 2, both Vitropore 442T and Dia Schumalith F40 clay bonded silicon carbide filters tend to lose bulk material strength within the first 227 hours of 900 °C CFBC test operation.

For the Vitropore 442T matrix crystallization results along the outer surface of the binder phase that coats the silicon carbide grains, as well as along the binder ligaments that bond adjacent silicon carbide grains together. Frequently void formations are evident below the binder coating surface (i.e., adjacent to the silicon carbide grain). Crystallization of the binder phase surface results in the formation of ~1 μm silica-enriched grains (i.e., cristobalite) which follow the contour of the binder coated grains and/or ligament bond posts.

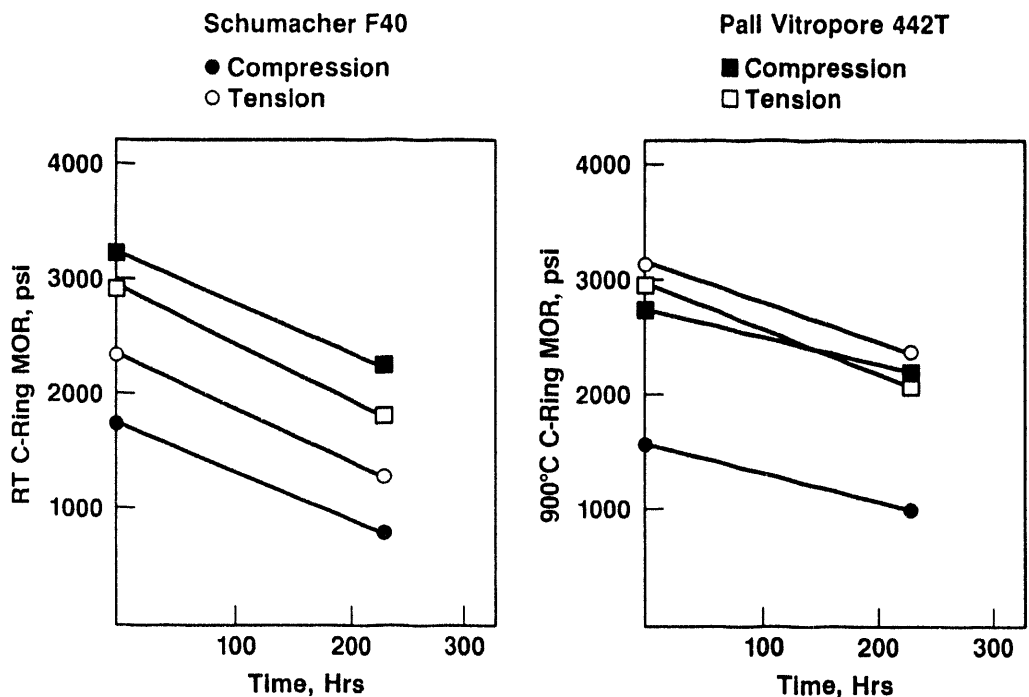
As identified by XRD analysis, the Vitropore 442T matrix initially consists of hexagonal α-SiC, mullite, and a trace of tridymite. When the Vitropore 442T matrix is ground in order to extract the binder phase for analysis, a glass phase and trace concentrations of cristobalite are observed in the powder patterns of the binder phase. After 227 hours of CFBC exposure at 900 °C, the Vitropore 442T matrix consists of α-SiC, mullite, cristobalite, and trace concentrations of tridymite. Extraction and analysis of the binder phase composition indicates the presence of mullite, cristobalite, and tridymite. Note that cristobalite is detected at a higher concentration in the Vitropore 442T matrix after 227 hours of exposure in the 900 °C CFBC gas phase environment. A simultaneous increase in the mullite concentration in the binder phase is also observed. The formation of cristobalite and mullite are expected to result from crystallization of the binder phase as opposed to oxidation of the SiC grain surface.

Similar crystallization of the binder phase occurs within the Dia Schumalith F40 matrix that experienced 227 hours of operation in the 900 °C CFBC gas environment. Silica-enriched phase formations (i.e., cristobalite) are evident along the binder coated silicon carbide grains. Coalescence of the binder phase is also evident, leading to what appears to be the formation of a mullite or rod-like structure. As crystallization of the cristobalite and/or mullite phases occurs, voids or areas of limited binder phase exist along the surface of the silicon carbide grains. Frequently bubble-like, raised pockets of the binder coating are evident along the silicon carbide grains. Out-gassing of the bubble formations is clearly evident by the jagged crack-like features that result from perhaps diffusion of the process gas through the binder to the surface of the silicon carbide grain where reactions lead to the formation of silica and release of carbon dioxide.

**Table 8. Percent Strength Retention in CFBC-Exposed Clay Bonded Silicon Carbide Candle Filters**

Candle ID	Temp., °C	Time, Hrs	Room Temp. Strength		Hot Strength, 830°C		Hot Strength, 900°C	
			C-Ring Compression	C-Ring Tension	C-Ring Compression	C-Ring Tension	C-Ring Compression	C-Ring Tension
S2855/374C (T12)	900	227 (a)	46	55	—	—	63	76
R3-0061 (M19)	900	227 (a)	71	63	—	—	80	69
R4-135 (B22) *	830	512 (b)	59	57	61	64	—	—
R1-147 (B4) **	830	512 (b)	53	58	58	64	—	—
R5-140 (B8) ***	830	1026 (b,c)	67	66	79	75	—	—
R2-151 (B19) ***	830	1026 (b,c)	55	67	68	69	—	—
R6-142 (B20) ***	830	1026 (b,c)	60	71	73	73	—	—
R2-118 (B37)	830	1026 (b,c)	53	63	71	67	—	—
R7-134 (B6)	830	514 (c)	65	61	72	72	—	—
R2-110 (B17)	830	514 (c)	71	78	80	90	—	—

- \* Intact Candle But Bowd
- \*\* Candle Broke During Shipment; Cracks on OD Near Flange; Bow Evident
- \*\*\* Cracks on OD Near Flange; Tilt or Bow Evident; 12-17 mm Elongation of Body
- (a) Test Segment 1
- (b) Test Segment 2
- (c) Test Segment 3



**Figure 2. Residual Bulk Strength of the Vitropore 442T and Dia Schumalith F40 Candle Filters After Exposure in the CFBC Karhula Gas Environment in Test Segment 1**



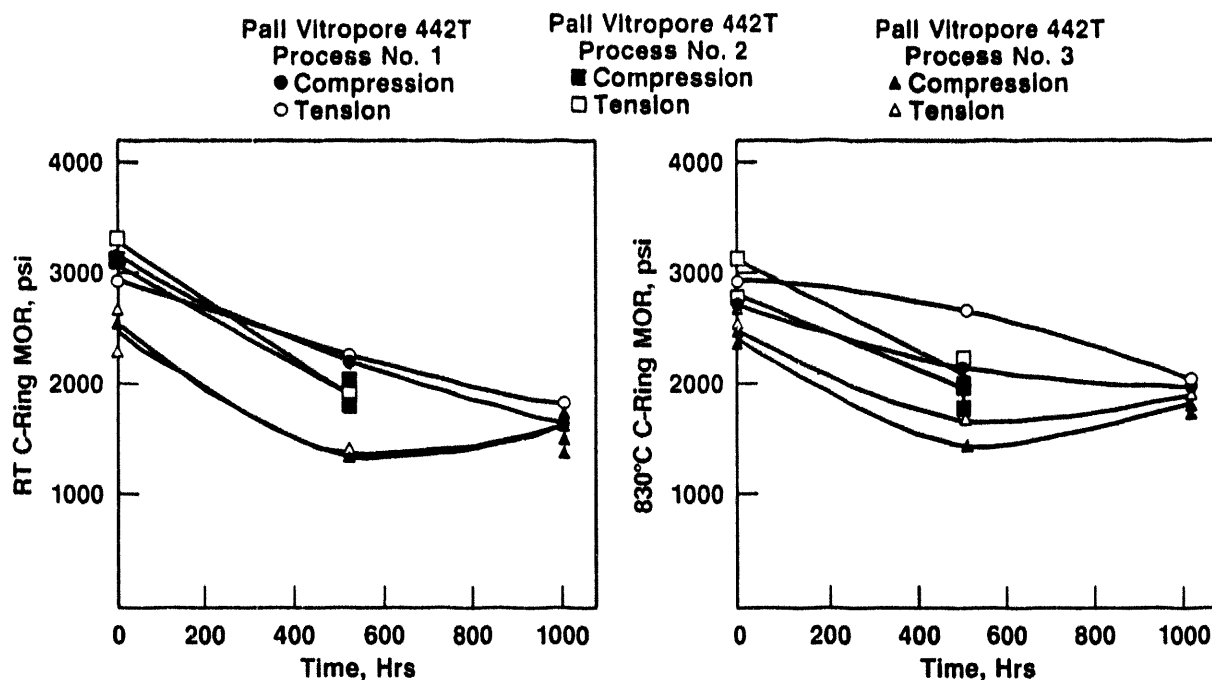
**Characterization of the Vitropore 442T Clay Bonded Silicon Carbide Candle Filters after Exposure in the W-APF System at the Ahlstrom CFBC Test Facility in Karhula, Finland (Test Segments 2 and 3).** The W-APF vessel was entirely re-candled in the fall of 1993 with new Vitropore 442T candle filters for use in testing in Test Segment 2. Testing was initiated on November 6, 1993, and continued for 512 hours at nominal temperatures of 830° C (Table 7). Post-test inspection of ten (10) Vitropore 442T candle filters from the bottom plenum indicated that candle R1-147 which had been located in position B-4 showed evidence of creep crack growth in the area of the body directly below the hemispherical flange. All ten candles subsequently underwent strength and microstructural characterization at Westinghouse, Ahlstrom, SRI, and Pall. Ten new Vitropore 442T candle filters were installed in the W-APF prior to reinitiation of testing in Test Segment 2.

Testing was reinitiated in February 1994, and continued until March 1994, accumulating an additional 514 hours of operation at nominal temperatures of 830° C. Post-test inspection of the Vitropore 442T candles included the removal of fourteen (14) candles from the bottom plenum, six (6) of which showed evidence of creep crack growth (i.e., crack formation and elongation of the candle filter body). Since these candles were identified to have been manufactured via a known production method, and similarly processed candles remained within the bottom array, all bottom plenum candles were removed, and the unit was re-candled once again with new Vitropore 442T candle filters. Post-test inspection of the removed candles indicated that eighteen (18) of the fifty-two (52) bottom plenum candles had developed cracks below their flanges. Based on the manner in which the Vitropore 442T candles had been manufactured, elongation of the candle body ranged between 0-2 mm (i.e., manufacturing process No. 1 and No. 2), and 11-12 mm (i.e., manufacturing process No. 3) for

candles which had been operated in the later 514 hours of CFBC operation. Similar elongation measurements were made for candles that had experienced the entire 1026 hours of 830° C CFBC operation. These measurements indicated that candles which were produced via manufacturing process No. 1 elongated by 1-12 mm, while candles produced via manufacturing process No. 2 elongated by 9-14 mm, and candles produced via manufacturing process No. 3 elongated by 13-26 mm.

Figure 3 illustrates the residual bulk strength of the Vitropore 442T candle filters as a function of filter manufacturing process, and operating time in the CFBC process gas environment. Irrespective of how the Vitropore 442T candle filters were manufactured, all candles appear to lose bulk material strength as a function of time at 830° C. Although we did not have the opportunity to characterize the Vitropore 442T filter matrix after 50-100 hours of test operation, as had been done for the alumina/mullite filter matrix, a general trend of matrix conditioning is evident after 500-1,000 hours of operation in the 830° C CFBC gas environment. As shown in Table 8, a nearly equivalent loss of bulk material strength is indicated along both ID and OD surfaces of the Vitropore 442T matrix. This is in contrast with the residual bulk material strength of the Vitropore 442T matrix after 227 hours of exposure in the 900° C CFBC gas phase environment.

SEM/EDAX analyses illustrate the progression of the changes that occurs within the microstructure of the Vitropore 442T candle filter matrix as a function of process operating time. After 512 hours of exposure in the 830° C CFBC gas phase environment, holes and voids are evident in the binder ligaments that bond adjacent silicon carbide grains together. Crystallization of the binder phase that coats the fine silicon carbide grains in the Vitropore 442T OD membrane



**Figure 3. Residual Bulk Strength of the Vitropore 442T Clay Bonded Silicon Carbide Candle Filters After Exposure in the CFBC Karhula Gas Environment in Test Segments 2 and 3**

occurs, leading to the formation of silica-enriched (i.e., cristobalite) areas. Similarly crystallization of the binder phase occurs along the coarse support silicon carbide grains. Along either the fine or coarse support silicon carbide grains, areas that are adjacent to the crystalline formations appear to either have a thinner binder coating layer, or an exposed grain surface. Typically along the coarse support grains where silica-enriched crystalline phases form are associated depressions which often mark the initiation site for microcrack formation(s) along the binder coating surface.

Bubble formations in the binder phase are evident at the silicon carbide grain-binder ligament post areas. Frequently cracks occur along the surface of the bubbles, indicating that out-gassing has occurred from what was expected to be the release of carbon dioxide from reaction of diffused oxygen and/or steam through the

binder coating with the surface of the silicon carbide grain.

Infrequently "patchy" rod-like crystalline phases (i.e., mullite) are evident within the binder phase that coats the coarse support silicon carbide grains of the Vitropore 442T filter matrix that was exposed for 512 hours to the 830 °C CFBC gas environment. The presence of the rare earth additives used in the manufacture of the Vitropore 442T filter matrix is also evident in the rod-like mullite features.

Moving toward the ID surface of the 512 hour, 830 °C CFBC-exposed matrix are areas of the binder phase that have undergone extensive crystallization. In these areas the crystallized binder appears to be lifted and is somewhat detached from the underlying binder phase and/or surface of the silicon carbide grain. XRD analysis of the Vitropore 442T matrix after 512 hours of

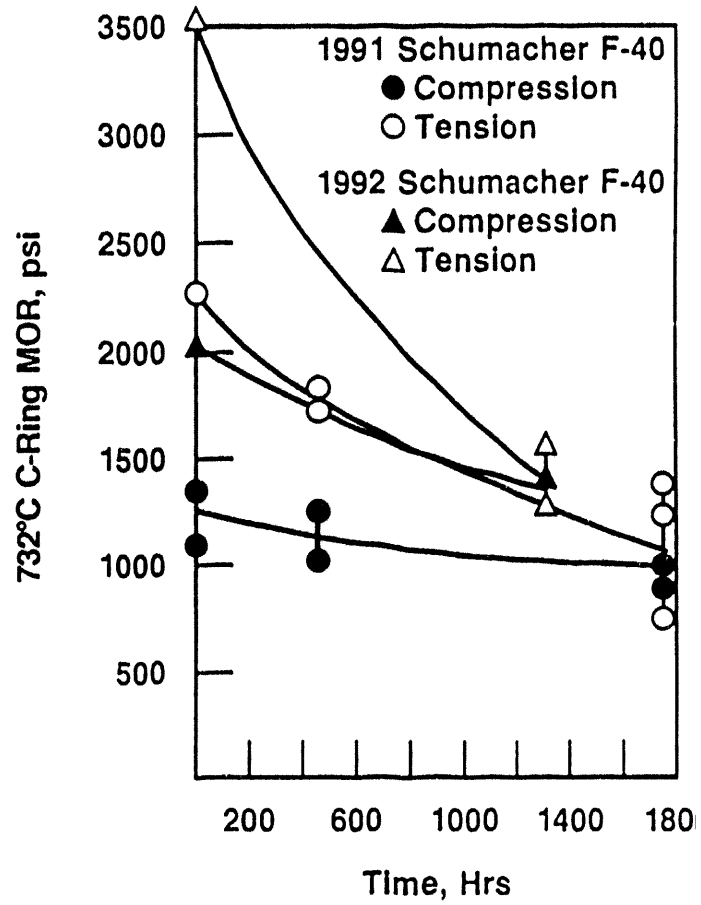
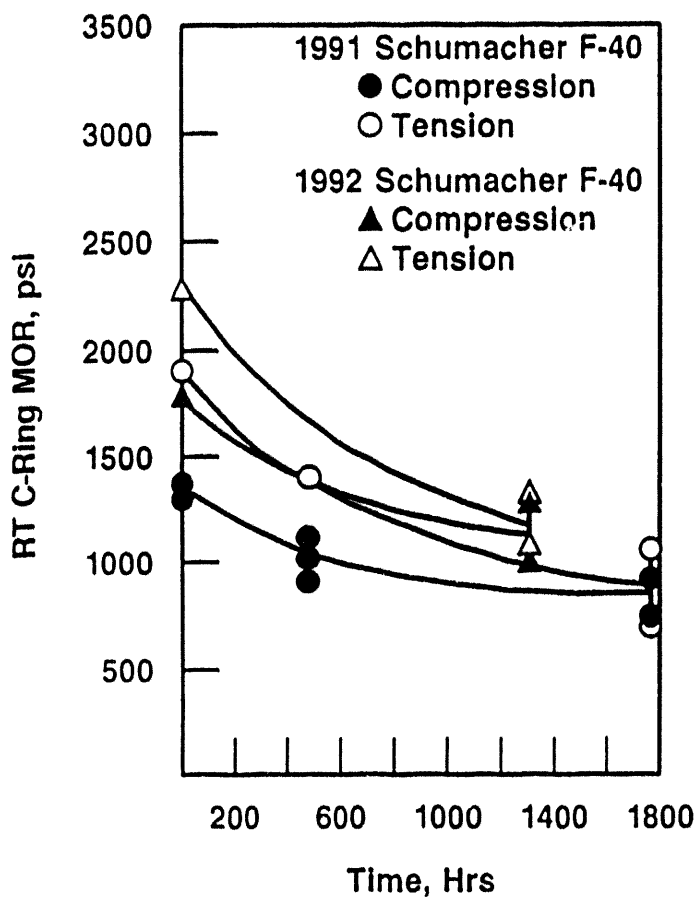
exposure to the CFBC gas environment identifies the presence of  $\alpha$ -SiC as a major phase, traces of cristobalite (<5%), mullite (~5%), and very little glass.

After 1,026 hours of exposure to the 830° C CFBC gas environment, extensive surface crystallization of the Vitropore 442T binder phase is evident. Crystallization continues to result in the formation of a silica-enriched phase (i.e., cristobalite). Void formations are readily evident along the surface, as well as throughout the binder ligament posts. Surface and internal ligament holes are expected in part to reduce the bulk strength of the CFBC-exposed Vitropore 442T matrix.

Due to existence of creep crack growth and elongation of the Vitropore 442T filters in the bottom plenum after 512 and 1,026 hours of exposure in the 830° C CFBC gas environment, all candles were removed and replaced with new Vitropore 442T candles. The W-APF system was then operated for an additional 315 hours at nominal temperatures of 830° C. Candle filters located in the top and middle plenums accumulated 1,341 hours of test operation. Post-test inspection of the W-APF Vitropore 442T candle arrays indicated that one candle from the top plenum, and another candle from the middle plenum had failed as a result of creep crack growth (i.e., crack formation(s) below the hemispherical flange; absence of ash bridging), while an additional ten (10) candles from the top and middle plenums showed evidence of creep growth cracks below the flange of each filter body. Elongation of the candles (i.e., 2-35 mm) resulted within the top and middle plenum filters. Characterization of the residual filter bulk material strength, changes within the microstructure, and creep crack propagation will be initiated when the 315 and 1,341 hour CFBC-exposed Vitropore 442T candles become available.

**Characterization of the Dia Schumalith F40 Clay Bonded Silicon Carbide Candle Filters after Exposure in the W-APF System at the AEP PFBC Test Facility in Brilliant, Ohio (Test Segments 1, 2, and 3).** Hot gas filter operation of the W-APF system at the AEP PFBC Demonstration plant was conducted in three test campaigns in which Schumacher Dia Schumalith F40 clay bonded silicon carbide candle filters were exposed to PFBC process gases at temperatures of 732° C for periods of 464, 1,296, and 1,278 hours (i.e., 3,038 hours of total test operation). Irrespective of the manner in which the Dia Schumalith F40 matrix was manufactured (i.e., higher initial bulk material strength as a result of high firing procedures, etc.), the clay bonded silicon carbide matrix tends to initially lose bulk material strength, and stabilize to a conditioned strength after extended operating time (Figure 4). Preliminary bulk material strength characterization as determined by room temperature and process temperature C-ring compressive and tensile testing indicates comparable strengths of the matrix after 3,038 hours of hot gas filtration as after 1,760 hours of hot gas filtration in the 732° C PFBC gas environment (Table 9). Typically a greater loss of bulk material strength appears to occur along the ID surface of the 15 mm Dia Schumalith F40 filter wall (Table 10), implying that the matrix has undergone thermal fatigue as a result of contact with the cold pulse cleaning gas.

During the course of 3,038 hour PFBC exposure, the fibrous aluminosilicate OD membrane remained intact, preventing fines penetration into the candle filter wall. SEM/EDAX characterization of the matrix indicates that with prolonged filtration at temperatures of 732° C, the clay bonded silicon carbide matrix undergoes extensive crystallization of the binder phase that coats the silicon carbide grains. A silica-enriched phase (i.e., cristobalite) forms as a continuous layer or "crust" (i.e., ~5  $\mu$ m) along the binder coating that



**Figure 4. Residual Bulk Strength of the Dia Schumalith F40 Clay Bonded Silicon Carbide Matrix After Exposure in the AEP PFBC Gas Environment**

**Table 9. Residual Bulk Strength of the Dia Schumalith F40 Matrix as a Function of Exposure Time in the AEP PFBC Gas Environment**

Production Lot	PFBC Temp., °C	PFBC Time, Hrs	Room Temperature Strength, psi		Hot Strength (730° C), psi	
			C-Ring Compression	C-Ring Tension	C-Ring Compression	C-Ring Tension
1991	732	464	1083±148	1438±108	1137±101	1778±246
			1140±120	1424±162	1132±112	1873±174
1991	732	1760	908± 72	1117± 91	1064±72	1418±122
			794± 50	709± 71	1028±94	973±121
			793± 58	711± 89	989±77	885± 54
			793± 39	1016±134	968±96	1252±241
1991	732	3038	720± 57	944±172	890±65	1284±199

**Table 10. Percent Strength Retention in PFBC-Exposed Dia Schumalith F40 Clay Bonded Silicon Carbide Candle Filters**

Production Lot	Temp., °C	Time, Hrs	Test Segment	Room Temperature C-Ring		Hot Strength, 732°C C-Ring	
				Compression	Tension	Compression	Tension
F40 1991	732	464	1	85	75	83	78
F40 1991	732	1,760	1,2	64	47	72	49
F40 1991	732	3,038	1,2,3	56	50	63	55
F40 1992	732	1,294	2	64	54	69	43
F40 1992	732	2,574	2,3	63	51	75	49

originally encapsulated the silicon carbide grains. Below the silica-enriched phase are what appears to be mullite-like rod formations. Mullite is considered to result from the crystallization of alumina and silica that was originally present in the binder. Further effort will be required to determine if oxidation of the silicon carbide grain has occurred, as well as any variations in the microstructure that may have resulted throughout the 15 mm Dia Schumalith F40 candle filter wall.

The complete or nearly complete crystallization of the binder phase, leading to what appears to be a stable binder phase, is considered to be responsible for the bulk material strength conditioning that results after extended exposure of the Dia Schumalith F40 matrix in the 732 °C PFBC gas environment.

**Summary.** Each of the first generation porous ceramic candle filters have performed as complete barrier filters, preventing fines penetration into the clean gas passages of the W-APF systems. As complex ceramic matrices, the alumina/mullite and clay bonded silicon carbide filters undergo numerous phase changes (i.e., crystallization) that result from contact with the high temperature process gas environment. The progressive phase changes and/or crystallization that occurs is considered in part to lead to the early conditioning or stabilization of the bulk material strength. For both the alumina/mullite and Vitropore 442T matrices at 900 °C, thermal fatigue appears to initially reduce the bulk material strength along the candle filter ID surface. The effects of thermal fatigue appears to be mitigated in the Vitropore 442T matrix at CFBC operating temperatures of 830 °C, while our most recent information indicates that thermal fatigue occurs along the ID surface of the Dia Schumalith F40 matrix at PFBC temperatures of 732 °C. Evidence of microcrack formations through the structural matrix of the alumina/mullite filter material can be readily seen, while difficulty is encountered in attempting to establish

the existence of extensive microcrack formation within the clay bonded silicon carbide filter materials.

The manner in which each of the candle filter elements has failed during process operation is relatively unique.<sup>(1,2)</sup> For the alumina/mullite candle filters, thermal shock events resulting from process system transient (i.e., fuel ignition events, etc.), led to thermal-mechanical fracture of the brittle ceramic matrix through the formation of longitudinal cracks which traversed large lengths of the candle filter body. For the Vitropore 442T matrix, creep crack growth and elongation of the filter body at temperatures of 830 °C led to failure of the candle body directly below the hemispherical flange. Frequently bowing or bending of the Vitropore 442T matrix was observed in the absence of ash accumulation or bridging. For the Dia Schumalith F40 matrix, extensive bowing of the matrix can be observed at temperatures of 732 °C when extensive ash bridges form between adjacent candle filters. The accumulation of ash frequently causes failure of the filter elements during cool-down of the W-APF units, resulting in fresh fracture of the filter elements immediately below the hemispherical flange of the candles.

As technology moves forward to mitigate ash bridging within the filtration systems, and process operations are maintained to reduce the impact of thermal transients, operation of the filtration systems can be expected to surpass the number of hot gas filtration hours already accumulated via the existing alumina/mullite and clay bonded silicon carbide filters. It will then be possible to demonstrate the actual filter operating life of these materials, in view of the phase changes that occur during the initial hours of hot gas filtration. Long-term effects of gas phase alkali if released into the filtration system, gaseous sulfur dioxide, trioxide, carbonyl, and hydrogen sulfide, and volatile chlorides, as well as ash or char particulates, can then be

investigated in order to demonstrate the ultimate stability and operating life of the porous ceramic filter materials.

**Surveillance Coupons.** Two separate filter sample surveillance baskets were fabricated for installation at the AEP PFBC site, each of which contained samples of the following porous ceramic filter materials: Coors P-100A and P-100A-1 alumina/mullite; Refractron 505; Schumacher Dia Schumalith F40; Coors mullite; GTE cordierite and cordierite-silicon carbide; and AirResearch sintered silicon nitride. Each material was positioned along an 0.5 in SS316 rod, and was separated via either a Fiberfrax layer or a Nextel 312/MM mat gasket. Inconel 600 metal end plates and a surrounding wire mesh cage were used to fabricate the surveillance baskets which contained the filter material coupons.

One of the surveillance baskets was positioned above the freeboard area in the AEP combustor, while the second surveillance basket was positioned above the tubesheet in the W-APF vessel at AEP. All material samples appeared to remain intact, retaining their physical integrity after ~6,000 hours of exposure in the 815-843 °C (1500-1550 °F) freeboard area, as well as after ~3,038 hours of exposure in the ~732 °C (1350 °F) W-APF tubesheet area. Characterization of these materials will be initiated after the surveillance baskets have been removed from the freeboard and tubesheet areas during the scheduled September-October 1994 maintenance period.

A similar surveillance coupon array was fabricated and placed above the W-APF tubesheet during the first week of June 1994. This array contained samples of the second generation porous ceramic filter materials which included: DuPont filament wound PRD-66; DuPont SiC-SiC continuous fiber ceramic composite (CFCC); 3M CVI-SiC; Pall Vitropore 442T and clay

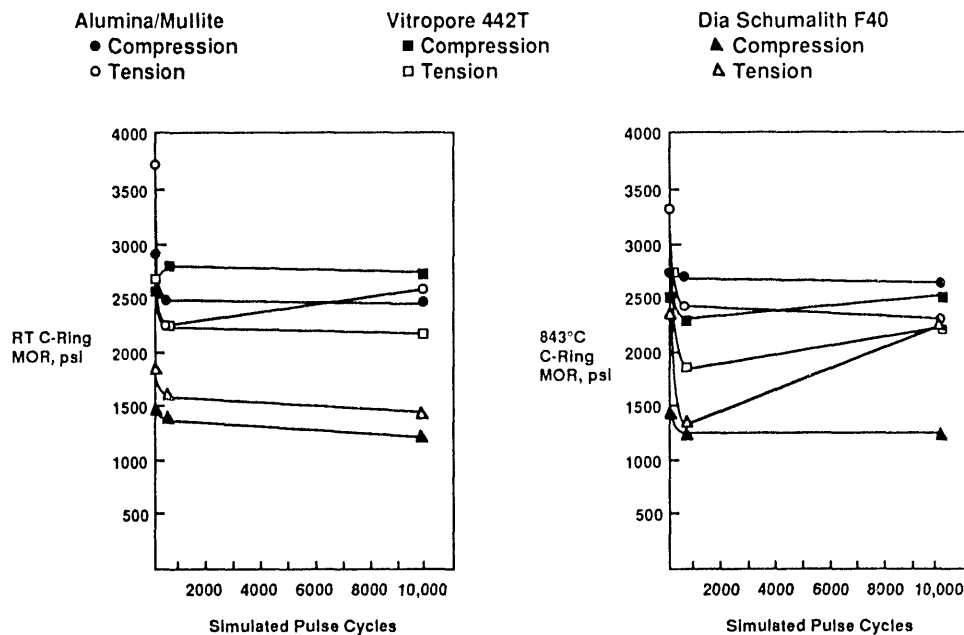
bonded mullite; Brunswick calcium aluminosilicate; Ultramet CVI-SiC reticulated foam; Third Millennium sintered silicon carbide; and swatches of the 3M Nextel 312 and 550 cloth which are utilized in the manufacture of ceramic filter bags or which serve as the structural support fiber or woven braid in the 3M CVI-SiC ceramic composite candle.

### **Accelerated Thermal Fatigue Testing**

Accelerated pulse testing of as-manufactured Coors alumina/mullite, Schumacher Dia Schumalith F40 clay bonded silicon carbide, and Pall Vitropore 442T clay bonded silicon carbide 1.5 m candle filters was completed under high temperature, high pressure (HTHP), simulated pressurized fluidized-bed combustion (PFBC) gas conditions at Westinghouse. In addition two alumina/mullite candle filters which had been exposed for 227 and 716 hours in the circulating fluidized-bed combustion (CFBC) gas environment in the W-APF system in Karhula, Finland, were also tested with the as-manufactured candle filters. Each filter was subjected to temperatures of 843 °C (1,550 °F), system pressures of 100 psig, and either 771 or 10,000 simulated pulse cleaning cycles. Air was utilized as the pulse gas media. During HTHP testing, the pulse duration was 0.4 sec, with 1.5 minute intervals between each delivered pulse. The thermal duty of the pulse gas was 25-50 Btu/pulse-ft<sup>2</sup>, typical of the pulse gas thermal load used in the W-APF system at AEP. All of the first generation monolithic candle filters remained intact during the entire 10,000 pulse cycle test. Cracks were not visibly evident along any of the filters after testing had been completed.

As shown in Figure 5, a greater loss of bulk material strength in the alumina/mullite, Vitropore 442T and Dia Schumalith F40 materials generally resulted along the ID surface of the candle filters, implying thermal fatigue of the matrices had occurred primarily as a result of

### 10,000 Simulated Pulse Cycles



**Figure 5. Accelerated Thermal Fatigue Testing**

contact with the cold pulse gas. Conditioning of the matrices appears to rapidly occur during the accelerated thermal cycling life of the filters (i.e., <771 pulse cycles). Similar conditioning of the filter matrices was evident in the field-tested filter materials.

Candle filters which were subjected to the entire 716 hours of test operation in the CFBC gas environment, including the initial fuel ignition event, in Karhula, Finland, were similarly exposed to 10,000 additional pulse cycles in the W-HTHP test facility. As shown in Table 11, the room temperature OD surface strength of the CFBC-exposed, 10,000 pulse cycled candle appears to be comparable with that of the 716 hour CFBC-exposed candle filter. The data indicate that the additional 10,000 pulse cycles further reduces the room temperature ID wall strength by 30%. At process temperatures of 843 °C, the CFBC-exposed, 10,000 pulse cycled candle appears to strengthen along its OD surface,

while a 15-25% reduction in bulk material strength is observed along the alumina/mullite ID wall.

Cross-sectioning of the 716 hour CFBC-exposed, 10,000 accelerated pulse cycled alumina/mullite candle filter revealed the existence of a circumferential crack that was located at ~1 mm from the candle's ID surface. The crack morphology typically resembled spalling of the surface of a ceramic component which is initially at high temperature and is then rapidly cooled by contact with water or room temperature air. The location of the resulting circumferential crack confirms the Westinghouse thermal stress model calculations which identifies the location of maximum stress within the porous ceramic filter matrix during pulse cleaning as 1-2 mm from the ID surface.<sup>(2)</sup>

Candles that were installed in the later segment of the hot gas filtration test at Karhula did not experience the fuel ignition event. The



**Table 11. Accelerated Thermal Fatigue Testing**

**CFBC-Exposed Alumina/Mullite  
10,000 Simulated Pulse Cycles**

Candle ID	CFBC Process		Simulated Pulse Cycling		Room Temperature Strength		Hot Strength	
	Temp., °C	Time, Hrs	Cycles	Temp., °C	C-Ring Compression, psi	C-Ring Tension, psi	C-Ring Compression, psi	C-Ring Tension, psi
AC-128 RA (T3)	900	572*	—	—	2195 ± 147	1512 ± 181	2118 ± 129(a)	1283 ± 139(a)
AC-122RB (M27)	900	227*	10,000	843	2380 ± 117	2445 ± 299	2652 ± 196(b)	1916 ± 239(b)
AC-135RB (M26)	900	227*	10,000	843(c)	2137 ± 97	2005 ± 133	2354 ± 246(b)	1808 ± 269(b)
AC-274 (B19)	900	716**	—	—	2369 ± 147	2469 ± 503	2254 ± 208(a)	1900 ± 170(a)
AC-162 (M32/M31)	900	716**	10,000	843	2389 ± 106	1766 ± 188	2617 ± 152(b)	1601 ± 250(b)
AC-206 (M28/M32)	900	716**	10,000	843(c)	2474 ± 120	1701 ± 118	2565 ± 150(b)	1407 ± 111(b)

\* Later Portion of CFBC Test Segment, Excluding Fuel Ignition Event

\*\* Complete CFBC Test Segment, Including Fuel Ignition Event

(a) Hot Strength Testing at 900°C

(b) Hot Strength Testing at 843°C

(c) Fallsafe/Regenerator Device Included

post-fuel ignition event candle that was used as our background material strength had 572 hours of CFBC operation, while our post-fuel ignition event CFBC-exposed candles which underwent an additional 10,000 pulse cycles had only experienced 227 hours of CFBC operation. The difference in CFBC operation of 227 versus 572 hours is expected to provide some variation in the strength of the ceramic matrix, making any comparison of the CFBC-exposed and CFBC-exposed, 10,000 pulse cycled candle strength results somewhat difficult. Clearly the 572 hour CFBC-exposed candle had initially lower room temperature and hot strengths along both ID and OD surfaces in comparison to the 227 hour CFBC-exposed, 10,000 pulse cycled candle.

**Interrupted Static and Dynamic Fatigue Testing**

The propensity for flaws to grow in ceramics at applied stress intensities ( $K_{app}$ ) well below the critical stress intensity ( $K_{IC}$ ) has been established.

For dense silicon carbides and nitrides, as well as ceramic fibers, a threshold stress intensity ( $K_{Th}$ ) above which cracks grow and below they do not grow can be established. These values have been estimated using a combination of two experimental techniques – the stressing rate dependence of strength (dynamic fatigue) and interrupted static fatigue testing. The first technique provides an estimate of the stress level required for crack growth and, using this value, the effects of sustained static stress in the range of this estimate on the strength distribution of sample materials subjected to static stress and then rapidly fractured provides a realistic estimate of the threshold stress and, thus  $K_{Th}$ , if  $K_{IC}$  is known. For opaque, complex materials, the crack growth cannot be measured directly, so indirect techniques such as these must be used.

**Approach.** Dynamic fatigue testing was conducted at 800 °C and 900 °C to assess the stressing rate dependence of strength for the

alumina/mullite and Vitropore 442T clay bonded silicon carbide filter matrices. If materials exhibit slow crack growth, then strength increases will be observed with increasing stressing rates. At faster stressing rates, there is less time for slow crack growth (SCG) to occur and thus higher strengths are observed.

The crack growth velocity ( $v$ ) has been shown to be described empirically by the following exponential relationship:

$$v = A K_I^N \quad (1)$$

where  $K_I$  is the stress intensity is raised to the power of  $N$  (the susceptibility for SCG), and  $A$  is the pre-exponential constant. In the absence of a threshold stress intensity for SCG, the dependence of the strength ( $\sigma_f$ ) of the material on stressing rate ( $\dot{\sigma}$ ) is given by the following expression:

$$\sigma_f = \left( B (N+1) \dot{\sigma} S_i^{(N-2)} \right)^{1/(N+1)} \quad (2)$$

where  $S_i$  is the inert strength (strength in the absence of any SCG), and  $B$  is a constant that is related to the crack geometry ( $Y$ ), the critical stress intensity or fracture toughness ( $K_{Ic}$ ), and the two SCG parameters ( $A$  and  $N$ , given in equation 1) via the expression:

$$B = \frac{2}{A Y^2 (N-2) K_{Ic}^{(N-2)}} \quad (3)$$

By measuring the strength of a material using a range of stressing rates, typically several orders of magnitude, and plotting the natural log of the average strength as a function of the natural

log of the stressing rate, the SCG susceptibility parameter  $N$  can be determined from the slope of the regression line which is equal to  $1/(N+1)$ .  $B$  and subsequently  $A$  are then evaluated based on the intercept of the regression.

If a material exhibits a threshold stress intensity for SCG, then the dependence of stress on stressing rate is expressed as follows:

$$\sigma_f = \frac{K_{Th}}{Y \sqrt{c_i}} \left[ 1 + \frac{2 Y (N+1) \dot{\sigma} c_i^{3/2}}{A (N-2) K_{Th}^{(N+1)}} \right]^{1/(N+1)} \quad (4)$$

where  $c_i$  is the initial crack size prior to slow crack growth. The initial crack size, when possible, should be measured directly from the fracture surface of sample. However, this can prove to be a very formidable, if not impossible, task for highly porous materials such as the candle filter materials.

While the dynamic fatigue tests are used to estimate the SCG parameters, the interrupted static fatigue tests are needed to identify the existence of a threshold stress intensity for SCG. The interrupted static fatigue technique involves loading samples to and holding the stress level constant such that the average nominal stress intensity is below the critical stress intensity, but near the threshold stress for SCG, for a certain length of time (typically 1 to several hundred hours).

The choice of static stress levels can be approximated from the results of dynamic fatigue testing. For example, if the dynamic fatigue tests yield decreasing strengths with decreasing stressing rates followed by an apparent strength independence or strength increase at the slowest stressing rates, then a threshold for SCG and/or other mechanisms (i.e., crack blunting) may be operative. If the dynamic fatigue results exhibit

such a behavior, then stress levels in the range of the lowest stresses that exhibited a dependence of stressing rate should be used for the static fatigue tests.

The objective of the interrupted static fatigue tests is to identify stress levels at which some of the material samples survive for the entire hold period and are subsequently loaded to failure, and some of the material samples fail during the static hold period. Stress levels where mixed behavior (i.e., survival and failure) is observed indicate the possible existence of a threshold stress intensity for SCG. Several stress levels and possibly several hold times are needed to accurately determine the threshold stress intensity for SCG. In parallel with this effort, models are being developed to assure that the true thresholds are being determined.

Finally, the critical stress intensity was measured using a single edge-notched C-ring technique at both 800 °C and 900 °C. A notch

was machined into the outer surface of the C-ring at the location of the maximum tensile stress. The notch depth was approximately 20% of the thickness of the C-ring. The sample was then heated to the test temperature, held for fifteen minutes to thermally equilibrate the porous ceramic filter matrix, and was subsequently loaded to failure. The failure load, notch depth and sample dimensions were then used to determine the critical stress intensity or fracture toughness for each material. The ratio of the threshold stress intensity to the critical stress intensity is directly related to the ratio of the threshold stress to critical fracture stress. These ratios can be used as the basis for filter design criteria.

## Results

**Dynamic Fatigue.** Strengths as a function of stressing rates were measured for both materials at 800 °C and 900 °C in air. Table 12

**Table 12. Average Strength and Weibull Parameters for Alumina/Mullite and Vitropore Candle Filter Materials at 800 °C and 900 °C**

STRESS RATE (psi/sec)	800°C				900°C			SLOW CRACK GROWTH PARAMETERS			
	AVERAGE STRENGTH (ksi) (MPa)	m*	$\sigma_0^\dagger$ (ksi) (MPa)	AVERAGE STRENGTH (ksi) (MPa)	m*	$\sigma_0^\dagger$ (ksi) (MPa)	N	B	A	$K_{IC}^\ddagger$ (MPa√m)	
ALUMINA/MULLITE											800°C
10 <sup>+3</sup>	3.1±0.4 21.2±3.0	11.6	3.4 23.1	2.4±0.2 16.6±1.4	23.9	2.5 17.1	48	3.43	25.08	0.84	
10 <sup>+2</sup>	2.9±0.2 20.0±1.6	19.1	3.1 21.1	2.8±0.3 19.5±2.2	12.9	3.0 20.9	900°C				
10 <sup>+1</sup>	2.8±0.1 19.3±0.9	32.0	2.9 20.1	2.6±0.3 17.7±2.4	10.2	2.7 18.8	15	8.47	0.01	1.00	
1	2.9±0.3 19.7±2.0	10.1	3.0 20.8	2.1±0.1 14.6±1.1	15.9	2.2 15.5					
VITROPORE											800°C
17,000	3.3±0.1 22.7±1.0	22.8	3.4 23.2				18	0.79	1.67	0.82	
1,700	3.4±0.1 23.6±1.0	27.9	3.5 24.0	2.7±0.2 18.6±1.4	17.5	2.8 19.2					
850				2.6±0.2 17.8±1.2	17.7	2.7 18.3	900°C				
170	3.1±0.1 21.1±0.8	34.6	4.0 27.5	2.7±0.3 18.8±1.9	10.9	2.8 19.2	13	0.98	2.86	0.75	
17	2.7±0.3 18.5±2.3	8.7	2.8 19.6								

\* Weibull modulus based on maximum likelihood regression method

† Characteristic strength based on maximum likelihood regression method

‡ Single Edge Notched C-ring Technique

lists both the average stresses and Weibull parameters obtained for each stressing rate. In addition, Table 12 also identifies the slow crack growth parameters based on Equations 2 and 3. Figures 6 and 7 show the strength dependence as a function of stressing rate for the alumina/mullite and Vitropore 442T clay bonded silicon carbide C-rings, respectively. At 800° C the alumina/mullite matrix exhibited decreasing strength with decreasing stressing rate for the three fastest stressing rates, followed by a strength increase for the slowest stressing rate. This strength increase may be indicative of crack blunting suggesting the possible existence of a threshold stress intensity. Based on these results, interrupted static fatigue studies were conducted using stress levels ranging between 2,500 and 3,000 psi.

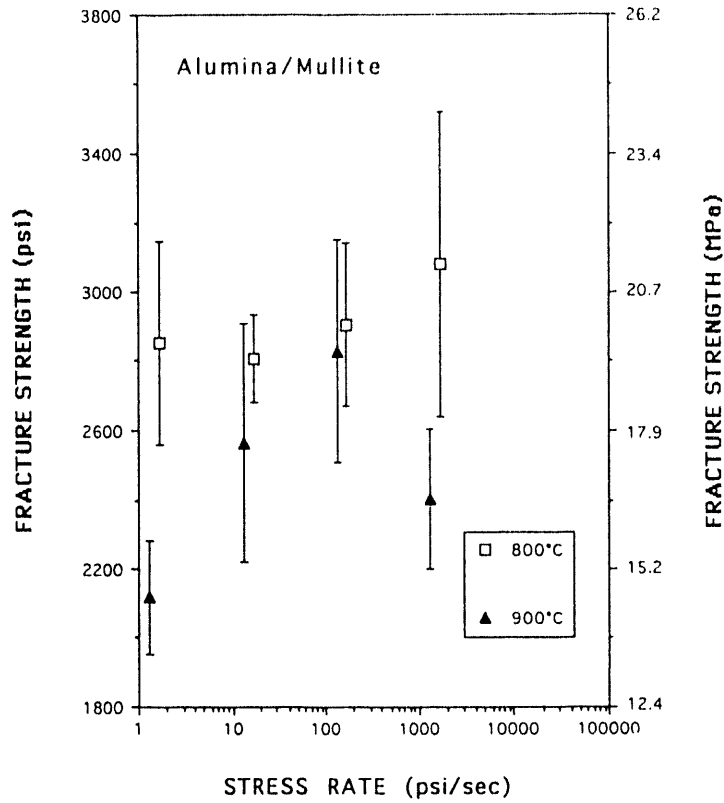
At 900° C, decreasing strengths with decreasing stressing rates were observed for the alumina/mullite filter matrix for the three slowest stressing rates. Unlike the 800° C results, no strength increase was observed at the slowest stressing rate. However, an apparent strength decrease was observed for the highest stressing rate. The exact cause of this behavior is not known. It is likely that the decrease in strength may in part be due to the electronic limitations of the Instron testing machine. The Instron may not be able to capture the true peak load at failure for C-rings tested using the fastest stressing rate. However, this does not explain why similar behavior was not observed for the alumina/mullite C-rings tested at 800° C using the fastest stressing rate. Due to the uncertainty of the failure loads at the highest stressing rate, only the data for the slowest three stressing rates at 900° C were used to estimate the SCG parameters. At 800° C, the data for the slowest stressing rate was not used to estimate the SCG parameters, since a crack blunting mechanism may be operative and dominant over any SCG mechanisms.

Based on these results (Figure 6) and Equations 2 and 3 (assuming no threshold stress intensity), N values of 48 and 15, were estimated at 800° C and 900° C, respectively. The higher the N value the less susceptible a material is to SCG. Thus, increasing the temperature by 100° C significantly increases the susceptibility for SCG for the alumina/mullite material.

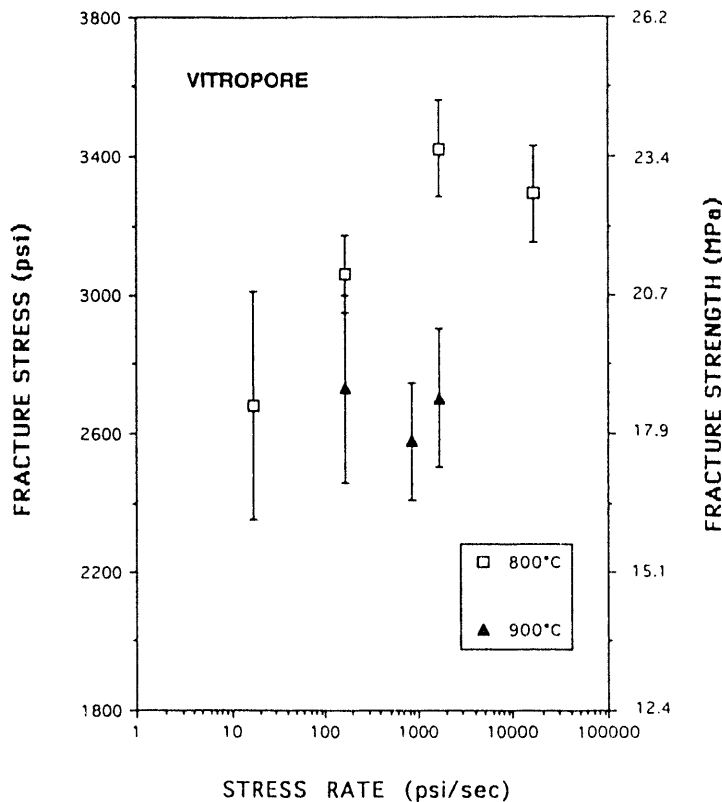
Dynamic fatigue tests conducted at 800° C and 900° C for the Vitropore 442T material (Figure 7) were complicated by the onset of creep, which was observed using the slowest stressing rate at 800° C and the two slowest stressing rates at 900° C. Due to the electronic limitations of the Instron, higher stressing rates did not yield accurate stress measurements (i.e., strengths decrease for fastest stressing rate at 800° C). Thus, one-half order of magnitude increments in stressing rates were used to test the Vitropore 442T clay bonded silicon carbide C-rings at 900° C.

The estimated SCG parameters listed in Table 12, were obtained using the middle three stressing rates (slowest rate not shown because material crept), and the fastest two rates at 900° C. Unlike the alumina/mullite material, the Vitropore 442T material did not exhibit a large reduction of N with an increase in temperature from 800° C to 900° C. However, the creep behavior at the slower stressing rates limited the estimate of the N value to strengths obtained at two stressing rates. The SCG parameters are being used to model the static fatigue behavior of the clay bonded silicon carbide Vitropore 442T candle filter matrix.

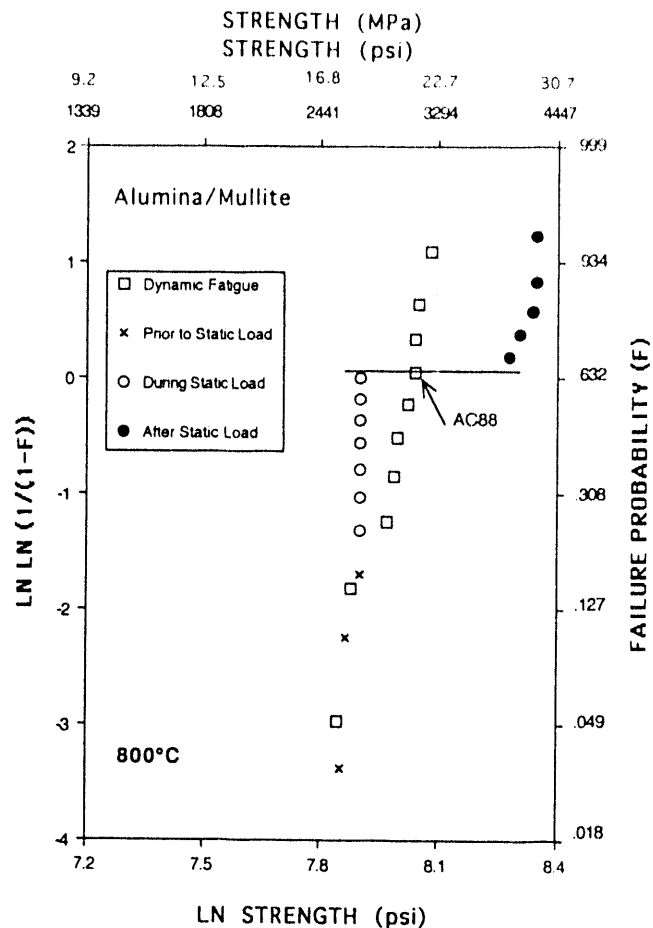
**Static Fatigue.** The preliminary indications of a threshold for sub-critical crack growth for the alumina/mullite material at 800° C are shown in Figure 8. Superimposed on this figure are the results of the static fatigue tests from the previous Weibull strength distribution plot for the dynamic fatigue data (open squares) obtained using the



**Figure 6. Dynamic Fatigue Strength Data as a Function of Temperature for Alumina/Mullite. Errors Bars Represent One Standard Deviation Based on 10 Samples/Stress Rate**



**Figure 7. Dynamic Fatigue Strength Data at 800°C and 900°C for Vitropore 442T C-Rings Loaded in Compression. Error Bars Represent One Standard Deviation Based on 10 Samples/Stress Rate**



**Figure 8. Weibull Strength Distribution of Both the Dynamic Fatigue C-Rings Tested Using a Loading Rate of 0.05 in/min, and the Results of Interrupted Static Fatigue Tests for the Alumina/Mullite Matrix at 800° C Using a Stress of 18.6 MPa (2700 psi)**

same loading rate (0.05 in/min). Samples that failed while loading to the static stress (represented by the symbol x) exhibited a strength distribution equivalent to the dynamic fatigue data. In contrast, material samples that survived (solid circles) exhibited significant apparent strengthening (~40%) as compared to the strength distribution from the dynamic fatigue tests. The material samples that failed during the 10 hour static hold (open circles) represent the transition between those samples that exhibited fast fracture (stress intensities greater than the threshold for SCG), and those material samples that survived (stress intensities less than the threshold for SCG). These results indicate that a stress of 2,700 psi (18.6 MPa) yields stress intensities that straddle  $K_{Th}$  for the size C-rings used in this study at 800° C. Future modeling efforts will attempt to confirm these conclusions.

The cross-over point for this transition in strength behavior of the alumina/mullite filter matrix, illustrated by the horizontal line in

Figure 8, is represented by the alumina/mullite filter matrix obtained from candle filter AC88. Based on the measured fracture toughness ( $K_{Ic}$ ) of 0.84 MPa√m and the fracture stress ( $\sigma_f$ ) of 20.8 MPa (3,013 psi) for the alumina/mullite filter matrix, a flaw size (C) of ~1,060 μm was determined via the following expression:

$$K_{Ic} = 1.24 \sigma_f \sqrt{C} \quad (5)$$

where 1.24 is a constant for the crack geometry (Y) based on the elliptical integral of the second kind for a semi-elliptical surface flaw. Flaws greater than 1,060 μm presumably grew and caused catastrophic failed, while flaws smaller than 1,060 μm were blunted. Thus, the initial estimate of the  $K_{Th}$  is 0.75 MPa√m based on the flaw size of 1,060 μm and the static stress of 18.6 MPa (2,700 psi). The value is a very high fraction of the critical stress intensity (fracture toughness,  $K_{Ic}$ ) indicating that slow crack

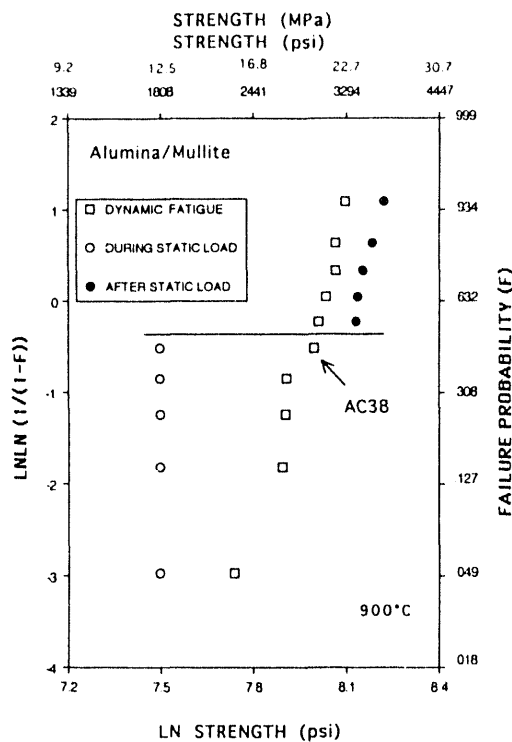
growth will apparently only occur at stresses that are a high fraction of the short term fracture stress ( $\sigma_{Th}/\sigma_{Ic} = K_{Th}/K_{Ic}$ ).

To date, static fatigue tests on ten alumina/mullite C-rings at 900° C using a static stress to 1,800 psi (12.4 MPa) exhibited mixed behavior: five material samples survived, and five failed during the 10 hour static hold with time-to-failure ranging from 45 to 356 minutes. For those material samples that survived and were subsequently loaded to failure, the average strength was  $3,450 \pm 133$  psi ( $23.7 \pm 0.9$  MPa), which represent an apparent strength increase of ~22%.

The indications of a threshold for sub-critical crack growth in the alumina/mullite material at 900° C are shown in Figure 9. Superimposed on this figure are the results of the static fatigue tests from the previous Weibull strength distribution plot for the dynamic fatigue data (open squares)

obtained using the same loading rate (0.05 in/min). Material samples that survived (solid circles) exhibited significant strengthening (~22%) as compared to the strength distribution from the dynamic fatigue tests. Material samples that failed during the 10 hour static hold (open circles) represent the transition between those samples that exhibited fast fracture (stress intensities greater than the threshold for SCG), and those samples that survived (stress intensities less than the threshold for SCG). These results indicate that a stress of 1,800 psi (12.4 MPa) yields stress intensities that straddle  $K_{Th}$  for the size C-rings used in this effort at 900° C. Future modeling will attempt to confirm these results.

The cross-over point for this transition in the strength behavior of the alumina/mullite filter matrix, illustrated by the horizontal line in Figure 9, is represented by the alumina/mullite filter matrix obtained from candle filter AC38. Based on the measured fracture toughness ( $K_{Ic}$ ) of 1.0 MPa√m, and the fracture stress ( $\sigma_f$ ) of



**Figure 9. Weibull Strength Distribution of Both the Dynamic Fatigue C-Rings Tested Using a Loading Rate of 0.05 in/min, and the Results of Interrupted Static Fatigue Tests for the Alumina/Mullite Matrix at 900° C Using a Stress of 12.4 MPa (1800 psi)**

19.5 MPa (2,827 psi) for the alumina/mullite filter matrix, a flaw size ( $C$ ) of  $\sim 1,710 \mu\text{m}$  was determined via Equation 5. Flaws greater than  $1,710 \mu\text{m}$  presumably grew and caused catastrophic failure, while flaws smaller than  $1,710 \mu\text{m}$  were blunted. Thus, the initial estimate of the  $K_{Th}$  is  $\sim 0.51 \text{ MPa}\sqrt{\text{m}}$  based on the flaw size of  $1,710 \mu\text{m}$  and the static stress of 12.4 MPa (1,800 psi). The value, which is  $\sim 50\%$  of the critical stress intensity (fracture toughness,  $K_{IC}$ ) indicates that slow crack growth will apparently only occur at stresses that are about half of the short-term fracture stress.

As for the Vitropore 442T clay bonded silicon carbide matrix, creep behavior is observed under static load condition for temperatures ranging from  $800^\circ\text{C}$  to  $900^\circ\text{C}$ . Testing was aborted after  $\sim 2$  hours because the C-ring completely collapsed the notch (initial notch width of  $\sim 0.5$  in). Examination of the sample revealed no visible evidence of creep crack formation on the outer surface.

Even at  $800^\circ\text{C}$ , significant creep occurs so as to render the elastic stress solutions invalid. This was evident after re-examining the extent of crosshead travel during the  $800^\circ\text{C}$  testing. In contrast to the alumina/mullite material, which exhibited essentially no travel ( $0-0.001$  in) during the static hold, the Vitropore 442T material tested at  $800^\circ\text{C}$  exhibited two orders of magnitude greater crosshead travel than the alumina/mullite material. Although a constant load was maintained at  $800^\circ\text{C}$  for the Vitropore 442T material, the extent of crosshead travel indicates that creep was operative. The stress redistribution associated with creep, yields a non-constant stress, despite a constant applied load.

In addition to the problem associated with creep, further complications are involved due to apparent material variations among the rings

machined from the various candle filters which demonstrated different resistances to crack growth.

**Fracture Toughness.** Fracture toughness measurements were made using the single edge notched C-ring technique for both materials at  $800^\circ\text{C}$  and  $900^\circ\text{C}$  (Table 12). While the Vitropore 442T material exhibited a slight decrease in toughness with increasing temperature, the alumina/mullite showed a slight strength increase.

**Conclusions.** The results of the dynamic fatigue tests indicate that both materials exhibit slow crack growth. The alumina/mullite material shows a large change in susceptibility to SCG (N) from  $800^\circ\text{C}$  to  $900^\circ\text{C}$ , while the clay bonded silicon carbide material (Vitropore 442T), which is more susceptible to SCG, shows only a small change in susceptibility from  $800^\circ\text{C}$  to  $900^\circ\text{C}$ .

The interrupted static fatigue tests for the alumina/mullite material have yielded encouraging results which suggest the existence of a threshold stress intensity. Modelling efforts to more accurately determine the threshold stress intensity at both  $800^\circ\text{C}$  and  $900^\circ\text{C}$  are continuing.

Due to complications of both creep behavior under static load conditions and apparent candle-to-candle variations for the Vitropore 442T material, accurate assessment of a threshold for crack growth will involve considerably more effort. The most appropriate direction to proceed in this area is currently being addressed.

## FUTURE WORK

Similar to the accelerated thermal fatigue testing which was conducted in this program, the



as-manufactured alumina/mullite and clay bonded silicon carbide Vitropore 442T and Dia Schumalith F40 candle filters will be subjected to a series of thermal shock exposures in which the number and/or the severity of the thermal shock (i.e., temperature change) delivered to the filter array will be increased. Field-tested alumina/mullite candle filters which have experienced up to 716 hours of CFBC exposure at temperature at process operating temperatures of 900° C will also be included in this effort.

First generation candle filter surveillance coupons which have been exposed above the freeboard area of the AEP combustor, as well as above the W-APF tubesheet will be characterized in order to demonstrate the possible impact of long-term exposure to PFBC process temperatures, transients, ash and gas compositions on filter material stability. Similarly the Dia Schumalith F40 and Vitropore 442T candles which have been operated in W-APF systems at AEP and at Ahlstrom will continue to be characterized as materials become available. The information generated during both the coupon and candle filter characterization efforts will provide the underlying basis for filter life model development.

## ACKNOWLEDGEMENTS

We wish to acknowledge Ted McMahon and Richard Dennis at DOE/METC for their continued support.

We also wish to acknowledge the efforts of Bob Walko for performing the destructive strength testing on the various candle filter elements. In addition we wish to extend our sincere thanks to Richard Kunkle, George Schneider, Art Fellers, Bill Kuba, Bud Kittle, John Meyer, and Joe Condle for performing the extensive bench-scale and HTHP filtration testing subtasks in this program. We also wish to acknowledge the efforts of Bob Palmquist

and Tom Mullen who have performed the analytical microscopy work throughout all of our material characterization programs. A special acknowledgement is extended to J. P. Singh at the Argonne National Laboratory for his efforts in providing the CFBC-exposed alumina/mullite fracture toughness measurements. And finally we express our thanks to David L. Shelleman at Penn State for performing the static and dynamic fatigue testing.

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## 8a.4

## Properties of Ceramic Candle Filters

### CONTRACTOR INFORMATION

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**METC Project Manager** Thomas P. Dorchak

**Period of Performance** September 9, 1993 to August 22, 1994

**Schedule and Milestone**

### FY94 Program Schedule

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	S	O	N	D	J	F	M	A	M	J	J	A
Material Characterization												
Topical Report												

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### BACKGROUND

The mechanical integrity of the ceramic filter elements is a key issue for hot gas cleanup systems. To meet the demands of the advanced power systems the filter components must sustain the thermal stresses of normal operations (pulse cleaning), of start-up and shut-down conditions and of unanticipated process upsets, such as excessive ash accumulation, without catastrophic

failure; they also must survive the various mechanical loads associated with handling and assembly, normal operations, and process upsets. For near-term filter systems, these elements should survive at operating temperatures of 1550°F for three years.

Ceramic filter materials have proven to be less inert and durable than originally supposed. The monolithic ceramic materials are susceptible to thermal stresses and possibly slow crack growth. The clay-bonded silicon carbide particle

filters have shown substantial creep and chemical degradation of the binder material. In-service failures have occurred for both monolithic alumina mullite and clay-bonded silicon carbide materials.

## **OBJECTIVE**

The objectives of this program have been: (1) the post-test evaluation of candle filters, (2) to measure the material properties of current filter materials, destructively and non-destructively, and to relate these properties and behaviors to in-service performance, (3) to develop an understanding of the material requirements for hot gas filter elements, (4) to develop material property goals for filter materials, and (5) to establish test matrices and a protocol to evaluate candidate filter materials. This last objective is particularly critical to the government and to potential material suppliers. An effective protocol for evaluating candidate materials would increase the likelihood of success when a commitment was made by the government for large scale testing in one of the process development units such as Tidd or Karhula. It would also decrease the financial burden for material manufacturers because they would not scale-up fabrication and/or manufacturing on materials until they had reasonable chance for success in large scale evaluations. The program objectives were defined based on a materials characterization philosophy to (1) measure intrinsic material properties, (2) use test methods, test conditions, and test matrices that are sensitive to applications and the particular material being evaluated, (3) support system designers by providing predictive properties and properties for performance assessment, and (4) support materials manufacturers.

## **PROJECT DESCRIPTION**

The objectives of this program have been pursued by measuring the basic material properties of current materials. The fundamental properties

are tensile stress-strain responses at ambient and elevated temperature, thermal expansion, compressive stress strain responses at room and elevated temperature, thermal conductivity versus temperature and tensile creep. Macro and microstructural evaluations have been performed and various nondestructive inspection techniques have been used to look at bulk response of material, for quality control, for property changes, and for flaws or defects. All of the measurements have been made on new and exposed materials.

The information developed from these tests has been combined with post-exposure failure analyses and calculations of stress states using engineering models to give an overall understanding of the material requirements of a hot gas filtration system. Macrostructurally based material models have been developed to understand fundamental limitations of current materials and to generate information that can be used to develop better materials and assess them with minimum expenditure of time and money.

## **RESULTS**

For the three current HGCU materials - Refractron and Schumacher clay-bonded materials and the Coors alumina-mullite material - limited data packages have been developed. While both the clay-bonded materials most likely experience local microcracking because of thermal stresses during pulse cleaning, the materials are somewhat "damage tolerant." Individual binder bridges are probably cracked, but extensive crack propagation is difficult particularly for the Schumacher material. Results from a single METC/SRI patch test specimen showed that slow crack propagation may be possible in the Refractron materials, but more data are needed.

For both of the clay-bonded materials the high temperature creep of the binder and the degradation of the binder with temperature, time

and environment for operating times up to ~20,000 hours are the critical issues. The two materials appear to be somewhat different with respect to their binder properties and macrostructure. These differences do affect performance somewhat but it is more a matter of degree rather than fundamental differences. The Refractron material (442T) may have a slightly higher temperature capability, 50-100°F, but shows more variability than the Schumacher material. Both materials have limited life above ~1400°F. Life is affected by both creep rates and binder degradation. Hot tears have been observed in the Refractron material after ~1000 hours in Karhula. With some realistic engineering models it may be possible to set creep rate limits for lifetimes up to 5000 hours, but more data are needed on both materials; creep and effects of environmental exposure are interactive. To achieve lifetimes of up to 20,000 hours basic changes in the binder materials are required.

The Coors alumina-mullite material definitely has microcracking during pulse cleaning. The tensile strain-to-failure and thermal expansion data show that temperature difference in the filter on the order of 180°F will cause microcracking. Microcracking from thermal stresses have been verified in a single METC/SRI patch test specimen and by microstructural observations on the ID of rings removed from used filters. The basic material issue at present is slow crack growth from the microcracks. In high temperature creep tests, creep has not been observed but specimens have failed at relatively low stresses after short times. One specimen at 1700°F and 1000 psi failed after <1 hour. A second specimen at 1550°F and 200 psi had not failed after ~ 500 hours.

The previous brief discussions have been concerned with normal operating conditions. Process upsets such as unexpected thermal transients and ash bridging compound the problems creating higher loads and stresses.

Candles bowed by as much as 0.60 in. have been observed. These candles would eventually fail from creep if the lateral load from ash-bridging was not removed.

## **FUTURE WORK**

Evaluation will continue on the clay-bonded and alumina-mullite materials. Generating data and understanding requirements for life prediction will be the focus of the efforts. In addition preliminary assessments are underway on:

I F and P Fibrosic Filters

3M Filter Material

Dupont Lanxide PRD 66 Filters

Dupont Lanxide, SiC Ceramic Composite

Data being generated on these materials include tensile stress-strain curves at room and elevated temperatures and thermal expansion. Southern is working with METC and Westinghouse to develop some nominal property goals for future material manufacturers.

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Spain, J. D., Starrett, H.S., "Physical, Mechanical, and Thermal Properties of Schumacher SiC Filter Material," Technical Report to DOE/METC under DE-AC21-89MC26233, SRI-MME-94-253, March 1994.

Spain, J. D., Starrett, H.S., "Physical, Mechanical, and Thermal Properties of Refractron SiC Filter Material," Technical Report to DOE/METC under DE-AC21-89MC26233, SRI-MME-94-301, April 1994.

Spain, J. D., Starrett, H.S., "Physical, Mechanical, and Thermal Properties of Coors Alumina Mullite Filter Material," Technical Report to DOE/METC under DE-AC21-89MC26233, SRI-MME-94-513, July 1994.

**8a.5**

**Results of Patch Tests**

**CONTRACT INFORMATION**

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**Period of Performance** August 23, 1989 to August 22, 1994

**Schedule and Milestone**

**FY94 Program Schedule**

	S	O	N	D	J	F	M	A	M	J	J	A
Material Characterization	_____											
Patch Testing	_____											
Topical Report	_____											

**OBJECTIVES**

The objectives of this work were to construct, install, and operate a patch testing unit on a hot gas stream at a coal-fired fluidized-bed boiler. A 2000-hour "patch test" was conducted on ceramic disks of materials used in the fabrication of ceramic candles and ceramic cross-flow filters. The primary issues addressed in these tests were the long-term physical, thermal, and chemical stability of the ceramic materials; long-term pressure drop and filtration characteristics of the ceramic filters; potential for irreversible blinding of filter elements; and long-

term performance and reliability of auxiliary hardware, such as the tube sheet and pulse-cleaning systems.

**BACKGROUND INFORMATION**

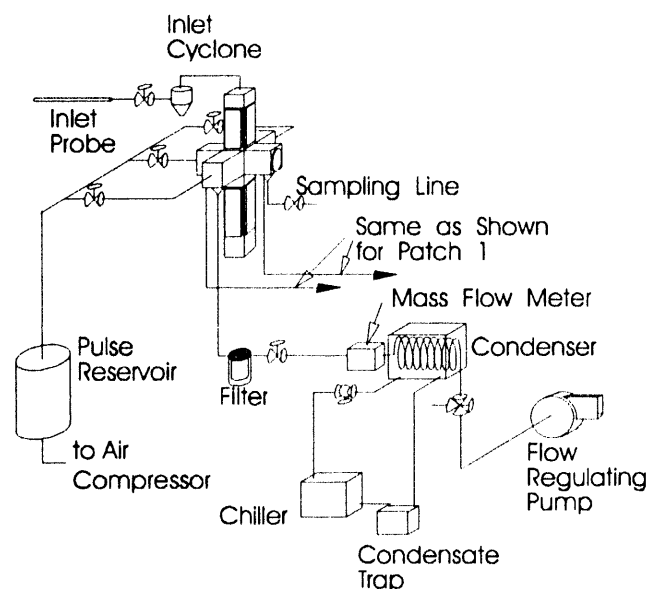
Ceramic filters are among the primary candidates for particulate removal from gas streams at high temperature and high pressure. There are, however, some concerns about the durability of such filters and their resistance to penetration of fine particles into the pores of the material. Because there are many possible sources of particulate matter, operating over a

range of conditions and a variety of filter configurations, tests of full-scale or pilot-scale filter systems are not economically favorable for screening materials for optimal filter characteristics. However, small-scale tests can be very effective, if care is taken to ensure that realistic gas and particulate characteristics are provided. Tests with resuspended dust are questionable because clusters and agglomerates of submicron particles cannot be effectively separated and dispersed, so the particles that would be the most challenging in practical applications are absent in such a test.

All of the above considerations favor the use of a sidestream from an active source of primary particles with appropriate gas chemistry and temperature.

## PROJECT DESCRIPTION

The apparatus (patch tester) developed for this program was designed to test three ceramic filter samples simultaneously and independently on a gas stream extracted from a full-scale system. Figure 1 is a schematic diagram of the device.



**Figure 1. Schematic diagram of Patch Tester**

The samples, or patches, were ceramic disks, approximately 10 cm in diameter, mounted on three sides of a rectangular stack. The fourth branch was used for making measurements of the inlet particle loading and size distribution.

The inlet side of the system was kept at the required temperature by a set of ceramic heaters. Because of the small filtration area, the total gas flow rate required to operate all three filters at a face velocity of 5.6 cm/s was only about 74  $l_{\text{actual}}/\text{min}$ . Independent flow controllers, flow meters, and positive displacement pumps were provided on all four branches of the patch tester. A heat exchanger and back-up filter were provided on each line to protect the moving parts from hot or dirty gases. The system was designed to operate automatically and unattended, and a dedicated computer system was included in the setup for continuous data acquisition. Remote access to the data was available by modem connection to a dedicated telephone line.

## Patch Material Characterization

Before the patch tests were carried out the patches were examined by various techniques, and their mechanical properties were measured. Nondestructive methods were used to determine bulk density, open porosity, and sonic velocity. Advanced methods of spectral acoustics were also applied, including Fast Fourier Transform techniques and measurements of attenuation as a function of frequency. X-rays were made of all disks to determine whether any large voids, cracks or other inhomogeneous features could be found.

The patches were made especially for this test program by the manufacturers of filter candles. Specifications, in terms of granularity, pore size, and membrane thickness corresponded to those of functioning candles, but it cannot be assumed that the manufacturing technique was

an exact analog of that used for candle production. Methods of forming the granules into the desired shape and consolidating the mass before sintering might very well be dependent upon the specific geometry of the product. Of course the manufacturers were aware of the purpose of this program, so we can assume that they made every effort to provide patches that would exemplify the properties and characteristics of their commercial products.

A few of the disks were sacrificed to make samples for measuring tensile strength, compressive properties, and coefficient of thermal expansion. These data provided benchmarks for comparison with measurements on used disks after the test, and they also verified that the disks used were reasonable representative of the corresponding candle filters.

### The Test Program

The patch tester was set up at the Unit 2 atmospheric fluidized-bed combustor on the campus of Iowa State University during the second half of 1992. The sidestream was extracted by methods similar to those used for stack sampling. A probe inserted into the gas stream was used to draw an aliquot of gas. The patches were characterized before the test, as mentioned above, to provide base line data on mechanical strength and permeability. The materials selected for the first test included two different types of clay-bonded SiC, provided by Refractron and Schumacher, and one patch made of an alumina mullite by Coors.

In September 1993 the patch tester was brought up to approximately 840 °C, and gas flow from the duct was initiated. After we completed preliminary measurements of operating parameters and took samples of the inlet particulate matter, we put the device into a timed pulse-cleaning mode, with an interval of 4

hr between cleaning pulses. The gas flow rate was controlled to maintain a constant filtering face velocity of 5.6 cm/s. for each of the three filters independently. The back pulse reservoir was maintained at 410 kPa (60 lb/in<sup>2</sup>) throughout the test. Nearly steady-state operation continued for more than 2300 hr. The system was monitored by computer link from SRI's offices in Birmingham.

### RESULTS

Figure 2 is a plot of pressure drop vs. time for two of the filter patches during a period of about 18 hours in mid-November, which was roughly in the middle of the test program. (The trace for the other SiC patch is not included in this figure because it is nearly identical with the one shown, and is therefore difficult to distinguish in this black and white representation.)

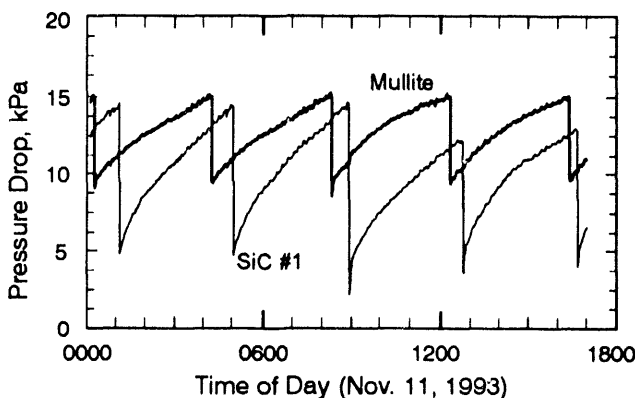


Figure 2. Selected  $\Delta P$  Traces for Two Patches

The mullite disk (as received) had a lower value of permeability than either of the SiC disks. This difference explains the lower values of  $\Delta P$  that can be seen in figure 2 for the SiC filter immediately after cleaning.

The slopes of the curves shown in figure 2 also seem to be slightly different, which is not expected to occur if the filtering face velocities

were the same. The slope is the rate of change in  $\Delta P$ , which should depend only upon the face velocity and the characteristics of the particulate matter (assuming the mass loading in the inlet gas remains constant). Because a planar filtering geometry is used, the  $\Delta P$  traces should be linear, but in every case, there is a perceptible decrease in the slope of the curve with increasing time.

The curvature of these data can be explained in part by the usual tendency for dust cake to come off the filter in patches, rather than as a monolithic whole. The pressure drop rises most rapidly immediately after cleaning, because there are isolated areas of low permeability that fill up quickly, resulting in a rapid rise in  $\Delta P$ . When the dust cake begins to smooth out over the filter surface, the rate of increase in  $\Delta P$  slows. If the gas flow rate is held constant the slope of  $\Delta P$  vs. time should always approach the same asymptotic value.

A downward curvature of the  $\Delta P$  trace could also result from leaks in the system downstream of the filter and ahead of the mass flow meter. As the pressure drop across the filter is increased to maintain a constant total gas flow rate through the flow meter, the rate of gas flow through such a leak would increase. The result would be a decreasing flow rate through the filter, with a concomitant reduction in the rate of dust cake accumulation. Both of these effects would contribute to an external appearance of decreasing dust cake permeability.

Since the asymptotic behavior of the  $\Delta P$  traces for the three filters is similar, we have concluded that the effects of inleakage were relatively small.

### Particulate Collection Efficiency

We measured total particulate loading in the gas at the inlet and outlet of each of the filters.

The inlet mass loading was approximately 1000 ppm ( $1.37 \text{ g/m}^3_{\text{normal}}$ ). Outlet mass loadings and collection efficiencies are given in Table 1.

A sampling time of 5 hours was used for the two SiC discs, and the time was extended to 15 hours for the Coors filter.

**Table 1. Outlet Mass Loading**

Filter	Outlet Mass $\text{mg/m}^3_{\text{norm}}$	ppm	Collection Efficiency
Refractron	19	14	98.6%
Schumacher	*	*	>99.99
Coors	0.015	0.012	>99.999

\*Less than measurement sensitivity.

The relatively low collection efficiency of the Refractron filter was attributed to cracks in the filter which were found when the system was opened and the filters were examined. Reviewing the data, we found a gradual increase in the pressure drop across the backup filter downstream of the Refractron disk. This trend appears to have begun approximately in mid-December. There were no associated process upsets or changes in operating parameters at that time.

The very high values of collection efficiency found for the Schumacher and Coors filters indicates that the gaskets and mountings remained secure throughout the test.

### Pulse Cleaning Effectiveness

During the last few days of the test period, we performed a limited evaluation of pulse cleaning pressure requirements. We gradually



reduced the pressure in the reservoir and observed the pressure drop across the filter disks after cleaning. With a pulse pressure of about 215 kPa (30 lb/in<sup>2</sup>) the clean value of  $\Delta P$  could be recovered consistently. When the pulse pressure was reduced to 110 kPa, the filtering pressure drop could no longer be controlled.

Although these results are specific to the configuration of the patch tester, they indicate that ceramic filters can be cleaned effectively by reverse pulses of gas at relatively low pressure.

### **Effects of Tests on Filter Materials**

Some problems with the ceramic filters were evident when they were examined before removing them from the system. One of the clay-bonded SiC disks (Refractron) was broken, although it remained held in place by the mounting. Subsequent detailed examination of the surfaces of the cracks indicates that the damage began near the center of the disk and propagated outward. It does not appear that excessive stresses arose from the supporting ridges around the periphery of the disc. No corresponding cracks or flaws had been indicated in advance by nondestructive testing.

The other SiC disk (Schumacher) was obviously bowed, the distortion being toward the direction of filtering gas flow. The axial displacement at the center was approximately 2.0 mm. The pressure over the face of the disk during filtration averaged approximately 10 kPa. Of course the pressure during reverse pulsing was considerably greater, but the duration of pulsing was infinitesimal in comparison with the duration of forward gas flow. The bowing indicates that this material is susceptible to creep under relatively small stress when operated at temperatures of about 840 °C or higher.

The creep rate calculated from this measurement confirms that the thermal and

mechanical behavior of this material is dominated by the vitreous binder, which will ultimately limit the range of applicability of this kind of ceramic.

### **FUTURE WORK**

A second test program has been developed for testing three diverse kinds of filter materials. This test, also be carried out at Iowa State University, is scheduled to be carried out this summer.

**8a.6**

**PFBC Dust Cake Studies**

**CONTRACT INFORMATION**

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**Period of Performance** Sept. 30, 1987 to Sept. 30, 1994

**Schedule and Milestones**

**FY94 Program Schedule**

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	S	O	N	D	J	F	M	A	M	J	J	A	S
<u>Phase III</u>													
Testing													
Evaluation													
Report													

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**OBJECTIVES**

The Westinghouse Electric Corporation, Science & Technology Center is developing an Integrated Low Emissions Cleanup (ILEC) concept for high-temperature gas cleaning to meet environmental standards, as well as to provide economical gas turbine life. The ILEC concept simultaneously controls particulate, sulfur, alkali, and other contaminants in high-pressure fuel gases, or combustion gases, at temperatures up to about 1700° F in advanced, coal-fired, power

generation systems. The overall objective of this program is to demonstrate, at a bench scale, the conceptual, technical feasibility of the ILEC concept for multi-contaminant control, and to provide test data applicable to the design of subsequent field tests. The current program objective is to conduct ceramic barrier filter testing under simulated PFBC conditions to deal with filter cake permeability and pulse cleaning issues that have been identified in recent PFBC filter field testing.

## BACKGROUND INFORMATION

Westinghouse filter field testing is currently being conducted at three PFBC installations at temperatures up to 1700° F. The characteristics of these hot gas ceramic barrier filter facilities are summarized in Table 1. These facilities provide critical data supporting the design of five new PFBC filter facilities:

- Foster Wheeler (FWDC), Phase 3, Advanced-PFBC facility (Livingston, NJ),
- Southern Company Services, PCD test facility (Wilsonville, Alabama),
- Foster Wheeler, Wilsonville Advanced-PFBC plant,
- Midwest Power, Pyropower Circulating-PFBC, DMEC-1, Clean Coal Technology Program,
- Air Products/Foster Wheeler Advanced-PFBC, FREMP, Clean Coal Technology Program.

Test observations made at the three PFBC filter facilities are summarized in Table 2, and indicate that issues with filter cake permeability, pulse cleaning, fly ash bridging, and fly ash bulk flow can arise in PFBC, depending on the nature of the fly ash and the filter operating conditions.

## PROJECT DESCRIPTION

The ILEC development program has a three phase structure:

- Phase 1 - Laboratory-Scale Testing
- Phase 2 - Bench-Scale Equipment Design and Fabrication

- Phase 3 - Bench-Scale Testing

Phases 1 and 2 have been completed (Newby, et al., 1990). ILEC feasibility testing is now being conducted in Phase 3. The current test program is focussing on PFBC filter cake issues. Six test sets have been defined, and are being carried out in the following sequence:

- 1) Cake Permeability Tests (impact of temperature)
- 2) Cake Pulse Removal Tests (impact of temperature)
- 3) Additive Tests
- 4) Pulse Intensity Tests
- 5) Continuous Operation Tests
- 6) Sulfur/Alkali Removal Tests

The first set of tests have nearly been completed, and are highly controlled tests examining changes in filter cake permeability primarily as a function of temperature. The results of these tests are discussed in this report. The second set of tests are directed toward measuring the impact of temperature on pulse cleaning effectiveness. Test set three is again directed toward difficult filter cake conditions, examining the performance of various filter cake additives and the additive application procedures. The impact of the pulse cleaning intensity is measured in test set four. Test set five will confirm observations for selected test conditions during several longer duration tests. Finally, test set six will measure sulfur removal and alkali removal performance in the filter vessel. This final test set will also note the influence of the injected sorbents on the filter cake behavior.

**Table 1. Westinghouse PFBC Filter System Characteristics**

	<u>AEP Tidd Brilliant, OH</u>	<u>Foster Wheeler Livingston, NJ</u>	<u>Ahlstrom Pilot Karhula, Finland</u>
Facility Size (MW <sub>e</sub> ):	30	1.2	10
Number Candles:	384	14-22	128
Gas Flow (acfm):	7500	300	3070
Face Velocity (ft/min):	7	2-8	7-8
Pressure (psig):	135	100-200	160
Temperature (° F):	1300-1550	1450-1700	1450-1650
Fuels/Sorbents:	Pgh #8/Plum Run dolomite	Several	Wide Range
Hours of Test Operation			
- Longest Continuous:	550	72	280
- Cumulative:	3000	800	2000

**Table 2. General PFBC Filter Cake Observations**

	<u>AEP Tidd Brilliant, OH</u>	<u>Foster Wheeler Livingston, NJ</u>	<u>Ahlstrom Pilot Karhula, Finland</u>
Fly Ash Size: ( $\mu\text{m}$ )	1-3	5 - 25	12-22
Dust Loading: (1000 ppmw)	0.5-1	2 - 30	4 - 18
Permeability:	Low Decreases T>1400F	Medium to High Decreases T>1500F	Medium to High
Pulse Frequency: (1/hr)	1-2	0.5-3	1-2
Pulse Intensity Required	Large	Medium to Low	Low
Occurrence of Bridging and Hard Residue	At T>1400F	At T>1600F	None
Cake Composition	Highly Sulfated and Carbonated High $\text{CaMg}_3(\text{SO}_4)_4$	Highly Sulfated and Carbonated Alkali Eutectic Sublayer	Little Information
Vessel Drainage Performance:	Periodic Difficulties	Periodic Difficulties	Good

## RESULTS

### Bench-Scale Facility Description

A natural gas-fired, bench-scale, high-pressure, high-temperature, ceramic barrier filter test facility is being used to study ILEC performance under simulated PFBC conditions. The objective of the bench-scale simulation is to produce a gas having pressure, temperature, gas composition (SO<sub>2</sub>, alkali content, and particulate content), and fly ash particulate characteristics similar to actual coal-fired PFBC. An assembly drawing of the facility is shown in Figure 1. A horizontal, natural gas combustor is attached to the vertical, filter pressure vessel. The combustor is a carbon steel pressure vessel, refractory-lined, with an internal, high-alloy steel liner. It is designed to operate entirely on natural gas, or with coal and natural gas combinations. Sulfur sorbents, alkali sorbents, or deposit additives are also injected into the secondary zone of the combustor.

The uncooled tubesheet, with a 31-inch plate diameter, has been designed with commercial candle holder features that included fail-safe/regenerator devices. The tubesheet, capable of holding up to 19 candles, supports four ceramic candle filter elements in this program, each 1.5 meter in length. The inlet gas enters the vessel horizontally, near the level of the base of the candles, with no baffle to deflect the inlet jet. The four candles are located so that direct impaction by the inlet gas stream is avoided. The candles are pulse cleaned simultaneously. The outlet piping section incorporates gas sampling for particulate content, alkali content, sulfur content, and other gas species of interest, such as CO<sub>2</sub>, as well as instrumentation to measure total gas flow and temperature.

The cake permeability tests have been conducted under the following nominal conditions:

- Face velocity: 7 ft/min (fixed gas volumetric flow at all conditions)
- Pressure: 100 psig

Key measurements made during the tests are:

- Gas and fly ash mass flow rates,
- Filter gas inlet and outlet temperature,
- Tubesheet  $\Delta P$ ,
- Gas CO<sub>2</sub> content.

The tests are conducted by heating the unit to 1300° F, at the test pressure (100 psig) and face velocity (7 ft/min). A filter cake, having specified pressure drop, is deposited on the ceramic candles, and fly ash feeding is halted. The system temperature is then increased in a series of steps to about 1650° F, holding at each step for about 1/2 to 1 hour. The system temperature is then reduced in steps down to 1300° F. Figure 2 is a plot of test data from a typical run, showing the temperature record, the face velocity, the filter cake weight deposited per unit area of the filter elements, and the tubesheet pressure drop record.

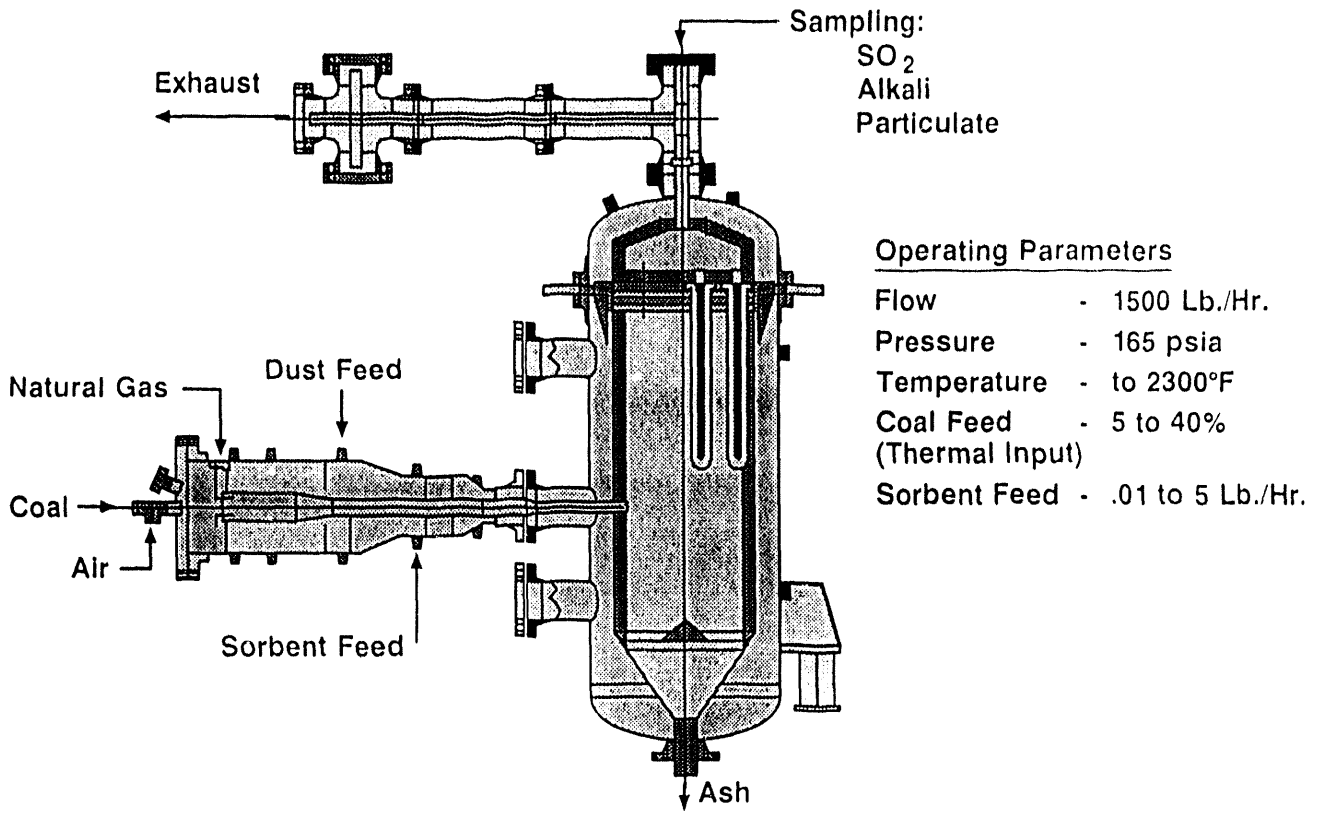
Filter cake permeabilities are extracted from the pressure drop data recorded during the tests. The total system pressure drop is contributed by several separate components:

$$\Delta P = \Delta P_f + \Delta P_r + \Delta P_c + \Delta P_p \quad (1)$$

where  $\Delta P$  is the total system pressure drop, and the component subscripts represent the filter elements (f), the filter cake permanent residue (r), the active filter cake (c), and the other system losses (p). The filter cake mass permeability ( $k_m$ ) is defined by the equation

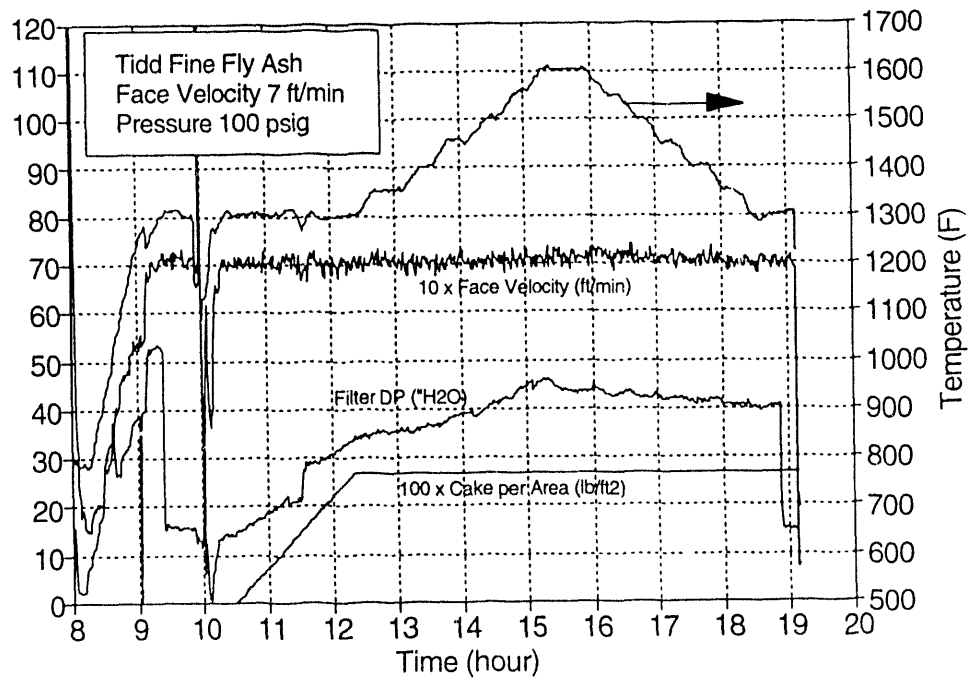
$$\Delta P_c = \mu U M / k_m \quad (2)$$

where  $\mu$  = gas viscosity,  $U$  = face velocity, and  $M$  = mass per unit area.



**Figure 1. Test Facility Assembly Drawing**

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**Figure 2. Typical Filter Cake Permeability Test Record**

In each test, it has been observed that the filter cake permeability falls as the temperature is initially increased above 1300° F, and it reaches a minimum value at the temperature where the calcium in the filter cake calcines from CaCO<sub>3</sub> to CaO. As the temperature is again reduced, the filter cake permeability increases slightly, but does not return to its original value because irreversible structure changes in the filter cake have occurred. This behavior is illustrated for a typical run in Figure 3, showing the mass permeability and temperature as a function of time.

Thirteen tests have been completed using the following PFBC fly ashes:

- Tidd bubbling-PFBC fine fly ash,
- Tidd bubbling-PFBC primary cyclone,
- Grimethorpe Red fly ash (Grimethorpe Run 129),
- Karhula circulating-PFBC fly ash.

All have shown this same behavior. This behavior is explained with respect to calcium-based sorbent material sintering phenomena. It is known from prior evidence (eg., studies of calcium sorbent surface area on injection into hot gas streams, and calcium-based deposits on boiler heat transfer surfaces) that sintering is a fast process at these temperatures. The rate and extent of calcium sintering increases with:

- increased temperature
- reduced particle size
- increased CO<sub>2</sub> and H<sub>2</sub>O in gas
- increased content of Na and K in the cake

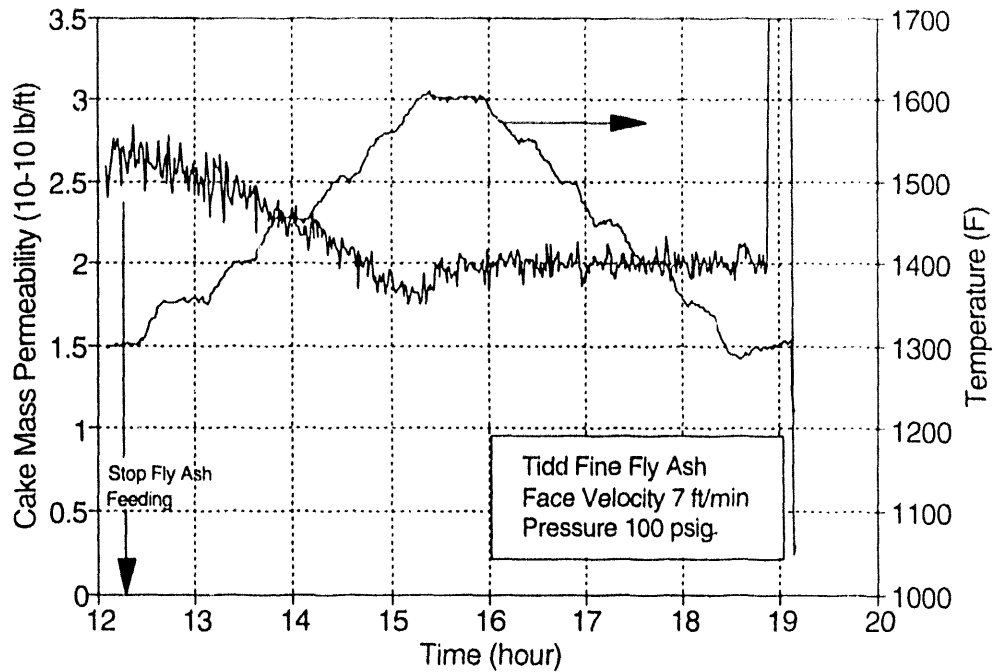
- increased content of CaCO<sub>3</sub> and CaSO<sub>4</sub> vs CaO and MgO
- occurrence of sulfation and carbonation reactions

Table 3 lists some of the test results obtained, showing variations with pressure, face velocity, and fly ash source. The results are consistent with field test trends, and direct comparison of the laboratory test and field test permeabilities are shown in Table 4. The laboratory permeabilities are generally greater than the field test permeabilities, and this may be because the CO<sub>2</sub> and SO<sub>2</sub> partial pressures are lower in the laboratory tests than in the field tests. Figure 4 plots the CO<sub>2</sub> partial pressure and filter operating temperature for Tidd PFBC filter tests, for Karhula CPFBC filter tests, for FWDC filter tests, and for the laboratory filter cake permeability tests.

Conclusions reached in the program are:

- Laboratory filter cake trends are consistent with field unit data trends;
- PFBC filter cake permeability is largely controlled by sintering of limestone constituents in the fly ash;
- Filter cake sintering is induced by CO<sub>2</sub> and SO<sub>2</sub> in the gas;
- Increased filter cake sintering results from:
  - higher CO<sub>2</sub> and SO<sub>2</sub> pressures,
  - higher temperatures,
  - finer fly ash particles,
  - calcitic limestone vs dolomitic limestone;

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**Figure 3. Typical Filter Cake Mass Permeability Result**

**Table 3. Filter Cake Permeability Test Results**

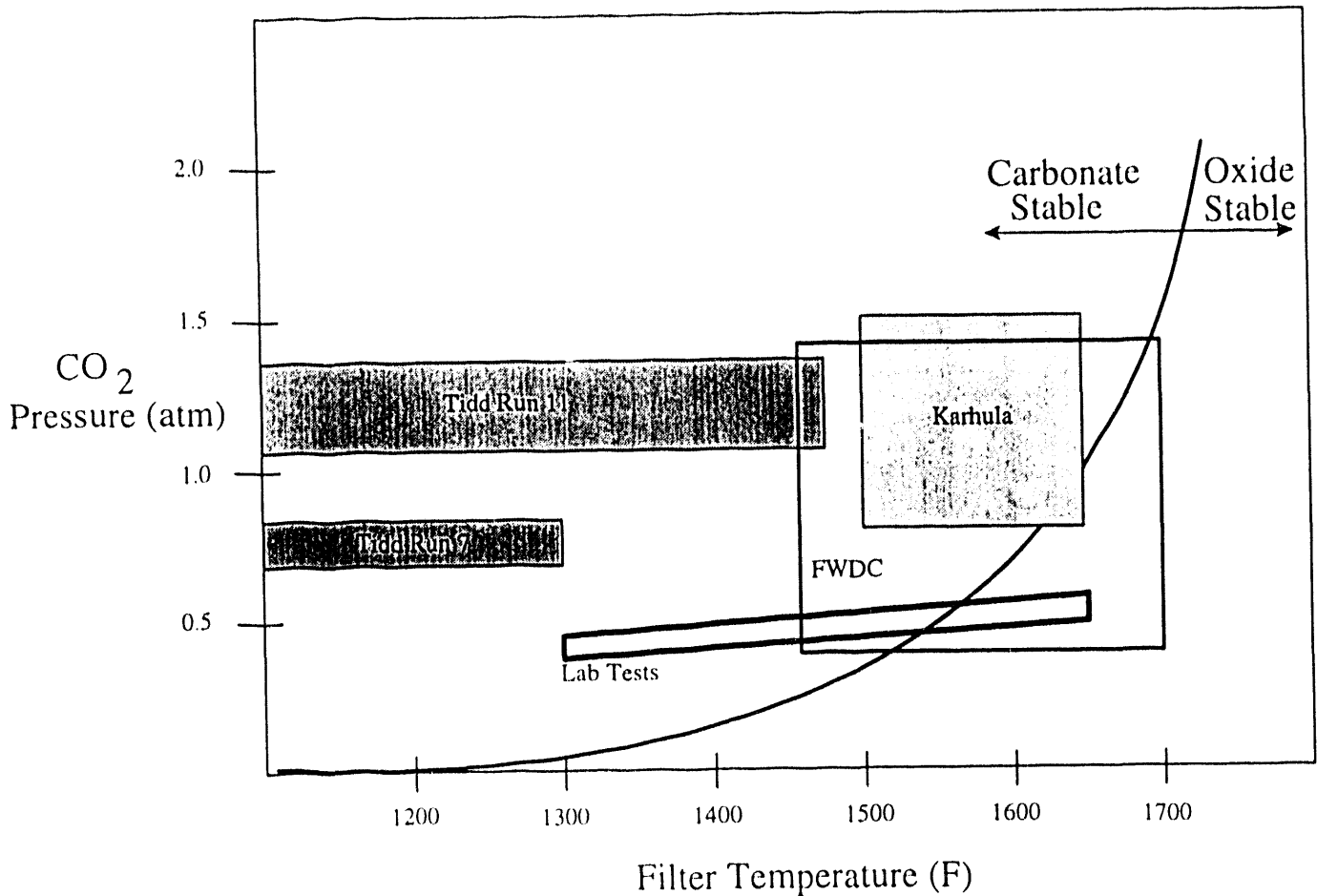
<u>Fly Ash Source</u>	<u>Pressure (psig)</u>	<u>Face Velocity (ft/min)</u>	<u>Cake Permeability at 1300° F (10<sup>-10</sup> lb/ft)</u>	<u>Minimum Permeability (10<sup>-10</sup> lb/ft)</u>	<u>Temperature at Minimum (° F)</u>
Tidd Fine	100	7	3.0	1.8	1550
Tidd Fine	150	7	3.2	1.6	1550
Tidd Fine	50	7	3.0	2.0	1500
Tidd Fine	100	12	2.4	1.6	1550
Tidd Cyclone + Fly Ash	100	7	4.3	3.8	1535
Grimethorpe Red	100	7	1.4	1.0	1550
Karhula	100	7	9.3	8.1	1600



**Table 4. Comparison of Laboratory and Field Permeabilities**

<u>Fly Ash Source:</u>	<u>Tidd</u>	<u>Karhula</u>	<u>Grimethorpe</u>	<u>FWDC</u>
Laboratory Permeability ( $10^{-10}$ lb/ft):	1.6-1.8	8	1.1	ND
Field Permeability ( $10^{-10}$ lb/ft):	0.2-0.6	2-6	ND	2-5

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 ND: Not Determined



**Figure 4. Comparison of Field Filter and Laboratory Filter Test Conditions**

- Control of PFBC cake permeability and bridging might be realized by:
  - limiting PFBC temperature,
  - limiting primary cyclone performance,
  - making proper PFBC sorbent selection.

## **REFERENCES**

R. A. Newby, et al., Integrated Low Emissions Cleanup System for Direct Coal Fueled Turbines, Phase I Final Report to DOE under Contract No. DE-AC21-87MC24257, May 1990 (DOE/MC/24257--2927).