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Filter Component Assessment

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Abstract

Advanced particulate filtration systems are currently being developed at Westinghouse for use in both coal-fired Integrated Gasification Combined Cycle (IGCC) and Pressurized Fluidized Bed Combustion (PFBC) systems. To date, Westinghouse has demonstrated 5855 hours of successful operation of first generation monolithic filter elements in PFBC applications when ash bridging or process thermal transient excursions are avoided.

Alternate advanced monolithic and second generation fiber reinforced, filament wound, and vacuum infiltrated filters are also being developed which are considered to have enhanced high temperature creep resistance, improved fracture toughness, or enhanced thermal shock characteristics, respectively. Mechanical and component fabrication improvements, as well as degradation mechanisms for each filter element have been identified by Westinghouse during exposure to simulated PFBC operating conditions and alkali-containing steam/air environments.

Additional effort is currently being focused on determining the stability of the advanced monolithic high temperature creep resistant clay bonded silicon carbide (SiC) materials, alumina/mullite, and chemically vapor infiltrated (CVI) SiC materials during operation in the Westinghouse Advanced Particulate Filtration (W-APF) system at Foster Wheeler's pressurized circulating fluidized-bed combustion (PCFBC) test facility in Karhula, Finland. Select advanced filter materials are being defined for additional long-term exposure in integrated gasification combined cycle (IGCC) gas streams. The results of these efforts are summarized in this paper.[†]

Introduction

Westinghouse in conjunction with DOE/METC has developed and established filter qualification and material surveillance programs related to the operation of hot gas filter systems in pilot plant and demonstration test units. This protocol has been a valuable aid in identifying and resolving filter performance and material stability issues. Recent experience has been focused primarily on the operation of hot gas filtration systems in oxidizing gas environments,

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with limited experience in reducing gas environments (Table 1). General findings from previous material surveillance programs have shown that:

- During operation under PFBC conditions, first-generation clay bonded silicon carbide candle filters experienced creep, while the oxide-based alumina/mullite filters exhibited limited resistance to thermal fatigue and/or shock during exposure to severe process thermal transient events.
- During process operation in the oxidizing PFBC environment, both clay bonded silicon carbide and alumina/mullite candles exhibited a loss of strength until a conditioned or stabilized matrix strength was achieved. Similarly, these materials experienced numerous phase and microstructural changes as a function of process operating time.
- A reduction in the strength of an as-manufactured, clay bonded silicon carbide candle filter from 1400-1800 psi to 650-770 psi after 5855 hours of operation in the American Electric Power (AEP) PFBC gas environment occurred without catastrophic damage being suffered by the filter elements.

In addition to characterization of field-tested filter elements, information obtained from coupon samples exposed to process gases at temperature for periods of ~3000 to ~10,000 hours has provided significant insight into the stability and/or changes which resulted in both the first-generation monolithic, and advanced, fiber-reinforced, second-generation filter materials.

In conjunction with field testing, development of the as-manufactured material properties, and identification of the mechanical, thermal, and chemical response of the filter materials during simulated PFBC and high temperature corrosion testing have demonstrated the possible long-term, life-limiting degradation mechanisms of the commercially available monolithic and recently developed second generation filter elements which are likely to occur during field service operation (Tables 2 and 3). For the advanced second generation materials which are the focus of this program, critical issues related to the stability of the 3M CVI-SiC and DuPont SiC-SiC matrices include oxidation along both surfaces of the deposited SiC, viability of the interface coating, stability of the fibers, and bonding of the fibers to the SiC encasement during process operation. Alternately retention of the DuPont PRD-66 membrane, mitigation of divot formations, strengthening of the flange, and production of a barrier vs bulk filter remain to be addressed. Similarly successful operation of the IF&P Fibrosic™ candles in Westinghouse's filter arrays during pulse cycling remains to be demonstrated.

Objectives

The objectives of the Base Program are to:

- Provide a more "ruggedized" filter system that utilizes porous ceramic filters which have improved resistance to damage resulting from crack propagation, thermal fatigue and/or thermal excursions during plant or process transient conditions, and/or mechanical ash bridging events within the candle filter array (Task 1).

Table 1

Westinghouse APF Field Experience

Process	Temperature °C	Operating Conditions	Operating Hours	Candle Filter Type	Test Facility
AEP PFBC	620 - 845	Oxidizing	5855 2815 2815 1705 1705 1705	SiC (a) Alumina/Mullite SiC (b) SiC Composite Filament Wound SiC (c)	Demonstration Plant
Karhula PCFBC	850 - 900 690 - 850 850 850	Oxidizing Oxidizing Oxidizing Oxidizing	716 1341 540 387	Alumina/Mullite SiC (b) SiC (c,d) Alumina/Mullite (e) SiC Composite	Pilot Plant
FW PFBC	700 - 870 700 - 870	Oxidizing Oxidizing	58 ~ 900	Alumina/Mullite SiC (a)	Pilot Plant
FW Carbonizer	<760	Reducing	63 ~ 400	Alumina/Mullite SiC (a)	Pilot Plant
Texaco Gasifier	700	Reducing	~ 400	SiC (a) Alumina/Mullite	Pilot Plant
IGT Biomass	760 - 915 675	Reducing Reducing	21 30	SiC (a) SiC (a)	Pilot Plant

(a) Schumacher Dia Schumalith F40
(b) Pall Vitropore 442T
(c) Schumacher Dia Schumalith FT20
(d) Pall 326
(e) Alumina/Mullite (1110 Hr PFBC-Exposed).

- Assess the effects of long-term (i.e., 1000-1500 hours) pilot-scale exposure under actual pressurized circulating fluidized-bed combustion (PCFBC) conditions on advanced candle filter failure modes and degradation mechanisms (Task 2).
- Assess the stability of select advanced filter materials when subjected to long-term exposure in actual integrated gasification combined cycle (IGCC) gas streams (Task 3).

Table 2

Hot Gas Filter Materials

Commercial or Near Commercial Filters	As-Manufactured Permeability	Membrane	Dust Cake Removal	Load Bearing Capacity	Creep Resistance (>600°C)	Thermal Fatigue/ Shock Resistance	Volatile Alkali Sorption (T>600-800°C)	Volatile Alkali Impact
Schumacher/Pall Clay Bonded SiC*	High	Present	High	High	Low	High/High	High	High; Silicate Eutectic Formation
Coors Alumina/Mullite	Moderate-High	Typically Not Present	High	High	High	High/ Susceptible	Moderate	Negligible; Crystallization and Strength Increase
3M CVI-SiC Composite	High	Open Woven Outer Structure	Moderate	Low	High	High/High	Negligible Initial Uptake; Accelerated Oxidation	Embrittlement; Eutectic Formation
DuPont PRD-66	High	Present	High	Low	High (TBD)	High/High	(TBD)	Membrane Cracking and Spalling
DuPont SiC-SiC	Moderate-High	Present	High	Low	High (TBD)	High/Low	Negligible Initial Uptake; Accelerated Oxidation	Embrittlement; Eutectic Formation
IF&P Fibrosic™	High	Present	High	Very Low	High	Low/High	Moderate-High	Phase Changes; Rigidization Spalling

* Original Matrices: Schumacher Dia Schumalith F40; Pall Vitropore 442T.
TBD: To Be Determined.

Table 3

High Temperature Filter Material Stability

Filter Matrix	Thermal Fatigue	Thermal Shock	Flow-Through Oxidation/Corrosion	
	Accelerated Pulse Cycling	Thermal Transient Testing	Steam/Air	Alkali/Steam/Air
Coors Alumina/Mullite	Intact	Susceptible	Conversion Of Amorphous Phase To Anorthite; Strength Increase	Conversion Of Amorphous Phase To Albite; Anorthite; Strength Increase
Schumacher F40	Intact	Intact	NT	Eutectic Formation; Plastic Deformation
Pall Vitropore 442T	Intact	Intact	NT	Eutectic Formation; Plastic Deformation Expected
3M CVI-SiC Composite	Intact	Intact	Strength Loss; Surface Oxidation	Strength Loss; Accelerated Oxidation; Eutectic Formation; Embrittlement
DuPont PRD-66	Flange Failure; Matrix Strengthening; Holder Redesign	Intact	Strength Increase	Strength Loss; Membrane Cracking and Spalling
DuPont SiC-SiC	Intact	Lapped Seam Rupture	Strength Loss; Surface Oxidation	Strength Loss; Accelerated Oxidation; Eutectic Formation; Embrittlement
IF&P Fibrosic™	Mid-Body Fracture; Hole Formations	Intact	Tearing; Rupture	Rigidization

NT: Not Tested.

Approach

In order to provide a more "ruggedized" filter system that utilizes porous ceramic filters, Westinghouse subjects full-body filter elements to qualification testing in its PFBC simulator test facility in Pittsburgh, PA, prior to considering any filter element for use in field operation. During filter qualification testing, filter elements are subjected to

- Simulated steady state PFBC operating conditions
- Accelerated pulse cycling to determine the impact of thermal fatigue primarily along the inner surface of the filter element
- Simulated thermal transient conditions which reflect possible rapid system start-up and shut-down cycles.

In addition to determining the response of the filter material to the simulated PFBC environment, qualification testing demonstrates

- Filtration efficiency during steady state operation
- Dust cake removal capability
- Conditioned filter pressure drop
- Sealing and mounting capability of the element(s) within the metal housing.

Post-test non-destructive evaluation of filter elements is generally conducted to assess the

- Retention of fines along the outer surface of the elements
- Integrity of the flange
- Overall integrity of the filter body.

Alternately post-test destructive characterization of the filter elements identifies the

- Integrity of the cross-sectioned filter matrix (i.e., crack formations)
- Microstructure of the resulting filter matrix
- Semi-quantitative elemental composition throughout the element
- Residual room and process temperature bulk strength
- Residual room temperature hoop strength
- Resulting elastic modulus and Poisson's ratio
- Thermal coefficient of expansion
- High temperature creep strain
- Affinity of ash or char fines to the membrane
- Penetration of fines through the filter wall
- Composition of the ash fines.

In order to demonstrate the long-term thermal/chemical stability of the various filter materials, high temperature corrosion testing is conducted utilizing either discs or mini-candles. These materials are exposed generally for a period of 400 hours at temperatures of 870ºC to a flow-through environment which contains either 5-7% steam/air or 20 ppm NaCl/5-7% steam/air.

The materials are also subjected to simulated pulse cycling at every 20 minute intervals throughout the course of flow-through testing. Post-test characterization of the material includes:

- Inspection for cracks
- Determining the residual bulk strength of the matrix along both gas stream and pulse cycled surfaces
- Identifying whether microstructural and/or compositional changes have occurred within the material.

Project Description

The focus of the Filter Component Assessment program in Task 1 has been to evaluate the filtration characteristics, mechanical integrity, and corrosion resistance of the following advanced or second generation candle filters for use in advanced coal-fired applications:

- 3M CVI-SiC composite — Chemical vapor infiltration of silicon carbide on an aluminosilicate Nextel™ 312 fiber preform
- DuPont PRD-66 — Filament wound candle containing corundum, cordierite, cristobalite, and mullite
- DuPont SiC-SiC composite — Chemical vapor infiltration of silicon carbide on a Nicalon™ felt or mesh screen support layer
- IF&P Fibrosic™ — Vacuum infiltrated oxide-based chopped fibrous matrix.

In order to assess the effects of long-term (i.e., 1000-1500 hours) pilot-scale exposure on advanced candle filter failure modes and degradation mechanisms in Task 2,

- Advanced monolithic, high temperature, creep resistant Schumacher Dia Schumalith FT20 candles
- Advanced monolithic, high temperature, creep resistant Pall 326 candles
- Coors P-100A-1 alumina/mullite candles, and
- 3M CVI-SiC composite candles

were installed in the Westinghouse Advanced Particulate Filtration (W-APF) system that was operated at Foster Wheeler's PCFBC test facility in Karhula, Finland. To date, two test campaigns have been completed in which 112 candles were operated for a period of 540 hours at temperatures of ~850ºC using Illinois No. 6, Sparta coal, as the feed material and Linwood limestone as the sulfur sorbent (Table 4).

Post-test characterization of PCFBC surveillance candles which has recently been completed included determining the resulting gas flow resistance of the filter elements, residual process temperature bulk strength, high temperature creep and thermal expansion properties, and residual microstructure and phase composition. In addition, characterization of ash samples that were removed from various locations within the three filter arrays was also conducted. These analyses included determining the bulk density, moisture content, bulk strength, thermal expansion, and identification of the morphology and composition of the ash materials. Continued testing at Karhula is planned to begin in August 1996, tentatively for an additional ~1000 hours

Table 4

Summary of PCFBC Filter Operation

	Test Segment No. 1	Test Segment No. 2
Number of Candles	112	112
Filter Operating Temperature, ºC	826-853	818-860
Filter Operating Pressure, bar	10.7-11.1	10.6-11.3
Nominal Face Velocity, cm/s	3.5-4.1	3.1-4.2
Inlet Dust Load, ppmw	11,400-15,000	11700
Coal Feed	Illinois No. 6 (Sparta)	Illinois No. 6 (Sparta)
Sulfur Sorbent	Linwood Limestone	Linwood Limestone
Operating Hours (Coal)	153	387

of service operation in Test Segment No. 3. The information obtained from the filter material and ash analyses will serve as the basis for determining continued use of the various filter elements in selected Clean Coal Technology Demonstration projects.

Additional effort is being initiated in Task 3, to assess the stability of select advanced filter materials when subjected to long-term exposure in actual IGCC gas streams. A pressurized mini-vessel will tentatively be installed downstream of the W-APF at the Sierra Pacific Piñon Pine IGCC Demonstration Plant in Reno, NV. There either mini-candles or coupons will be exposed at temperature to the process fuel gas for extended periods of time in the absence of char fines. Post-test characterization of either the mini-candles or coupon materials will be conducted as previously described.

Results

First generation monolithic porous ceramic filter materials have been shown by Westinghouse to experience thermal fatigue, high temperature creep, and loss of material strength when operated for extended periods of time in advanced coal-fired process applications.[1,2] In order to mitigate high temperature creep and/or creep crack growth, both Schumacher and Pall developed a creep resistant binder for use in the manufacture of the clay bonded silicon carbide candle filters. High temperature creep testing conducted at Westinghouse using the Schumacher Dia Schumalith FT20 and Pall 326 filter materials demonstrated the absence of creep when either material was subjected for ~500 hours to a 500 psi, 4-point bending load at ~845°C.

Additional effort at Westinghouse was conducted to assess the impact of thermal fatigue and/or thermal shock on the stability of the clay bonded silicon carbide and alumina/mullite filter materials. During simulated PFBC testing at Westinghouse, both the clay bonded silicon carbide and Coors P-100A-1 alumina/mullite candles were instrumented with high speed thermocouples in order to measure the axial and radial temperature profiles through the filter wall during pulse cleaning (i.e., thermal fatigue), and during exposure to simulated thermal transient conditions (i.e., thermal shock). As described in Appendix A, the resulting temperature profiles were

utilized to establish the stress intensity within the filter elements.[3] Clearly the use of the fail-safe/regenerator above the Coors P-100A-1 alumina/mullite candle filters significantly reduced the stress intensity experienced along the inner surface of the filter element during pulse cycling. In contrast, however, depending on the magnitude of the thermal transient event, stresses which exceeded the rupture strength of the Coors P-100A-1 alumina/mullite filter matrix were established along the outer surface of the candle. Under similar thermal transient conditions, stresses established within the clay bonded silicon carbide candles were calculated to be equal to, or below the rupture modulus of the filter matrix. As a result, the clay bonded silicon carbide filter elements were considered to have a greater likelihood for survival in process systems that experience rapid shut-down or start-up cycles in comparison to the Coors P-100A-1 alumina/mullite filter elements.

In order to develop a more "ruggedized" filter system, identifying the stability and life of candidate filter materials during operation in advanced coal-fired applications is essential. In the following sections, discussions are provided which summarize Westinghouse's material characterization and filter surveillance efforts that were conducted during the past year.

HTHP PFBC Filter Qualification Testing

Prior to selection, installation, and operation in the field, candle filter qualification testing is conducted at Westinghouse to assure that developmental, prototypic, or commercial filter elements demonstrate a >99.99% particle collection efficiency during steady state, simulated PFBC operation. Similarly the initial pressure drop across the elements at process temperature, dust cake removal efficiency, and the as-manufactured strength of the flange and mechanical sealing/mounting of the element within the filter holder are assessed. In addition, Westinghouse qualifies the performance of the various filters under extreme conditions as accelerated pulse cycling which monitors the performance of the matrix with respect to thermal fatigue, as well as exposure to thermal transient events which simulate rapid start-up or shut-down ramps experienced during process upset conditions.

In order to evaluate the performance of the advanced second generation filter elements, an array which included a DuPont PRD-66, a 3M CVI-SiC composite, and a DuPont SiC-SiC composite filter was installed in the Westinghouse high temperature, high pressure (HTHP) PFBC simulator test facility in Pittsburgh, PA.[2] Initially the filter array was heated to temperatures of ~845°C prior to initiating thermal transient testing. A series of seven increasing severity thermal transients were delivered to the array which reduced the outer surface temperature of the filter elements by 6-110°C and 20-240°C within the first five and sixty seconds, respectively, after transient initiation. Nine maximum severity thermal transients followed. Subsequently the array was subjected to ten accelerated pulse cleaning cycles, and a final maximum severity thermal transient.

After 42 hours of thermal transient testing, the filter array was slow cooled, and the elements were removed for destructive characterization. Characterization of the resulting morphology and phase composition via scanning electron microscopy/energy dispersive x-ray analysis (SEM/EDAX) and x-ray diffraction (XRD) analysis has not been completed for the thermal transient-tested DuPont PRD-66 and 3M CVI-SiC filter matrices. However, when the

DuPont SiC-SiC composite was subjected to SEM/EDAX analyses, several interesting changes were observed as a result of simulated PFBC thermal transient testing.[4] These included:

- Evidence of oxidation of the CVI-SiC outer surface
- Removal of the interface layer that was originally deposited around the Nicalon™ fibers in the single-ply felt layer of the DuPont SiC-SiC composite
- Removal of the interface layer particularly along the outer fiber bundle or tow, directly beneath the CVI-SiC encapsulating layer in the mesh support screen
- Melting or "sintering" of adjacent Nicalon™ fibers with the enhanced oxidation phase that was added during the manufacture of the mesh screen support layer.

Retention of the interface layer along the interior of the fiber bundle or tow in the mesh screen support layer remains to be determined.

Based on the resulting load versus deflection curves that were generated during compressive and tensile strength testing, the fracture toughness of the DuPont SiC-SiC composite appeared to be reduced after 42 hours of thermal transient testing.[2] Loss of fracture toughness was primarily attributed to removal of the interface layer in the single-ply felt and mesh screen support layers, and to the "sintering" of the Nicalon™ fibers in the mesh screen support layer.

In order to identify the thermal fatigue resistance of the advanced second generation filters, a second set of DuPont PRD-66, 3M CVI-SiC composite, and DuPont SiC-SiC composite candles were installed in the Westinghouse PFBC simulator and subjected to 3514 accelerated pulse cleaning cycles during a period of 197 hours. The temperature of the filter array was maintained at ~845°C while accelerated pulse cycling was conducted.

The interface layer that initially surrounded the Nicalon™ fibers in the single-ply felt layer of the DuPont SiC-SiC filter matrix was again seen to be removed after 197 hours of accelerated pulse cycle testing. Similarly crack formations were evident along the outer periphery of the Nicalon™ fibers.[4] Typically the cracks had rounded tips, as well as segmented "step-like" characteristics.

"Halo-like" areas were readily evident along the periphery of the Nicalon™ fibers in the single-ply felt. These areas were generally enriched with oxygen, and effectively demarcated the location to where the cracks penetrated. Bonding of the Nicalon™ fiber to the inner surface of the CVI-SiC encapsulating shell often resulted near the crack formations.

Within the mesh screen support layer, thin CVI-SiC bands which followed the contour of the Nicalon™ fibers, the enhanced oxidation phase, and perhaps the interface layer were evident. Near the periphery of the fiber bundle or tow (i.e., adjacent to the CVI-SiC encapsulating layer), as well as within the bundle, irregularly shaped Nicalon™ fibers were evident. Melting of the fibers was frequently observed, as well as mottling of the fiber surface. These were considered to result as a response or reaction of the Nicalon™ fibers with the enhanced oxidation phase that was included in the mesh screen support layer. Adjacent to the CVI-SiC encapsulating layer, the melted fibers formed an interconnected network which readily formed cracks during fast fracture. Void formations that were observed in the fractured mesh screen support layer may have resulted from fiber pull-out during sample preparation, or

alternately reflected removal of the interface phase during exposure to simulated PFBC process operating conditions. The Nicalon™ fibers in the mesh screen support layer of the DuPont SiC-SiC composite filter matrix did not exhibit crack formations along their periphery.

Oxidation of the outer surface of the CVI-SiC encapsulting layer was again evident after 197 hours of accelerated pulse cycling in the Westinghouse PFBC simulator test facility.

As previously discussed, the reduced fracture toughness of the DuPont SiC-SiC filter matrix after 197 hours of accelerated pulse cycling was primarily attributed to removal of the interface layer in the single-ply felt and mesh screen support layers, and to the "sintering" of the Nicalon™ fibers in the mesh screen support layer.

After ~800 hours of steady state, thermal transient, and accelerated pulsing of DuPont SiC-SiC candles in the simulated PFBC process environment which contained ash and 2 ppm gas phase sodium chloride (i.e., equivalent to 20 NaCl at 1 atm), further changes were evident within the filter matrix.[5] These included an apparent swelling of the Nicalon™ fibers within the single-ply felt layer. As a result, the fibers completely, or nearly completely filled the void in the CVI-SiC encapsulating shell. Bonding of the Nicalon™ fibers to the inner wall of the CVI-SiC shell was also apparent in the single-ply felt layer.

Efforts are required to conduct similar post-test characterization of the accelerated pulse cycled and thermal transient-tested 3M CVI-SiC composite and DuPont PRD-66 filter materials.

Characterization of Long-Term PFBC-Exposed Filter Coupons

Monolithic Filter Materials[3]

Porous ceramic monolithic filter material coupons (i.e., flat discs) were installed in an open metal structure and placed above the combustor freeboard area at the AEP Tidd Demonstration Plant in Brilliant, OH. These coupons were exposed to the PFBC process gases and fines in a flow-over fashion for ~10,000 hours (i.e., May 1992-January 1995). Both the ceramic filter coupons and metal structure remained intact during the prolonged exposure in the 815-843°C PFBC environment.

As shown in Table 5, numerous phase changes occurred within the porous ceramic monolithic filter materials after extended exposure to the high temperature PFBC environment. Although the filter materials were exposed as coupons in a flow-over fashion, the phase changes appeared to result throughout the entire matrix (i.e., not simply along the surface that was contacted with the process gas and/or particulate fines). The impact of phase changes on the residual bulk strength was not determined for the small coupons since insufficient material was available for evaluation.

Table 5

Stability of the Porous Ceramic Monolithic Filter Materials after ~10,000 Hours of Flow-Over Exposure at AEP

Filter Material	Microstructural Changes
Coors P-100A Alumina/Mullite	• Matrix remained intact • Extensive mullitization • Composition of the ligaments included oxygen, aluminum, and silicon, with secondary contributions of calcium • Composition of the pore cavity wall included oxygen, aluminum, and silicon, with secondary contributions of magnesium, calcium, and potassium • Migration of magnesium, calcium, etc., from the interior of the matrix to the pore cavity wall typically results during exposure to high temperature oxidizing environments
Coors P-100A-1 Alumina/Mullite	• Matrix remained intact • Extensive crystallization/mullitization • Migration of magnesium to the pore cavity wall
Coors Mullite	• Matrix remained intact • As-manufactured matrix contained ~50-100 μm agglomerates that were enriched with oxygen, silicon, and aluminum • "Melt-like" features were evident along the pore cavity walls which encapsulated a subsurface crystalline phase after exposure to the PFBC environment
GTE Cordierite	• Matrix remained intact • Crystallization along the pore cavity walls
GTE Cordierite-Silicon Nitride	• Matrix remained intact • Numerous sintered submicron particles were present throughout the PFBC-exposed matrix • Open porosity retained • Ligaments contained additional porosity, particularly near the pore cavity surface
AiResearch Sintered Silicon Nitride	• Matrix remained intact • Retention of extensive formation of the "whisker-like" α-Si_3N_4 mat formation along the pore cavity walls

In addition to the flat coupons discs, O-rings or cylindrical sections removed from alumina/mullite and clay bonded silicon carbide candle filters were exposed for ~10,000 hours to the 815-843°C PFBC environment above the combustor freeboard at AEP. The residual bulk strengths of the ~10,000 hour exposed Coors P-100A alumina/mullite, Refractron 505, and Schumacher Dia Schumalith F40 (production lot from 1991) materials were determined at temperatures of 732ºC.[a]

The residual high temperature, bulk strengths of the ~10,000 hour exposed filter coupons are shown in Table 6. Unfortunately, neither the as-manufactured Coors P-100A nor Refractron 505 matrices were available for characterization at nominal filter operating temperatures of 732°C. From preliminary data that were generated at Westinghouse for the as-manufactured Coors P-100A alumina/mullite candles that were used at the Texaco reentrained gasification plant in Montebello, CA, the alumina/mullite filter matrix was determined to have an initial C-ring compressive strength of 2203±215 psi at 870°C. The as-manufactured strength for the P-100A alumina/mullite filter matrix would be expected to be close to, or possibly slightly greater than 2203 psi at 732°C. The strength of the P-100A filter matrix appeared to be reduced after ~10,000 hours of exposure above the combustor freeboard area at AEP (i.e., 1655±149 psi).

Table 6

C-Ring Compressive Strength of the ~10,000 Hour Combustor Freeboard-Exposed Porous Ceramic Monolithic Filter Materials

Filter Matrix	Strength, psi (732°C)
Coors P-100A	1577.0 1694.8 1845.1 1503.1 Average ± 1σ: 1655 ± 149
Refractron 505	2041.9 2343.7 2173.2 2567.0 Average ± 1σ: 2282 ± 227
Schumacher Dia Schumalith F40	1103.3 1050.2 967.4 976.0 Average ± 1σ: 1002 ± 38

[a] Typical operating temperature of the W-APF throughout the five test campaigns at AEP. The Coors P-100A alumina/mullite and Refractron 505 matrices are no longer utilized to produce full body candle filters for use in field applications. These materials have been replaced by the Coors P-100A-1 alumina/mullite and Pall Vitropore 442T filter matrices, respectively.

Since the filter materials were not pulse cycled during flow-over exposure above the combustor freeboard, the reduction in strength is considered to result from microstructural changes within the P-100A alumina/mullite material as a result of exposure to the PFBC operating temperatures and flue gas chemistry.

Similar to the Coors P-100A alumina/mullite filter matrix, strength measurements for the Refractron 505 filter matrix were previously generated at Westinghouse at temperatures of 870°C. The high temperature C-ring compressive strength of the as-manufactured Refractron 505 filter matrix was 2507±229 psi. The as-manufactured strength of the Refractron 505 filter matrix would be expected to be close to, or possibly slightly greater than 2507 psi at 732°C. After ~10,000 hours of exposure above the combustor freeboard area at AEP, the residual bulk strength of the Refractron 505 matrix was determined to be 2282±227 psi. Once again the strength of the Refractron 505 filter sample which had not been subjected to pulse cycling, was reduced primarily as a result of exposure to the process gas environment (i.e., gas chemistry and process operating temperature).

Based on the information generated in Westinghouse's surveillance program with AEP,[1] the Schumacher Dia Schumalith F40 filter matrix which had been produced in 1991, had an as-manufactured C-ring compressive strength of 1416±127 psi at 732°C. After ~10,000 hours of exposure at AEP, the residual strength of the Schumacher Dia Schumalith F40 filter matrix was 1002±38 psi. These data also imply that exposure to the process gas environment (i.e., gas chemistry and process operating temperature) had an impact on the residual strength of the filter matrix. As with the Coors P-100A and Refractron 505 filter samples, the Schumacher Dia Schumalith F40 filter samples did not experience pulse cleaning.

Since only four C-rings of each PFBC-exposed material were subjected to compressive strength testing at the elevated process operating temperatures, the question of statistical significance of the residual bulk material strength data arises. Obviously only general material response trends can be identified.

Second Generation Porous Ceramic Filter Materials

Sections removed from 3M CVI-SiC, DuPont PRD-66, DuPont SiC-SiC (double-ply felt), and IF&P Fibrosic™ candles were exposed above the W-APF tubesheet at the AEP Tidd Demonstration Plant in Brilliant, OH. These materials were placed on a material surveillance coupon tree, and exposed to PFBC conditions during Test Segments 3, 4, and 5. Post-exposure characterization of each material was conducted in order to determine whether microstructural changes had occurred after extended static exposure to the PFBC environment. Highlights of this effort follow:

3M CVI-SiC Composite

- Oxidation resulted along the exterior and interior of the CVI-SiC encapsulating structure of the 3M CVI-SiC composite filter matrix.
- The thin ~2 μm CVI-SiC infiltrated layer that was originally formed within the interior of the triaxial support braid consisted of ~1 μm SiC and ~1 μm SiO_2. Cracks were evident which penetrated through the SiO_2-enriched encapsulating layer.

- The oxygen-enriched layer along the interior of the CVI-SiC encapsulating shell appeared to bond to the Nextel™ 312 fibers in the 3M composite filter matrix.
- Oxidation of the ~2 μm CVI-SiC layer along the outer confinement and filtration mat layers of the 3M composite filter matrix occurred after exposure to the PFBC environment, again leading to the formation of SiO_2. Spalling of the SiO_2-enriched layer may occur during thermal cycling of the filter, subsequently causing loss of material.
- After 2815 hours of exposure to static PFBC process operating conditions, ~21% of the initial strength of the triaxial support braid remained (Table 7). Neither the filtration mat nor outer confinement layers were expected to significantly contribute to the overall strength of the 3M CVI-SiC composite filter matrix.

DuPont PRD-66

- Crystallization of the fibers in the filament wound PRD-66 filter matrix was evident after exposure to flow-over PFBC conditions. Crystallization and grain growth were expected to lead to the lowered high temperature strength of the matrix after 4094 hours of exposure in the PFBC environment. Approximately 67% of the as-manufactured strength of the PRD-66 filter matrix remained after 4094 hours of exposure to static PFBC operating conditions.

DuPont SiC-SiC Composite

- Removal of the interface layer within the DuPont SiC-SiC double-ply felt filter matrix resulted during exposure to flow-over PFBC conditions.
- Longitudinal crack formations resulted along the Nicalon™ fibers in the DuPont SiC-SiC double-ply felt filter matrix after 4094 hours of exposure to flow-over PFBC conditions.
- Oxidation along the inner surface of the CVI-SiC shell resulted during exposure to flow-over PFBC operating conditions.
- In addition to crack formations, the Nicalon™ fibers experienced a volume increase due to oxidation. As a result of oxidation along the inner surface of the CVI-SiC shell and the Nicalon™ fibers, the void space between the fibers and shell decreased. The fibers ultimately become bonded to the encapsulating shell through contact with the oxygen-enriched (i.e., silica) “melt-like” phase. As a result, fracture toughness of the material was reduced.
- The strength of the DuPont double-ply SiC-SiC matrix appeared to slightly increase during flow-over exposure to PFBC operating conditions. This was considered to result from bonding of the residual fibers to the inner surface of the oxidized CVI-SiC encapsulating shell.

Table 7

Summary of the Strength and Morphology of the Second Generation Filter Materials after Exposure to PFBC Test Conditions above the W-APF Tubesheet at AEP

Filter Matrix	Exposure Time, Hrs	Initial Process Strength, psi	PFBC-Exposed Process Strength, psi	SEM/EDAX Characterization
3M CVI-SiC	2815	10652± 2184[a,b] (Composite Fracture)	2187[b] (0.4 lbs[c] Composite Failure)	Oxidation of CVI-SiC Outer and Inner Surfaces; Bonding of Nextel™ 312 Fibers to CVI-SiC-SiO_x
DuPont PRD-66	4094	988±86[a] (Brittle Fracture)	666 (6.8 lbs Brittle Fracture)	Crystallization and Grain Growth of the Polycrystalline Fibers; Limited Slurry Infiltration into Interior of Filament Bundles; Voids within Individual Fibers
DuPont SiC-SiC	4094	4703[d] (Brittle Fracture)	5867[d] (10.4 lbs; Brittle Fracture)	Removal of the Interface Layer; Oxidation of CVI-SiC Outer and Inner Surfaces; Oxidation of Nicalon™ Fibers; Bonding of Fibers to CVI-SiC
IF&P Fibrosic™	2815	ND	ND	Morphology Similar to As-Manufactured Filter Matrix

(a) Previously Reported Strengths From An Alternate Filter Production Lot.
(b) Triaxial Braid Wall Thickness Used To Calculated Resulting Bulk Strength.
(c) Ultimate Load To Failure.
(d) Double Ply Felt.
As-Manufactured and PFBC-Exposed Process Strengths Determined at 843°C.
ND: Not Determined.

IF&P Fibrosic™

- Virtually little change in the morphology of the IF&P Fibrosic™ fibers was observed after 2815 hours of exposure to the flow-over PFBC gas environment.

Characterization of the PCFBC-Exposed Candle Filter Materials

In order to assess the effects of long-term, pilot-scale exposure on the stability of the advanced monolithic and second generation fiber reinforced filter elements (Task 2), 112 candles were installed in the Westinghouse APF system at Foster Wheeler's test facility in Karhula, Finland (Table 4). An assessment of the performance and characterization of the material properties of the Schumacher Dia Schumalith FT20, Pall 326, Coors P-100A-1 alumina/mullite, and 3M CVI-SiC composite surveillance candles were conducted at the conclusion of Test Segments 1 and 2. Post-test filter element characterization included:

- Room temperature gas flow resistance measurements
- Scanning electron microscopy/energy dispersive x-ray analysis (SEM/EDAX)
- Compressive and tensile bulk strength testing at room and process temperature
- Hoop stress, elastic modulus, and Poisson's ratio
- Thermal expansion
- High temperature creep testing.

The results of these analyses follow.

Room Temperature Gas Flow Resistance

PCFBC surveillance candle filters were shipped to Westinghouse at the conclusion of Test Segment No. 2, and were initially subjected to room temperature gas flow resistance measurements. A partial dust cake remained along the outer surface of each filter element after shipment. Each candle was then brushed in order to remove the residual dust cake layer, and resubjected to room temperature gas flow resistance measurements. The as-received and brushed gas flow resistance measurements were generally comparable for each filter element type (Figure 1).

Scanning Electron Microscopy/Energy Dispersive X-Ray Analysis

Sections of material were removed from each filter element at the conclusion of Test Segment No. 2. Each section was fresh fractured and subjected to SEM/EDAX analyses in order to determine whether microstructural changes had occurred along either the outer (i.e., membrane surface) or pulse cycled surfaces, or throughout the cross-sectioned wall of the PCFBC-exposed filter elements.

Extensive crystallization of the high temperature creep resistant Schumacher Dia Schumalith FT20 filter matrix occurred after 540 hours of operation in the 850°C PCFBC environment. Negligible changes were detected within the fibrous outer membrane.

Similar extensive crystallization of the high temperature creep resistant binder resulted within the Pall 326 filter matrix after 540 hours of operation in the 850°C PCFBC environment. Changes within the fine silicon carbide grit membrane tended to indicate the presence of silica, implying oxidation of the grit, or localized (i.e., nondispersed) areas of the binder phase.

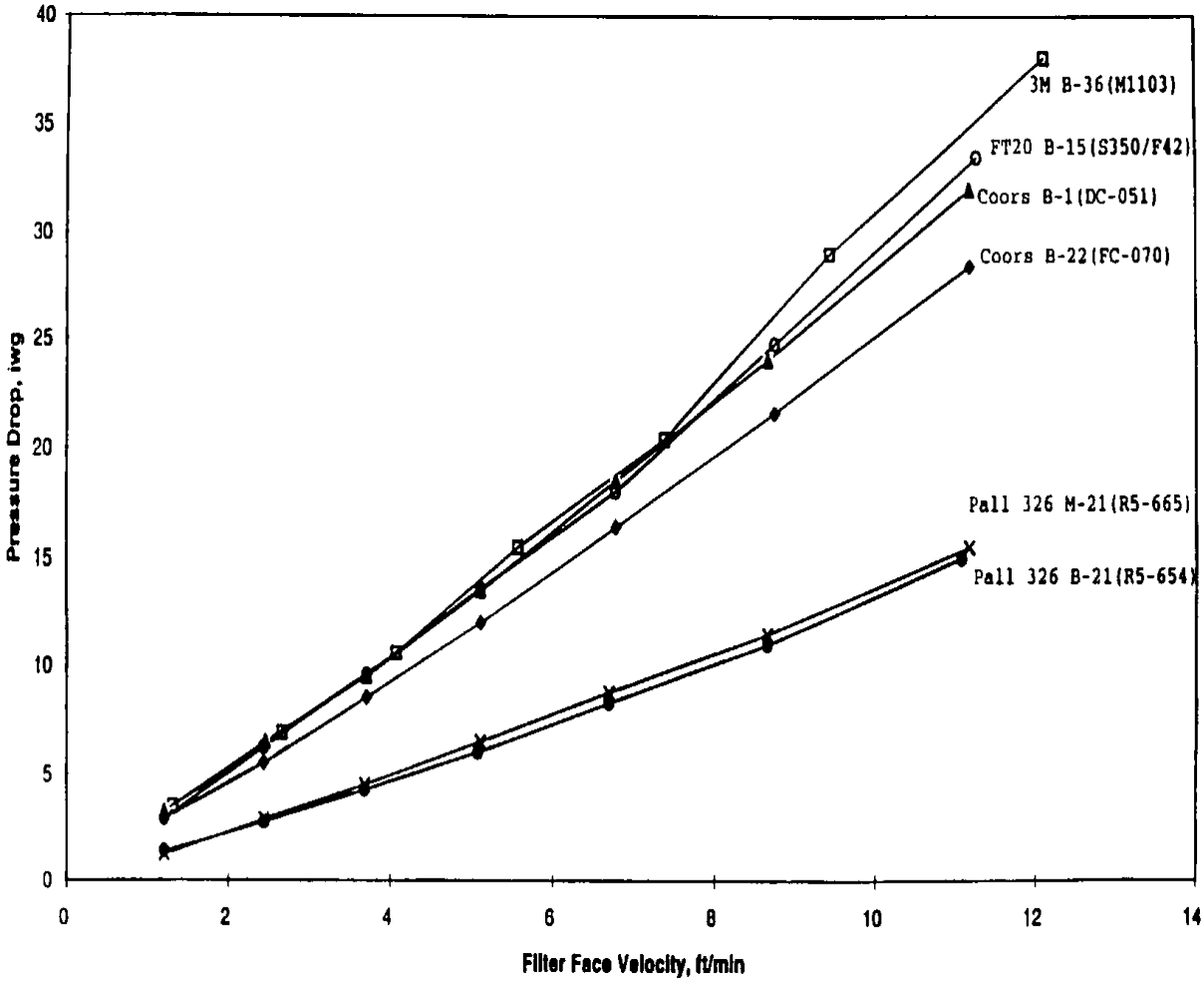


Figure 1 — Room Temperature Gas Flow Resistance of the PCFBC Surveillance Candle Filters (Test Segment No. 2)

Both newly manufactured Coors P-100A-1 alumina/mullite candles, as well as candles that had been operated in the W-APF system at AEP were installed prior to initiating PCFBC testing in Test Segment No. 1. Post-test SEM/EDAX characterization of the 505 hour PCFBC-exposed, and 1650 hour PFBC/PCFBC-exposed Coors P-100A-1 alumina/mullite filters indicated that crystallization resulted along the pore cavity walls, and throughout the structural ligaments.

When removed from the filter array after 387 hours of operation at 850°C in the PCFBC environment, a color change was readily evident along the outer confinement and possibly filtration mat layers of the 3M CVI-SiC composite candle filters. The original dark black color of the 2 μm CVI-SiC coating that encapsulated either the Nextel™ 312 fibers in the outer confinement layer, or the alumina-based fibers in the filtration mat layer was not retained along the majority of the filter body. Instead after 387 hours of operation, the outer confinement layer appeared to be white (i.e., excluding the presence of ash fines), while the filtration mat layer was generally a light to medium grey. A similar change in the color of the 3M CVI-SiC composite candle filters had been experienced at Westinghouse during qualification testing under simulated PFBC conditions. Initial consideration was that the "strand-like" appearance of the confinement layer exposed "bare" fibers as a result of removal of the CVI-SiC encapsulating shell during exposure in the high temperature oxidizing environment. Generally the triaxial support braid which consisted of an ~100 μm CVI-SiC layer that encapsulated twisted Nextel™ 312 filaments or fiber bundles, retained its as-manufactured dark black appearance.

SEM/EDAX characterization of the PCFBC-exposed 3M CVI-SiC composite filter matrix confirmed removal of the 2 μm SiC layer that initially encapsulated the Nextel™ 312 fibers in the outer confinement layer. Characterization of the lapped filtration mat indicated that oxidation of the 2 μm CVI-SiC shell had also occurred. As a result, an ~1 μm oxygen-enriched layer formed along the outer surface of the as-manufactured CVI-SiC coating. An oxygen-enriched region also formed along the inner surface of the CVI-SiC shell, bonding the shell to the filtration mat fibers. Bonding of the oxygen-enriched CVI-SiC shell to the fibers ultimately reduces fracture toughness and possibly increases strength within this layer of the composite filter matrix.

Characterization of the triaxial support braid identified oxidation and pitting along the outer surface of the CVI-SiC shell that encapsulated underlying Nextel™ 312 filaments. Within the filament or fiber bundles, a thin layer of CVI-SiC generally coated individual fibers. Frequently gaps were evident between the thin CVI-SiC layers and the fibers, as well as areas which clearly showed bonding of the shell to the surface of the contained fiber.

Compressive and Tensile Strength Testing

Strength characterization of the as-manufactured Schumacher Dia Schumalith FT20, Pall 326, Coors P-100A-1 alumina/mullite, and 3M CVI-SiC composite candle filters was conducted via compressive and tensile testing of C-rings at room temperature, 850°C, 870°C, and 900°C. In addition, Coors P-100A-1 alumina/mullite and 3M CVI-SiC composite candles removed after

completion of Test Segments 1 and 2, and Schumacher Dia Schumalith FT20 and Pall 326 filters removed after completion of Test Segment No. 2 were subjected to C-ring compressive and tensile strength testing at room temperature, 850°C, and 900°C. Process temperature strengths (i.e., 850°C) of sections from broken filter elements that were removed from the ash hopper at the conclusion of Test Segment No. 2 were also identified. The results of these analyses are presented in Table 8 and Figure 2.

In summary, the as-manufactured

- Schumacher FT20 filter matrix retained its brittle fracture characteristics during C-ring compressive and tensile testing up to temperatures of 900°C.
- Pall 326 filter matrix exhibited brittle fracture characteristics during C-ring compressive and tensile testing at room temperature and 850°C, but underwent plastic deformation at temperatures of 870-900°C.
- Coors P-100A-1 matrix exhibited brittle fracture characteristics during C-ring compressive and tensile testing up to temperatures of 900°C.
- 3M CVI-SiC composite matrix similarly retained its composite characteristics (i.e., room temperature to 900° C).
- Testing of the as-manufactured Schumacher FT20 and Pall 326 filter matrices at high temperature (i.e., 850°C, 870°C, and 900°C) resulted in higher compressive/tensile strengths in comparison to the calculated room temperature strengths. Although these materials were manufactured at substantially higher temperatures, the apparent enhanced strength was considered to reflect the response of the clay bonded silicon carbide filter matrix to the higher test temperatures. Flaws generated during cooldown from process operating conditions, or edge cracks/flaws created during sample preparation (i.e., cutting) may have "self-healed" when the clay bonded silicon carbide filter materials were re-exposed to high temperature (850-900°C).
- The resulting load bearing capability of each filter material at 850°C follows:
    - Schumacher Dia Schumalith FT20: 58.8±2.5 lb (compression); 36.4±0.2 lb (tension)
    - Pall 326: 95.7±1.2 lb (compression); 53.9±8.0 lb (tension)
    - Coors P-100A-1 alumina/mullite: 54.3±1.8 lb (compression); 39.5±2.1 lb (tension)
    - 3M CVI-SiC composite: 3.2±0.8 lb (compression); 2.1±0.4 lb (tension).

After exposure to PCFBC conditions,

- The Schumacher Dia Schumalith FT20, Pall 326, and Coors P-100A-1 alumina/mullite materials (i.e., intact, as well as broken filter elements) exhibited brittle fracture characteristics during C-ring compressive and tensile strength testing at room temperature, 850°C, and 900°C.

Table 8

As-Manufactured and Residual Process Strength of the PCFBC-Exposed Porous Ceramic Filter Materials

		C-Ring Compressive Strength, psi				C-Ring Tensile Strength, psi			
Candle ID No.	**Time, Hrs.**	**25°C**	**850°C**	**870°C**	**900°C**	**25°C**	**850°C**	**870°C**	**900°C**
Schumacher FT20									
S350F/108	---	2135±155 (8)	3147± 85 (2)	3409±205 (8)	3549±127 (4)	1727± 74 (6)	3070± 52 (2)	3023± 72 (6)	2937±141 (4)
S350F/8 (T12)	540	2140± 88 (8)	3716±426 (8)	ND	3602± 38 (5)	1957±146 (8)	2927±169 (8)	ND	3076±251 (6)
S350F/42 (B15)	505	2014± 72 (8)	3711±240 (8)	ND	3758± 93 (6)	1837±96 (8)	3185±187 (8)	ND	3343±212 (6)
Test Segment No. 2 Broken Section	540	ND	3242±454 (9)	ND	ND	ND	3001±205 (9)	ND	ND
Pall 326									
R3-676	---	2961±186 (8)	4713±133 (2)	4953±270 (8)	3942±345 (4)	2480±124 (6)	4019±576 (2)	4498±262 (6)	4057±203 (4)
R5-654 (B21)	505	2001±167 (8)	2998±215 (8)	ND	3078±236 (6)	1807±92 (8)	2816±361 (8)	ND	2848±243 (6)
R5-665 (M21)	540	1784±159 (8)	3126±221 (8)	ND	3342±237 (6)	1792±170 (8)	3309±426 (8)	ND	3088±313 (6)
Test Segment No. 2 Broken Section	540	ND	2979±210 (9)	ND	ND	ND	2824±329 (8)	ND	ND

Table 8 (Continued)

As-Manufactured and Residual Process Strength of the PCFBC-Exposed Porous Ceramic Filter Materials

		C-Ring Compressive Strength, psi				C-Ring Tensile Strength, psi			
Candle ID No.	**Time, Hrs.**	**25°C**	**850°C**	**870°C**	**900°C**	**25°C**	**850°C**	**870°C**	**900°C**
Coors P-100A-1									
FC-030	---	2587±268 (8)	2843± 76 (2)	2966±222 (8)	2668±345 (4)	2751±248 (6)	2927±851 (2)	2928± 69 (6)	3019±223 (4)
FC-043 (B17)	118	2166±127 (8)	2759±126 (2)	2658±253 (8)	2710±290 (4)	2344± 77 (6)	2325±232 (2)	2588±124 (6)	2367±171 (4)
FC-070 (B22)	505	1900±106 (8)	2540±188 (8)	ND	2338±119 (6)	2406±152 (8)	2516±203 (8)	ND	2560±111 (6)
DC-051 (B1)	1650[(a)]	1611±199 (8)	1921±112 (8)	ND	1950±117 (6)	2065±185 (8)	2161±197 (8)	ND	2321±70 (6)
Test Segment No. 2 Broken Section	505 or 540	ND	2236±105 (9)	ND	ND	ND	2382±117 (9)	ND	ND
3M CVI-SiC Composite									
M-51076	---	1903±362 (8) 12448±2620 (8)	2472±414 (2) 11194±4509 (2)	1823±532 (8) 12286±4014 (8)	2173±725 (4) 13987±4105 (4)	1715±281 (6) 10718±2035 (6)	1754±266 (2) 10816±1771 (2)	1781±124 (6) 10632±538 (6)	1119±238 (4) 6800±1335 (4)
M-51093 (B47)	35	1092±262 (8) 8799±1844 (8)	1296±518 (2) 9858±3321 (2)	1151±461 (8) 9303±3335 (8)	1575±322 (4) 11328±1489 (4)	1197±332 (6) 8397±2340 (6)	1095±134 (2) 7737±1273 (2)	911±227 (6) 6808±1038 (6)	1049±297 (4) 7182±1529 (4)
M-51103 (B36)	387	667±193 (8) 5880±1733 (8)	1491±292 (8) 12701±2561 (8)	ND	1185±639 (6) 9845±4545 (6)	657±198 (8) 5044±1582 (8)	878±156 (8) 6520±659 (8)	ND	979±255 (6) 7471±1478 (6)
Test Segment No. 2 Broken Section	387	ND	1841±308 (9) 11724±1598 (9)	ND	ND	ND	1277±436 (9) 7192±2045 (9)	ND	ND

(a) Element Was Exposed For 1110 Hrs To PFBC Test Conditions In Test Segment No. 5 At AEP (Location AB-32).

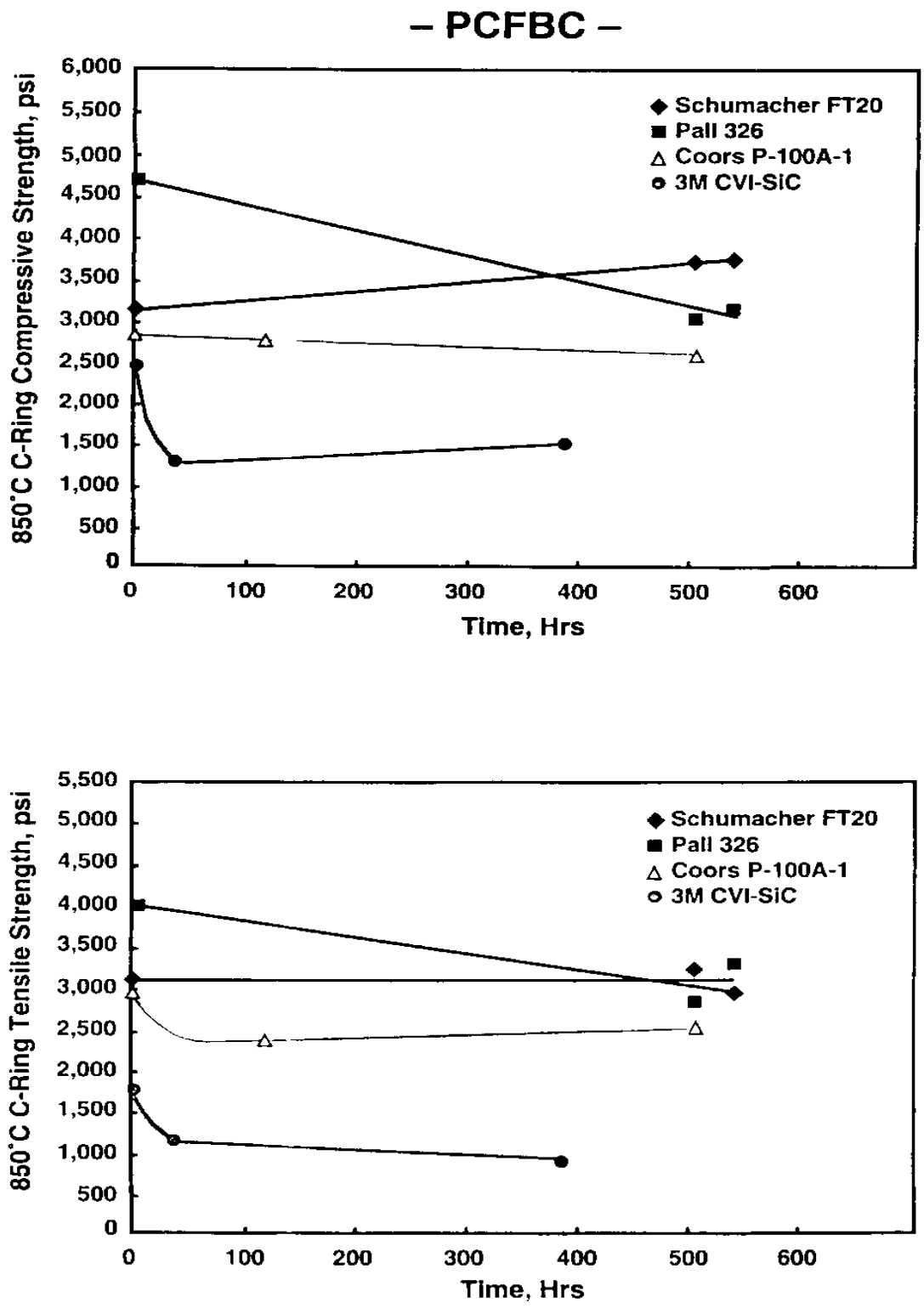


Figure 2 — Residual High Temperature Strength of the PCFBC-Exposed Candle Filters

- The 3M CVI-SiC composite filter matrix (i.e., intact, as well as broken filter elements) retained its composite failure characteristics during C-ring compressive and tensile strength testing at room temperature, 850°C, and 900°C.
- In general, either virtually no change, or an increase in strength of the Schumacher Dia Schumalith FT20 filters resulted along the OD and ID surfaces of the material after 505-540 hours of operation in the 850°C, PCFBC environment (i.e., elements located in the top and bottom arrays).[b]
- In contrast to the Schumacher Dia Schumalith FT20 filter material, the Pall 326 filters that were located in the middle and bottom arrays exhibited a loss of strength along both OD and ID surfaces after 505-540 hours of operation in the 850°C, PCFBC environment (i.e., 34-36% compressive strength reduction at 850°C; 18-30% tensile strength reduction at 850°C).
- Within the first 505 hours of PCFBC operation, the Coors P-100A-1 alumina/mullite filter elements that were located in the bottom array tended to experience a loss of strength along their OD surface (i.e., 3% compressive strength loss after 118 hours at 850°C; 11% compressive strength loss after 505 hours at 850°C). An initial reduction in strength was also apparent along the ID surface of the Coors P-100A-1 alumina/mullite filter elements during exposure to PCFBC operating conditions. A slight increase in strength may have resulted along the ID wall between 118 and 505 hours of operation (i.e., 21% tensile strength loss after 118 hours at 850°C; 14% tensile strength loss after 505 hours at 850°C). Continued operation of the Coors P-100A-1 alumina/mullite filters is required to demonstrate whether strengthening of the matrix is indeed occurring with time.
- The Coors P-100A-1 alumina/mullite filter elements that experienced 1110 hours of PFBC operation at the AEP Tidd Demonstration Plant in Brilliant, Ohio, remained intact after an additional 540 hours of operation in the Foster Wheeler PCFBC test facility in Karhula, Finland. As shown in Table 8, the compressive and tensile strength of the PFBC/PCFBC-exposed Coors P-100A-1 alumina/mullite matrix was lower than that of the PCFBC-exposed Coors P-100A-1 alumina/mullite elements.[c]
- When compared with the residual process temperature strengths of the clay bonded silicon carbide filter elements, a lower residual process temperature strength was evident for the Coors P-100A-1 alumina/mullite candle filters.
- In order to determine the strength of the 3M CVI-SiC composite filters, 15 mm C-rings were cut from intact filter elements or broken sections, and were tested in a similar fashion as the Schumacher, Pall, or Coors filter materials. In calculating the

[b] Compressive strength testing evaluates the residual strength along the OD surface of the candle filter that was in direct contact with the process gas. Tensile strength testing evaluates the residual strength along the ID surface of the candle filter that was in direct contact with the pulse cycled gas. If the porous ceramic filter matrix is susceptible to thermal fatigue, generally due to repetitive pulse cycling, a reduction in strength of the matrix would be evident along the ID or tensile-tested surface of the filter element.

[c] Comparison of the as-manufactured room temperature C-ring compressive and tensile strengths of the Coors P-100A-1 alumina/mullite filters with the reported PFBC/PCFBC-exposed filters indicates a 37% reduction in strength along the OD surface of the matrix, while a 24% loss of strength results along the ID surface of the material. Strength testing at 850°C, 870°C, and 900°C of the Tidd manufactured alumina/mullite P-100A-1 filters (i.e., "DC" series) was not conducted, and therefore comparison of the high temperature PFBC/PCFBC strengths can not be made.

residual strength, the wall thickness of the triaxial support braid was utilized, as well as the entire thickness of the three layer composite structure.[d] Depending on which wall thickness was used, strengths exceeding 5000 psi can be estimated (i.e., triaxial braid thickness only), or strengths ranging between ~600 and ~2500 psi can be determined (i.e., entire wall thickness).

- Using the entire wall thickness to calculate strength, the strength of the as-manufactured and PCFBC-exposed 3M CVI-SiC composite filter matrices was lower than either the Schumacher, Pall, or Coors as-manufactured or PCFBC-exposed filter materials.
- In general, the 3M CVI-SiC composite filter matrix exhibited a loss of strength after operation in the PCFBC environment (i.e., 48% and 40% compressive strength loss at 850°C after 35 and 387 hours, respectively; 38% and 50% tensile strength loss at 850°C after 35 and 387 hours, respectively).
- The load bearing capability of each PCFBC-tested filter material at 850°C follows:
    - Schumacher Dia Schumalith FT20: 61-73 lb (compression); 38-41 lb (tension)
    - Pall 326: 57-61 lb (compression); 35-42 lb (tension)
    - Coors P-100A-1 alumina/mullite: 34-43 lb (compression); 25-29 lb (tension)
    - 3M CVI-SiC composite: 2-2.5 lb (compression); 1.2-1.5 lb (tension).

Hoop Stress, Elastic Modulus, and Poisson's Ratio

A 254 mm (10 inch) section was removed from the Test Segment No. 2, PCFBC-exposed candle filters. Two 90° strain gage rosettes were installed along both inside and outside surfaces of the filter sections, at approximately the center of the test sample. A water filled bladder was inserted into the ID bore of each filter section, and was subsequently pressurized to determine the ultimate hoop strength of the filter matrix. The outer strain gage measurements for the 3M CVI-SiC composite filter matrix were somewhat in question, since delamination and shear were expected to have resulted between the layers of the composite structure during burst testing.

The pressure required to fail each filter section, as well as the ultimate hoop stress, elastic modulus, and Poisson's ratio for each of the Test Segment No. 2, PCFBC-exposed filter sections are presented in Table 9. Also included in Table 9 are similar material properties for the as-manufactured filter matrices.

Thermal Expansion

Sections of the PCFBC-exposed Schumacher Dia Schumalith FT20 and Pall 326 filter materials were subjected to thermal expansion testing. As shown Figure 3, the thermal expansion of the PCFBC-exposed Schumacher and Pall filter materials is nearly identical. Similarly the thermal expansion of the PCFBC-exposed clay bonded silicon carbide filter materials is nearly identical to that of the as-manufactured Coors P-100A-1 alumina/mullite and Schumacher Dia Schumalith F40 filter matrices.

[d] Entire wall thickness of the 3M CVI-SiC composite filter includes the thickness of the outer confinement layer, the middle filtration mat, and the structural support or triaxial braid.

Table 9

Material Properties of the PCFBC-Exposed Porous Ceramic Candle Filters —-Test Segment No. 2 —

Candle ID No.	Operating Time, Hrs	Burst Pressure, psi	Ultimate Hoop Stress, psi	Modulus, psi x 10^6	Poisson's Ratio
Schumacher Dia Schumalith FT20					
As-Manufactured	--	665	1703	7.3	0.17
S350F/108 (T12)	540	555	1496	7.44	0.208
S350F/42 (B15)	505	590	1584	7.39	0.152
Pall 326					
As-Manufactured	--	NE	NE	NE	NE
R5-655 (M21)	540	525	1369	5.00	0.162
R5-654 (B21)	505	520	1344	5.15	0.160
Coors P-100A-1 Alumina/Mullite					
As-Manufactured	--	860	2317	5.7	0.23
FC-070 (B22)	505	540	1503	4.84	0.205
DC-051 (B1)	1650[(a)]	505	1373	5.18	0.196
3M CVI-SiC Composite					
As-Manufactured	--	NE	1.01 ksi	2.96-3.38	0.14-0.27
M-51103 (B36)	387	133	1179	3.35	0.224

NE: Not Evaluated.
(a) PFBC/PCFBC-Exposed Candle Filter.

The thermal expansion of the porous ceramic filter materials is lower than the thermal expansion of the PCFBC or PFBC ash materials. The PCFBC ash sample that was used in the thermal expansion testing was representative of the dust cake layer that remained along the outer surface of the candle filters at the conclusion of Test Segment No. 2 at the Foster Wheeler test facility in Karhula, Finland. The PFBC ash sample was taken from a densely packed ash plug that formed within the inner bore of the filter elements during Test Segment No. 5 at the AEP Tidd Demonstration Plant in Brilliant, OH. The relatively wide range in the percent expansion of the ash materials as a function of temperature is expected to reflect the difference in the density, as well as the variation in the composition of the ash deposits.

High Temperature Creep

High temperature creep testing was conducted on 115 mm x 8.5 mm x 12 mm bars removed from PCFBC-exposed Schumacher Dia Schumalith FT20 and Pall 326 candle filters. As shown in Figure 4, these materials, unlike the Schumacher Dia Schumalith F40 and Pall

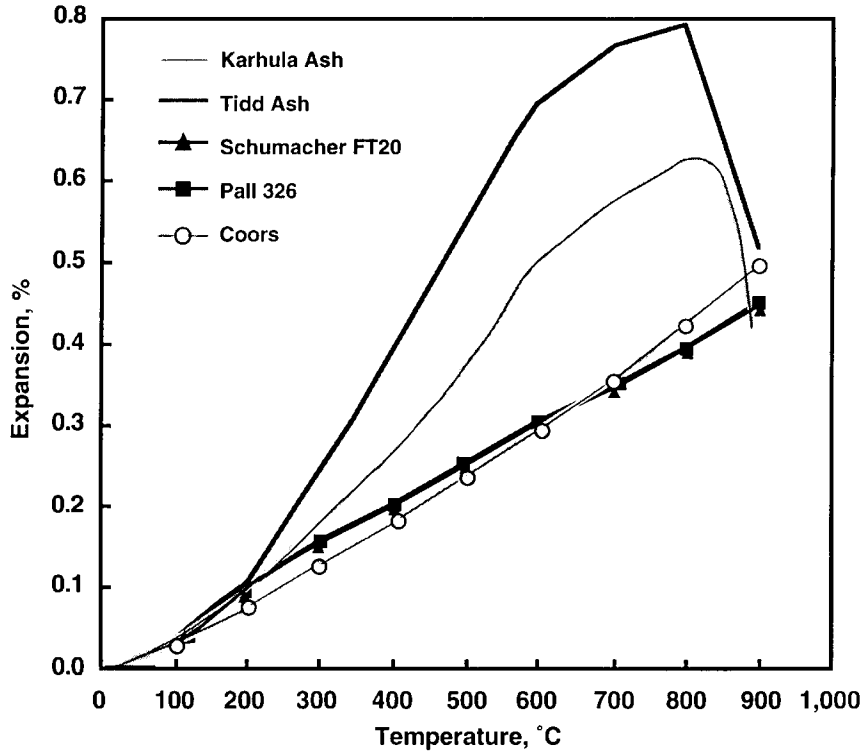
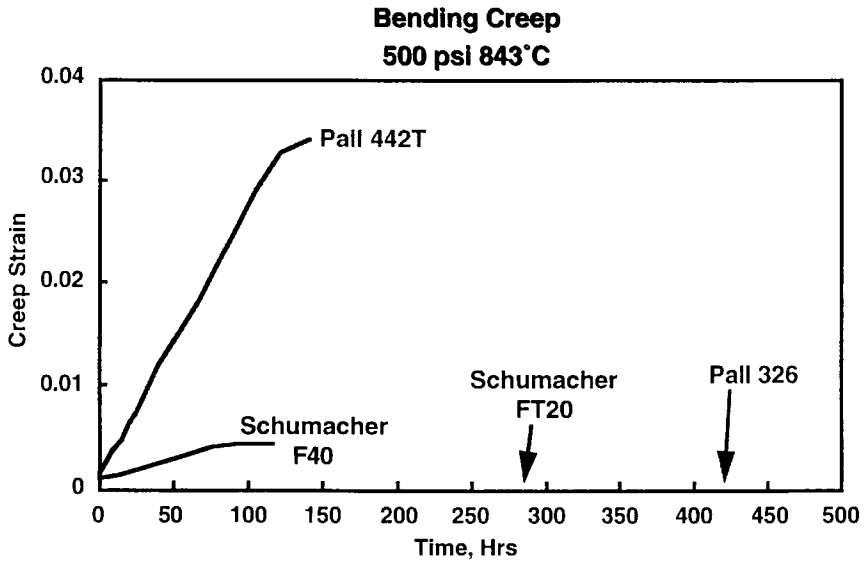


Figure 3 — Thermal Expansion of the Porous Ceramic Filter and Ash Materials



Schumacher Dia Schumalith F40 (S418/312B): 3038 Hrs, 732°C PFBC
Schumacher Dia Schumalith FT20 (S350F/106): 540 Hrs, 850°C PCFBC
Pall Vitropore 442T (R3-86): 1341 Hrs, 830°C PCFBC
Pall 326 (R5-665): 540 Hrs, 850°C PCFBC

Figure 4 — High Temperature Creep Strain as a Function of Time

Vitropore 442T materials, did not exhibit high temperature creep when a 500 psi, 4-point bend, flexural load was applied for ~300-500 hours at temperatures of 843°C (1550°F).

Similar high temperature creep testing was conducted using the 3M CVI-SiC composite filter matrix. Although delamination of the triaxial braid from the filtration and outer confinement layers resulted, testing indicated that the 3M CVI-SiC composite matrix did not exhibit creep strain after ~300-500 hours when a 500 psi, 4-point bend flexural load was applied to the material at temperatures of 843°C (1550°F).

Summary

With the exception of oxidation and removal of silicon carbide from the outer confinement and filtration mat layers of the 3M CVI-SiC composite filter matrix, degradation of the porous ceramic filter materials after operation in the PCFBC environment appeared to be limited. Additional operation in the PCFBC environment will be required to determine whether the advanced monolithic and fiber reinforced filter materials have achieved a conditioned residual bulk strength.

In addition to oxidation and removal of silicon carbide, the low load bearing capability of the 3M CVI-SiC composite filter elements, generally increases the potential for failure during ash bridging events, as well as crushing during handling and installation within the filter array, and maintaining adequate sealing around the filter flange.

Characterization of the PCFBC Ash (Test Segment No. 2)

Samples of the ash cake deposits were removed from various locations of the W-APF at the conclusion of Test Segment No. 2, and subjected to the following analyses:

- Scanning electron microscopy/energy dispersive x-ray analysis (SEM/EDAX)
- X-ray diffraction analysis (XRD)
- Moisture content
- Room temperature compression strength
- Density
- Thermal expansion.

Typically two types of particles were present within the ash cake layer that deposited along the outer surface of the candle filters in the top array. These include:

- Submicron and micron particulates that were enriched with aluminum and silicon. These particles were considered to be entrained fly ash fines that were released during the combustion of coal. “Melt-like” features were evident along the ash fines.
- Larger sulfated or partially sulfated calcium-based limestone or sorbent fines.

The aluminum and silicon-enriched coal ash fines were generally seen to adhere to the larger sulfated or partially sulfated sorbent fines. Extensive crystalline features were evident along the outer surface of the sulfated or partially sulfated sorbent fines. Agglomeration of crystalline

formations and/or "melt-like" features were detected, particularly along the surface of coal ash fines. In addition, spherical, ~10 μm particles enriched with silicon and calcium were also present within the deposited ash cake layer.

Dense packing of fines resulted within the ash cake layer that formed along the outer surface of the candle filters in the top array. Based on the analyses conducted, the cross-sectioned ash cake did not show evidence of fracturing of bonds or "melt-like" phases that may have originally been present between adjacent particles. This implies close packing of fines, with limited point contact between particles prior to fresh fracturing of the deposited ash cake layer.

Ash deposits that collected along the metal filter holders were also characterized. SEM/EDAX analysis indicated that porosity existed within the interconnective network of ash and sorbent particles that deposited around the metal filter holders. Within the deposit, a "melt-like" phase formed, sintering adjacent particles together. The sintered bond frequently formed point contact, necks, and channels between particles within the PCFBC deposit. The bond was identified by EDAX to be silicon and/or silicon-aluminum-enriched.

Sorbent particles were also present within the ash cake deposit that formed around the metal filter holders. Based on EDAX analyses, the limestone sorbent was considered to be completely or nearly completely sulfated. As in the filter cake deposit, the metal holder deposit contained discrete sorbent fines which were larger than the retained ash fines or agglomerates.

Similar analyses were conducted on ash removed from the filter hopper. A greater quantity of the larger calcium-containing sorbent and possibly the iron-enriched particles was detected in the ash hopper deposit in comparison to the quantity of these particles found within either the filter ash cake layer or metal holder deposits. When viewed at high magnification, the filter hopper ash was seen to contain extensive porosity. Porosity resulted from the interconnective network of fines that were held together through a silicon and/or aluminum-silicon-enriched "melt-like point contact, neck or channel phase.

X-ray diffraction (XRD) analysis was conducted to identify bulk composition of the PCFBC ash cake deposits. As shown in Table 10, the PCFBC ash consisted primarily of anhydrite and quartz, with secondary contributions of hematite, aluminosilicate phases, as well as an amorphous phase. Characteristically the PCFBC ash had a higher concentration of α-SiO_2 in comparison to the PFBC ash which formed within the W-APF at the AEP Tidd Demonstration Plant.[6] Although small, the concentration of the amorphous phase present in the PCFBC fines was greater than in the PFBC fines.

Comparison of the PFBC and PCFBC candle filter ash cake deposits indicated that

- The concentration of anhydrite was generally comparable in both materials.
- A higher concentration of hematite (Fe_2O_3) and quartz (α-SiO_2) resulted in the PCFBC ash cake layer.

Table 10

PCFBC Ash Composition
— Test Segment No. 2 —

X-Ray Diffraction Analysis, wt%	
Anhydrite ($CaSO_4$)	46-60%
Quartz (α-SiO_2)	21-40%
Hematite (Fe_2O_3)	9-11%
Aluminosilicate	3-8%
Anorthite ($CaAl_2Si_2O_8$)	
Margarite ($CaAl_2(Si_2OAl_2)O_{10}(OH)_2$_	
Kyanite (Al_2SiO_5)	
Amorphous	0-3%
* Additional Phases Present	

- Magnesium sulfate hydrates ($MgSO_4 \bullet xH_2O$), periclase (MgO), calcite ($CaCO_3$), and monticellite ($CaMgSiO_4$) were present in the PFBC ash which resulted from the utilization of a dolomitic limestone sorbent at Tidd vs limestone at Karhula.

With respect to the composition of the ash cake deposits that formed around the W-APF filter holders,

- The ash deposit that formed around the holders in the top filter arrays at Tidd was extremely hard and tenaciously bonded. This material had a substantially higher concentration of anhydrite (i.e., sulfated sorbent; $CaSO_4$) in comparison to either the deposits formed around middle and bottom holders at Tidd, or around the holders in the top array at Karhula. The tenacious nature of the ash cake deposit formed at Tidd decreased as the concentration of anhydrite and magnesium sulfate hydrate decreased, and the concentration of periclase (MgO), calcium carbonate ($CaCO_3$), monticellite ($CaMgSiO_4$), and calcium sulfate ($CaSO_4$) increased.[e]
- Hematite (Fe_2O_3) and quartz (α-SiO_2) were present at a higher concentration in the PCFBC fines that deposited around the holder in the top array at Karhula, in comparison to the fines that collected along the top array filter holders at Tidd.

PCFBC ash deposits that were removed from the W-APF cluster at the conclusion of Test Segment No. 2 were subjected to density, compressive strength, and moisture analyses. The resulting averaged density of the PCFBC ash samples was determined to be 0.587 ± 0.062 gm/cm^3.

[e] The filter holder ash deposit formed at Karhula was less tenaciously bonded in comparison to the ash that formed along the filter holders in the top arrays at Tidd. The consistency of the Karhula filter holder deposit along the top array was similar to the middle and bottom array filter holders at Tidd in Test Segment No. 5, as well as throughout all arrays in prior test segments at Tidd.

The compressive strength of the various ash deposits ranged between 0.7 and 18.8 psi (i.e., average compressive strength: 11.01±7.3 psi; 4.7±3.1 lbs to failure). Typically the ash deposits had an 0.02-0.07% moisture content.

Application

As a key component in advanced coal- or biomass-based power applications, hot gas filtration systems protect the downstream heat exchanger and gas turbine components from particle fouling and erosion, cleaning the process gas to meet emission requirements. When installed in either PFBC or IGCC plants, lower downstream component costs are projected, in addition to improved energy efficiency, lower maintenance, and elimination of additional and expensive fuel or flue gas treatment systems. As a critical component, long-term performance, durability, and life of the porous ceramic filter elements are essential to the successful operation of the hot gas filtration system in advanced combustion and gasification applications.

Future Activities

Efforts will be focused on continuing testing of the advanced monolithic and second generation fiber reinforced candle filters in the W-APF system at Foster Wheeler's PCFBC test facility in Karhula, Finland (Task 2).

Effort is being initiated in Task 3, to assess the stability of select advanced filter materials (Table 11) when subjected to long-term exposure in actual IGCC gas streams. A pressurized mini-vessel will tentatively be installed downstream of the W-APF system at the Sierra Pacific Piñon Pine IGCC Demonstration Plant in Reno, NV. There either mini-candles or coupons will be exposed to the process fuel gas at temperature for extended periods of time in the absence of char fines. Post-test characterization of either the mini-candles or coupons will be conducted .

Based on the information obtained during PCFBC and IGCC testing, additional component or material improvements will be identified and discussed with the various candle filter suppliers. If available, modified elements will be resubjected to qualification testing in Westinghouse's PFBC simulator (Task 1), or included within the Piñon Pine mini-vessel (Task 3).

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Table 11

Candidate Porous Ceramic Candle Filters and Material Coupons for Testing under IGCC Conditions

Mini-Filters	Coupons
Schumacher Dia Schumalith F40	DuPont SiC-SiC
Schumacher Dia Schumalith FT20	Ultramet CVI-SiC Reticulated Foam
Pall Vitropore 442T	Kaiser Carbon-Based Matrix
Pall 326	Kaiser Pyrolyzed Carbon-Nicalon
Coors P-100A-1 Alumina/Mullite	INEX Siliconized Silicon Carbide
3M CVI-SiC Composite	Textron Nitride Bonded Silicon Carbide
DuPont PRD-66	Textron Nitride Bonded Silicon Nitride
Pall Iron Aluminide	Triad Corporation Calcium Aluminosilicate
Blasch Alumina	Phoenix Oxide-Based Zirconate
Blasch Alumina/SiC	Corning Cordierite
3M Oxide-Based CFRCC	Scapa Aluminosilicate-Based Fibers
Techniweave Oxide-Based CFRCC	
B&W Oxide-Based CFRCC	

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Appendix A

Thermal Stress Modeling

Under simulated PFBC process conditions, Westinghouse monitored the temperature profile through various porous ceramic filter elements to determine the axial and radial temperature gradients that resulted along the entire length of the candle filter during pulse cleaning, or when a thermal transient event occurred. During pulse cycling, a maximum temperature drop of ~420°C (750°F) was shown to result along the inner surface of the filter body, near the mid-section of the element, 0.35 sec after delivery of the pulse. The extent of cooling of the filter matrix was limited to ~2 mm from the inner surface, similar to the area where circumferential cracking was observed after PCFBC and accelerated pulse cycle testing of the alumina/mullite candle filters. Within 5 sec after delivery of the pulse, the inner surface of the element returned to within ~30ºC (50°F) of the initial operating temperature (Figure A-1).

In order to reduce the impact of thermal stress induced along the inner surface of the filter elements, Westinghouse installs a fail-safe/regenerator device above each candle. During pulse cycling with the inclusion of the fail-safe/regenerator device, a maximum temperature drop of ~170°C (300°F) was shown to result along the mid-section of the filter element, 0.35 sec after delivery of the pulse.

The axial and radial temperature measurements generated along and through the P-100A-1 filter wall during pulse cycling were utilized to identify a corresponding stress intensity profile throughout the matrix via finite element modeling. Along the mid-section of the candle filter, the stress intensity was calculated to be ~10,000 psi, primarily within the first 0.5 mm from the inner surface of the candle, at 0.35 sec after delivery of the pulse. The stress intensity resulting from pulse cleaning exceeded the rupture modulus of the as-manufactured P-100A-1 alumina/mullite filter matrix (i.e., 2500-3000 psi). As a result of high stress in this area of the filter element, microcrack formations are likely to occur. With the inclusion of the fail-safe/regenerator device, stress intensities as high as 3600 psi were calculated within the first 1 mm along the inner surface of the candle, at 0.35 sec after initiation of the pulse cycle. Clearly the use of the fail-safe/regenerator device sufficiently heats the incoming pulse gas to the extent that thermal stress within the matrix approaches or is below the rupture modulus of the as-manufactured P-100A-1 alumina/mullite filter matrix. As a result, the formation of circumferential and/or radial microcracks along the inner surface of the filter element is expected to be mitigated.

Temperature gradients were also measured along the outer surface of the P-100A-1 alumina/mullite and clay bonded silicon carbide filter elements during simulated thermal transient testing. These measurements were utilized to project the temperature profile, hoop stress, and hoop strain through each filter wall. Using a simple axisymetric heat transfer model, the projected temperature through the wall as a function of time after transient initiation was shown to closely correspond to the temperature measurements from high-speed thermocouples that were embedded within the filter wall.

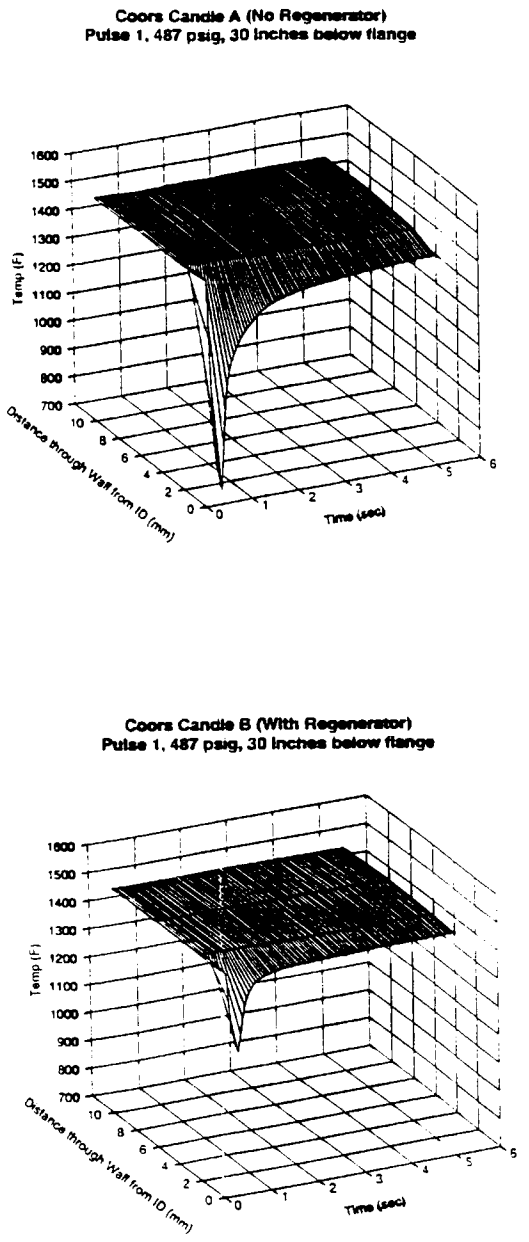

Figure A-1 — Axial and Radial Temperature Profile During Pulse Cycling of Porous Ceramic Candle Filters

During exposure to simulated maximum thermal transient conditions, the P-100A-1 alumina/mullite filter matrix maintained the original operating temperature along the inner surface of the filter element while the outer surface rapidly cooled (Figure A-2). Between 10 and 60 sec after initiation of the event, the projected maximum hoop stress along the outer surface of the candle approached the rupture modulus of the filter matrix. For process-aged materials which have reduced bulk strengths, the stresses resulting during transient testing would be sufficient to induce microcrack formations along the outer surface of the filter element. In

conjunction with thermal fatigue along the inner surface of the alumina/mullite filter matrix which results from pulse cycling without the inclusion of the fail-safe/regenerator, the matrix is expected to be highly susceptible to slow crack growth, particularly in the presence of steam. As a result subcritical crack extension may occur during thermal excursions, leading to failure of the filter element.

As shown in Figure A-3, a more suppressed temperature gradient resulted through the wall of the clay bonded silicon carbide filter elements in comparison to the P-100A-1 alumina/mullite candle filters during exposure to thermal transient conditions. A maximum stress of ~450 psi was projected along the outer surface of the clay bonded silicon carbide filter during exposure to a maximum severity thermal transient event. This is substantially lower than the 2500-3000 psi bulk strength of the as-manufactured clay bonded silicon carbide filter matrix.

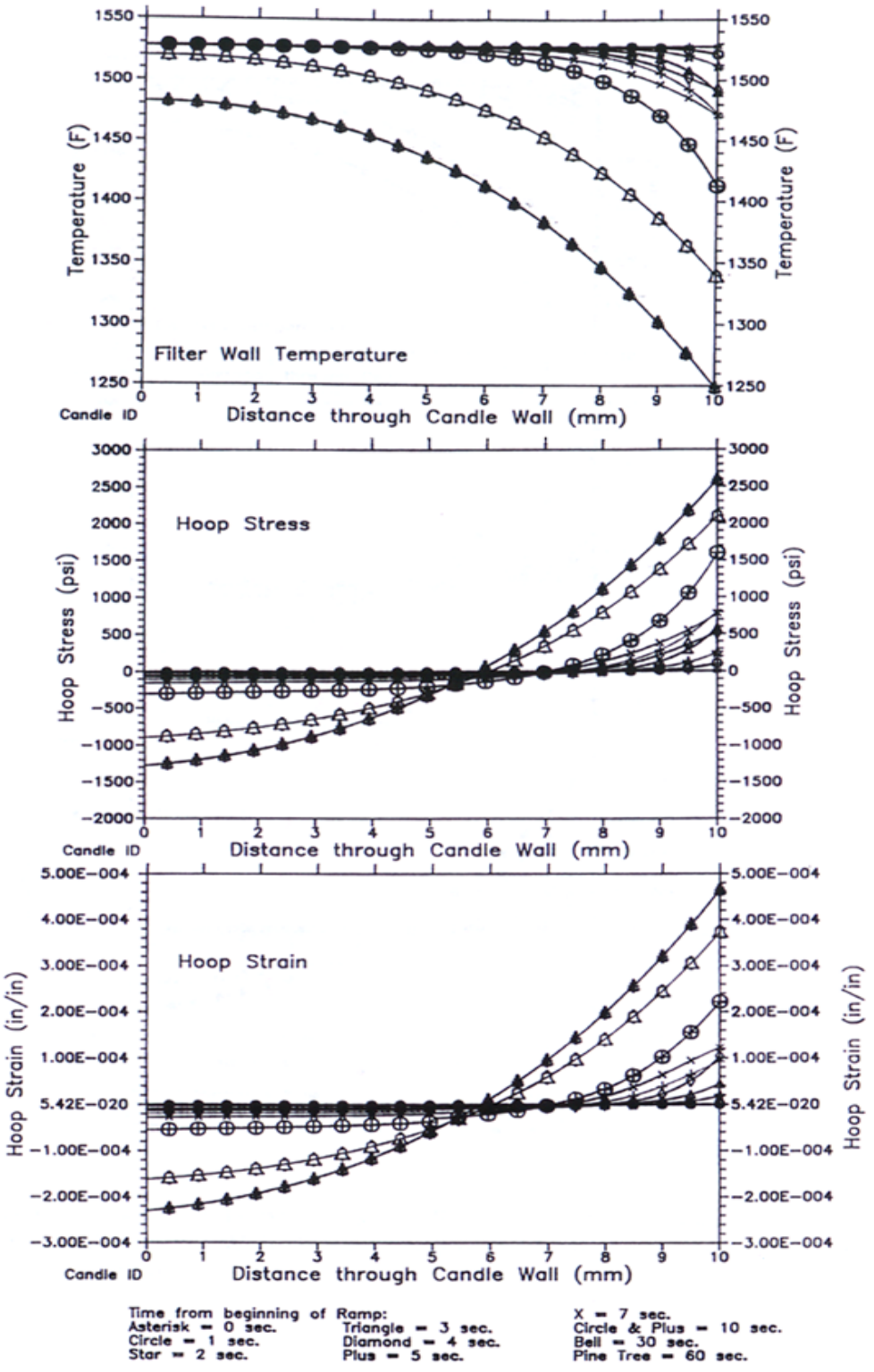

Figure A-2 — Projected Temperature, Hoop Stress, and Strain through the Coors P-100A-1 Alumina/Mullite Filter Wall during Exposure to Maximum Severity Thermal Transient Conditions

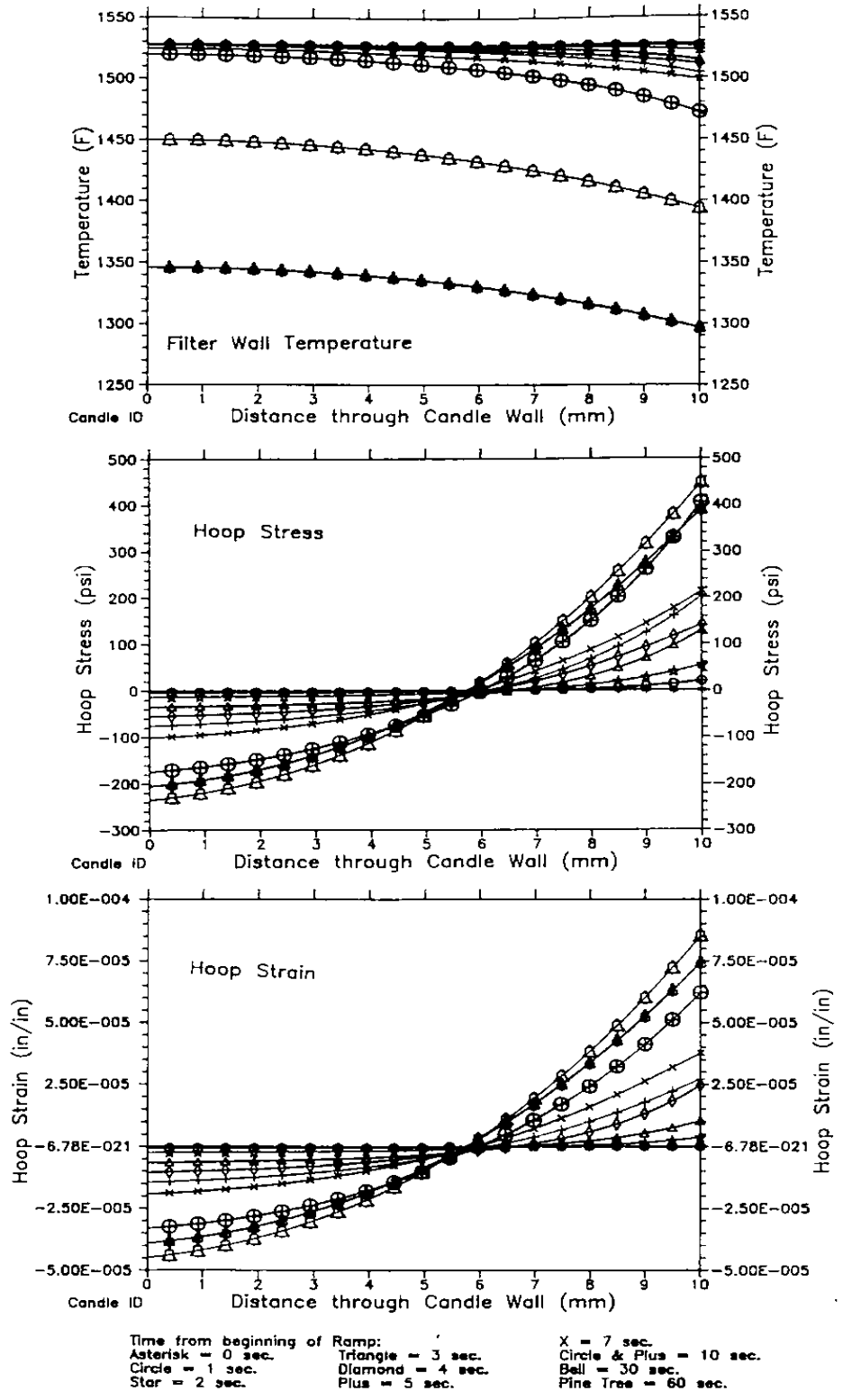

Figure A-3 — Projected Temperature, Hoop Stress, and Strain through the Pall Vitropore 442T Filter Wall during Exposure to Maximum Severity Thermal Transient Conditions