ADVANCED SECOND GENERATION CERAMIC CANDLE FILTERS

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by

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ABSTRACT

Through sponsorship from the Department of Energy's National Energy Technology Laboratory (DOE/NETL), development and manufacture of advanced second generation candle filters was undertaken in the early 1990's. Efforts were primarily focused on the manufacture of fracture toughened, 1.5 m, continuous fiber ceramic composite (CFCC) and filament wound candle filters by 3M, McDermott, DuPont Lanxide Composites, and Techniweave.

In order to demonstrate long-term thermal, chemical, and mechanical stability of the advanced second generation candle filter materials, Siemens Westinghouse[1] initiated high temperature, bench-scale, corrosion testing of 3M's CVI-SiC and DuPont's PRD-66 mini-candles, and DuPont's CFCC SiC-SiC and IF&P Fibrosic™ coupons under simulated, pressurized fluidized-bed combustion (PFBC) conditions. This effort was followed by an evaluation of the mechanical and filtration performance of the advanced second generation filter elements in Siemens Westinghouse's bench-scale PFBC test facility in Pittsburgh, Pennsylvania. Arrays of 1.4-1.5 m 3M CVI-SiC, DuPont PRD-66, DuPont SiC-SiC, and IF&P Fibrosic™ candles were subjected to steady state process operating conditions, increased severity thermal transients, and accelerated pulse cycling test campaigns which represented ~1760 hours of equivalent filter operating life.

Siemens Westinghouse subsequently participated in early material surveillance programs which marked entry of the 3M CVI-SiC and DuPont PRD-66 candle filters in Siemens Westinghouse Advanced Particulate Filtration (APF) system at the American Electric Power (AEP) Tidd Demonstration Plant in Brilliant, Ohio. Siemens Westinghouse then conducted an extended, accelerated life, qualification program, evaluating the performance of the 3M, McDermott, and Techniweave oxide-based CFCC filter elements, modified DuPont PRD-66 elements, and the Blasch, Scapa Cerafil™, and Specific Surface monolithic candles for use in our APF system at the Foster Wheeler pressurized circulating fluidized-bed combustion (PCFBC), pilot-scale, test facility in Karhula, Finland. This report presents a summary of these efforts, defining the stability of the various porous ceramic filter materials, as well as component performance and extended life for use in advanced coal-based power systems.

[1] Formerly the Westinghouse Electric Corporation.

LIST OF ACRONYMS

AEP	American Electric Power
APF	Advanced Particulate Filtration
CFCC	Continuous fiber ceramic composite
CVD	Chemical vapor deposition
CVI	Chemical vapor infiltration
DOE	Department of Energy
EDAX	Energy dispersive x-ray analysis
IF&P	Industrial Filter and Pump
IGCC	Integrated gasification combined cycle
PCFBC	Pressurized circulating fluidized-bed combustion
PFBC	Pressurized fluidized-bed combustion
NETL	National Energy Technology Laboratory
SEM	Scanning electron microscopy
SiC	Silicon carbide

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1. INTRODUCTION

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 pressurized fluidized-bed combustion (PFBC) or integrated gasification combined cycle (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 operating at temperatures of $\leq$850°C ($\leq$1560°F), long-term performance, durability, and life of the porous ceramic filter elements are essential to the successful operation of hot gas filtration systems in advanced combustion and gasification applications.

Efforts during the late 1970's through the early 1990's were focused on the development of filter systems which utilized monolithic clay bonded silicon carbide candle filters. As a result of operation in combustion gas environments, oxide-based, porous ceramic filter matrices were developed in order to achieve the chemical stability requirements for materials during long-term operation. Both oxide, as well as nonoxide porous ceramic filters were installed and operated in Siemens Westinghouse's Advanced Particulate Filtration (APF) system[1] at the American Electric Power (AEP) PFBC Tidd Demonstration Plant in Brilliant, Ohio, and at the Foster Wheeler pressurized circulating fluidized-bed combustion (PCFBC) test facility in Karhula, Finland (Figure 1.1). During operation of the 384 candle filter system at AEP between 1992 and 1995, 1710 and 5855 hours of operation were achieved with the Coors P-100A-1 alumina/mullite and Schumacher Dia Schumalith F40 surveillance candles, respectively. At Karhula, 2201-3331 hours of operation were achieved with the Coors P-100A-1 alumina/mullite surveillance filter elements, and 2201 hours with the Schumacher Dia Schumalith FT20 and Pall 326 surveillance filters between 1995 and 1997.

AEP PFBC Tidd Demonstration Plant

Foster Wheeler PCFBC Facility

Figure 1.1 — Siemens Westinghouse Advanced Particulate Filtration systems.

[1] Formerly the Westinghouse Electric Corporation.

Siemens Westinghouse conducted extensive filter material surveillance programs for candles removed from both the AEP and Karhula test facilities.[1, 2, 3] These efforts demonstrated that typically a reduction in filter material strength resulted and numerous microstructural changes occurred within each matrix as a response to operation in the high temperature, oxidizing, PFBC or PCFBC process gas environment. Microcrack formations were identified frequently within the alumina/mullite filter matrix, and were also expected to occur within the binder phase and through the silicon carbide grains in the clay bonded filter materials. Information generated at Siemens Westinghouse supported the use of a pulse regenerator device to 'preheat' the pulse cleaning air prior to entering the candle i.d. bore in order to mitigate thermal fatigue and microcrack formation of the ceramic matrices.

To provide a more 'ruggidized' filter system, emphasis was focused on the development and manufacture of advanced second generation porous ceramic filter elements which had significantly improved resistance to damage resulting from crack propagation and thermal fatigue, thermal excursions during plant/process upsets, and/or mechanical ash bridging within the cluster or array design (Figure 1.2). Several manufacturers developed chemically vapor-infiltrated (CVI) candle filters which were expected to have improved fracture toughness characteristics. Improved toughness characteristics were believed to mitigate crack propagation through the porous ceramic matrix, ultimately minimizing catastrophic failure, particularly under thermal fatigue and/or shock conditions during process operation. CVI-toughened materials typically included the use of both oxide and nonoxide continuous fiber ceramic composites (CFCCs).

Figure 1.2 — First generation monolithic and advanced second generation candle filters.

In the developmental and prototype CFCC filter-manufacturing efforts which involved the use of nonoxide CFCCs, the stability and oxidation resistance of the fiber/matrix interfaces continued to be key technical issues limiting high temperature use. Although the as-manufactured nonoxide CFCCs exhibited nonbrittle failure characteristics and had sufficient room temperature mechanical properties, the mechanical properties of the nonoxide CFCCs degraded as a result of oxidation of the interface during exposure to high temperature (>800°C; >1470°F) in an oxidizing environment. When this occurred, brittle failure resulted. In developing an oxide-based CFCC, the key issues which remained to be resolved were the production of oxide fibers with improved high-temperature capabilities, particularly under low stress loads (i.e., 5 to 15 ksi), as well as development of functional interface coatings which toughened the nonbrittle oxide CFCC matrix.

Manufacturers alternately developed fracture-toughened materials which utilized sol-gel and/or chemical vapor composite (CVC) techniques to form potentially less expensive fiber, felt or mat, whisker or particulate-reinforced oxide and nonoxide ceramic composites. The concept of utilizing a single-phase matrix also had merit in the production of porous ceramic filter elements, since phase transformations frequently resulted during process operation which potentially lead to mismatch in the thermal coefficients of expansion of the various resulting phases. Alternatively, the inclusion of conductive phases (i.e., silicon carbide, SiC) within oxide-based ceramic matrices had also been considered.

Similar to the commercially available filter elements, the advanced second generation filter materials were required to achieve reliable long-term service use, and be reproducibly manufactured in quantities of >500-1000 filter elements/year. The criteria for reliable, long-term service operation included:

- A minimum of one year continuous, maintenance-free, operating life (i.e., $\geq$8000 hours)
- Viability to withstand thermal shock conditions during 10-20 emergency process plant shutdowns, as well as through >100 controlled process startups and shutdowns
- Reliable performance in the event of exposure to temperature excursions of ~540-650°C (~1000-1200°F)
- Resistance to fracture and catastrophic failure during an ash-bridging event
- Maintenance chemical and phase stability during exposure to corrosive alkali, sulfur, or chloride gas, liquid, or condensed phase species.

During the mid 1990's, an assessment of the performance and the thermal/chemical/mechanical stability of following four, advanced second generation, candle filters was addressed at Siemens Westinghouse:

- 3M CVI-SiC — Chemical vapor infiltration of silicon carbide into an aluminosilicate Nextel™ 312 fiber preform
- DuPont Lanxide Composites SiC-SiC — Chemical infiltration of silicon carbide Nicalon™ fiber mat or felt preform
- DuPont Lanxide Composites PRD-66 — Filament wound structure containing corundum, cordierite, cristobalite, and mullite
- IF&P Fibrosic™ — Industrial Filter and Pump, vacuum infiltrated, oxide-based, chopped fibrous matrix.

Selection of the 3M, DuPont,[2] and IF&P filter elements was primarily based on the perceived near-term commercial manufacturing capabilities of each supplier, as well as considered benefits of the non-monolithic, advanced, second generation materials. The following sections discuss the bench-scale and qualification efforts conducted at Siemens Westinghouse to assess the thermal, chemical, and mechanical stability of the advanced, second generation filters, and their initial utilization in pilot and demonstration plant facilities.

[2] DuPont Lanxide Composites was acquired by Allied Signal, then Honeywell, and currently is a unit of GE Power Systems.

2. ADVANCED SECOND GENERATION POROUS CERAMIC FILTERS

The 1.5 m, 3M CVI-SiC, continuous fiber ceramic composite (CFCC) filter consisted of three layers — an outer open mesh confinement layer, a middle lapped filtration mat, and an inner triaxial braid fabric layer that formed the structural support matrix of the filter element (Figure 2.1). Within the confinement and filtration mat layers, an ~1-2 μm layer of silicon carbide was deposited which encapsulated the Nextel™ 312 or alumina-based fibers, while an ~100 μm layer of silicon carbide was deposited along the Nextel™ 312 triaxial braid in the support matrix. In order to strengthen the flange and end cap areas of the 3M candles, a second layer of the Nextel™ 312 triaxial braid was applied to each filter element.

The oxide-based, filament wound, DuPont PRD-66 filter matrix consisted of winding alumina coated S-glass along a mandrel, forming an open chevron pattern in the structural support wall (Figure 2.2). Subsequently an alumina coated single filament layer was circumferentially applied to the outer surface of each element, forming the external hoop wrapped membrane. Nominally the wall thickness of the DuPont PRD-66 filter wall was ~7 mm. A densified insert was added to the interior of the flange, in order to provide a thicker flange surface for gasketing and sealing of the elements in the Siemens Westinghouse filter system. An additional slurry coating was applied to the flange and end cap of each element for densification and increased strength. After high firing, the PRD-66 filament wound matrix consisted of a layered cordierite, mullite, cristobalite, and corundum microstructure, with minor concentrations of an amorphous phase. Due to the difference in thermal expansion of mullite, cordierite, and corundum, a microcracked structure resulted in the 1.5 m DuPont PRD-66 filter elements.

The first generation DuPont SiC-SiC candles were fabricated from a two-ply Nicalon™ felt that was formed as a 1.4 m cylindrical tube. A plug was inserted into one end of the cylinder to form a closed end cap, while additional felt was wrapped around the other open end of the cylindrical tube to from the flange. Silicon carbide was chemically vapor infiltrated (CVI) throughout both Nicalon™ felt layers, forming a strengthened matrix, as well as bonding the felt wrap and/or plug to the filter body. Fine grain silicon carbide grit was applied with polymeric resin slurry along the outer surface of the filter element, forming the external membrane.

Subsequently DuPont manufactured SiC-SiC composite candle filters utilizing a hybrid architecture (Figure 2.3). An open mesh Nicalon™ screen served as the candle filter support structure. A Nicalon™ single-ply felt was layered over the mesh, and the unit was subsequently subjected to silicon carbide CVI. A silicon carbide grit was applied to the outer surface of the CVI-SiC felt in order to form an effective particulate barrier filter membrane. Since Nicalon™ is primarily a silicon carbide fiber which contained oxygen, and silicon carbide was deposited along the fiber rigidizing the preform, the material was designated as a SiC-SiC composite filter matrix.

The second generation Industrial Filter & Pump (IF&P) Fibrosic™ filter matrix consisted of vacuum infiltrated alumina and aluminosilicate chopped fibers (Figure 2.4).

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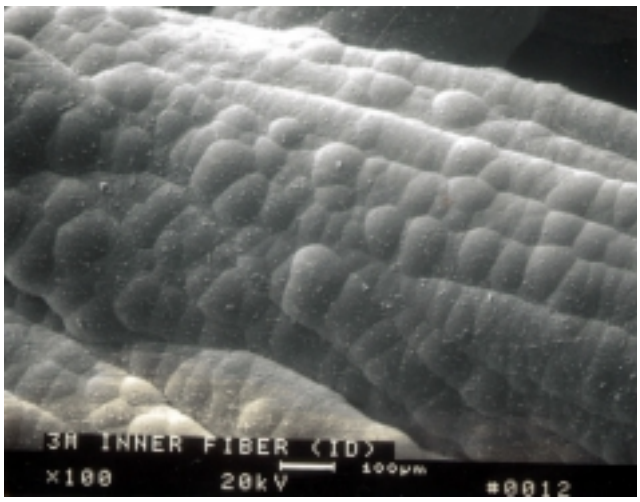
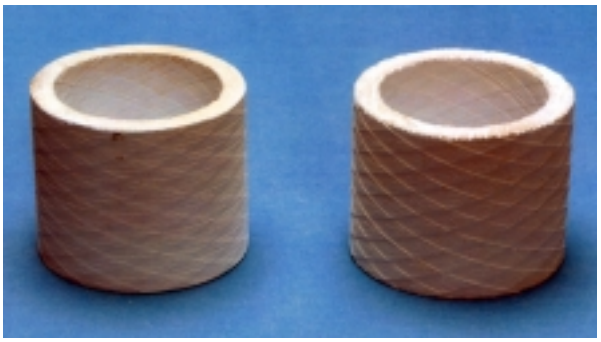


(b)

(c)

Figure 2.1 — 3M CVI-SiC CFCC composite filter matrix: (a) Composite structure: Outer confinement layer; Middle lapped filtration layer; Inner triaxial braid structural support; (b) External CVI-SiC surface; (c) CVI-SiC coated Nextel™ 312 fiber.



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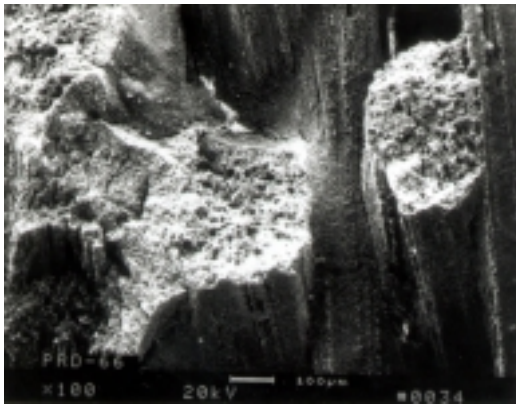

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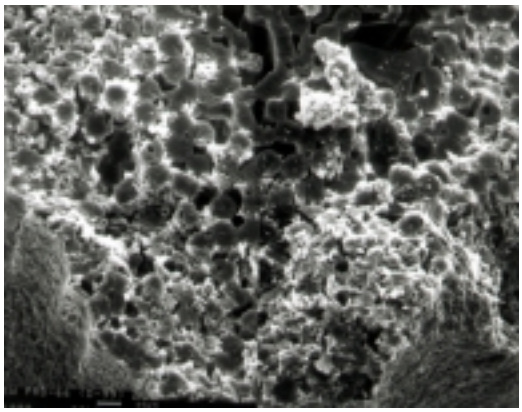

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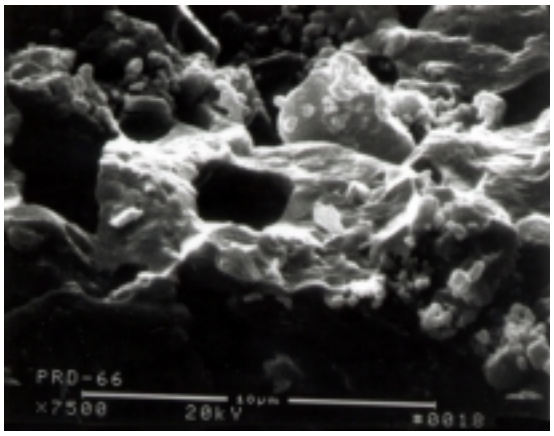

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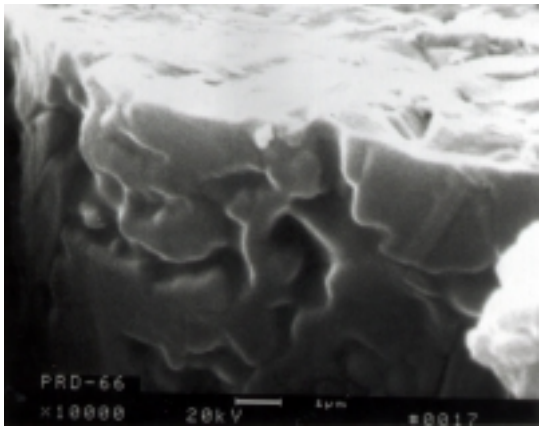
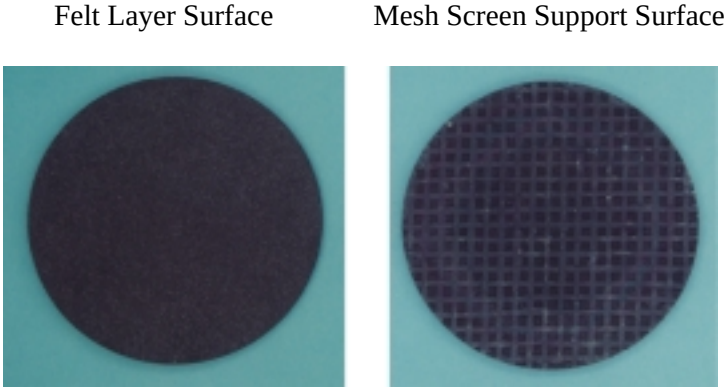


(e)

Figure 2.2 — DuPont PRD-66 filament wound filter matrix: (a) Section of the filament wound architecture; (b) Cross-sectioned as-manufactured matrix; (c) Higher magnification of the cross-sectioned fibers and polycrystalline phase; (d) Cross-sectioned fiber; (e) Fiber outer surface.



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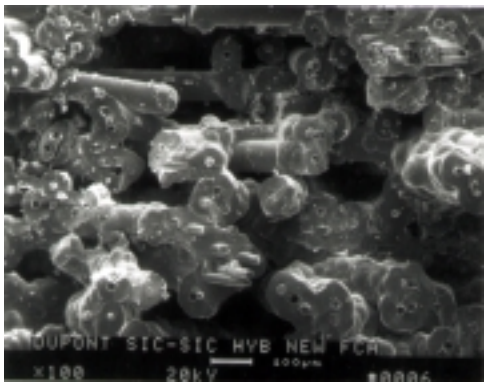

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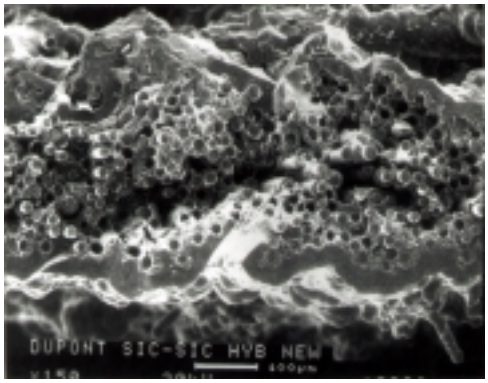

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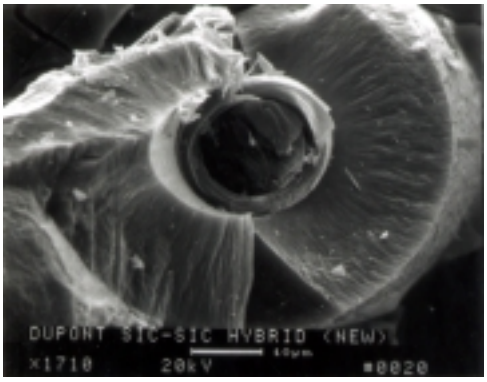
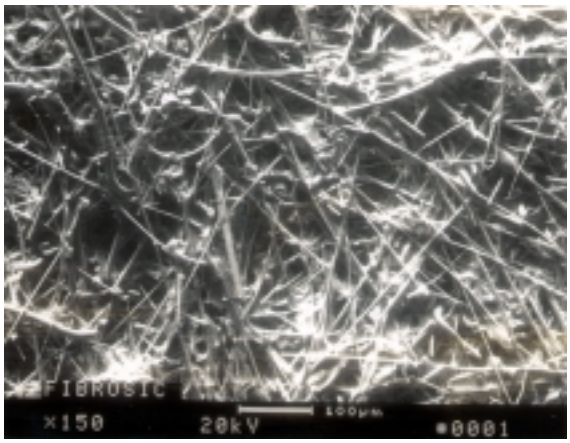


(d)

Figure 2.3 — DuPont SiC-SiC CFCC filter matrix: (a) Hybrid structure; (b) As-manufactured single-ply felt layer; (c) As-manufactured mesh screen support layer; (d) Presence of the interface layer encapsulating Nicalon™ fibers in the as-manufactured single-ply felt layer.



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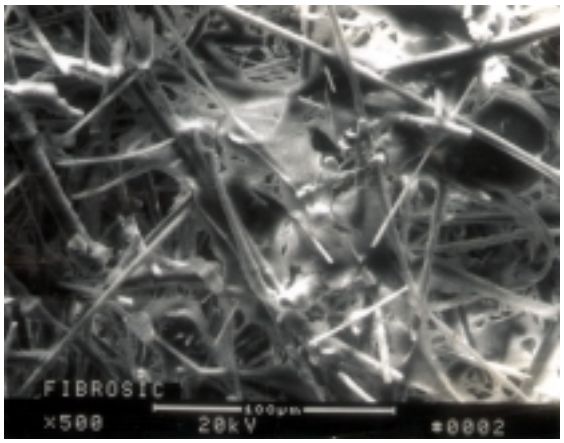

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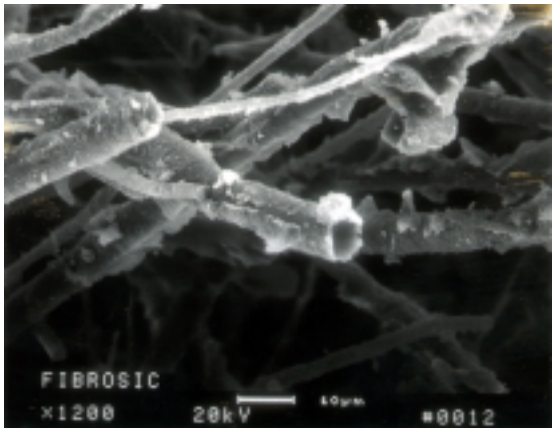

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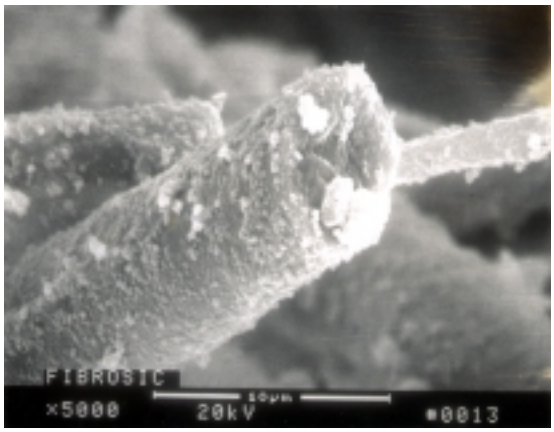


(d)

Figure 2.4 — IF&P Fibrosic™ filter matrix: (a) Vacuum infiltrated alumina and aluminosilicate-chopped fibers; (b) Higher magnification of the filtration media outer surface; (c) Chopped fibers contained within the cross-sectioned filter wall; (d) Higher magnification illustrating the morphology of the cross-sectioned fibers.

2.1 Thermal/Chemical Stability

Siemens Westinghouse demonstrated the relative thermal and chemical stability of the oxide-based Coors P-100A-1 alumina/mullite filter matrix during exposure to high temperature, oxidizing conditions which contained gas phase alkali and steam.[4-6] In order to demonstrate the stability of the oxide-based DuPont PRD-66 and IF&P Fibrosic™ materials, PRD-66 mini-candles which were manufactured with an ~75 mm section of open filtration area, and 70 mm diameter x 6.4 mm thick membrane coated IF&P discs were exposed for 400 hours at 870°C (~1600°F) to 5-7% steam/air and 20 ppm NaCl/5-7% steam/air flow-through testing in our bench-scale system shown in Figure 2.5. During the 400 hour exposure in 1995, both mini-candles and filter discs were subjected to pulse cleaning cycles at every 20 minute intervals, reflecting nominal process operating conditions.

High temperature, flow-through testing was similarly conducted in 1995 for the nonoxide-based, advanced, second generation filters using mini-candles of the 3M CVI-SiC composite matrix, and discs of the DuPont SiC-SiC composite matrix. Post-test characterization of each material included c-ring compression testing at 870°C (~1600°F) of the as-manufactured and flow-through-tested mini-candles, and 4-point bend, ¼-point flexural strength testing at 870°C (~1600°F) of the as-manufactured and flow-through-tested filter discs. A summary of these results follows.

Figure 2.5 — Bench-scale, high temperature, flow-through test facility.

2.1.1 3M CVI-SiC Composite Filter Matrix[7]

The 3M CVI-SiC composite filter consisted of three layers — an outer open mesh confinement layer, a middle filtration mat, and an inner triaxial braided fabric layer which formed the structural support matrix of the filter element. Within the confinement and filtration mat layers, an ~1-2 µm layer of silicon carbide encapsulated Nextel™ 312 or alumina-based fibers, while an ~100 µm layer of silicon carbide was deposited along the Nextel™ 312 triaxial braid in the support matrix.

After exposure to either the high temperature steam/air or alkali/steam/air flow-through environment, the 3M CVI-SiC mini-candles appeared to be intact, retaining their initial configuration (Figure 2.6). What was readily apparent was the very brittle nature of the outer confinement layer along the alkali/steam/air-exposed 3M CVI-SiC composite matrix. In several locations, the fiber bundles of the confinement layer weave were broken or missing.

Figure 2.6 — 3M CVI-SiC mini-candles after exposure to high temperature flow-through testing.

During preparation of the 3M CVI-SiC matrix for strength testing, two as-manufactured mini-candles were cut into 15 mm or 25.4 mm sections for high temperature c-ring compression testing. These sections were removed from the open filtration area of the mini-candle. Similarly, a 25.4 mm o-ring section was removed from the reinforced filter matrix directly below the flange. All three layers of the as-manufactured 3M CVI-SiC composite matrix remained bonded together during preparation of either the 15 mm and 25.4 mm c-ring, or 25.4 mm o-ring samples.

C-ring and o-ring samples were removed from the steam/air and alkali/steam/air-exposed 3M CVI-SiC mini-candles. During sample preparation, the three composite layers of the steam/air-exposed 3M CVI-SiC matrix remained bonded together, while debonding occurred along both the 15 mm and 25.4 mm c-ring sections that were removed from the alkali/steam/air-exposed 3M CVI-SiC filter matrix.

What was also apparent for both the steam/air and alkali/steam/air-exposed 3M mini-candles was the extreme difficulty that was encountered during cutting of the reinforced matrix directly below the flange. As shown in Table 2.1, the applied compressive load required to fail the flange reinforced o-ring sections, and the resulting diamteral strength generally decreased after the 3M CVI-SiC composite matrix was exposed for 400 hours at temperatures of 870°C (~1600°F) to either the steam/air or alkali/steam/air flow-through environment.

As shown in Table 2.2, the applied compressive load required to fail c-rings that were removed from the 3M CVI-SiC composite filtration area, and the resulting strength also decreased after 400 hours of exposure in the 870°C (~1600°F) steam/air or alkali/steam/air flow-through environment. The resulting load vs deflection curves that were generated during c-ring compression testing tended to indicate that a reduction in the fracture toughness of the embrittled, 3M CVI-SiC composite matrix resulted after exposure at high temperature to the steam/air and alkali/steam/air test conditions.

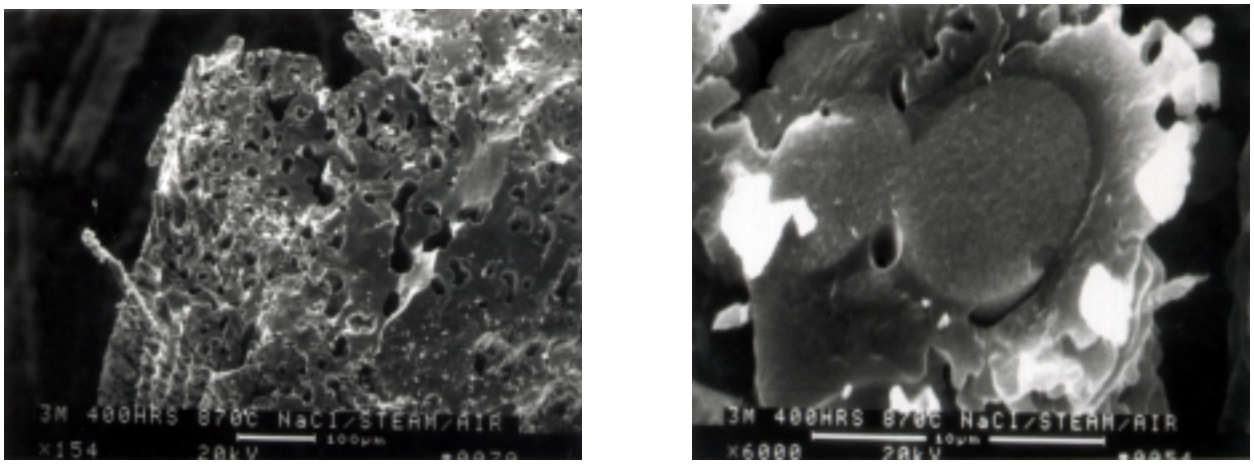
Scanning electron microscopy/energy dispersive x-ray analyses (SEM/EDAX) of the steam/air and alkali/steam/air-exposed 3M CVI-SiC filter matrices (Figure 2.7) indicated that the ~1-2 μm SiC layer that had been deposited within the outer confinement layer and triaxial braid support fiber bundles, and along the alumina-based filtration mat fibers, had generally been removed. The ~100 μm CVI-SiC deposited layer which coated the triaxial support braid was intact, but was enriched with oxygen. Due to the very thin nature of the interface layer that initially coated the Nextel™ 312 and alumina-based fibers, resolution and identification of its presence in either the as-manufactured or flow-through-tested mini-candles could not be made using SEM/EDAX analytical techniques. Alumina and silicon-rich, micron, nodular formations were evident along the Nextel™ 312 and alumina-based fibers in all three layers of the steam/air-exposed 3M CVI-SiC composite matrix.

TABLE 2.1
DIAMETRAL COMPRESSIVE STRENGTH OF THE 3M CVI-SiC COMPOSITE MINI-CANDLE REINFORCED FLANGE

Filter Identification Number	Exposure	Time, Hrs	Temperature, °C	Diametral Testing at 870°C (O-ring Compression)	
				Load, lbs	Strength, psi
3M-453707	As-Manufactured (25.4 mm)	—	—	30.5	2247
3M-453715	As-Manufactured (25.4 mm)	—	—	32.7	1907
3M-453710	Steam/Air (25.4 mm)	400	870	19.9	1975
3M-453708	Alkali/Steam/ Air (25.4 mm)	400	870	18.5	1375

TABLE 2.2
STRENGTH CHARACTERIZATION OF THE STEAM/AIR AND ALKALI/STEAM/AIR-EXPOSED 3M CVI-SiC COMPOSITE MINI-CANDLES

Filter Identification Number	Exposure	Time, Hrs	Temperature, °C	Compressive Load, lbs	C-Ring Compressive Strength at 870°C, psi	
					Composite Matrix	Triaxial Support Braid
3M-453707	As-Manufactured (15 mm)	—	—	3.3±0.36	1739±259	11784±1476
3M-453715	As-Manufactured (25.4 mm)	—	—	4.0±0.14	1183±78	8561±463
3M-453710	Steam/Air (15 mm)	400	870	2.10±0.62	987±250	8533±1546
3M-453708	Alkali/Steam/Air (15 mm)	400	870	1.7	717	6298
3M-453708	Alkali/Steam/Air (25.4 mm)	400	870	2.85±0.64	762±151	5808±695



(a) (b)

Figure 2.7 — Oxidation of the steam/air and alkali/steam/air-exposed 3M CVI-SiC filter matrix: (a) Melt formation of the Nextel™ 312 and CVI-SiC matrix after 400 hours of exposure at 870°C to 20 ppm NaCl/steam/air; (b) Silica-enriched melt formed between the alumina fibers and the residual CVI-SiC matrix.

2.1.2 DuPont PRD-66 Filament Wound Filter Matrix[7]

The oxide-based, filament wound, DuPont PRD-66 filter matrix consisted of a layered cordierite, mullite, cristobalite, and corundum microstructure. An amorphous phase was also present in the as-manufactured PRD-66 filter matrix. Due to the difference of thermal expansion of mullite, cordierite, and corundum which were present in the PRD-66 matrix, a microcracked structure was formed after high firing.

The diamond pattern weave of the polycrystalline refractory oxide-based fibers formed the ~7 mm structural support layer of the DuPont PRD-66 filter matrix. A thin membrane layer was wrapped along the outer surface of the support matrix, producing a light weight, bulk filter element.

After 400 hours of exposure in the 870°C (~1600°F) steam/air and alkali/steam/air environment, the DuPont PRD-66 mini-candles generally remained intact. However, along the outer membrane surface of the alkali/steam/air-exposed mini-candle, numerous longitudinal cracks were evident (Figure 2.8). In addition, an ~1 cm x ~1.5 cm section of the membrane and several of the underlying structural support fibers were removed along the alkali/steam/air-exposed mini-candle.

As shown in Table 2.3, the high temperature, c-ring compressive strength of the filtration area in the brittle PRD-66 filter matrix appeared to slightly increase after 400 hours of exposure to the steam/air flow-through environment, while a slight decrease in strength was observed after 400 hours of exposure in the alkali/steam/air environment. In contrast, virtually no change in strength was observed along the slurry infiltrated flange or end cap areas of the PRD-66 mini-candles after exposure to either the high temperature steam/air or alkali/steam/air environment (Table 2.4).

Figure 2.8 — Cracks and spalled areas of the o.d. surface membrane of the 400 hour, 870°C, 20 ppm NaCl/5-7% steam/air-exposed DuPont PRD-66 filter matrix.

TABLE 2.3
STRENGTH CHARACTERIZATION OF THE STEAM/AIR AND ALKALI/STEAM/AIR-EXPOSED DuPONT PRD-66 MINI-CANDLES

Filter Identification Number	Exposure	Time, Hrs	Temperature, °C	C-Ring Compression at 870°C	
				Load, lbs	Strength, psi
D-282	As-Manufactured (15 mm)	—	—	9.65±0.72	1352±135
D-289	Steam/Air (15 mm)	400	870	11.88±0.71	1541±146
D-288	Alkali/Steam/ Air (15 mm)	400	870	8.58±1.70	1093±265

TABLE 2.4
STRENGTH OF THE REINFORCED FLANGE AND END CAP SECTIONS OF THE DuPONT PRD-66 MINI-CANDLES AFTER FLOW-THROUGH TESTING

Filter Identification Number	Exposure	Time, Hrs	Temp., °C	Diametral O-Ring Compression Testing			
				Flange		End Cap	
				Load, lbs	Strength, psi	Load, lbs	Strength, psi
D-282	As-Manufactured (25.4 mm)	—	—	81.3	2341	56.3	1625
D-289	Steam/Air (25.4 mm)	400	870	74.0	1997	60.5	1665
D-288	Alkali/Steam/ Air (25.4 mm)	400	870	84.2	1819	50.5	1619

2.1.3 DuPont SiC-SiC Composite Filter Matrix[7]

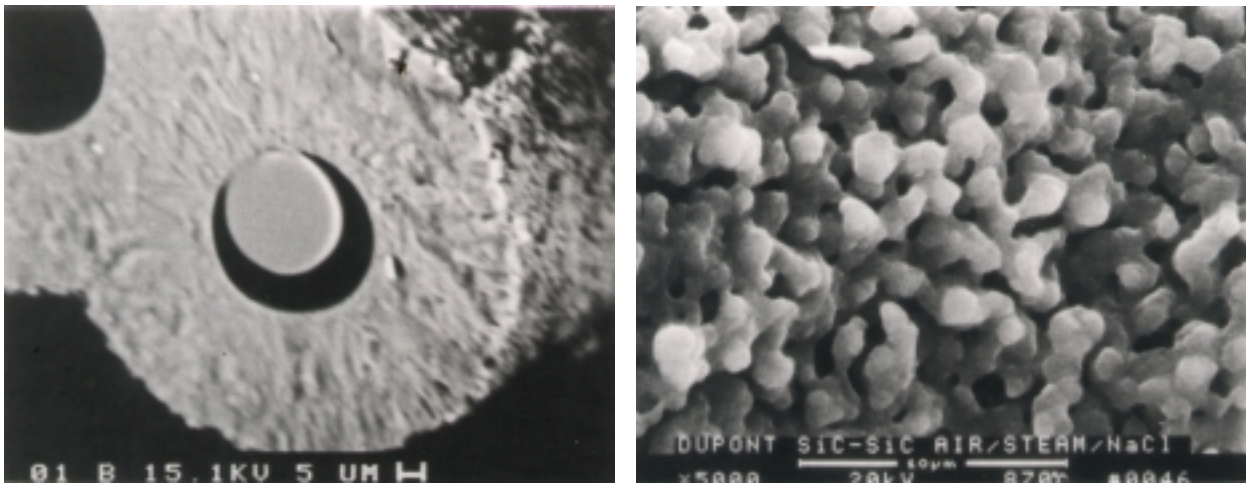
The DuPont SiC-SiC composite matrix consisted of an ~10-20 µm silicon carbide layer that was chemically vapor infiltrated (CVI) along an ~2-5 µm interface coating layer which encapsulated ~15 µm Nicalon™ fibers. Several issues were raised as to the oxidative stability of the dual single-ply or hybrid (i.e., single-ply felt; single-ply open mesh screen) DuPont SiC-SiC composite matrix, and in particular the stability of the interface coating and Nicalon™ fibers.

After 400 hours of exposure of the DuPont SiC-SiC matrix to either the 870°C (~1600°F), flow-through, steam/air or alkali/steam/air environment, depletion of the interface coating resulted (Figure 2.9). A mottled crystalline phase formed along the SiC-SiC surface which was identified by EDAX analysis to consist of silicon and oxygen (i.e., on a 1:1 atomic percent basis). The Si-O phase formed a noncontinuous layer along the surface of the SiC-SiC matrix. The presence of gas phase sodium appeared to enhance surface oxidation of the SiC-SiC matrix, leading to areas that were enriched with SiO_2. Sorption of sodium into the SiO_x or SiO_2 layer which covered the SiC-SiC matrix was not

detected by EDAX analysis after 400 hours of exposure of the DuPont SiC-SiC composite matrix to the 870°C (~1600°F), 20 ppm NaCl/steam/air, flow-through environment. Continued exposure may be needed to promote sorption of alkali, and possibly formation of the sodium silicate eutectic phase. In this manner, the relatively inert SiC-SiC matrix appeared to exhibit a slower oxidation or corrosion rate in comparison to the clay bonded silicon carbide filter materials.

In contrast, the fine grained SiC membrane coating of the DuPont matrix tended to form a glaze during the high temperature, alkali/steam/air exposure, which reduced gas flow permeability through the filter disc (Figure 2.10). The glazed surface that was enriched with sodium could serve as a potential site for collection and adherence of ash fines at process operating conditions, which may ultimately cause blinding of the filter element surface.

The residual strength of the hybrid DuPont SiC-SiC matrix (i.e., single-ply felt; single-ply open mesh screen) was determined at 870°C (~1600°F) via 4-point bend, ¼-flexural strength testing of bend bars that had been removed from the alkali-exposed filter disc. Approximately 60% of the as-manufactured strength remained along the surface of the matrix that had been subjected to pulse cleaning, while only 42% of the as-manufactured strength remained along the membrane coated surface that sorbed alkali during the 400 hour alkali/steam/air, flow-through test exposure (Table 2.5). Based on the load vs deflection curves that were generated during high temperature, flexural strength testing, the fracture toughness of the low fiber volume, DuPont SiC-SiC filter matrix appeared to decrease after 400 hours of exposure in either the 870°C (~1600°F) steam/air or alkali/steam/air environment.



(a) (b)

Figure 2.9 — DuPont SiC-SiC composite matrix after 400 hours of exposure at 870°C to the 20 ppm NaCl/5-7% steam/air flow-through test environment: (a) Depletion of the interface coating; (b) Oxidation of the CVI-SiC surface.

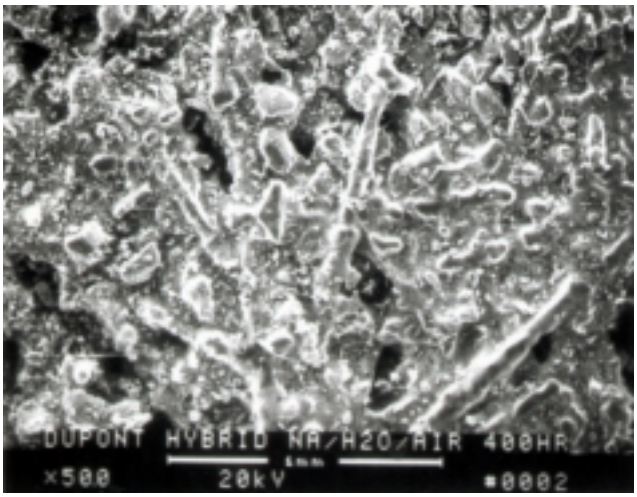


Figure 2.10 — Glazed sodium-enriched surface membrane of the DuPont SiC-SiC composite candle filter matrix after 400 hours of exposure to the 870°C, 20 ppm NaCl/5-7% steam/air flow-through test environment.

TABLE 2.5
4-POINT BEND, ¼-POINT FLEXURAL STRENGTH OF THE STEAM/AIR AND ALKALI/STEAM/AIR-EXPOSED DuPONT SiC-SiC FILTER MATRIX

Filter Matrix	Membrane Tested in Compression, psi[(a)]		Membrane Tested in Tension, psi[(b)]	
	25°C	870°C	25°C	870°C
As-Manufactured	13118±1697[(c)]	12068±3128[(c)]	7269±272[(c)]	4981±1801[(c)]
Steam/Air	NA	5427±749[(d)]	NA	4450±541[(d)]
Alkali/Steam/Air	NA	3953±90[(d)]	NA	3092±304[(d)]
Alkali/Steam/Air	NA	7269±557[(c)]	NA	2106±454[(c)]

Span: 1.57 in; Crosshead speed: 0.02 in/min.
(a) Pulse cycled surface strength; (Open mesh screen).
(b) Membrane coated surface strength; (Single-ply felt mat).
(c) Bend bars were cut parallel to the open mesh screen ribs.
(d) Bend bars were cut on a diagonal to the open mesh screen ribs.
NA: Not applicable.

2.1.4 IF&P Fibrosic™ Filter Matrix[(7)]

The vacuum infiltrated alumina and aluminosilicate chopped fiber IF&P Fibrosic™ matrix was similarly subjected to 400 hours of flow-through testing at 870°C (~1600°F) in both the steam/air and alkali/steam/air environment. After 400 hours of exposure in the 870°C (~1600°F) steam/air

environment, the membrane coated IF&P Fibrosic™ disc was torn as a result of repeated pulse cleaning cycles (Figure 2.11). In contrast, the IF&P Fibrosic™ disc that had been exposed for 400 hours at 870°C (~1600°F) to the alkali/steam/air environment remained intact, and appeared to have rigidized.

In order to cut bend bars for four-point bend, ¼-point flexural testing of the IF&P Fibrosic™ filter matrix, the as-manufactured filter disc was positioned along a metal support block with double-backed tape, and dry cut with a diamond wheel. Unfortunately during removal of the bend bars, tearing resulted, and strength characterization of the as-manufactured and alkali/steam/air-exposed discs was suspended.

Microstructural analyses were performed on the 400 hour, 870°C (~1600°F) alkali/steam/air-exposed IF&P Fibrosic™ filter matrix. EDAX analysis identified sorption of alkali into the aluminosilicate-enriched fibers vs sorption into the alumina-enriched bond phase. Generally a greater concentration of sodium was sorbed along the membrane coated surface of the IF&P Fibrosic™ filter disc in comparison to throughout the remainder of the filter wall.

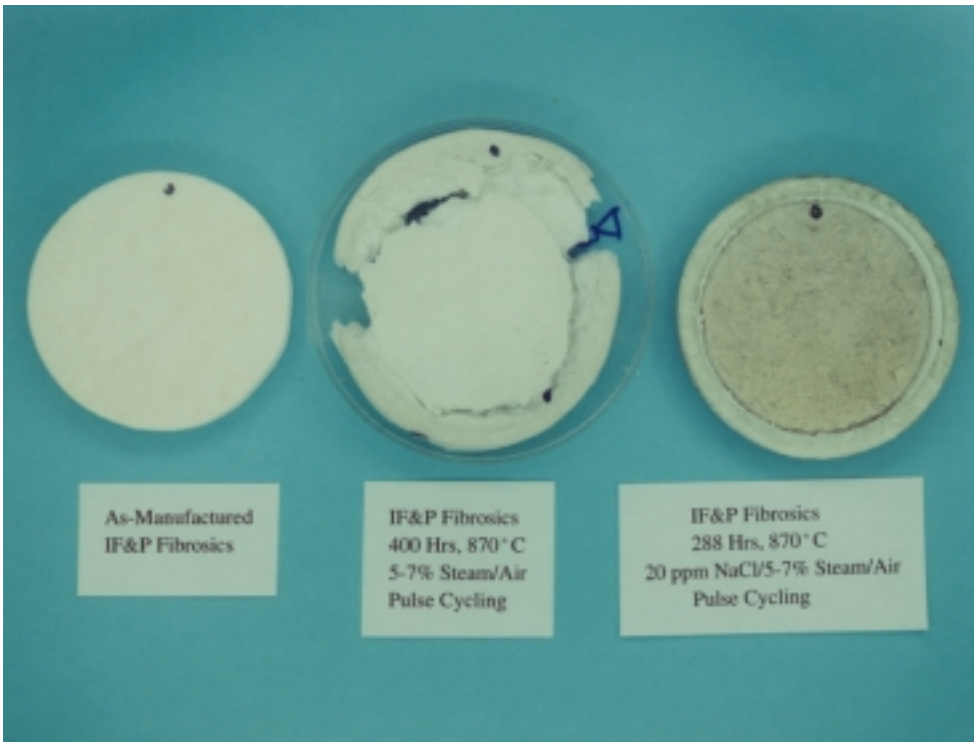


Figure 2.11 — IF&P Fibrosic™ filter discs after 400 hours of exposure at 870°C to the steam/air and alkali/steam/air flow-through test environment.

2.2 Mechanical Properties of the As-Manufactured Second Generation Filter Elements

In conjunction with first generation monolithic filter elements, the room temperature, gas flow resistance of the as-manufactured, 1.5 m, 3M CVI-SiC composite, DuPont PRD-66, and IF&P Fibrosic™ candle filters, and 1.4 m DuPont SiC-SiC candle filter is shown in Figure 2.12. These data indicate the relatively high initial permeability of the porous, light weight, second generation filter elements.

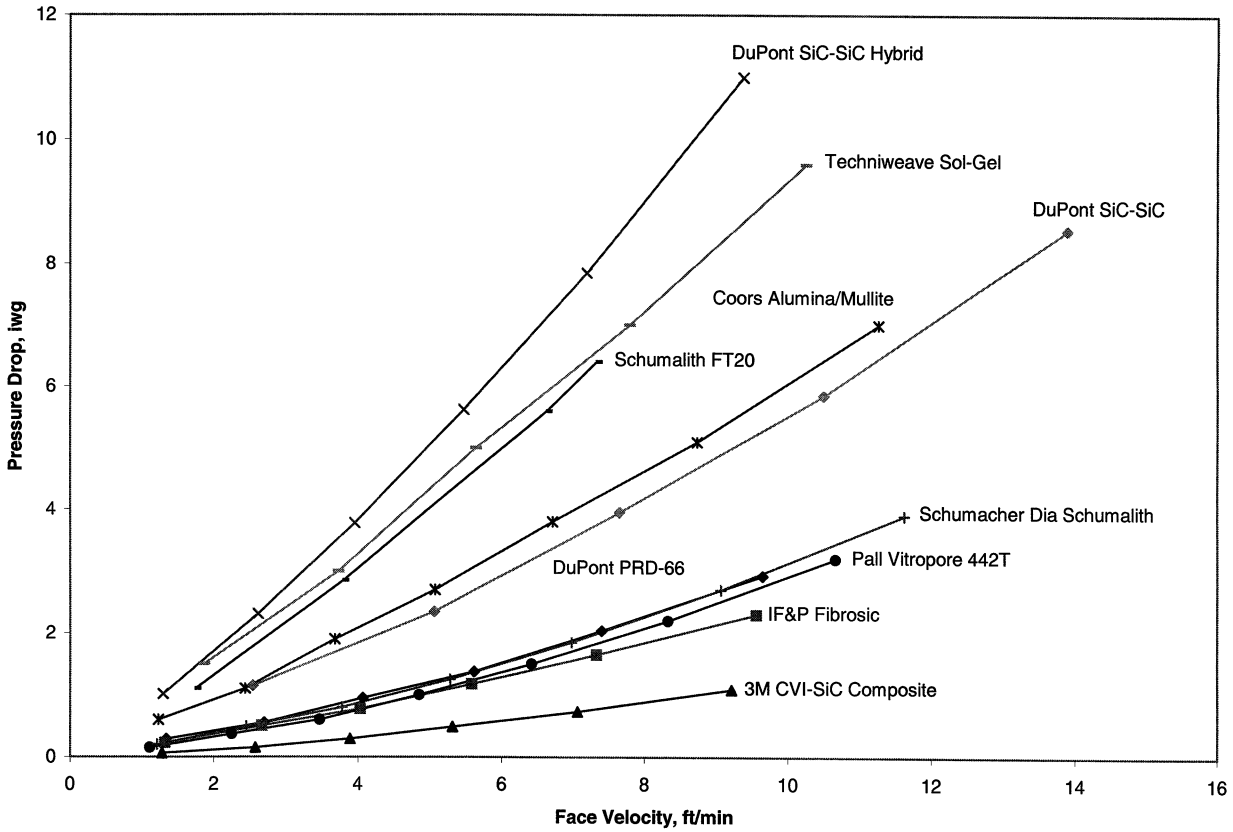


Figure 2.12— Room temperature gas flow resistance of the first generation monolithic and advanced second generation candle filters.

All advanced second generation candle filters were characterized in terms of their as-manufactured compressive and tensile strengths via c-ring and/or o-ring testing at room temperature and at various elevated temperatures that reflected specific process operating conditions. For example, 732°C (1350°F) represented the nominal Siemens Westinghouse advanced particulate filtration (APF) system operating temperature at the American Electric Power (AEP) Tidd Demonstration Plant during testing in 1994 (Test Segment 4). Temperatures of 843C (1550°F) reflected the operating temperatures for the Siemens Westinghouse pressurized fluidized-bed combustion (PFBC) test facility during accelerated pulse cycling and/or process transient testing. Similarly, 870°C (1600°F) represented temperatures utilized by Siemens Westinghouse to conduct bench-scale, flow-through, alkali corrosion testing. Typically 15 mm or 25.4 mm cylindrical sections were cut from each candle filter using a diamond wheel. The orientation of the c-ring sample was rotated around the filter body in order to eliminate any uni-axial processing or manufacturing effects.

As shown in Table 2.6, the as-manufactured strengths of the advanced second generation candle filters varied from being relatively low for the IF&P Fibrosic™ matrix, to moderately strong for the filament wound DuPont PRD-66 filter matrix, to extremely strong for the 3M CVI-SiC triaxial braid and DuPont SiC-SiC composite matrices. Both the applied load-to-failure and resulting calculated strength varied slightly depending on whether the sample length was 15 mm or 25.4 mm, and whether c-ring or o-ring testing was conducted. By adopting a standard sample length (i.e., 15 mm or 25.4 mm) and test methodology, the as-manufactured and field-tested filter strength data could be compared. Siemens Westinghouse elected to continue testing 15 mm samples, utilizing primarily c-ring compressive and tensile testing to monitor changes along the gas contacted outer surface and pulse cycled inner surface of the filter element, respectively, both at room temperature, as well as at process operating temperatures.

TABLE 2.6
LOAD AND ULTIMATE STRENGTH OF THE AS-MANUFACTURED SECOND GENERATION CANDLE FILTERS

Sample Identification Number	Room Temperature		843°C		870°C	
	Load, lbs	Strength, psi	Load, lbs	Strength, psi	Load, lbs	Strength, psi
3M CVI-SiC Composite						
43-1-2 C-ring Compression (15 mm)	2.5	1343 (13187)[a]	2.6[b]	1352 (13444)[b]	ND	ND
43-1-2 C-ring Compression (25.4 mm)	5.4	1635 (17012)	5.1[b]	1425 (16857)[b]	ND	ND
43-1-2 O-ring Compression (15 mm)	7.7±0.84	1315±186 (13276±1417)	5.98±1.03[b]	1104±350[b] (11250±3595)[b]	ND	ND
43-1-2 O-ring Compression (25.4 mm)	13.61±1.90	1314±254 (14026±2012)	10.8±1.7[b]	1060±219[b] 11012±1795[b]	ND	ND
DuPont PRD-66						
D-259 C-Ring Compression (15 mm)	10.84±0.41	1325.01±71.11	12.69±1.72	1543.35±196.99	13.29±0.95	1629.41±142.14
D-259 C-Ring Tension (15 mm)	7.54±1.77	1195.95±280.56	7.53±0.58	1213.25±93.22	8.73±1.85	1393.56±280.96
D-249 O-Ring Compression (15 mm)	27.93±1.93	1064.89±84.21	30.21±4.02	1160.89±197.40	30.08±3.19	1144.78±139.90
D-249 O-Ring Compression (20.4 mm)	37.21±3.00	1040.44±106.19	37.48±4.75	1033.56±129.84	41.60±6.52	1148.56±173.54
DuPont SiC-SiC						
D/O C-Ring Compression (15 mm)	4.12±0.38	11905±1556	5.90±0.80	18518±2378	5.53±1.40	16585±5220
D/O C-Ring Tension (15 mm)	6.48±0.84	20210±4139	5.80±0.86	18811±3617	6.48±1.67	20242±5560
D/O O-Ring Compression (15 mm)	18.57±2.18	16303±1249	19.73±2.57	18810±1374	21.37±1.12	18902±652
D/O O-Ring Compression (25.4 mm)	32.63±8.24	18064±2156	26.53±5.78	14887±271	33.70±6.61	16226±252
IF&P Fibrosic™						
RX9502-1709 C-Ring Compression (15 mm)	0.64±0.12	28.13±6.70	2.11±0.41	98.86±18.69	2.14±0.59	95.70±17.58
RX9502-1709 C-Ring Tension (15 mm)	0.71±0.15	50.90±11.96	1.03±0.16	70.70±11.71	1.04±0.36	74.45±25.59
RX9502-1708 O-Ring Compression (15 mm)	2.62±0.23	53.22±5.45	3.24±0.55	66.56±9.90	3.62±0.49	71.67±12.54
RX9502-1708 O-Ring Compression (20.4 mm)	4.03±0.24	59.33±7.11	4.74±0.90	67.56±15.70	5.36±0.42	78.67±9.64

(a) Three layer composite strength; (Triaxial braid support).
(b) Strength measured at 732°C
ND: Not determined.
Crosshead speed of 0.05 in/min was consistently used during conduct of c-ring compressive and tensile strength testing.

This test methodology was shown to be adequate for maintaining the integrity of the ceramic matrices for all four advanced, second generation filter materials. Limited additional testing was, however, conducted at Siemens Westinghouse utilizing either 15 mm or 25.4 mm o-rings.

Due to the relative thin wall construction of the 3M CVI-SiC and DuPont SiC-SiC composite matrices (i.e., ~3.6 mm 3M composite thickness; ~1.3 mm 3M triaxial support braid thickness; and ~1.6 mm DuPont composite thickness), the resulting strength of these materials was calculated to be extremely high in comparison to the monolithic or first generation, and alternate advanced or second generation filter elements. In contrast, however, the applied load required to fail the 3M CVI-SiC and DuPont SiC-SiC composite materials was substantially lower than the applied load required to fail the first generation monolithic materials, but was higher than the applied load needed to fail the IF&P Fibrosic™ matrix (Table 2.7). Failure of either the first generation monolithic or advanced second generation candles would however, be expected to result if ash bridging were to occur within a candle cluster or array during process operation.

TABLE 2.7
FIRST GENERATION MONOLITHIC AND ADVANCED SECOND GENERATION CANDLE FILTER MATERIAL STRENGTH

Filter Matrix (15 mm)	25°C C-Ring Strength, psi		25°C C-Ring Ultimate Load, lbs	
Coors Alumina/Mullite	2575±182	2721±415	42.8±2.4	31.4±4.9
Pall Vitropore 442T	2857±186	2574±177	60.5±5.9	35.3±1.8
Schumacher Dia Schumalith F40	1300±213[(a)] 1790±112[(b)]	1907±111 2308±275	74.5±14.2 102.1±9.4	53.4±3.7 63.8±8.2
Schumacher Dia Schumalith FT20	2296±261	2268±167	44.2±4.8	29.2±2.9
3M CVI-SiC Composite	1343[(c)] 13187[(d)] 1315±186[(c)*] 13276±1417[(d)*]		2.5 2.5 7.7±0.84* 7.7±0.84*	
DuPont SiC-SiC	11905±1566 16303±1249*	20210±4139	4.12±0.38 18.57±2.18*	6.48±0.84
DuPont PRD-66	1219±162	1265±188	9.2±1.0	7.3±1.3
IF&P Fibrosic™	28.1±6.7 53.2±5.5*	50.9±12.0	0.64±0.12 2.6±0.2*	0.71±0.15

(a) 1991 production lot.
(b) 1992 production lot.
(c) Composite strength (Containment layer, filtration mat, and triaxial support braid).
(d) Triaxial support braid.
* 15 mm o-ring diametral compressive strength or load.

3. ASSESSMENT OF PROTOTYPE ADVANCED SECOND GENERATION CANDLE FILTER HIGH TEMPERATURE PERFORMANCE

3.1 Bench-Scale Testing[7]

Prior to selection, installation, and operation in the field, candle filter qualification testing was conducted at Siemens Westinghouse in 1995 to assure that prototype or developmental filters demonstrated >99.99% particle collection efficiency during steady-state, simulated pressurized fluidized-bed combustion (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 were assessed. In addition, Siemens Westinghouse qualified the performance of the various filters under accelerated pulse cycling conditions to monitor the performance of the matrix with respect to thermal fatigue, as well as exposure to thermal transient events which simulated rapid startup or shutdown cycles experienced during process operation.

In order to evaluate the performance of the advanced second generation filter elements, an array which included a 1.5 m DuPont PRD-66 filter, a 1.5 m 3M CVI-SiC composite filter, a 1.4 m DuPont SiC-SiC composite filter, and a 1.5 m IF&P Fibrosic™ candle filter was installed in the Siemens Westinghouse PFBC simulator test facility in Pittsburgh, Pennsylvania (Figure 3.1). Initially, the filter array was heated to temperatures of 843°C (1550°F) 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 6-100°C and 20-240°C within the first 5 and 60 sec, respectively, after transient initiation. After each transient, a rapid reheat was conducted in order to return to processing operating temperatures of 843°C (1550°F) within one hour after transient initiation. Nine maximum thermal transients followed. Subsequently the array was subjected to ten accelerated pulse cleaning cycles and a final maximum severity thermal transient.

Figure 3.1 — Siemens Westinghouse PFBC simulator test facility.

After 42 hours of thermal transient testing, the filter array was slow cooled and the elements were removed for destructive characterization. With the exception of the DuPont SiC-SiC candle, all filter elements remained intact. The longitudinal seam partially opened (i.e., <1 mm gap; ~40 cm rupture length; Figure 3.2), permitting fines to penetrate through, and to be released into the clean gas stream. During handling, the candle fractured into two segments.

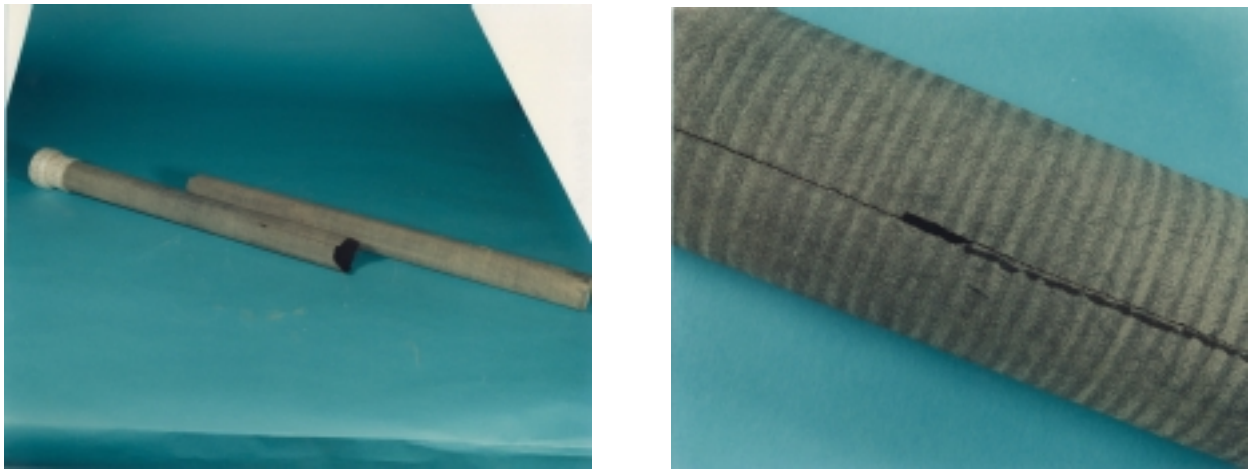


Figure 3.2 — Ruptured seam of the DuPont SiC-SiC composite candle filter after process transient testing.

When the DuPont SiC-SiC composite matrix was subjected to scanning electron microscopy/ energy dispersive x-ray analysis (SEM/EDAX), several changes were observed to have resulted within the thermal transient-tested material. 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.
- Bonding or 'sintering' of adjacent Nicalon™ fibers with the enhanced oxidation phase that was added during the manufacture of the mesh screen support layer.

Based on the resulting load vs deflection curves that were generated during c-ring strength testing, the fracture toughness of the DuPont SiC-SiC composite appeared to have been reduced after

42 hours of thermal transient testing. 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 bonding or sintering of the Nicalon™ fibers in the mesh screen support layer.

In order to identify the viability and thermal fatigue resistance of the advanced second generation filters, a second set of filter elements which included a DuPont PRD-66 filter, a 3M CVI-SiC composite filter, a DuPont SiC-SiC composite filter, and an IF&P Fibrosic™ candle was installed in the Siemens Westinghouse PFBC simulator test facility. Simulated PFBC testing was conducted at temperatures of 843°C (1550°F) and pressures of 100 psig, with a pulse cleaning cycle delivered every 2.5 minutes. After 1645 pulse cleaning cycles, dust was detected in the clean gas stream, indicating that a breach had occurred within the filter system. Upon disassembly, the IF&P Fibrosic™ candle was observed to have fractured along the mid-body (Figure 3.3). A large hole (~75 mm long; ~40 mm wide) was evident below the flange, and a smaller through-wall hole (i.e., ~10 mm) was evident along the filter body. The longitudinal fractured surface of the Fibrosic™ matrix followed matrix texture lines along the outer surface of the filter element. Striated layers of chopped fiber matrix were evident throughout the fractured IF&P Fibrosic™ filter wall.

Figure 3.3 — Fractured IF&P Fibrosic™ candle filter after exposure to 1645 accelerated pulse cycles in the simulated PFBC operating environment.

Gas flow stream lines were evident along the i.d. wall of the fractured IF&P Fibrosic™ candle. These lines which were ~180° from the large hole that resulted below the flange, were considered to have resulted from repeated pulse cycling. Further inspection of the i.d. surface near the flange indicated that the surface of the infiltrate that was used to provide additional strength along the flange had partially separated from the support matrix.

After removal of the failed IF&P Fibrosic™ candle from the filter array, testing was reinitiated at 843°C (1550°F), and continued until 3514 pulse cleaning cycles (i.e., 1757 hours of equivalent operating life) had been delivered to the filter array. Subsequently ash was fed for an additional eight hours in order to develop a dust cake layer along the outer surface of each of the remaining filter elements. Post-test inspection indicated that with the exception of the 3M CVI-SiC composite filter, all of the remaining filter elements were intact. Failure at the base of the flange of the 3M CVI-SiC composite matrix resulted from extended pulse cycling which partially dislodged the primary gasket from its original position in the filter holder mount.

3.1.1 Microstructural Characterization of the DuPont SiC-SiC Candle Filter Matrix

SEM/EDAX analysis of the bench-scale-tested, DuPont SiC-SiC filter matrix indicated that the interface layer that initially surrounded the Nicalon™ fibers in the single-ply felt layer was removed after 197 hours of accelerated pulse cycle testing (Figure 3.4). Similarly, crack formations resulted along the outer periphery of the Nicalon™ fibers. 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 which 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 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™ fiber with the enhanced 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 encapsulating layer was again evident after 197 hours of accelerated pulse cycling in the Siemens 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 bonding or sintering of the Nicalon™ fibers in the mesh screen support layer.

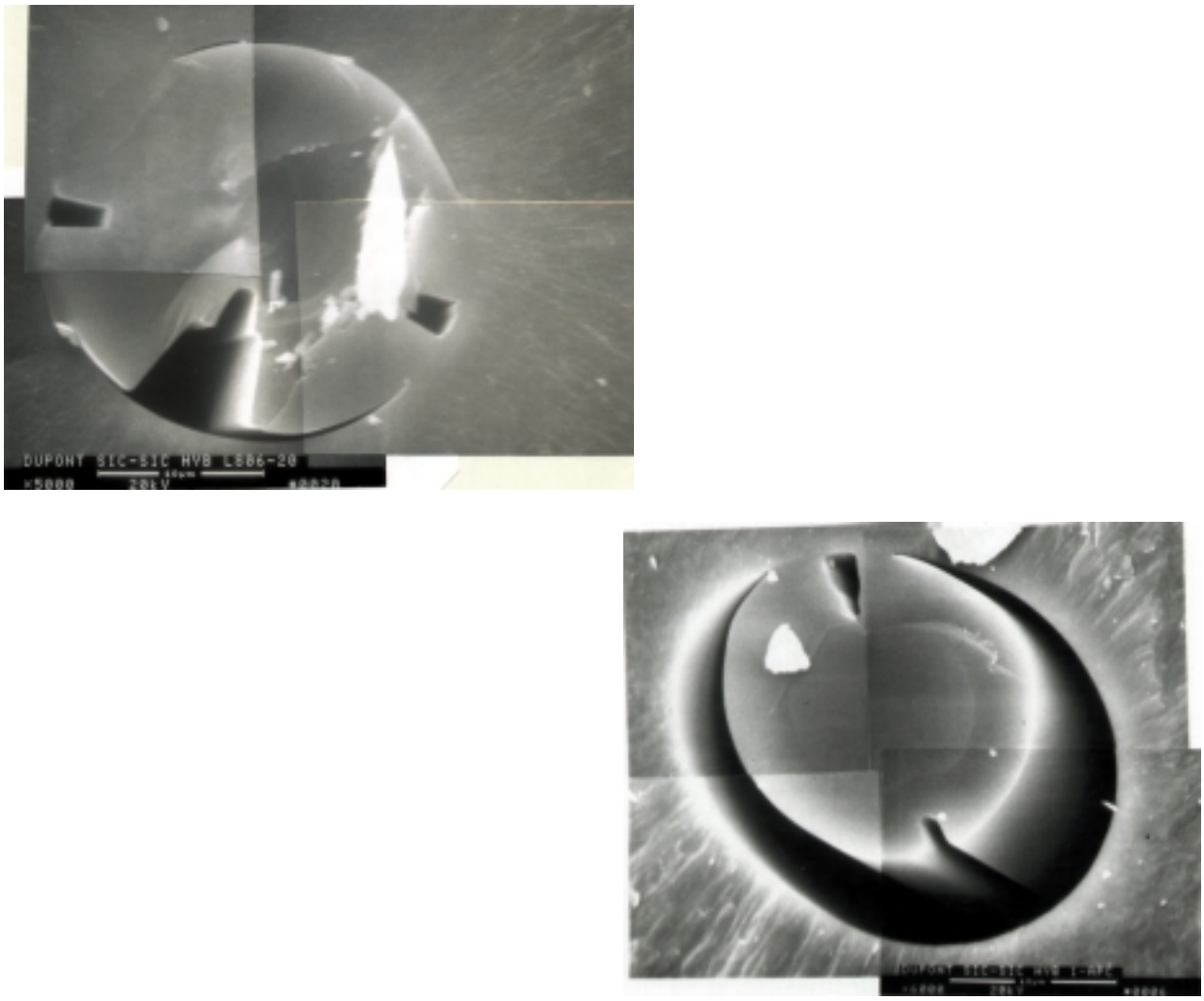


Figure 3.4 — Removal of the interface layer and bonding of the Nicalon™ fibers to the silicon carbide structural matrix in the DuPont SiC-SiC filter material after exposure to simulated, accelerated life, PFBC testing.

3.2 Demonstration Plant Testing[8, 9]

With PFBC nearing a commercialization status through several operating and planned demonstration plants throughout the world, Siemens Westinghouse had the opportunity to demonstrate the feasibility of utilizing advanced hot gas filtration systems to reduce coal-fired particulate emissions, enabling the application of gas turbines into solid fuel cycles. Between 1992 and 1995 this opportunity was realized at the American Electric Power (AEP) Tidd Demonstration Plant in Brilliant, Ohio, when Siemens Westinghouse's 10-MW_e slipstream hot gas cleaning system was incorporated into the plant configuration. Initially AEP utilized seven parallel strings of primary and secondary cyclones to remove approximately 98% of the particulate fines released into the turbine expansion gas, followed by an electrostatic precipitator (ESP) to clean the particulates from the exhaust gas to meet environmental

regulations. The Siemens Westinghouse Advanced Particulate Filtration (APF) system replaced a primary and secondary cyclone string, and ultimately produced an expansion gas that was acceptable for use with high performance industrial gas turbines, eliminating the need for further conventional ESP or baghouse cleaning.

In addition to demonstrating feasibility of the hot gas filtration technology at AEP, Siemens Westinghouse conducted an in-depth filter material surveillance program in conjunction with the Department of Energy (DOE) at Morgantown, West Virginia, identifying the long-term stability and/or degradation of various first and second generation porous ceramic filter elements that were utilized throughout five test campaigns. Although porous ceramic hot gas filters generally experienced a loss of material strength and underwent numerous phase and microstructural changes during PFBC operation, the elements achieved a stabilized conditioned strength and remained intact during normal plant operation.

3.2.1 Siemens Westinghouse Advanced Particulate Filtration System

The Siemens Westinghouse APF utilized at AEP consisted of a pressure vessel which contained a maximum of 384, 1.5 m porous ceramic candle filters (Figure 3.5). At AEP three identical clusters were supported from a high alloy uncooled tubesheet and an inverted thermal expansion cone. Each cluster contained three plenums of candles arranged vertically, with 38 candles in the top and middle plenums, and 52 candles in the bottom plenum. The individual candles were supported from the tubesheet in each plenum using bolted metal collars and high temperature ceramic gaskets.

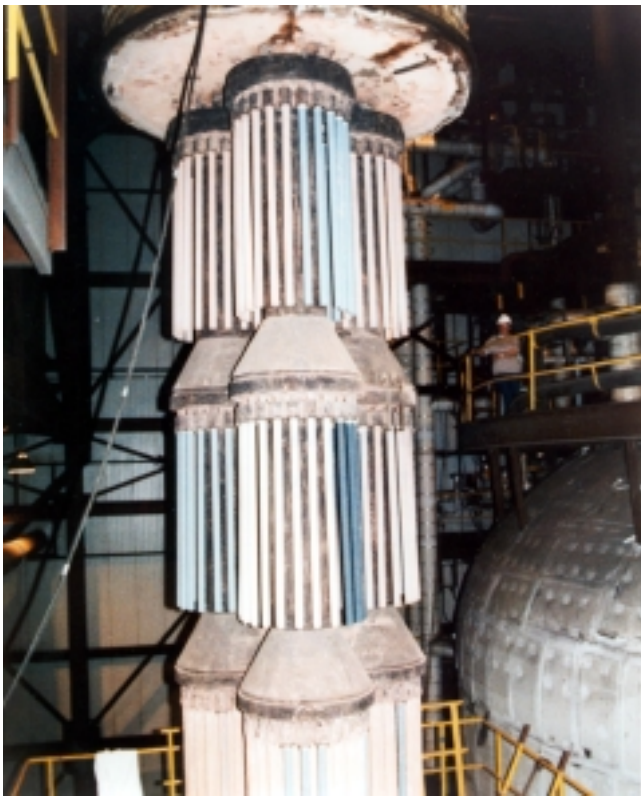


Figure 3.5 — Siemens Westinghouse Advanced Particulate Filtration system.

Five test campaigns were conducted at AEP in which the porous ceramic filters were exposed to process gas temperatures ranging between 620 and 845°C (~1150 and 1555°F; Table 3.1). During the first three test campaigns, 384 Schumacher Dia Schumalith F40 clay bonded silicon carbide candle were installed within the filter vessel. Ash bridging resulted within the various arrays leading to deformation (i.e., bowing and/or tilting) of the candles. In addition, localized failure of the filter elements resulted primarily at the base of the filter flange during cool-down of the bridged filter system. In order to mitigate ash bridging, the primary cyclone was detuned during Test Segment 3, permitting ash with a larger mass mean particle size to enter the filter vessel.

TABLE 3.1
SUMMARY OF SIEMENS WESTINGHOUSE APF TESTING UNDER PFBC CONDITIONS AT AEP

Test Segment	1 10/92-12/92	2 7/93-9/93	3 1/94-4/94	4 7/94-10/94	5 1/95-3/95
Coal	Pittsburgh No. 8	Pittsburgh No. 8	Pittsburgh No. 8	Pittsburgh No. 8	Pittsburgh No. 8; Minnehaha; Consol
Sorbent	Plum Run Greenfield Dolomite	Plum Run Greenfield Dolomite; Delaware Limestone	Plum Run Greenfield Dolomite	Plum Run Greenfield Dolomite; National Lime Limestone	Plum Run Greenfield Dolomite; Mulzer Dolomite Delaware Limestone
Number of Candles	384	384	384	288	288
Schumacher F40	384	384	384	258	5
Schumacher FT20	—	—	—	8	—
Pall Vitropore 442T	—	—	—	8	153
Coors Alumina/Mullite	—	—	—	8	98
3M CVI-SiC Composite	—	—	—	3	10
DuPont PRD-66	—	—	—	3	22
Primary Cyclone	In-Service	In-Service	Detuned	Detuned	Inactive
Operating Hours (Coal)	464.4	1295	1278.8	1705.8	1110.4
Operating Temperature, °C (°F)	730-790 (1345-1455)	620-790 (1150-1455)	650-780 (1200-1435)	660-760 (1220-1400)	760-845 (1400-1555)
Nominal Face Velocity, cm/s	2.4-3.3	2.3-3.3	2.3-3.3	3.0-4.4	4.5
Inlet Dust Loading, ppmw	600	600	3,200	3,200	18,000
Ash Mass Mean, µm	1-3	1-3	5-7	5-7	27
Ash Formation	Bridging	Bridging	Bridging	Bridging	Absence of Ash Bridging

Operation of the system continued with the primary cyclone detuned in Test Segment 4. In addition, the face velocity of the gas was increased by reducing the total number of filter elements installed in the vessel. Although the Schumacher Dia Schumalith F40 candle filters were principally used to fill the various arrays, alternate first generation and advanced fiber reinforced elements were installed within the Siemens Westinghouse APF. These included high temperature creep resistant Schumacher Dia Schumalith FT20 filter elements, Pall Vitropore 442T clay bonded silicon carbide candles, Coors P-100A-1 alumina/mullite candle filters, 3M CVI-SiC composite candles, and DuPont filament wound PRD-66 filter elements. Although the primary cyclone was detuned, the mass mean particle size was not significantly altered, and bridging continued to occur with the APF vessel.

During Test Segment 5, the primary cyclone was completely inactivated, and a full cluster of Pall Vitropore 442T, and a nearly full cluster of Coors P-100A-1 alumina/mullite elements, and an array of DuPont PRD-66 elements were installed within the Siemens Westinghouse APF. Five 3M CVI-SiC composite elements were installed in both a top and bottom array. After 1110 hours of operation, ash bridging between the candles and/or internal metal support structures had been mitigated.

3.2.2 Coupon Testing

Sections of the advanced second generation 3M CVI-SiC, DuPont PRD-66, DuPont SiC-SiC (double-ply felt), and IF&P Fibrosic™ candle filter materials were placed on a coupon tree that was positioned above the Siemens Westinghouse APF tubesheet at AEP. These materials were exposed to PFBC flow-over conditions during Test Segments 3, 4, and 5. Post-test characterization of each material was conducted in order to determine whether microstructural changes had occurred after extended exposure to the PFBC environment.

During manufacturing, an ~1-2 µm layer of silicon carbide was deposited along the outer confinement and filtration mat layers of the 3M CVI-SiC composite filter matrix. In contrast, an ~100 µm layer of silicon carbide was deposited along the outer surface of the triaxial braid which formed the support structure of the filter element. After 2815 hours of exposure in the PFBC environment, oxidation resulted along the external and internal surfaces of the CVI-SiC layers. As a result of oxidation along the external surface of the CVI-SiC layers, spalling of the SiO_2-enriched layer was identified as a potential degradation mechanism of the 3M composite filter matrix, particularly during exposure to thermal transient operating conditions. As a result of the internal surface oxidation, the CVI-SiC encapsulating layers appeared to be bonded to the underlying Nextel™ 312 fibers (Figure 3.6). This lead to embrittlement and a reduction in the fracture toughness of the outer confinement and filtration mat layers of the PFBC-exposed 3M CVI-SiC composite filter matrix.

The 2 µm CVI-SiC layer that infiltrated into the triaxial braid, coating individual fibers during the manufacturing process, similarly formed an ~1 µm SiO_2 enriched layer that frequently contained cracks (Figure 3.6). After 2815 hours of exposure to PFBC conditions, ~21% of the initial strength of the triaxial support braid remained (Table 3.2). 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.

A sample of the DuPont SiC-SiC composite matrix was also installed above the Siemens Westinghouse APF tubesheet at AEP. The DuPont SiC-SiC composite consisted of a double-ply felt wrap which contained ~15 µm diameter Nicalon™ fibers. The fibers were initially coated with an ~2-5 µm interface layer, and subsequently with an ~10-20 µm CVI-SiC layer. Fine grain silicon carbide grit was applied with a polymeric resin slurry to form an outer membrane.

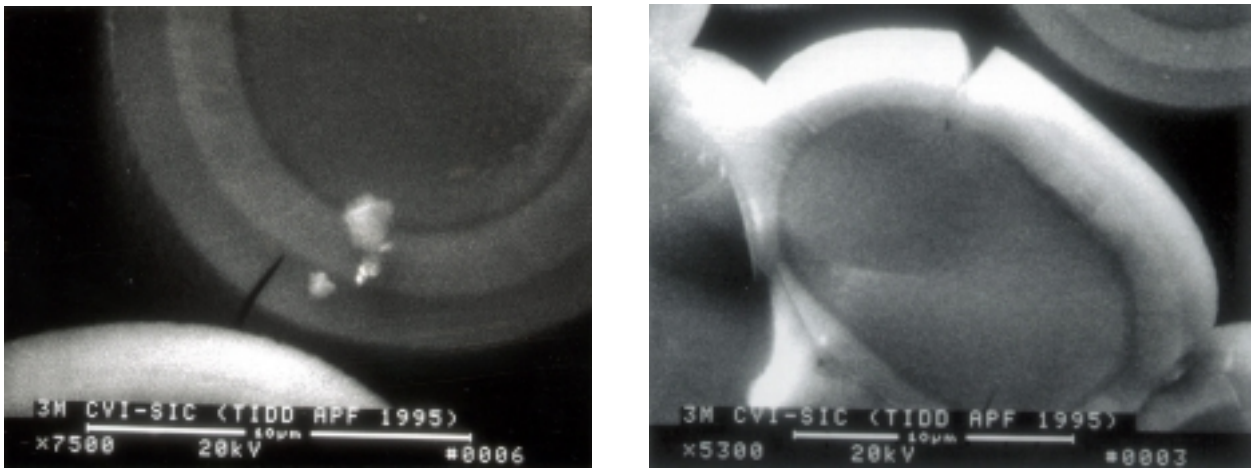


Figure 3.6 — Crack formation along the silica-enriched CVI-SiC infiltrated layers that surrounded the Nextel™ 312 fibers in the triaxial support braid of the 3M composite filter matrix after 2815 hours of exposure above the Siemens Westinghouse APF system tubesheet at AEP.

TABLE 3.2
SUMMARY OF THE C-RING COMPRESSIVE STRENGTH AND MICROSTRUCTURAL CHANGES RESULTING IN THE SECOND GENERATION FILTER MATERIALS AFTER EXPOSURE ABOVE THE SIEMENS WESTINGHOUSE APF TUBESHEET AT AEP

Filter Matrix	Exposure Time, Hrs	Initial Strength, psi (843°C)	PFBC-Exposed Strength, psi (843°C)	SEM/EDAX Characterization
3M CVI-SiC	2815	10652 ± 2184 [a, b] Composite Fracture	2187[b] (0.4 lbs) [c] Composite Fracture	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 the 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) Two production lots were utilized to manufacture the initial and PFBC-exposed materials.
(b) Triaxial braid wall thickness was used to calculate resulting bulk strength.
(c) Ultimate load to failure.
(d) ND: Not determined.

Several changes were evident within the microstructure of the DuPont SiC-SiC filter matrix after 4094 hours of exposure to PFBC flow-over conditions. These included:

- Removal of the interface layer which lead to the formation of voids between the silicon carbide encapsulating layer and the underlying Nicalon™ fibers.
- Surface oxidation and the formation of longitudinal cracks along the Nicalon™ fibers (Figure 3.7).
- Oxidation along the inner surface of the CVI-SiC encapsulating layer. The underlying fibers ultimately bonded to the encapsulating layers through contact with an oxygen-enriched (i.e., silica (SiO_2) or silica precursor (SiO_x)) 'melt-like' phase. As a result, the strength of the DuPont SiC-SiC matrix increased, while the fracture toughness of the material decreased.

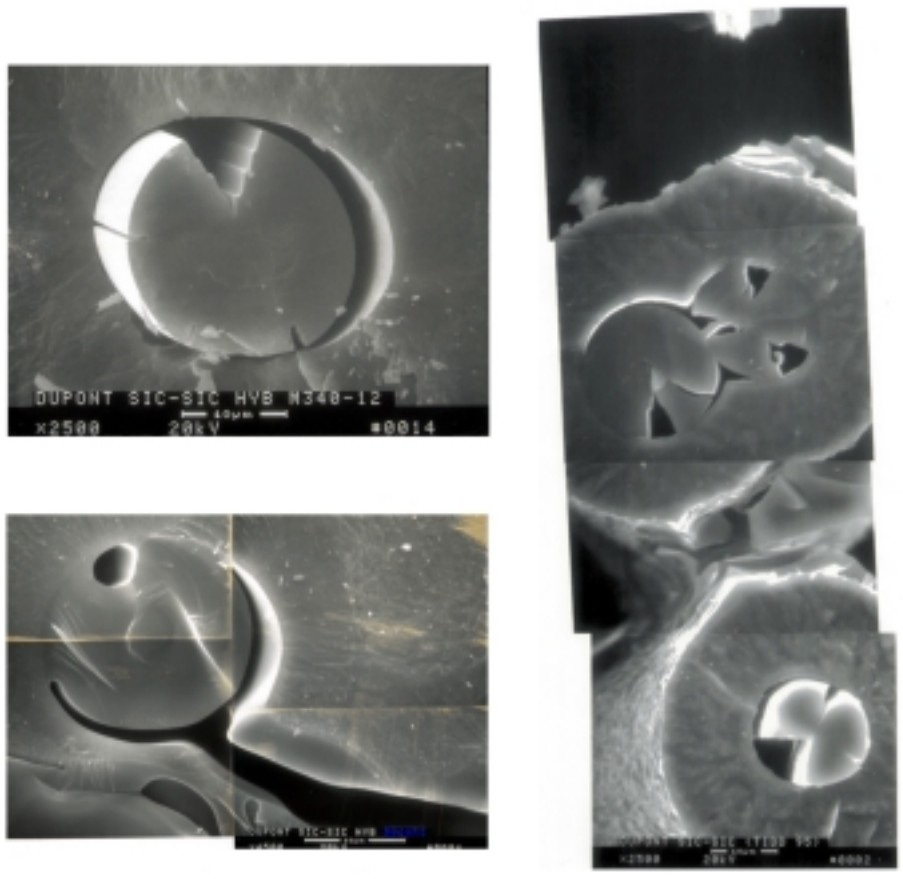


Figure 3.7 — Crack formations and bonding of the Nicalon™ fibers to the oxygen-enriched inner wall of the CVI-SiC encapsulating layers in the DuPont SiC-SiC double-ply felt composite filter matrix after 4094 hours of exposure above the Siemens Westinghouse APF system tubesheet at AEP.

In contrast to either the 3M CVI-SiC or DuPont SiC-SiC composite matrices, the oxide-based, filament wound, DuPont PRD-66 filter matrix consisted of cordierite fiber replicas in a polycrystalline slurry infiltrated matrix. Crystallization of the cordierite-enriched fiber replicas was evident after 4094 hours of exposure to flow-over conditions. A loss of 33% of the as-manufactured filter strength was considered to have resulted from crystallization and grain growth within the DuPont PRD-66 filter matrix.

Alternately the vacuum infiltrated IF&P Fibrosic™ matrix that was included in the sample array consisted of alumina and aluminosilicate chopped fibers, and an aluminosilicate binder phase. Rigidizers were added to the flange and end cap areas of the filter element, while colloidal mullite precursors were used to form the candle filter body. After 2815 hours of exposure in the flow-over PFBC environment, virtually no change in the microstructure of the IF&P Fibrosic™ matrix was evident.

3.2.3 3M CVI-SiC Composite Filter Testing

Three 3M CVI-SiC composite filters were initially installed in the Siemens Westinghouse APF in Test Segment 4, and after 1015 hours of operation, two elements failed at the base of their flange due to ash bridging. The third 3M filter element, which remained intact, achieved 1705 hours of PFBC test operation prior to termination of Test Segment 4.

Post-test room temperature, gas flow resistance measurements of the intact PFBC-exposed 3M filter indicated that the pressure drop across the element was ~220 inches of water gauge (in-wg) at a gas face velocity of 10 ft/min. The room temperature pressure drop across the ash coated 3M filter was relatively high in comparison to alternate filter elements that were exposed to similar PFBC test conditions. This was attributed to the adherence of the dust cake along and within the outer confinement layer, as well as through the filtration mat and triaxial support braid. Variation in the color of the filter matrix was observed along the outer surface of the filter element (Figure 3.8). This was attributed to removal of the external CVI-SiC outer coating exposing the underlying confinement fibers along the top two-thirds of the filter element.

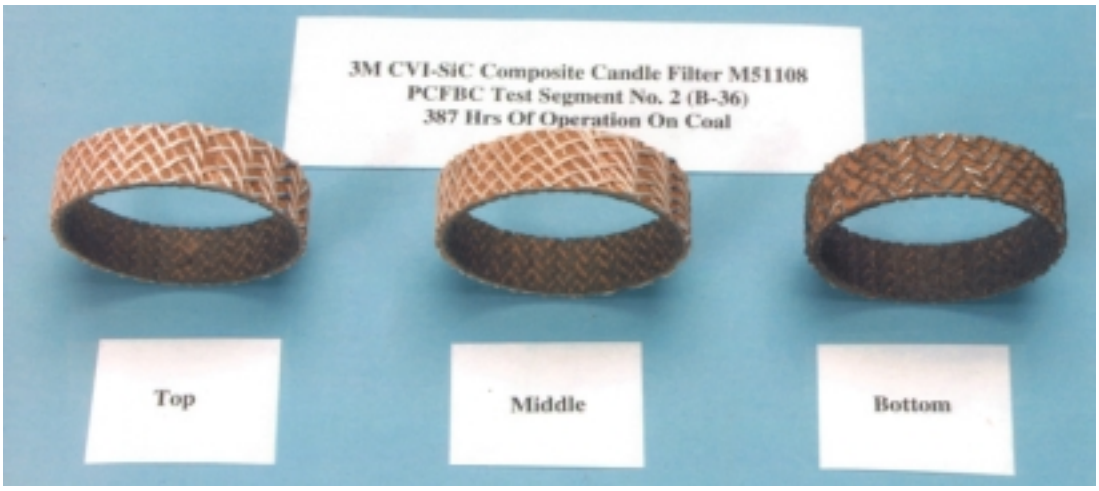
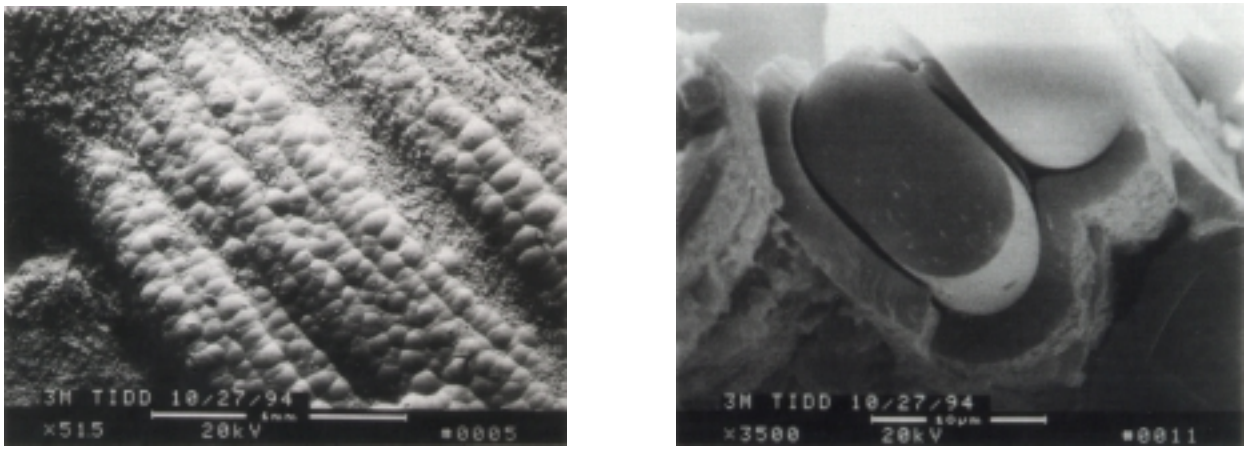


Figure 3.8 — Removal of the CVI-SiC coating along the outer confinement layer of the 3M CVI-SiC composite filter element after 387 hours of operation at AEP.

Characterization of the 3M CVI-SiC matrix via scanning electron microscopy/energy dispersive x-ray analysis (SEM/EDAX) indicated that minor changes in the morphology of the filter matrix had occurred after 1705 hours of operation in the 660-760°C (1220-1400°F) Siemens Westinghouse APF system at AEP (Figure 3.9). Due to deposition of the <1 μm interface coating, it was frequently difficult to discern whether the interface coating had remained intact. A gap or separation was considered to exist between the CVI-SiC coating and the underlying Nextel™ 312 fibers in several areas of the PFBC-exposed 3M composite filter matrix. In the high temperature PFBC environment, oxidation of the interface coating was expected to have occurred.



(a) (b)

Figure 3.9 — Morphology of the 3M CVI-SiC composite matrix after 1705 hours of operation in the 732°C PFBC environment: (a) External CVI-SiC coating; (b) Fractured CVI-SiC coated Nextel™ 312 fibers.

Post-test diametral compressive strength testing indicated that the strength of the PFBC-exposed 3M CVI-SiC composite matrix was greater than that of the as-manufactured filter matrix (Table 3.3). Due to the accumulation of fines within the PFBC-exposed matrix, 'wedging' of fines in between the SiC coated fibers may require a higher load to be applied to the matrix prior to failure, thus generating what appeared to be a strengthened composite matrix. Alternate mechanisms which include phase changes within the matrix were considered during process operation which would provide additional bulk strength to the 3M CVI-SiC composite matrix. During diametral compressive strength testing, the relatively low load bearing, PFBC-exposed, 3M CVI-SiC matrix generally retained the graceful fiber 'pull-out' characteristics of the fracture toughened, as-manufactured matrix.

Testing was reinitiated at AEP in Test Segment 5 during which time ten newly manufactured, 3M CVI-SiC composite filters were installed in the Siemens Westinghouse APF system. After 1110 hours of filter operation at temperatures ranging between 760 and 845°C (1400 and 1555°F), testing was completed. Post-test inspection indicated that all ten of the 3M CVI-SiC composite filter elements generally remained intact. A 25.4 mm diameter hole was evident along the outer confinement layer of one of the 3M filter elements. The location of the removed confinement layer was ~75 mm below the flange.

TABLE 3.3
3M AND DuPONT CANDLE FILTER MATRIX STRENGTH

Filter Matrix	Candle Filter Location	Test Segment No.	Operating Time, Hrs	Room Temperature C-Ring Testing		High Temperature (732°C) C-Ring Testing	
				Compressive	Tensile	Compressive	Tensile
3M CVI-SiC Composite							
43-1-2	—	—	—	1341±254[(a)]	NT	1060±219[(a)]	NT
43-1-6	B/M-15	4	1705	1696±195[(a)]	NT	1429±159[(a)]	NT
43-18-02	C/T-18	5	1110	2333±415[(a)]	NT	2225±361[(a)]	NT
DuPont PRD-66							
D-99	—	—	—	1219±162	1265±188	1277±178	1304±327
D-132	B/M-7	4	1705	1830±238	1725±320	1884±142	1642±401
D-237	B/M-8	5	1110	1533±202	1380±188	1897±256	1356±104

(a) Diametral o-ring strength reported for the entire composite wall (i.e., outer confinement layer, filtration mat, and triaxial braid.).
(b) NT: Not tested.

Assuming that the strength of the as-manufactured 3M filters that were used for testing at AEP in Test Segment 5 was equivalent to the strength of the as-manufactured filter elements that were used in Test Segment 4, characterization of the 1110 hour, PFBC-exposed, 3M CVI-SiC composite filter elements indicated that the strength of the ash-filled matrix once again appeared to increase.

3.2.4 DuPont PRD-66 Filament Wound Filter Testing

During operation of the Siemens Westinghouse APF in Test Segment 4, three DuPont PRD-66 filter elements were installed in a middle filter array. After 1705 hours of operation, all three filters remained intact. During c-ring preparation of the PFBC-exposed PRD-66 filter element, magnesium sulfate crystallized along the outer surface of the filter as a result of contact of the ash that was present within the filter i.d. bore and the wall with water during wet cutting with a diamond wheel. Post-test characterization of the PRD-66 matrix indicated that the bulk strength of the ash-filled matrix tended to increase after 1705 hours of operation at AEP in Test Segment 4 (Table 3.3).

Twenty-two newly manufactured DuPont PRD-66 candle filters were installed in a top array in the Siemens Westinghouse APF prior to operation in Test Segment 5. After ~232 hours of operation, sections of the PRD-66 matrix were identified in the ash hopper discharge, indicating that failure had occurred. Testing continued, and after ~775 hours of operation, additional sections of the PRD-66 filter matrix were found in the ash hopper discharge.

Testing was completed at AEP after 1110 hours of operation in Test Segment 5. Only two of the DuPont PRD-66 filter elements remained intact, four had suffered either mid-body fracture or failure at a location that was ~1125 mm below the flange, and 16 filters had fractured at the base of the flange. The outer surface of the intact and fractured filters was generally ‘ash free’, particularly along the portion of the body that was adjacent to the plenum support pipe, and to approximately mid-way down the length of the filter element. Alternately, a 1-2 mm ash deposit remained along the outer surface of the PRD-66

candles, near the bottom end cap. Areas of the outer membrane and underlying support fibers were removed in 'divot-like' line formations along the surface of the remaining intact and fractured filter elements (Figure 3.10). Localized 'divoting' was also observed below the gasket sleeve that was installed around the filter flange, and along alternate isolated areas of the filter body.

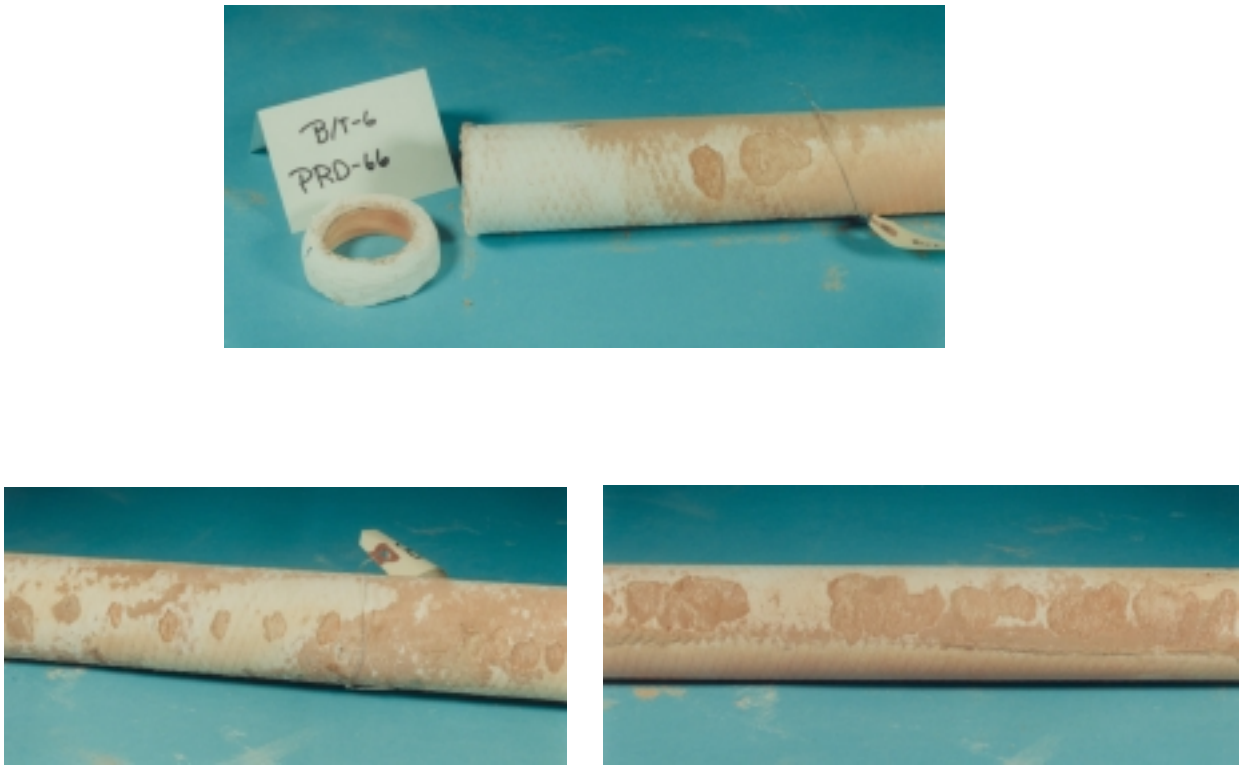


Figure 3.10 — Divot formations along the length of the DuPont PRD-66 filter elements after testing at AEP.

The mechanisms leading to 'divoting' and mid-body failure of the DuPont PRD-66 filter elements in Test Segment 5 were considered to be primarily related to delamination areas that were present within the wall of the filament wound matrix (i.e., uneven winding and/or localized drying or positioning of the elements during manufacturing of the elements; Figure 3.11), and were not expected to directly result from reactions of the process gas environment with the PRD-66 filter matrix. Post-test inspection indicated that fines were present within the ~7 mm PRD-66 filter wall. These may have resulted from penetration of submicron fines through the PRD-66 outer membrane, or fines that were back pulsed into the matrix after failure of an alternate candle had occurred. PFBC ash fines which have a higher thermal coefficient of expansion in comparison to the porous ceramic filter matrices may have induced localized internal failure within the wall during the numerous plant shutdown and startup cycles in Test Segment 5. Mid-body failure of the elements conceivably resulted once the wall had sufficiently weakened or thinned. Failure at the base of the PRD-66 filter flange was attributed to the low load bearing capability of the PRD-66 candle filters in conjunction with the high thermal coefficient of expansion ash that surrounded the filter holder mounts, when ash became 'wedged' in between the outer surface of the filter elements and the metal holders.

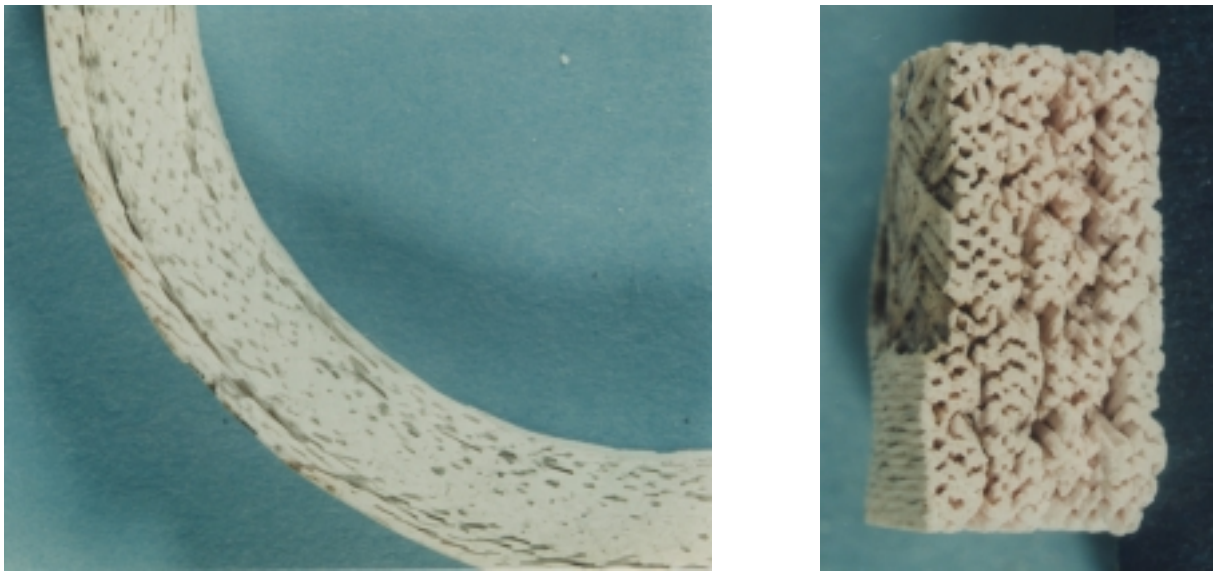


Figure 3.11 — Delamination within the as-manufactured, filament wound, DuPont PRD-66 filter matrix.

Assuming that the strength of the PRD-66 candles used in Test Segment 5 was equivalent to the strength of the PRD-66 candles used in Test Segment 4, post-test strength characterization of the 1110 hour PFBC-exposed PRD-66 filter matrix indicated that an increase in strength appeared to have occurred along the ash-filled o.d. surface, while virtually no change in strength was detected along the i.d. or pulse cycled surface of the filament wound matrix. Both as-manufactured and field-tested matrices exhibited brittle failure characteristics during compressive or tensile strength testing. Although fines were present throughout the field-tested PRD-66 filter wall, the morphology of the matrix after 1110-1705 hours of PFBC operation closely resembled the morphology of the as-manufactured filter matrix.

3.2.5 Comment

Post-test inspection of the candles which were utilized in Test Segment 5 indicated that after failure of isolated elements or an array of elements had occurred, ash was carried into the i.d. bore of many elements within a common filter cluster. As a result of candles filling with ash, cracks were evident at ~6-8 inches above the end cap of a Coors P-100A-1 alumina/mullite, a Schumacher Dia Schumalith F40, and a 3M CVI-SiC composite filter. The difference in thermal expansion of the ash in the internal plug and the porous ceramic filter matrices was considered to be responsible for crack initiation within the various filter elements during the numerous plant shutdown and startup cycles in Test Segment 5.

4. QUALIFICATION TESTING OF ADVANCED SECOND GENERATION CANDLE FILTERS

4.1 Material and Component Modifications

During 1994-1995, Siemens Westinghouse introduced the use of the oxide-based, filament wound, DuPont PRD-66 candle filter at the American Electric Power (AEP) Tidd demonstration plant in Brilliant, Ohio. Initial testing of the PRD-66 element demonstrated viability of the candle filter after 1705 hours of operation in the pressurized fluidized-bed combustion (PFBC) environment. Continued testing of the PRD-66 element in the final segment of PFBC testing at AEP identified the instability of the outer membrane layer of the element which resulted in the formation of divots and failure of the filter body. Low strength along the flange contributed to failure of the elements during 1110 hours of PFBC operation in the final segment of testing at AEP in 1995. Modifications to the membrane and strengthening of the flange were efforts undertaken by DuPont in 1995-1996 to improve the durability and performance of the PRD-66 candle filter.

Similar to the DuPont initiatives in 1995-1996, McDermott (i.e., formerly B&W), and Techniweave continued to develop alternate oxide-based, continuous fiber ceramic composite (CFCC) candle filters. Independent of the Department of Energy (DOE) initiatives, 3M transitioned its CVI-SiC filter technology to the development of an oxide-based CFCC candle filter. Instead of chemically vapor infiltrating silicon carbide along a Nextel™ 720 triaxial braid support layer which was covered with an outer filtration mat and Nextel™ 550 confinement layer, an oxide-based matrix was infiltrated along various layers of the candle preform. Densification of the flange and end caps was undertaken to provide additional strength to the filter element. Alternately McDermott utilized a filament winding process to fabricate an Almax and Nextel™ 610 preform which was subsequently infiltrated with chopped Saffil fibers and an alumina-enriched sol-gel binder.

Similarly, development of the Techniweave candle filter underwent several iterations. Initially Techniweave considered fabricating the candle as a 3-D woven integral composite structure, however, primarily driven by both material and labor cost, a 2-D wrapped fabric architecture was developed. After fabrication of a cylindrical tube, a flange and end cap were added to form the nonintegral candle configuration. Mullite sol was subsequently infiltrated along the entire Nextel™ 720 or Nextel™ 610 preform to form both an external surface membrane, and to bond subsurface fiber bundles to each other, ultimately providing additional strength within the filter wall.

During 1995-1996, Siemens Westinghouse supported Blasch Precision Ceramics in their development of a oxide-based, monolithic, cross flow filter element. During testing in Siemens Westinghouse's PFBC simulator test facility in Pittsburgh, Pennsylvania, failure of the Blasch cross flow filter element occurred at the base of the flange primarily as a result of low strength of the matrix and flange, and the relatively high stress induced during filter mounting. Due to on-going pilot and demonstration plant testing which utilized porous ceramic candle filter elements, Siemens Westinghouse encouraged Blasch to transition utilization of their injection molding process from fabricating cross flow filters to manufacturing candle filters. Even though the Blasch matrix had a lower strength than the monolithic, oxide-based, Coors P-100A-1 alumina/mullite filter matrix, the microstructure of the Blasch matrix was considered to be somewhat more resistant to thermal fatigue due to the use of a mullite bond or ligament to hold adjacent alumina grains to each other within the filter wall (i.e., analogous to the clay binder that held silicon carbide grains together in the Schumacher or Pall filter matrix). Transitioning the

Blasch injection molding process from the previous cross flow filter initiatives, readily led to the production of integral, nonmembrane coated, oxide-based candle filters.

Siemens Westinghouse explored the option of utilizing Specific Surface's porous ceramic Taperflow™ filter concept which was manufactured via a 3-D printing process. If produced as a 1.5 m filter element, the Taperflow™ candle would provide enhanced filtration surface area per unit volume in comparison to commercially available candle filters. Similarly, Siemens Westinghouse considered utilizing Scapa's vacuum infiltrated chopped fiber Cerafil™ filter in coal-based applications. Having transitioned from the former Foseco filter element, Cerafil™ elements which resembled IF&P's Fibrosic™ elements, provided both significant cost advantages and the possibility of using light weight filter elements within the filter array.

Prior to recommending the installation and operation of the advanced monolithic or composite filter elements in coal-fired pilot or demonstration plants, Siemens Westinghouse conducted qualification testing in the PFBC simulator test facility in Pittsburgh, Pennsylvania, to resolve many of the component design and material issues that may be encountered during use of the elements in PFBC, pressurized circulating fluidized-bed combustion (PCFBC), or integrated gasification combined cycle (IGCC) operating systems. A discussion of the qualification test program conducted by Siemens Westinghouse in 1997 which lead to the selection and ultimate installation of advanced porous ceramic filter elements in Foster Wheeler's PCFBC test facility in Karhula, Finland, is presented in the following section.

4.2 Bench-Scale Testing[10]

A candle filter qualification program was undertaken by Siemens Westinghouse in March 1997. Testing continued through May 1997, evaluating the retrofitability and performance of the 3M, McDermott, and Techniweave oxide-based CFCC candles, DuPont's PRD-66 filter element, and Blasch, Specific Surface, and Scapa Cerafil™ filters in Siemens Westinghouse's PFBC simulator test facility in Pittsburgh, Pennsylvania. Elements which remained intact after qualification testing would be considered as possible candidates for installation and operation within Siemens Westinghouse's filter cluster at the Foster Wheeler test facility in Karhula, Finland, in the fall of 1997. A newly manufactured Coors P-100A-1 alumina/mullite candle, and an AEP PFBC-exposed Coors candle were also included within the filter array during qualification testing. The inclusion of the Coors elements was to not only demonstrate the response of the oxide-based monolithic matrix under qualification test conditions, but also to serve as a background matrix for comparison of the response of the alternate filter materials.

One candle of each filter type was installed within the array (Figure 4.1). The filter array was initially subjected to 120 hours of steady state filtration testing, and subsequently 2200 accelerated pulse cycles (i.e., 1100 equivalent exposure hours), and 12 thermal transients typical of commercial plant operation. Based on the dust loading detected via isokinetic sampling after completion of 5 thermal transients, testing was suspended and the array was slow cooled in order to determine whether an element and/or gasket seal had failed. Inspection of the array indicated that the 1 m Techniweave Nextel™ 610 filter element had failed at the base of the flange. The failed element was removed from the array, its position was blanked, testing was reinitiated, and the array was subjected to 7 additional thermal transient events. After slow cooling the array and opening of the pressure vessel, all elements were removed for visual inspection, and subjected to permeability measurements and residual bulk strength characterization.

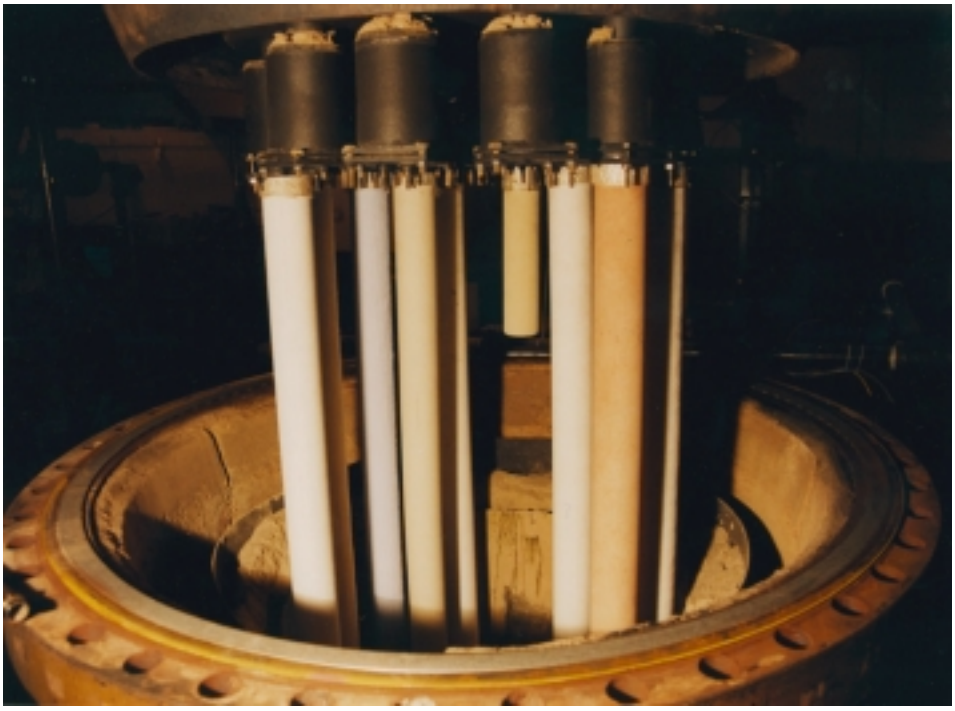


Figure 4.1 — Filter array used in the Siemens Westinghouse qualification test program.

Qualitative comments regarding the texture and thickness of the residual dust cake layer that remained along the surface of each filter element at the conclusion of the qualification test program prior to cleaning are provided in Table 4.1 and Figure 4.2. Figure 4.3 identifies the room temperature gas flow resistance of the filter elements prior to, and after qualification testing. The gas flow resistance across the PFBC-exposed Coors element and newly manufactured Techniweave Nextel™ 720 and Nextel™ 610 elements, and Specific Surface filters exceeded the Siemens Westinghouse performance and design specifications for as-manufactured filter elements. The gas flow resistance across these elements after qualification testing remained relatively high, while the alternate elements experienced effective cleaning and generally maintained a gas flow resistance of $\leq$10 in-wg/10 fpm after completion of the qualification test program.

In preparation for determining the bulk strength of the as-manufactured and qualification-tested candle filters, 15 mm wide o-rings were cut from the vacuum cleaned elements, and were subsequently notched. The c-ring sections from each element were subjected to compressive and tensile strength testing at room temperature and at ~845°C (1550°F). Figure 4.4 illustrates the variation in the wall thickness and contour of the flange of the various monolithic and advanced filter elements.

The room temperature and process temperature compressive and tensile strength of the as-manufactured and qualification-tested, advanced, second generation, porous ceramic candle filter matrices are presented in Table 4.2. As shown in the process temperature c-ring compressive strength information presented in Figure 4.5 the residual bulk strength of the McDermott (B&W), 3M, Techniweave Nextel™ 720, and newly manufactured Coors P-100A-1 alumina/mullite filter matrices tended to decrease as a result of the elements being subjected to simulated PFBC operating conditions.

TABLE 4.1
RESIDUAL FILTER DUST CAKE LAYER AFTER QUALIFICATION TESTING

Filter Element	Array Location	Surface Ash Deposit
DuPont PRD66-m	1	More texture to the deposited ash layer than on the coarse membrane
DuPont PRD66-c	3	—
3M CVI-SiC Oxide	5	Rippled uneven cake; No pin-holing of ash; Bottom intact
McDermott Oxide	7	—
Techniweave 610	8	(a)
Techniweave 720	9	—
Blasch	10	More rippled texture to surface ash
Coors New	12	—
Coors Tidd-Exposed	13	—
Specific Surface	15	—

(a) Element failed prior to completing thermal transient testing.

Figure 4.2 — Filter array at the conclusion of Siemens Westinghouse qualification testing.

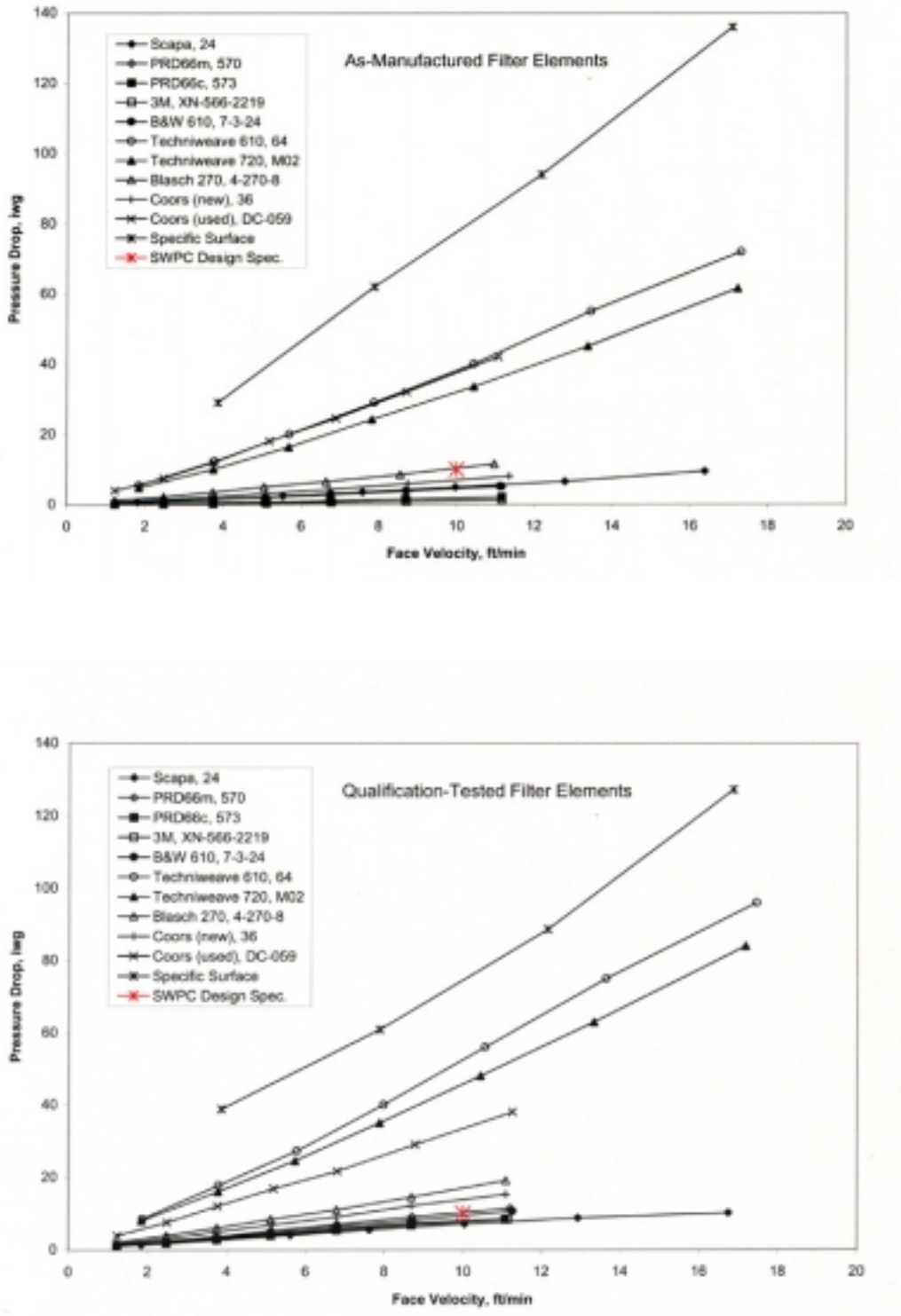


Figure 4.3 — Gas flow resistance of the qualification-tested filter elements prior to and after exposure in the Siemens Westinghouse PFBC simulator test facility.

Figure 4.4 — Variation in the wall thickness and flange contour of the monolithic and advanced filter elements.

Alternately the bulk strength of the DuPont PRD-66 and Techniweave Nextel™ 610, and PFBC-exposed Coors P-100A-1 alumina/mullite candles tended to increase after being subjected to qualification testing under simulated PFBC operating conditions. Retention of ash fines within the matrix (i.e., penetration and wedging of submicron and micron fines between the filaments and/or fibers; accumulation of fines along the candle i.d. bore during pulse cleaning after failure of an element or elements within the filter array; etc.), may be responsible for the apparent increase in process temperature strength for these elements. Although divot formations along the outer membrane of the DuPont PRD-66 candle did not occur during the qualification test program, the potential may exist during extended field operation, particularly if thermal expansion of the ash fines within the filter wall occurs during plant startup cycles, or hydration of the ash results during shutdown cycles.

Tables 4.3 and 4.4 identify the load bearing capabilities and additional material properties of the as-manufactured and qualification-tested, advanced, second generation, porous ceramic filter matrices. Figure 4.6 illustrates that a significant difference exists between the load bearing capabilities of the monolithic filter elements (i.e., Coors, Schumacher, and Pall), and the alternate advanced composite and

TABLE 4.2
ROOM TEMPERATURE AND PROCESS TEMPERATURE STRENGTH OF THE AS-MANUFACTURED AND QUALIFICATION-TESTED POROUS CERAMIC CANDLE FILTERS

Candle Identification Number	Status	C-Ring Compressive Strength, psi		C-Ring Tensile Strength, psi	
		25°C	843°C	25°C	843°C
DuPont PRD-66 (Coarse Membrane)					
D-563c	As-Manufactured	955±62 (9)	962±92 (8)	809±154 (9)	1008±103 (7)
D-573c	Qualification-Tested	1214±67 (9)	1210±86 (9)	990±82 (9)	1195±166 (9)
DuPont PRD-66 (Medium Membrane)					
D-564m	As-Manufactured	990±130 (9)	883±79 (9)	846±105 (9)	918±104 (9)
D-570m	Qualification-Tested	1021±127 (9)	1019±88 (9)	973±165 (9)	1193±149 (8)
McDermott Oxide-Based CFCC					
7-3-21	As-Manufactured	547±58 (9)	492±81 (9)	675±91 (9)	642±87 (9)
7-3-24	Qualification-Tested	414±60 (9)	368±67 (9)	514±151 (9)	405±153 (9)
3M Oxide-Based CFCC					
XN-566-2214	As-Manufactured	483±162 (9) 1929±648 (9)	722±162 (9)* 2885±648 (9)**	624±154 (9) 2389±590 (9)	657±102 (9) 2513±389 (9)
XN-566-2219	Qualification-Tested	177±42 (9) 605±171 (9)	235±88 (8) 880±287 (8)	146±63 (9) 561±255 (9)	241±91 (8) 956±361 (8)
Techniweave Oxide-Based CFCC (Nextel™ 610)					
T-65	As-Manufactured	2324±548 (9)	1714±548 (9)	1190±223 (9)	877±289 (9)
T-64	Qualification-Tested	2430±751 (9)	2102±536 (9)	1536±282 (9)	1008±200 (9)
Techniweave Oxide-Based CFCC (Nextel™ 720)					
T-M01	As-Manufactured	2215±431 (9)	1714±416 (9)	1377±413 (9)	980±249 (9)
T-M02	Qualification-Tested	1567±307 (8)	1311±526 (9)	772±242 (9)	735±164 (9)
Blasch Monolithic Mullite Bonded Alumina					
B-4-270-3	As-Manufactured	625±148 (9)	614±163 (9)	656±92 (9)	604±103 (9)
B-4-270-8	Qualification-Tested	433±137 (9)	477±111 (9)	630±66 (8)	510±128 (9)

* Entire Wall Thickness.
** Triaxial Braid Layer Thickness.

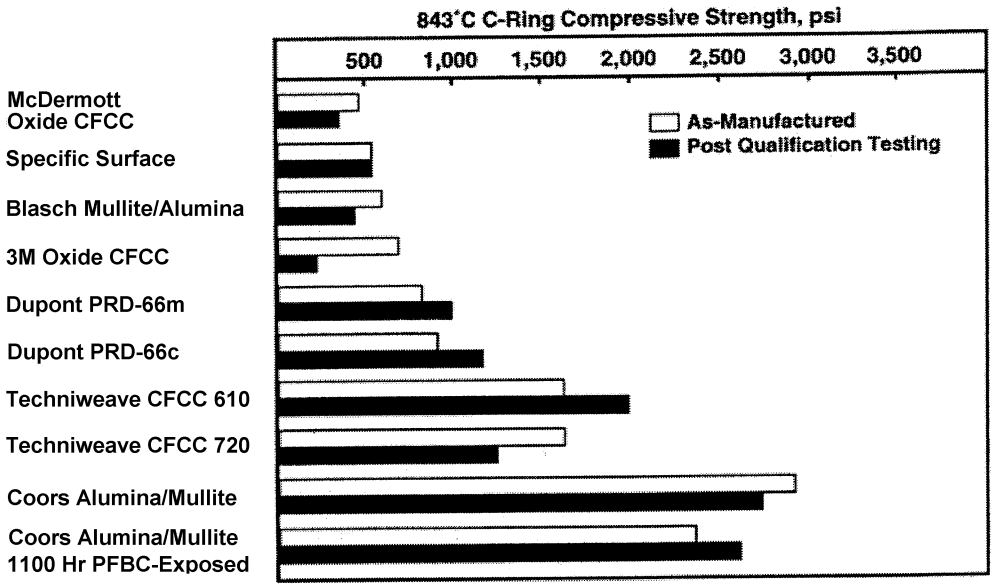


Figure 4.5 — Bulk strength of the as-manufactured and qualification-tested advanced monolithic and composite candle filters.

monolithic candles. Should an ash bridging event occur, or ash fines become wedged between the filter holder mount and the candle flange, failure during process operation or on removal of the elements from the holder would be more likely to occur for the lower load bearing advanced composite and monolithic candles. In the event that ash becomes significantly bridged between the elements and/or passive metal structures, all porous ceramic filter elements would be expected to experience catastrophic failure due to the high thermal expansion of the ash relative to the ceramic filter matrices and the support metal structures.

4.2.1 Filter Element Down-Selection

In order to select candles for possible inclusion in the Siemens Westinghouse filter cluster at the Foster Wheeler PCFBC test facility in Karhula, Finland, any element which did not remain intact during qualification testing, or could not be manufactured to Siemens Westinghouse's dimensional tolerance and design/performance specifications was eliminated from further consideration for testing in the fall of 1997. As a result, the Specific Surface element which exhibited an initial high gas flow resistance, and did not achieve either the flange or filter body dimensional tolerances was eliminated. When the qualification-tested Specific Surface element was subjected to c-ring compressive or tensile strength characterization, the 3-D printing process inherently established extensive *in-situ* delamination sites within the filter matrix. During final high firing of the elements at Specific Surface, the filters typically formed several cracks that needed to be prepared (i.e., bonding and sealing) prior to consideration for possible use.

The Scapa Cerafil™ element similarly was not recommended to be included in the Foster Wheeler PCFBC fall 1997 test campaign due to the low strength and load bearing capabilities of the vacuum infiltrated matrix, and the formation of the longitudinal crack along the filter body after being subjected to qualification testing (Figure 4.7). In addition, the standard Scapa Cerafil™ element lacked the appropriate flange contour for retrofit into Siemens Westinghouse's metal filter holders, and

TABLE 4.3
ULTIMATE LOAD APPLIED DURING STRENGTH CHARACTERIZATION OF THE AS-MANUFACTURED AND QUALIFICATION-TESTED POROUS CERAMIC CANDLE FILTERS

Candle Identification Number	Status	C-Ring Compressive Strength, psi		C-Ring Tensile Strength, psi	
		25°C	843°C	25°C	843°C
DuPont PRD-66 (Coarse Membrane)					
D-563c	As-Manufactured	8.2±0.5 (9)	8.2±0.9 (8)	5.2±1.1 (9)	6.7±0.7 (7)
D-573c	Qualification-Tested	10.3±0.6 (9)	10.3±0.6 (9)	6.4±1.2 (9)	7.6±1.0 (9)
DuPont PRD-66 (Medium Membrane)					
D-564m	As-Manufactured	8.0±0.9 (9)	7.3±0.6 (9)	5.2±0.6 (9)	5.7±0.6 (9)
D-570m	Qualification-Tested	8.3±1.0 (9)	8.3±0.8 (9)	6.1±0.9 (9)	7.4±0.8 (8)
McDermott Oxide-Based CFCC					
7-3-21	As-Manufactured	2.4±0.2 (9)	2.1±0.3 (9)	2.3±0.3 (9)	2.2±0.3 (9)
7-3-24	Qualification-Tested	1.4±0.1 (9)	1.3±0.2 (9)	1.4±0.4 (9)	1.1±0.4 (9)
3M Oxide-Based CFCC					
XN-566-2214	As-Manufactured	0.41±0.14 (9)	0.60±0.13 (9)	0.50±0.13 (9)	0.51±0.09 (9)
XN-566-2219	Qualification-Tested	0.11±0.33 (9)	0.16±0.05 (8)	0.13±0.05 (9)	0.21±0.06 (8)
Techniweave Oxide-Based CFCC (Nextel™ 610)					
T-65	As-Manufactured	7.7±1.5 (9)	6.0±1.8 (9)	3.3±1.3 (9)	2.5±0.7 (9)
T-64	Qualification-Tested	6.2±1.2 (9)	5.0±1.0 (9)	3.2±0.5 (9)	2.2±0.4 (9)
Techniweave Oxide-Based CFCC (Nextel™ 720)					
T-M01	As-Manufactured	6.2±0.6 (9)	4.7±0.8 (9)	3.1±0.9 (9)	2.3±0.6 (9)
T-M02	Qualification-Tested	5.1±1.2 (9)	4.3±1.0 (9)	2.1±0.3 (9)	2.0±0.3 (9)
Blasch Monolithic Mullite Bonded Alumina					
B-4-270-3	As-Manufactured	13.3±3.6 (9)	12.9±3.7 (9)	9.2±1.5 (9)	8.2±1.3 (9)
B-4-270-8	Qualification-Tested	10.3±3.2 (9)	11.1±2.5 (9)	9.4±1.1 (8)	7.4±1.4 (9)

individual hand cutting and densification of elements was not considered by Siemens Westinghouse to be efficient or cost effective.

Similarly shear failure (Figure 4.8) along the Techniweave Nextel™ 610 candle flange raised concern as to associated risks involved with utilizing a nonintegrally constructed filter element in long-term field test campaigns. In an attempt to identify the shear strength of the as-manufactured Techniweave filter elements, and assess the impact of qualification testing on the resulting strength of the nonintegral flange and end cap, bench-scale, shear testing was conducted. The results of this effort indicated that shear strength of the as-manufactured Techniweave filter flanges ranged between 138 and 259 psi, while the shear strength of the as-manufactured filter end caps ranged between 227 and 273 psi (Table 4.5). After being subjected to qualification testing, the shear strength of the end caps of both the Techniweave Nextel™ 720 and Nextel™ 610 candles was shown to decrease by 15% and 27%, respectively. The flange shear strength of the qualification-tested filters was not measured due to *in-situ* failure of the Nextel™ 610 element, and cross-sectioning of the Nextel™ 720 element for inspection purposes.

TABLE 4.4
MATERIAL PROPERTIES OF THE AS-MANUFACTURED AND QUALIFICATION-TESTED ADVANCED MONOLITHIC AND COMPOSITE CANDLE FILTERS

Candle Identification Number	Status	Burst Pressure, psi	Ultimate Hoop Stress, psi	Modulus, psi x 10^6	Poisson's Ratio
DuPont PRD-66 (Coarse Membrane)					
D-563c	As-Manufactured	148	555	7.96	0.86
D-573c	Post-Test	158	597	6.11	0.82
DuPont PRD-66 (Medium Membrane)					
D-564m	As-Manufactured	180	691	7.09	0.84
D-570m	Post-Test	170	653	5.42	0.84
McDermott Oxide-Based Composite					
7-3-21	As-Manufactured	188	998	1.25	0.95
7-3-24	Post-Test	136	776	1.17	0.85
3M Oxide-Based Composite					
XN-566-2214	As-Manufactured	52	586	1.36	0.73
XN-566-2219	Post-Test	29	334	1.35	0.73
Techniweave Oxide-Based Composite (Nextel™ 610)					
T-65	As-Manufactured	ND	ND	ND	ND
T-64	Post-Test	ND	ND	ND	ND
Techniweave Oxide-Based Composite (Nextel™ 720)					
T-M01	As-Manufactured	ND	ND	ND	ND
T-M02	Post-Test	ND	ND	ND	ND
Blasch Mullite Bonded Alumina					
B-4-270-3	As-Manufactured	170	410	2.12	0.09
B-4-270-8	Post-Test	155	376	1.60	0.09
Specific Surface Taperflow™					
SS-0	As-Manufactured	ND	ND	ND	ND
SS-1	Post-Test	ND	ND	ND	ND
Scapa Cerafil™					
S-24	As-Manufactured	ND	ND	ND	ND

ND: Not determined.

In addition to the low shear strength and failure of the Techniweave Nextel™ 610 filter element during qualification testing, the gas flow resistance of the Techniweave elements exceeded the Siemens Westinghouse design and performance specifications for as-manufactured filter elements. In view of the nonintegral construction of the filter elements, low flange and end cap shear strength, and high gas flow resistance, several process and design modifications were proposed and implemented. Although the gas flow resistance tolerances were achieved, and the flange was additionally densified, production of a nonintegrally fabricated body (i.e., absence of seams; inserts; etc.) remained a principle concern for successful use during long-term PCFBC operation.

Although the 3M oxide-based CFCC element (i.e., Nextel™ 550 outer confinement layer; Nextel™ 720 triaxial braid) remained intact after qualification testing at Siemens Westinghouse, the relatively low load bearing capability and strength of the element raised concern as to the long-term stability of the candles during field operation. In addition, when the 3M element was subjected to

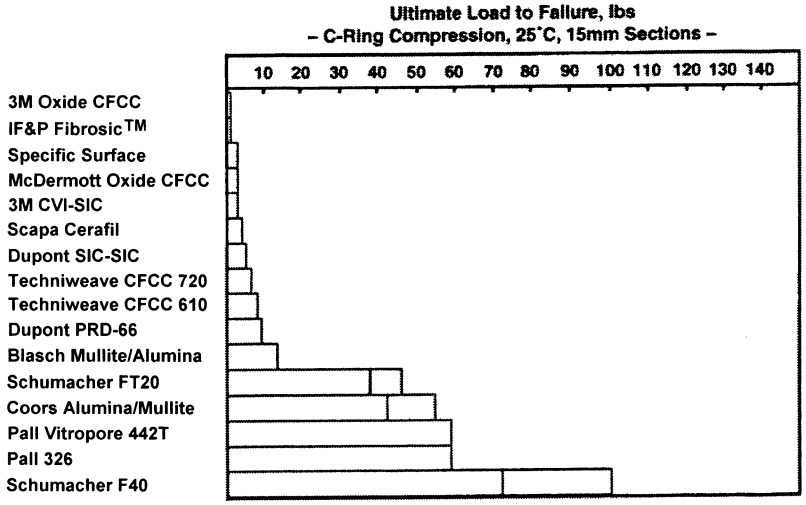


Figure 4.6 — Load bearing capabilities of the as-manufactured porous ceramic candle filters.

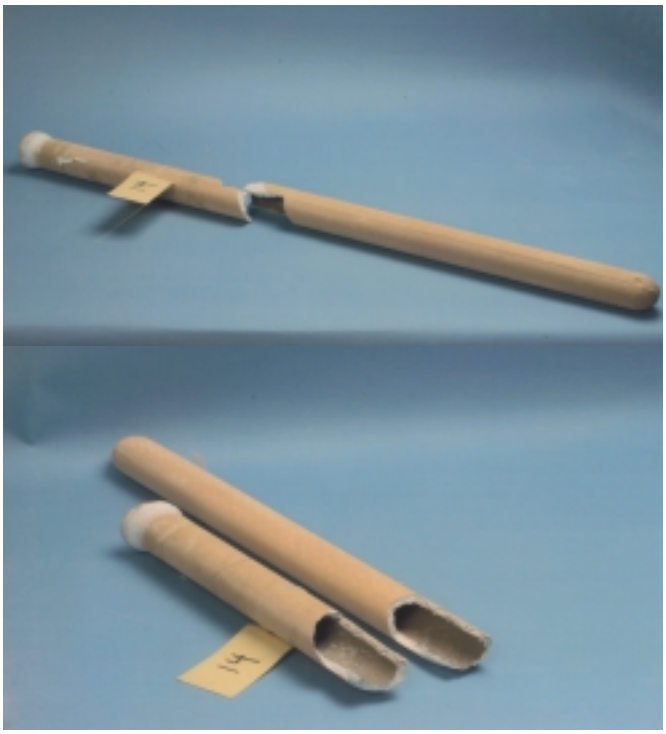


Figure 4.7 — Failure of the Scapa Cerafil™ filter element during Siemens Westinghouse simulated PFBC qualification testing.

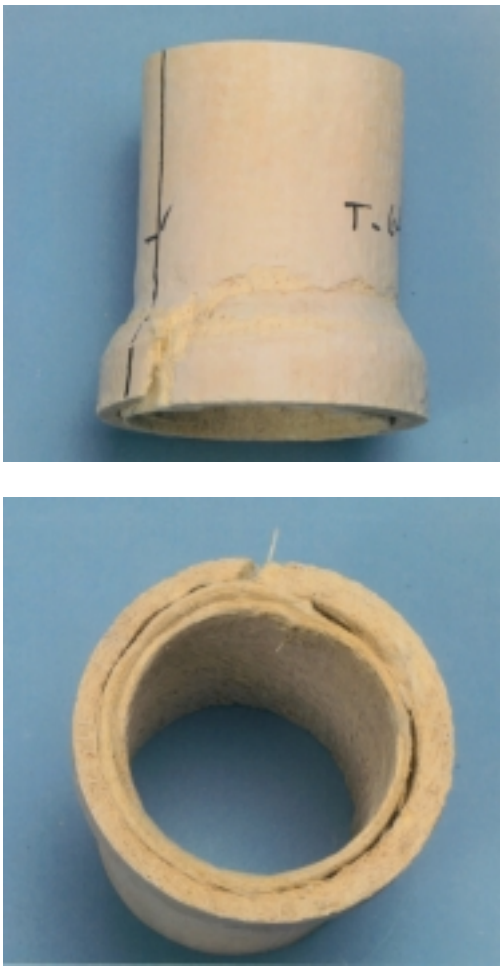


Figure 4.8 — Shear failure of the Techniweave filter element during Siemens Westinghouse simulated PFBC qualification testing.

TABLE 4.5
SHEAR STRENGTH OF THE AS-MANUFACTURED AND QUALIFICATION-TESTED TECHNIWEAVE CANDLE FILTER FLANGE AND END CAP

Element Identification Number	Fiber	Status After Qualification Testing	Shear Strength, psi	
			Flange	End Cap
T-64	Nextel™ 610	Failed	Not Tested Due To Shear Failure	166
T-65	Nextel™ 610	Not Tested	259	227
MO-1	Nextel™ 720	Not Tested	138	273
MO-2	Nextel™ 720	Intact	Cross-Sectioned For Inspection	232

strength characterization, the three layered matrix was identified to be relatively brittle, and easily fractured or torn. This raised the question of the residual fracture toughness characteristics of the 3M oxide-based filter element. In an attempt to circumvent failure and improve the long-term viability of the filter, 3M proposed and manufactured candles with and external Nextel™ 550 outer confinement layer, and an internal Nextel™ 610 triaxial braid support layer. 3M had elected to utilize the Nextel™ 610 matrix as a result in-house testing which demonstrated improved strength and performance over that of the Nextel™ 550 and Nextel™ 720 fibers.

4.2.2 Recommendations for Foster Wheeler PCFBC Testing

As a result of qualification testing, Siemens Westinghouse recommended the use of the following advanced and composite filters in the Foster Wheeler PCFBC test program:

- Eight 1.5 m DuPont PRD-66 filter elements. When manufactured with a coarse membrane, the elements appeared to perform as bulk filters during qualification testing at Siemens Westinghouse, and had a lower initial pressure drop in comparison to elements that were manufactured with a less coarse (i.e., medium), outer surface membrane. Similarly a lower pressure drop was retained along the coarse membrane PRD-66 filter elements in comparison to the medium membrane PRD-66 filter elements after a conditioned ash layer had been formed along the outer surface of the candles. The dimensional tolerance along the i.d. bore of the DuPont PRD-66 candles was to be maintained as identified by Siemens Westinghouse design specifications.

- Eight 1.5 m McDermott oxide-based CFCC filter elements. Although tufting of the fibers was observed along the flange of the McDermott oxide-based CFCC candles after qualification testing, the existing manufacturing protocol was viewed to be acceptable for use in production of the elements for PCFBC testing. The base fiber used to construct the McDermott filter elements was identified to be Nextel™ 610. Similar to alternate thinner walled advanced composite or filament wound filter elements, maintaining Siemens Westinghouse dimensional tolerance specifications along the i.d. bore of the McDermott candles was required.

- Eight 1.5 m 3M oxide-based CFCC filter elements. These candles were to be manufactured with a Nextel™ 610 triaxial support braid layer, an alumina-based fiber filtration layer, and a Nextel™ 550 open mesh outer confinement layer. The mixed Nextel™ 550/610 element were manufactured since only a fine mesh Nextel™ 610 outer confinement layer was available.[3] The fine mesh outer confinement layer configuration was not subjected to qualification testing, and therefore was not recommended for field test use.

- Two 1.5 m Techniweave oxide-based CFCC filter elements. Modification of the Techniweave filter element was required prior to utilization at Karhula. Techniweave was to manufacture 1.5 m filters with integral, densified/strengthened flanges. Additional changes included manufacturing the elements with a flat sealing surface along the top of the flange;

[3] The use of the Nextel™ 550/610 matrix was based on discussions with 3M during June, as well as delivery of the elements to site by August 1, 1997. As per discussions with 3M on July 7, 1997, a Nextel™ 610/610 would be available with the open mesh outer confinement layer, but delivery to site would be by August 18, 1997.

maintaining a dimensional tolerance along the i.d. bore; incorporating an integral end cap; achieving acceptable gas flow resistance; achieving acceptable dimensional tolerances.

- Eight 1.5 m monolithic Blasch mullite bonded alumina candle filters. The dimensional tolerances of these filter elements were to be maintained for acceptance/utilization at Karhula.

The candle filters described above were manufactured by DuPont, McDermott, 3M, Techniweave, and Blasch. Dimensional tolerance inspection sheets were received and reviewed in July 1997, with all candles being accepted for installation in the Siemens Westinghouse APF system at the Foster Wheeler PCFBC test facility in Karhula, Finland. Shipment of the candles, as well as hardware and gaskets from Siemens Westinghouse were to arrive at site by August 1, 1997. When received at site, each filter element underwent a visual and dimensional tolerance inspection, as well as permeability measurements, prior to further consideration and/or installation in the filter array. Testing of the filter array was scheduled to begin during the first week of September 1997, and to continue for a period of 500 hours at operating temperatures of 760°C (1400°F).

4.3 Foster Wheeler PCFBC Testing[2, 3]

Table 4.6 provides a general summary of the PCFBC filter operating experience utilizing the Siemens Westinghouse APF system at the Foster Wheeler test facility in Karhula, Finland. During testing in 1995-1996, 1166 hours of PCFBC operation were conducted at 820-860°C (1510-1580°F) using Coors P-100A-1 alumina/mullite, Schumacher F20, and Pall 326 candle filters. 3M CVI-SiC elements were also initially included in the filter cluster. A similar combination of elements was installed and operated within the filter cluster assembly during the first test segment in 1997. During this phase of testing, the operating temperature of the filter array remained at 800-850°C (1470-1560°F), while the Lakeland coal and sorbent were used as feed materials.

During the second test segment in 1997, the advanced composite and monolithic candles were installed in the bottom array (Figure 4.9). These elements included the oxide-based CFCC 3M, McDermott, and Techniweave filter elements, the oxide-based monolithic Blasch candles, and the DuPont PRD-66 filter elements. The filter was initially operated for a period of 40 hours at temperatures of 700-720°C (1290-1330°F; i.e., projected Lakeland operating conditions) utilizing the Lakeland feed materials, prior to detecting dust in the outlet stream. Testing was subsequently terminated and the vessel was cooled. Post-test inspection of the filter array indicated that the two Techniweave Nextel™ 720 oxide-based CFCC and seven 3M oxide-based Nextel™ 610 CFCC elements had experienced severe damage during the 40 hours of PCFBC operation. Close inspection of the Techniweave filter elements indicated that sections of the outer membrane, through-thickness fibers were removed, and debonding of the outer seam and unwrapping of the 2-D fabric wrap or layered architecture resulted. Pinholes as a result of through-thickness fiber removal permitted ash fines to pass from the o.d. to i.d. surfaces of the PCFBC-exposed filter elements. Similarly the 3M oxide-based CFCC elements experienced removal of sections from both the outer confinement and filtration mat layers, again permitting fines to pass from the o.d. to i.d. surfaces of the PCFBC-exposed filter elements. Sections of material beneath the confinement layer were also seen to be removed along the end caps of the 3M oxide-based CFCC filter elements. Although the Techniweave and 3M elements suffered severe damage during the 40 hours of PCFBC operation, all elements remained attached to the metal filter holder mounts.

TABLE 4.6
SUMMARY OF PCFBC TESTING

Test Segment (Date)	1 (11/95-12/95)	2 (2/96-4/96)	3 (8/96-10/96)
Coal	Illinois No. 6 (Sparta)	Illinois No. 6 (Sparta)	Illinois No. 6 (Sparta)
Sorbent	Linwood Limestone	Linwood Limestone	Linwood Limestone; Iowa Industrial Limestone; Resized Linwood Limestone
Number of Candles	112	112	128
Schumacher FT20	32-35	35	46
Pall 326	32-35	35	45
Coors Alumina/Mullite	24 (5) - 42 (5)[(a)]	33 (5)[(a)]	32 (4)[(a)]
3M CVI-SiC Composite	24-0	9	5
Operating Hours (Coal)	153[(b)]	387	626
Operating Temperature, °C (°F)	826-853 (1519-1567)	818-860 (1504-1580)	838-860 (1540-1580)
Operating Pressure, bar	10.7-11.1	10.6-11.3	10.5-10.7
Inlet Dust Loading, ppmw	12,000-13,500	12,000-15,500	11,000-12,500
d_{50}, µm (Malvern)	NA	NA	23 (20-26)

(a) Number of installed PFBC-exposed Coors filter elements (AEP Test Segment 5) shown in parentheses.
(b) Thirty-five hours of initial operation prior to removal of the 3M CVI-SiC composite filters, followed by 118 hours of continuous operation.
NA: Not available.

All of the damaged Techniweave and 3M PCFBC-exposed filters, and one as-manufactured element of each filter type were returned to Siemens Westinghouse for examination. Initially each element was subjected to room temperature gas flow resistance measurements. Figure 4.10 illustrates the gas flow resistance measurements for all 3M elements that were manufactured and/or tested during 1997 (i.e., Siemens Westinghouse: Nextel™ 550/Nextel™ 720; Karhula 1: Nextel™ 550/Nextel™ 610 (these failed after 40 hours of PCFBC exposure); Karhula 2: Nextel™ 610/Nextel™ 610 (fabricated for possible replacement and use in the final phase of testing in 1997)). With the exception of one candle (i.e., 714), all 3M elements maintained a gas flow resistance of $\leq$10 in-wg/10 fpm that was required by Siemens Westinghouse for newly manufactured filter elements. In contrast, variable gas flow resistance measurements were observed for the Techniweave Nextel™ 720 candles after 40 hours of PCFBC operation.

Due to the severe damage experienced by the Techniweave and 3M elements during the 40 hours of PCFBC operation, the filters were subjected to burst strength testing to identify the burst pressure of the elements, hoop stress, modulus, and Poisson's ratio for the oxide-based CFCC materials. Neither filter type was subjected to c-ring compressive or tensile testing to determine the initial and/or residual bulk strength of the elements.

As shown in Table 4.7, the burst strength of the Techniweave Nextel™ 720 elements decreased after 40 hours of PCFBC operation. Characterization of the PCFBC-exposed Techniweave elements indicated that significant material had been debonded and/or removed from the outer surface of the elements, primarily as a result of the manufacturing process used to achieve an acceptably smooth outer surface finish, and to achieve acceptable gas flow resistance measurements for newly manufactured filter

TABLE 4.6 (Cont'd.)
SUMMARY OF PCFBC TESTING

Test Segment (Date)	1 (4/7/97-4/27/97)	2 (5/12/97-6/2/97)	1 (9/4/97-9/8/97)	2 (9/13/97-9/22/97)	3 (10/14/97-11/7/97)
Coal	Eastern Kentucky (Beech Fork)	Eastern Kentucky (Beech Fork)	Eastern Kentucky (Beech Fork)	Eastern Kentucky (Beech Fork)	Eastern Kentucky (Beech Fork)
Sorbent	Florida Limestone (Gregg Mine)	Florida Limestone (Gregg Mine)	Florida Limestone (Gregg Mine)	Florida Limestone (Gregg Mine)	Florida Limestone (Gregg Mine)
Number of Candles	128	128	90	90	112
Coors P-100A-1	72	72	28	31	33
Pall 326	28	28	16	19	22
Schumacher FT20	28	28	16	19	28
3M Oxide CFCC	—	—	7	—	—
McDermott CFCC	—	—	6	6	7
Techniweave CFCC	—	—	2	—	—
Blasch (Monolith)	—	—	4	4	6
DuPont PRD-66	—	—	7	7	7
Other	—	—	4	4	9
Operating Hours (Coal)	275	179	40	199	342
Temperature, °C (°F)	830-850 (1526-1562)	830-850 (1526-1562)	700-720 (1292-1328)	700-750 (1292-1382)	700-750 (1292-1382)
Pressure, bar	10-11	11	10	10-11	9.5-11
Face Velocity, cm/sec	2.7-3.5	2.4-3.9	4.0	3.7-4.0	2.8-4.0
Inlet Dust Loading, ppmw	7,000-11,000	7,000-10,000	8,000-9,000	7,000-9,000	5,700-8,200
d_{50}, µm (Malvern)	Not Reported	Not Reported	Not Reported	Not Reported	Not Reported

elements. Insufficient matrix was seen to be present throughout the filter wall which promoted delayering of the wrapped fabric layers. Removal of the poorly bonded through-thickness fibers permitted penetration of fines from the o.d. to i.d. surfaces of the filter element. The lack of matrix bond also permitted the outer seam along the lapped brittle fabric edge to be removed, torn, or debonded from the underlying filter wall.

Although shear failure had not occurred along the flange or end caps of the Techniweave Nextel™ 720 filter elements after 40 hours of PCFBC operation, Siemens Westinghouse strongly recommended that elements subsequently manufactured by Techniweave be constructed as an integral body in order to mitigate the potential for failure during extended field operation. Similarly the application of a thicker and better bonded matrix and membrane along the outer surface of the filter elements was also recommended.

Once received at Siemens Westinghouse, the 3M elements were subjected to x-ray diffraction analysis (i.e., phase/compositional characterization), and layer adherence testing. Table 4.8 identifies the results of these analyses for various 3M elements that were provided to Siemens Westinghouse for use in the qualification test program, as well as the modified materials that were manufactured in August 1997.

Figure 4.9 — Advanced composite filter element testing at the Foster Wheeler test facility in Karhula, Finland.

The coarse open mesh outer confinement layer on the qualification-tested and August 1997, PCFBC-tested elements was constructed from Nextel™ 550, with an underlying alumina-enriched filtration mat layer. The triaxial support braid for the qualification-tested filter elements consisted of Nextel™ 720 which was primarily composed of mullite-enriched fiber bundles. The matrix applied to bond the various layers was predominantly alumina-based. For the 3M oxide-based CFCC filter elements that were supplied in August 1997, the triaxial braid consisted of Nextel™ 610 fiber bundles which were principally alumina-based fibers.

Due to the severe damage encountered by the 3M oxide-based CFCC elements after 40 hours of PCFBC operation, a layer adherence test was devised at Siemens Westinghouse in which a 2.54 cm x 2.54 cm metal coupon was bonded to either the outer confinement layer, or the outer confinement and filtration mat layers at various locations along the candle body. The metal coupon was shaped to precisely fit the contour of the 3M filter.

Layer adherence testing was conducted on not only the 3M qualification test candles, but also on elements from the August 1997 production lot. In addition, 3M CVI-SiC composite filters (i.e., newly manufactured and PCFBC-exposed elements), and newly manufactured 3M oxide-based Nextel™ 610/Nextel™ 610 candles that were produced as a replacement set for the failed 3M Nextel™ 550/ Nextel™ 610 were also subjected to layer adherence testing. The 3M Nextel™ 610/Nextel™ 610 replacement candles had additionally been modified to incorporate the use of a finer mesh outer confinement layer along the external surface of the filter element as a result of what was perceived to be

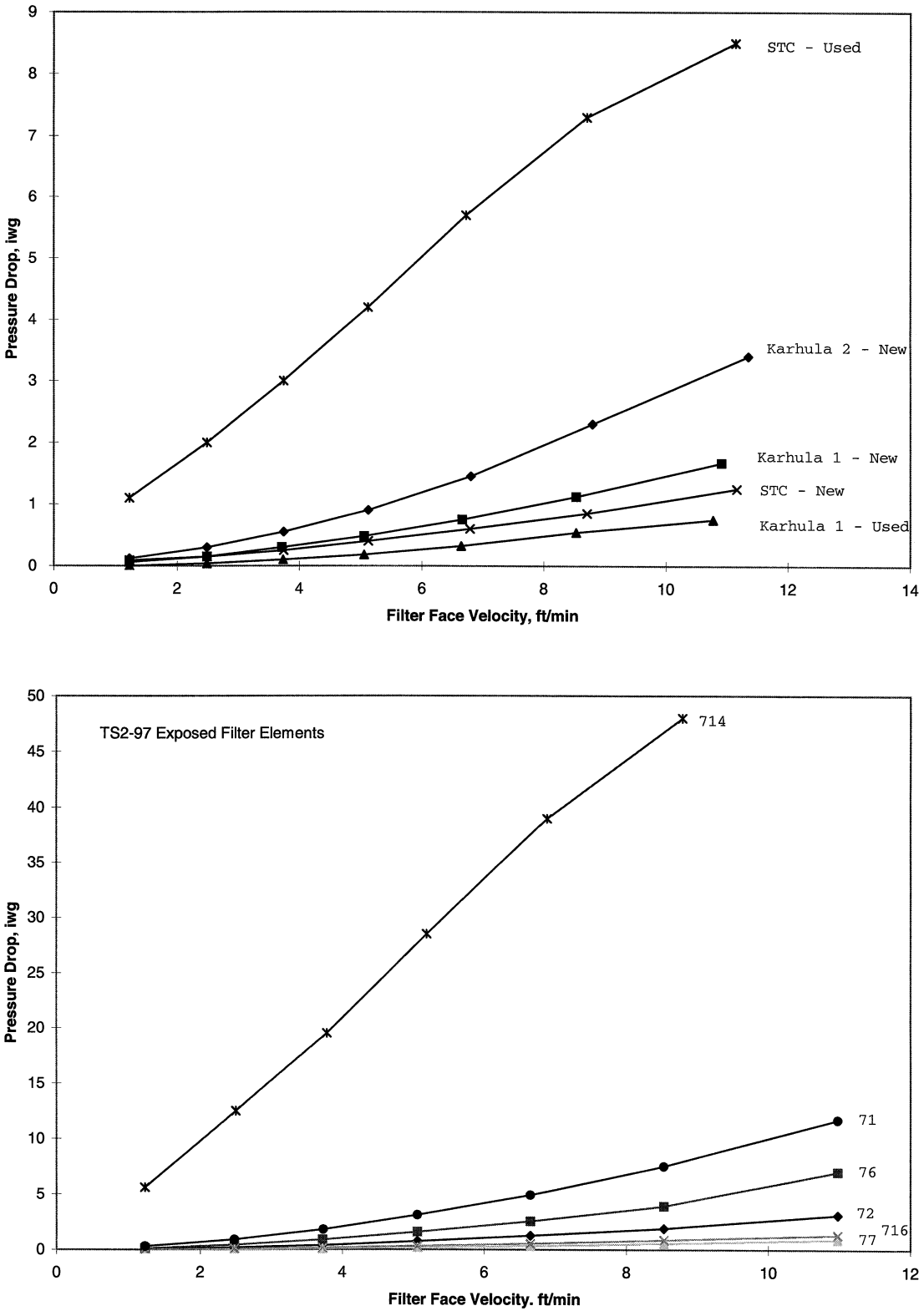


Figure 4.10 — Gas flow resistance of the 3M CVI-SiC candle filters.

TABLE 4.7
TECHNIWEAVE NEXTEL™ 720 CFCC CANDLE FILTER MATERIAL PROPERTIES

Filter Element	Burst Pressure, psi	Ultimate Hoop Stress, psi	Modulus, psi x 10^6	Poisson's Ratio
As-Manufactured	284	3512	12.4	Not Reported
PCFBC-Tested Element	202	2092	15.7	0.24

TABLE 4.8
X-RAY DIFFRACTION ANALYSIS OF THE
3M OXIDE-BASED CFCC CANDLE FILTERS

Test Program	Siemens Westinghouse Qualification Testing February-April 1997	Karhula-1 August 97 FW-PCFBC	Karhula -2 September 97 FW-PCFBC
Outer Confinement Layer	N550 - Coarse Major: α-Al_2O_3 Minor: γ/δ- Al_2O_3 Trace: Glass	N550 - Coarse	N610 - Fine
Filtration Mat	Al_2O_3-Based Minor: Glass Trace: Mullite	Al_2O_3-Based	Al_2O_3-Based
Triaxial Support Braid	N720 Major: α-Al_2O_3; Mullite Trace: Glass	N610	N610

a smoother surface which prevented ash adherence, and facilitated removal of fines during pulse gas cleaning.

As shown in Table 4.9, variable layer adherence strengths were observed along any given section of the 3M oxide-based, composite filter elements. Detachment of the outer confinement layer conceivably could easily occur along the 3M CVI-SiC composite elements, as well as along the Nextel™ 550/Nextel™ 610 elements that were manufactured in August 1997. A more strongly adherent outer confinement layer had been applied on both the Nextel™ 550/Nextel™ 720 qualification-tested filter elements, and on the Nextel™ 610/Nextel™ 610 replacement set of elements.

Similarly adherence strength of the outer confinement and filtration mat layers along the triaxial braid was nearly comparable for the 3M CVI-SiC composite elements, and the qualification-tested filter elements. A reduced adherence strength between these layers was evident for the 3M Nextel™ 550/Nextel™ 610 elements that had been supplied to Karhula in August 1997. A substantially enhanced adherence strength between these layers was identified for the replacement set of elements (i.e., Nextel™ 610/Nextel™ 610). The somewhat enhanced adherence of layers after PCFBC exposure may be attributed to ash present within the matrix, or alternately additional reactions which promoted bonding between the matrix and various fiber compositions (i.e., diffusion and/or sintering mechanisms).
As a final characterization effort conducted at Siemens Westinghouse, the various 3M filter elements were subjected to burst strength testing (Table 4.10). Clearly a reduction in bulk strength of the 3M CVI

and oxide-based materials resulted after either qualification testing at Siemens Westinghouse, or after exposure of the elements at Karhula.

TABLE 4.9
LAYER ADHERENCE TESTING OF THE
3M OXIDE-BASED CFCC CANDLE FILTERS

Filter Element	Outer Confinement Layer, lbs (psi)	Outer Confinement Layer & Filtration Mat, lbs (psi)
CVI-SiC Composite		
As-Manufactured	19[(a)]	61 (102), 80 (82)
Hopper TS1-95	5 (46)	53 (76), 67 (33)
W-STC Qualification-Tested	15[(a)], 16[(a)]	35[(b)]
Oxide-Based CFCC		
W-STC Qualification — As-Manufactured N550/720	37 (184)	56 (71), 85 (211)
Karhula-1 — As-Manufactured N550/610	22[(a)]	36 (20), 68 (85)
Karhula-1 — TS2-97 Tested (35 Hrs) N550/610	19 (93), 23 (46)	55 (55), 59 (296)
Karhula-2 — As-Manufactured N610(fine)/N610	34[(a)], 78[(a)]	107[(c)], 128[(c)]

(a) Outer mesh failed without any removal of the outer confinement layer.
(b) Area not identified due to the overall failure of the specimen during testing.
(c) Deformation of the cylindrical sample without debonding during testing.

TABLE 4.10
3M OXIDE-BASED CFCC CANDLE FILTER MATERIAL PROPERTIES

Filter Element	Burst Pressure, psi	Ultimate Hoop Stress, psi	Modulus, psi x 10^6	Poisson's Ratio
CVI-SiC Composite				
As-Manufactured		1.01 ksi	2.96-3.38	0.14-0.27
TS2-96 — 387 Hrs	133	1179	3.35	0.22
TS2-96 — 626 Hrs	105	946	5.59	0.34
Oxide-Based CFCC				
As-Manufactured Filter Elements for Qualification Testing	52	586	1.36	0.73
Qualification-Tested Filter Element	29	334	1.35	0.73
Karhula-1 As-Manufactured	64	572	6.0	0.27
Karhula-1 TS2-97 Tested	54	443	1.8	0.61
Karhula-2 As-Manufactured	118	1460	6.3	0.32

Based on the information generated at Siemens Westinghouse, as well as at 3M, it became apparent that the 3M oxide-based CFCC candles provided for PCFBC testing in August 1997, were manufactured in such a manner that limited 'uptake' of the matrix via the Nextel™ 610 triaxial support braid to provide adequate bonding of the fibers and adherence of the filtration mat and outer confinement layers. As a result during pulse cleaning, gas passed through the triaxial braid, and further debonded the filtration mat in localized areas along the filter wall. Detachment of the filtration mat layer and removal through the weakened open mesh outer confinement layer lead to tearing of the confinement layer, and ultimately removal of large sections of both brittle surface layers along the filter body.

Although the Nextel™ 610/Nextel™ 610 candles which had been manufactured as the replacement set of elements for use in the final PCFBC test segment were considered to be significantly improved in terms of surface layer adherence, the perceived risk of possible additional failure precluded installation and continued testing of the 3M elements during the final test segment in 1997.
Since ash fines had been detected in the outlet gas stream after 40 hours of PCFBC operation, all elements in the bottom array were removed, and cleaned prior to reinstallation. During removal, one of the DuPont PRD-66 filter elements was broken at the base of the flange. This resulted due to the tight fit when ash became wedged in between the flange and filter holder mount. The broken element was replaced with an alternate, newly manufactured DuPont PRD-66 filter. Post-test inspection of the McDermott elements indicated that localized areas of the Saffil and alumina-enriched sol-gel matrix were removed adjacent to and below the outer Nextel™ 610 filament surface. During cleaning of the McDermott elements, the relatively soft matrix led to "pull-out" of material and/or removal of broken Nextel™ 610 filaments. No apparent damage was experienced by the Blasch candles during either PCFBC testing or cleaning of the elements.

Once the bottom filter array and candle were cleaned (i.e., vacuum brushing; water washing), the elements were reinstalled within the array. Coors P-100A-1 alumina/mullite, Schumacher Dia Schumalith FT20, and Pall 326 elements were installed as replacements for the failed Techniweave and 3M candles. PCFBC testing was then reinitiated and continued for an additional 199 hours of operation at 700-750°C (1290-1380°F) using the Lakeland feed materials.

After 239 hours of service operation, testing was terminated and the unit was slow cooled, prior to inspection of the three filter arrays. Ash bridging was not observed. During this planned outage, additional candles were installed to fill the bottom array. Testing was reinitiated and continued for an additional 342 hours of operation at temperatures of 700-750°C (1290-1380°F), again utilizing the Lakeland feed materials.

After completion of the PCFBC test campaign in 1997, the vessel was slow cooled, opened, and the filters were subsequently inspected. Post-test inspection of the filter arrays clearly indicated that ash bridging had not occurred when the Lakeland feed materials were utilized, and when the filter vessel was operated at lower temperatures (Figure 4.11). The thickness of the dust cake layer along the surface of the top array filter elements was ~2-3 mm, while an ~2-5 mm thick dust cake layer remained along the outer surface of the middle array elements, and an ~2-3 mm dust cake layer remained along the outer surface of the bottom array elements. All elements were removed from the various arrays, permeability tested, cleaned, and subjected to dimensional inspection, and time-of-flight measurements.

With the exception of crack formation along the densified insert plug along the end cap of a Pall 326 filter element, and scratches along the membrane of the clay bonded silicon carbide filter elements due to contact with the false bottom metal plates, all elements were intact at the conclusion of PCFBC testing in 1997. Although divot formations were not observed along the outer surface of the DuPont

Figure 4.11 — Siemens Westinghouse Advanced Particulate Filtration system at the conclusion of testing at the Foster Wheeler PCFBC test facility in 1997.

PRD-66 filter elements, cleaning and handling frequently lead to the formation of minor abrasions along the outer surface of the DuPont, as well as McDermott elements.

Once again, localized removal of the matrix and fibers along the outer surface of the McDermott candles was identified at the conclusion of PCFBC testing in 1997. In addition, fibers along the i.d. wall of the McDermott elements were infrequently seen to be torn, dangling into the i.d. bore of the elements, most likely as a result of insufficient bonding and adherence during pulse cleaning. Possible thinning along the PCFBC-exposed Blasch end cap tips resulted in the vicinity of the plug inserts used to cap and seal the filter elements.

Select commercially available Coors P-100A-1, Pall 326, and Schumacher FT20 monolithic filter elements, and advanced McDermott composite, DuPont PRD-66 filament wound, and advanced monolithic Blasch filter elements were sent to Siemens Westinghouse for use in our material surveillance characterization effort. One element of each filter type was subjected to permeability testing, residual strength characterization, and phase composition and microstructural analyses. Extended life testing was conducted on one element of each PCFBC-exposed filter type in order to demonstrate their performance during continued operation under simulated PFBC steady state operating conditions. Each element was subsequently subjected to accelerated pulse cycling and thermal transient conditions which were representative of extended commercial operation. In this manner, the thermal fatigue characteristics of the various monolithic and composite matrices were identified.[3]

5. PROGRAM SUMMARY

As efforts were focused by the Department of Energy's National Energy Technology Laboratory (DOE/NETL) on development, and by industry on commercialization of the hot gas cleaning technology for use in advanced coal-based systems, development and manufacture of the critical component — the candle filter element — progressed from fabrication of porous ceramic nonoxide-based monolithic matrices, through generation of porous ceramic oxide-based monolithic matrices, to production of advanced, second generation, fracture toughened oxide- and nonoxide-based continuous fiber ceramic composites and filament wound architectures. In order to demonstrate long-term thermal, chemical, and mechanical stability of the advanced second generation candle filter materials, Siemens Westinghouse initiated high temperature, bench-scale, corrosion testing of 3M's CVI-SiC and DuPont's PRD-66 mini-candles, and DuPont's CFCC SiC-SiC and IF&P Fibrosic™ coupons under simulated, pressurized fluidized-bed combustion (PFBC) conditions in 1994. Testing was conducted at temperatures of 870°C (~1600°F) and at atmospheric pressure, for periods of 400 hours in which 5-7%steam/air or 20 ppm NaCl/5-7% steam/air flowed-through each porous ceramic filter matrix. Each matrix was similarly subjected to pulse cleaning cycles representative of typical plant operating conditions.

The high temperature strength and fracture toughness characteristics of the 3M CVI-SiC and DuPont SiC-SiC CFCC matrices decreased during bench-scale corrosion testing. This primarily resulted from

- Removal of the interface layer that was deposited between either the 3M Nextel™ or DuPont Nicalon™ fibers, and the SiC structural support overlayer
- Oxidation of the encapsulating SiC structural support, as well as oxidation and volume expansion of the DuPont Nicalon™ fibers
- Bonding of the 3M Nextel™ and oxidized DuPont Nicalon™ fibers to the overlying oxidized SiC structural support matrix via formation of an amorphous silica melt which lead to embrittlement of the nonoxide CFCC filter matrices.

Gas flow resistance through the DuPont SiC-SiC CFCC filter matrix also increased as a function of high temperature, bench-scale, corrosion test exposure time as a result of pore closure through the formation of a silica-enriched melt which coalesced the fine oxidized SiC grit contained in the outer surface membrane.

A slight increase in strength was observed when the DuPont filament wound PRD-66 filter matrix was exposed to bench-scale corrosion testing at Siemens Westinghouse. Although the stability of the oxide-based DuPont PRD-66 matrix was retained, numerous longitudinal cracks and removal of sections of the hoop wrapped membrane resulted during bench-scale corrosion testing of the DuPont mini-candles.

In both the high temperature, flow-through, bench-scale, corrosion and simulated pressurized fluidized-bed combustion (PFBC) test efforts, tearing of the IF&P Fibrosic™ vacuum infiltrated, chopped fibers and/or failure of the filter element resulted. During 400 hours of exposure in the high temperature steam/alkali/air environment, gas phase sodium tended to be sorbed within the aluminosilicate-enriched fibers contained along the membrane coated surface of the IF&P Fibrosic™ filter matrix. Although a chemical gradient resulted across the porous filter disc, negligible microstructural changes were observed within the IF&P Fibrosic™ chopped fiber matrix.

In order to evaluate the mechanical and filtration performance of the advanced second generation filter elements, arrays of 1.4-1.5 m 3M CVI-SiC, DuPont PRD-66, DuPont SiC-SiC, and IF&P Fibrosic™ candles were subjected to steady state process operating conditions, increased severity thermal transients, and accelerated pulse cycling test campaigns which represented ~1760 hours of equivalent filter operating life in Siemens Westinghouse's bench-scale PFBC test facility in Pittsburgh, Pennsylvania.

As a result of the filter component architecture, the longitudinal seam of the DuPont SiC-SiC candle ruptured, permitting fines to penetrate through, and to be released into the clean gas stream. Based on the resulting load vs deflection curves that were generated during c-ring strength testing, the fracture toughness of the DuPont SiC-SiC filter element appeared to have been reduced after 42 hours of PFBC thermal transient testing. Loss of fracture toughness was once again primarily attributed to removal of the interface layer in the single-ply felt and mesh screen support layers, and to the bonding of the Nicalon™ fibers to the adjacent oxidized encapsulating DuPont SiC-SiC structural support. Since construction of an integral DuPont SiC-SiC filter element was not deemed to be feasible, and the stability of the silicon carbide matrix in high temperature PFBC environments was clearly limited, further development of the DuPont SiC-SiC filters was terminated.

During accelerated life testing, through-hole formation and fracture of the IF&P Fibrosic™ candle was observed after 1645 pulse cleaning cycles (~823 hours of equivalent operating life) had been delivered to the filter array. Further inspection of the i.d. surface of the IF&P Fibrosic™ candle near the flange indicated that the surface of the infiltrate that was used to provide additional strength along the flange had partially separated from the support matrix. In contrast with the high temperature, bench-scale, flow-through, corrosion testing where the pulse gas directly contacted the nonmembrane coated surface of the IF&P Fibrosic™ matrix, a baffled plenum prevented direct impingement of the pulse gas along the i.d. bore of the filter element during simulated PFBC testing. Based on these results, Siemens Westinghouse recommended that if low load bearing, chopped fiber matrices as the IF&P Fibrosic™, Foseco or Scapa Cerafil™ candles were to be used in our Advanced Particulate Filtration (APF) systems, changes would be required to accommodate lower intensity pulse cleaning, as well as delivery of the pulse jet into the i.d. bore of the filter elements. This would limit incorporation of the "soft" matrix candles within our standard array containing either first generation monolithic or advanced second generation CFCC and filament wound filter elements.

After removal of the failed IF&P Fibrosic™ candle from the filter array, testing was reinitiated at 843°C (1550°F), and continued until 3514 pulse cleaning cycles (i.e., 1757 hours of equivalent operating life) had been delivered to the filter array. Subsequently ash was fed for an additional eight hours in order to develop a dust cake layer along the outer surface of each of the remaining filter elements. Post-test inspection indicated that with the exception of the 3M CVI-SiC composite filter, all of the remaining filter elements were intact. Failure at the base of the flange of the 3M CVI-SiC composite matrix resulted from extended pulse cycling which partially dislodged the primary gasket from its original position in the filter holder mount.

Siemens Westinghouse subsequently participated in material surveillance programs during operation of our Advanced Particulate Filtration (APF) system at the American Electric Power (AEP) Tidd Demonstration Plant in Brilliant, Ohio.[4] During early test campaigns at AEP, failure of the nonoxide- and oxide-based monolithic filter elements resulted from ash bridging within the filter arrays. In July 1994, three fractured toughened 3M CVI-SiC CFCC and three DuPont filament wound PRD-66 candles were installed within the Siemens Westinghouse APF. After 1015 hours of operation, two of the

[4] Effort conducted by Siemens Westinghouse under separate contract with the American Electric Power Service Corporation, DOE Contract No. DE-FC21-89MC26042.

3M filter elements failed at the base of their flange as a result of ash bridging. The third 3M filter element remained intact, and achieved 1705 hours of PFBC operation.

Post-test room temperature, gas flow resistance measurements of the intact PFBC-exposed 3M filter indicated that the pressure drop across the element was ~220 inches of water gauge (in-wg) at a gas face velocity of 10 ft/min. The room temperature pressure drop across the ash coated 3M filter was relatively high in comparison to alternate filter elements that were exposed to similar PFBC test conditions. This was attributed to the adherence of the dust cake along and within the outer confinement layer, as well as through the filtration mat and triaxial support braid.

Characterization of the 3M CVI-SiC matrix via scanning electron microscopy/energy dispersive x-ray analysis (SEM/EDAX) indicated that minor changes in the morphology of the filter matrix had occurred after 1705 hours of operation in the 660-760°C (1220-1400°F) Siemens Westinghouse APF system at AEP. Due to deposition of the <1 μm interface coating, it was frequently difficult to discern whether the interface coating had remained intact. A gap or separation was considered to exist between the CVI-SiC coating and the underlying Nextel™ 312 fibers in several areas of the PFBC-exposed 3M composite filter matrix. In the high temperature PFBC environment, oxidation of the interface coating was expected to have occurred.

After 1705 hours of operation, all three DuPont filament wound PRD-66 filter elements remained intact. Post-test characterization of the PRD-66 matrix indicated that the bulk strength of the ash-filled matrix tended to increase after 1705 hours of operation at AEP.

Prior to continued testing at AEP, modifications were made by Siemens Westinghouse to the gasketing and filter holder mount assembly for sealing the 3M CVI-SiC candle filters, in order to mitigate failure of the composite filter flange. In January 1995, ten 3M CVI-SiC CFCC and twenty-two DuPont filament wound PRD-66 candle filters were installed in the Siemens Westinghouse APF. After 1110 hours of filter operation at temperatures ranging between 760 and 845°C (1400 and 1555°F), testing at AEP was completed. Post-test inspection indicated that all ten of the 3M CVI-SiC composite filter elements generally remained intact. A 25.4 mm diameter hole was evident along the outer confinement layer of one of the 3M filter elements. The location of the removed confinement layer was ~75 mm below the flange.

In contrast, only two of the twenty-two DuPont PRD-66 filter elements remained intact, four had suffered either mid-body fracture or failure at a location that was ~1125 mm below the flange, and 16 filters had fractured at the base of the flange. The outer surface of the intact and fractured filters was generally ‘ash free’, particularly along the portion of the body that was adjacent to the plenum support pipe, and to approximately mid-way down the length of the filter element. Alternately, a 1-2 mm ash deposit remained along the outer surface of the PRD-66 candles, near the bottom end cap. Areas of the outer membrane and underlying support fibers were removed in ‘divot-like’ line formations along the surface of the remaining intact and fractured filter elements. Localized ‘divoting’ was also observed below the gasket sleeve that was installed around the filter flange, and along alternate isolated areas of the filter body.

Based on these results, and characterization of the 2815 hours of exposure of 3M coupons above the Siemens Westinghouse tubesheet at AEP which identified oxidation of the CVI-SiC matrix, 3M introduced their oxide-based CFCC candle for consideration in future advanced coal-based test programs. Similarly, DuPont focused their efforts on development of a mechanically enhanced o.d. surface membrane layer which not only limited fines penetration into the bulk filter wall, but also remained

intact, mitigating divot formation during process operation. Strengthening of the flange and end cap areas was additionally undertaken at DuPont.

In parallel test efforts, advanced coal-fired testing continued at Foster Wheeler's pressurized circulating fluidized-bed combustion (PCFBC) test facility in Karhula, Finland. Prior to installation of the advanced second generation filter elements at Karhula, Siemens Westinghouse conducted an extended, accelerated life, qualification program in the spring of 1997, evaluating the performance of the 3M, McDermott, and Techniweave oxide-based CFCC filter elements, modified DuPont PRD-66 elements, and the Blasch, Scapa Cerafil™, and Specific Surface monolithic candles in our bench-scale PFBC simulator in Pittsburgh, Pennsylvania. In order to accommodate for variation in the flange geometry and proper sealing, Siemens Westinghouse designed filter holder mount inserts and/or gasket sets tailored to fit each of the advanced second generation filter elements for use in our existing APF test facilities.

One candle of each advanced second generation filter type was installed within the Siemens Westinghouse PFBC simulator array. The filter array was initially subjected to 120 hours of steady state filtration testing, and subsequently 2200 accelerated pulse cycles (i.e., 1100 equivalent exposure hours), and 12 thermal transients typical of commercial plant operation. Based on the dust loading detected via isokinetic sampling after completion of five thermal transients, testing was suspended and the array was slow cooled in order to determine whether an element and/or gasket seal had failed. Inspection of the array indicated that a 1 m Techniweave Nextel™ 610 filter element had failed at the base of the flange. The failed element was removed from the array, its position was blanked, testing was reinitiated, and the array was subjected to seven additional thermal transient events. After slow cooling of the array and opening of the pressure vessel, all elements appeared to be intact.

In order to select candles for possible inclusion in the Siemens Westinghouse filter cluster at the Foster Wheeler PCFBC test facility in Karhula, Finland, any element which did not remain intact during qualification testing, or could not be manufactured to Siemens Westinghouse's dimensional tolerance and design/performance specifications was eliminated from further consideration for testing in the fall of 1997. As a result, the Specific Surface element which exhibited an initial high gas flow resistance, and did not achieve either the flange or filter body dimensional tolerances was eliminated. When the qualification-tested Specific Surface element was subjected to c-ring compressive or tensile strength characterization, the 3-D printing process inherently established extensive *in-situ* delamination sites within the filter matrix. During final high firing of the elements at Specific Surface, the filters typically formed several cracks which needed to be prepared (i.e., bonding and sealing) prior to consideration for possible use.

The Scapa Cerafil™ element similarly was not recommended by Siemens Westinghouse to be included in the Foster Wheeler PCFBC fall 1997 test campaign due to the low strength and load bearing capabilities of the vacuum infiltrated matrix, and the formation of the longitudinal crack along the filter body after being subjected to qualification testing. In addition, the standard Scapa Cerafil™ element lacked the appropriate flange contour for retrofit into Siemens Westinghouse's metal filter holders, and individual hand cutting and densification of elements was not considered by Siemens Westinghouse to be efficient or cost effective. Similarly shear failure along the Techniweave Nextel™ 610 candle flange raised concern as to associated risks involved with utilizing a nonintegrally constructed filter element in long-term field test campaigns.

Based on the qualification testing conducted by Siemens Westinghouse in 1997, and implementation of production modifications by the various candle filter suppliers, eight 1.5 m DuPont filament wound PRD-66, eight 1.5 m McDermott oxide-based CFCC, eight 1.5 m 3M oxide-based CFCC,

two 1.5 m Techniweave oxide-based CFCC, and eight 1.5 m oxide-based monolithic Blasch candle filters were manufactured and delivered for use in Karhula.

During the second PCFBC test segment at Karhula in 1997, the advanced composite and monolithic candles were installed in the bottom array. The filter was initially operated for a period of 40 hours at temperatures of 700-720°C (1290-1330°F; i.e., projected Lakeland operating conditions) utilizing the Lakeland feed materials, prior to detecting dust in the outlet stream. Testing was subsequently terminated and the vessel was cooled. Post-test inspection of the filter array indicated that the two Techniweave Nextel™ 720 oxide-based CFCC and seven 3M oxide-based Nextel™ 610 CFCC elements had experienced severe damage during the 40 hours of PCFBC operation. Close inspection of the Techniweave filter elements indicated that sections of the outer membrane, through-thickness fibers were removed, and debonding of the outer seam and unwrapping of the 2-D fabric wrap or layered architecture resulted. Pinholes as a result of through-thickness fiber removal permitted ash fines to pass from the o.d. to i.d. surfaces of the PCFBC-exposed filter elements. Similarly the 3M oxide-based CFCC elements experienced removal of sections from both the outer confinement and filtration mat layers, again permitting fines to pass from the o.d. to i.d. surfaces of the PCFBC-exposed filter elements. Sections of material beneath the confinement layer were also seen to be removed along the end caps of the 3M oxide-based CFCC filter elements. Although the Techniweave and 3M elements suffered severe damage during the 40 hours of PCFBC operation, all elements remained attached to the metal filter holder mounts.

Since ash fines had been detected in the outlet gas stream after 40 hours of PCFBC operation, all elements in the bottom array were removed, and cleaned prior to reinstallation. During removal, one of the DuPont PRD-66 filter elements was broken at the base of the flange. This resulted due to the tight fit when ash became wedged in between the flange and filter holder mount. The broken element was replaced with an alternate, newly manufactured DuPont PRD-66 filter. Post-test inspection of the McDermott elements indicated that localized areas of the Saffil and alumina-enriched sol-gel matrix were removed adjacent to and below the outer Nextel™ 610 filament surface. During cleaning of the McDermott elements, the relatively soft matrix led to “pull-out” of material and/or removal of broken Nextel™ 610 filaments. No apparent damage was experienced by the Blasch candles during either PCFBC testing or cleaning of the elements.

Once the bottom filter array and candle were cleaned (i.e., vacuum brushing; water washing), the elements were reinstalled within the array. Coors P-100A-1 alumina/mullite, Schumacher Dia Schumalith FT20, and Pall 326 elements were installed as replacements for the failed Techniweave and 3M candles. PCFBC testing was then reinitiated and continued for an additional 199 hours of operation at 700-750°C (1290-1380°F) using the Lakeland feed materials.

After 239 hours of service operation, testing was terminated and the unit was slow cooled, prior to inspection of the three filter arrays. Ash bridging was not observed. During this planned outage, additional candles were installed to fill the bottom array. Testing was reinitiated and continued for an additional 342 hours of operation at temperatures of 700-750°C (1290-1380°F), again utilizing the Lakeland feed materials.

After completion of the PCFBC test campaign in 1997, the vessel was slow cooled, opened, and the filters were subsequently inspected. Post-test inspection of the filter arrays clearly indicated that ash bridging had not occurred when the Lakeland feed materials were utilized, and when the filter vessel was operated at lower temperatures

6. CONCLUSIONS

Many of the filter elements discussed in this report continued to undergo further development, as well as additional field test exposure. However, with respect to the development and introduction of the advanced second generation filter elements between 1994 and 1997, the oxide-based McDermott CFCC and DuPont filament wound PRD-66 filter matrices demonstrated the most promise for extended use in advanced coal-based power systems. As oxides, limited microstructural changes would be expected to occur during extended exposure to high temperature PFBC or PCFBC environments. Provided that each filter is manufactured with sufficient flange strength, complete sealing of the elements within the filter holder mounts is maintained during process operation, and mitigation of membrane spalling can be accomplished, successful long-term operation of the advanced second generation filter elements in commercial, high temperature, particulate removal systems can be realized.

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