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Mechanical Analysis of a Cross Flow Filter

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*In memory of Dr. David F. Ciliberti and
Mr. Randall J. Dellefield who pioneered the
development of the cross flow filter technology.*

Mechanical Analysis of a Cross Flow Filter

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Advanced Fossil Energy Technologies

January 31, 1995

ABSTRACT

At Westinghouse, the ceramic cross flow filter was developed for particulate control in applications for high temperature, high pressure (HTHP) systems such as pressurized fluidized-bed combustion (PFBC), and integrated gasification combined cycle (IGCC) power generation. The objectives of this program were to improve the reliability and mechanical integrity of the basic porous ceramic filter element. The scope of the activities included improving the filter manufacturing process, the filter element design configuration, and the ability to effectively mount and seal the filter element in an operating system. The effort was focused primarily on the development and manufacture of the Coors cross flow filter which was fabricated from an alumina/mullite matrix using a stacked-plate construction.

Manufacture and design iterations performed as part of this program were evaluated, in part, in companion durability and qualification programs, as well as in field test programs which have been reported separately. Durability and field test results are summarized for clarity, where appropriate.

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MECHANICAL ANALYSIS OF A CROSS FLOW FILTER

SUMMARY

Development of the cross flow filter element was initiated at Westinghouse in the late 1970's. The cross flow filter concept emerged from two existing market segments — the 3M and Grace internal combustion engine exhaust catalytic support structures, and the Coors Ceramics Company and GTE regenerative heat exchanger technology (Figure S.1). In order to demonstrate concept feasibility, sub-scale 6x6x2 inch cross flow filters were initially manufactured and tested in pressurized fluidized-bed combustion (PFBC) test facilities at the Argonne National Laboratory (ANL), in the DOE/METC gasification and in the Westinghouse high temperature, high pressure (HTHP) combustion simulator test facilities.⁽¹⁾

Scaleup of the 6x6x2 inch cross flow to a 12x12x4 inch unit was initiated during 1986 and 1987. During manufacturing at Coors, several issues arose. These included manufacturing the 12x12x4 inch filter element with minimal distortion, deformation or slumpage; achievement of the final geometric tolerances accounting for shrinkage of the ceramic filter body during high firing; and achieving strong and continuous bonds between the stacked rib-to-plate configuration. Subsequent efforts at Coors focused on modifying the cross flow filter design and manufacturing process to produce commercial filter elements. In parallel with the efforts at Coors, production of full-scale 12x12x4 inch GTE cross flow filters was undertaken. Structural analyses were also conducted to identify potentially high stress regions in the cross flow filter configuration, particularly during pulse cycling.

The performance of the 12x12x4 inch cross flow filter was assessed by Westinghouse in various test programs conducted at the Science and Technology Center, as well as in the field. Testing indicated that the cross flow filter performed as an absolute barrier for removal of ash fines, and had acceptable pressure drop characteristics. Testing also indicated that the cross flow filter could be effectively cleaned by simple reverse pulse jet methods. The performance of the cross flow would, however, be compromised should the filter delaminate, or if a breach in the gasket or mounting seal occurred during process operation. Effort in this program focused on resolving the delamination and associated gasketing issues which were identified during conduct of various Westinghouse qualification and/or field test programs.

Figure S.1 — Ceramic Cross Flow Filter Concept

As efforts continued at Coors to manufacture commercial-scale cross flow filter elements, the mid-rib bond concept emerged. This design moved the plate bonds to low stress areas and improved the dimensional tolerances of the entire cross flow filter unit. During 1988, manufacturing process parameters as firing temperature, firing schedule, and filter weighting were explored to improve bond strength within the filter element. Testing both at Westinghouse and at the New York University PFBC test facility showed improvement, but a lingering susceptibility to delamination. Subsequent filter manufacturing modifications during 1989 included eliminating stress riser areas along the flange and channel corners, and grinding or machining the surfaces of the filter elements to tighter tolerances for improved dimensional compatibility with the mounting fixture.

Additional process modifications continued to be made at Coors in 1990 which included refining the firing cycle to improve bond strength, as well as exploring the use of alternate pore former materials. Filters which had been manufactured using the 1989-1990 process improvements underwent qualification testing at Westinghouse and field operation in the Texaco reentrained gasification system. Still plagued with the potential for delamination, efforts in 1991-1992 focused on improving the mid-rib bond strength, enhancing the as-manufactured strength of the matrix, and ultimately eliminating the use of pore former materials during production of the full-scale cross flow filter elements. In-house testing at Westinghouse during 1992-1993 demonstrated successful operation of the cross flow under steady state simulated PFBC conditions, and during exposure to thermal transient events. With the apparent improvements in bonding, particularly between the mid-rib bonds and plate seams, the cross flow filter was targeted for future installation and testing in advanced coal-fired demonstration sites.

As a direct transfer of the cross flow filter materials technology, Coors Ceramics successfully manufactured 1.0 m and 1.5 m candle filters during 1990-1991. Pre-qualification testing at Westinghouse provided the basis for utilization of the Coors candle filters in the pressurized circulating fluidized-bed combustion (PCFBC) gas environment at the Ahlstrom test facility in Karhula, Finland. Testing of the alumina/mullite material at pilot plant facilities not only provided the opportunity to accelerate development of the porous ceramic filters, but also provided exposure of the material to actual process gas operating conditions where the response of the matrix could be evaluated, and if necessary, modified.

In conjunction with these efforts, a ceramic materials characterization effort was undertaken at ANL in 1990-1991 to develop the fundamental material properties of the as-manufactured cross flow filters, and similarly for filters which had been operated in the Texaco gasification environment. In 1992-1993, ANL conducted a second materials property evaluation of the Coors filter matrix, utilizing candles which had been subjected to operation in the Karhula PCFBC environment.

In parallel with these efforts, direction was focused on the manufacture of a monolithic cross flow filter element. Based on existing alternate technologies, an injection molding process developed at Blasch Precision Ceramics, and the chemically vapor infiltrated (CVI) fiber reinforced ceramic composite technology developed by DuPont Lanxide were considered to be viable approaches to mitigate the delamination issues experienced in the Coors sintered stacked-plate configuration. Efforts at Blasch during 1992-1993 demonstrated the relative ease of manufacturing a dense 12x4x1 inch prototype monolithic cross flow filter section. Similarly, DuPont in 1992-1993 manufactured a porous 6x6x2 inch cross flow unit. Ease of scaleup to the 12x12x4 inch full-scale cross flow filter element is expected via both manufacturing approaches.

During the later stages of development of the cross flow filter at Coors, nondestructive evaluation techniques were utilized to assess the process modifications which were implemented during production of the filter element. Similarly, the addition of a membrane coating was developed at Coors, emphasizing application of a finer grain, contiguous layer along the outer surface of the candle filter. With many of the advancements which occurred during the manufacture and design optimization of the cross flow and candle filters, an alternate sheet or second generation filter concept emerged (Figure S.2). As a simple design, the sheet filter offers many attractive advantages which include improved packaging within a pressure vessel, ease of manufacturing, and potentially robust mechanical integrity based on its compact geometry.

As porous ceramic barrier filters, the cross flow, candle and yet to be qualified sheet filter concept provide viable alternatives for high temperature particulate removal in advanced coal-fired combustion or gasification process applications.

Figure S.2 — Porous Ceramic Alumina/Mullite Filter Elements

1. INTRODUCTION

The porous ceramic cross flow filter was developed by Westinghouse for use in high temperature, high pressure (HTHP) particulate control in applications as Pressurized Fluidized Bed Combustion (PFBC), and Integrated Gasification Combined Cycle (IGCC) Power generation systems. In the development of the cross flow filter element at Coors Ceramics, GTE, and AiResearch, thin porous ceramic plates which contained perpendicular ribs, formed the gas path channel network in the filter (Figure 1.1). After machining, the porous ceramic plates were stacked and fired at high temperature to form the porous cross flow filter structure (Figure 1.2).

During hot gas filtration, process gas containing particulates pass into the short, dirty side channels of the cross flow filter. Gas then passes through the porous ceramic plates that form the "roof" and "floor" of the channels and into the longer channels that form the clean gas passage (Figure 1.3). One end of the clean side channels is sealed to force the filtered gas to flow to a central collection plenum where the filter is mounted. Dust collected in the short side channels is periodically removed by reverse pulse cleaning. The major attributes of this filter concept are its absolute particulate filtration efficiency, and its capability to be operated at relatively low pressure drop. Since each filter plate represents a filter surface, the compact cross flow configuration has a very high filter surface area to volume ratio and is potentially economical.

In the late 1980's several ceramic materials were identified to have excellent mechanical properties (i.e., high temperature strength), and thermal shock and corrosion resistance for use in construction of the cross flow filter.⁽²⁾ These included the following oxide and nonoxide materials which were utilized to manufacture either 6x6x2 inch or 12x12x4 inch cross flow filter elements:

- Coors alumina/mullite
- GTE cordierite
- GTE cordierite-silicon nitride
- Foseco aluminosilicate foam
- AiResearch reaction bonded silicon nitride
- AiResearch sintered silicon nitride.

Figure 1.1 — Thin Porous Ceramic Plates in the Cross Flow Filter

Figure 1.2 — Commercial-Scale Cross Flow Filter

Figure 1.3 — Schematic Representation of the Cross Flow Design and Use as a Filter Element

The Coors alumina/mullite cross flow filter was fabricated by isostatically pressing 1/2-3/4 inch thick plates of the powdered raw materials, and then machined to form the ribbed structure of the plate configuration. High firing of the stacked plates ultimately bonded the ribs and seams, forming the dirty and clean gas channels of the sub-scale or full-scale cross flow filter units. The manufacturing process utilized by Coors provided the added flexibility of incorporating a flange into the "green" body, forming an integral body-flange configuration.

In contrast GTE and AiResearch manufactured cross flow filters via an extrusion process. After extrusion and machining, the porous ceramic plates were subsequently fired at high temperature to bond adjacent plates together. With respect to the GTE cross flow, the flange was bonded to the filter body during final high firing, while the AiResearch cross flow filter was manufactured with an integral body-flange configuration.

Alternately Foseco utilized vacuum infiltration to fabricate blocks of aluminosilicate foam. Parallel and perpendicular channels were then machined into the foam to form the cross flow filter configuration.

With many of the delamination issues which were encountered when the stacked-plate design was utilized in either simulated or field applications, it became readily apparent that development of a monolithic cross flow structure was essential. Several alternatives which were developed included the use of an injection molding process developed at Blasch Precision Ceramics, as well as the chemical vapor infiltration (CVI) process which deposited silicon carbide along ceramic felt preforms at the DuPont Lanxide Corporation. With both the injection molding and CVI-SiC processes, seamless monolithic cross flow filter elements could be manufactured which mitigate the delamination issues experienced along the rib and/or seams in the original stacked-plate cross flow filter design.

2. CROSS FLOW FILTER CONCEPT DEVELOPMENT AND TESTING

The ceramic cross flow filter was developed to provide a viable, highly efficient, particulate control option for use in advanced high temperature Pressurized Fluidized-Bed Combustion (PFBC) and Integrated Gasification Combined Cycle (IGCC) power generation systems. The cross flow filter consists of thin porous ceramic plates that contain channels formed by ribbed sections. The plates are stacked and fired to form a near monolithic porous structure.

Typically Westinghouse focused its attention on the development and manufacture of cross flow filters that were fabricated from an alumina/mullite-based matrix. Development efforts included scaleup of the filter elements from a 6x6x2 inch configuration which contained rib-to-plate bonds, to a 12x12x4 inch body that utilized a mid-rib bond configuration (Figure 2.1). The mid-rib bond provided a symmetric plate design which was perceived to have improved firing characteristics, and which simultaneously eliminated high stress, sharp channel corners in the rib-to-plate bond.

Modifications were also made in the fabrication and manufacturing of the cross flow filter elements to ensure retention of the base material strength and porosity while maintaining a crack-free, dimensionally stable, plate assembly with improved bond strength and uniformity. An additional feature incorporated into the cross flow filter design was the inclusion of a radiused flange section and gas channel corners which eliminated stress risers, and provided a more delamination-resistant filter body.

During the development of the cross flow filter, efforts were focused on material qualification testing which characterized the microstructure and mechanical properties of both as-fabricated and field-tested filters. These data, when used in conjunction with filter element stress analysis, parametrically identified "allowable" filter pulse gas cleaning conditions. Fractographic analysis of field-tested cross flow filter elements also provided insight into potential material related crack propagation mechanisms which served as the basis for critical design modifications, and standardizing production and quality control procedures.

Efforts at Westinghouse were also focused on identifying the long-term, high temperature stability of the cross flow filter materials for use in advanced coal-fired combustion or gasification applications.⁽²⁾ In comparison with alternate porous ceramic cross flow filter materials (i.e., cordierite, aluminosilicate foam, cordierite-silicon nitride, and reaction bonded silicon nitride), the alumina/mullite-based matrix was considered to exhibit a broad range of chemical stability for use in

Figure 2.1 — Mid-Rib Bond Cross Flow Filter Configuration

either an oxidizing or reducing gas environment, to be more chemically resistant to gas phase reactions (i.e., alkali, chloride, or sulfur species); and to maintain its chemical stability in the presence of adhering ash or char fines.

The principal objectives of this program were to improve the reliability, uniformity, and mechanical integrity of the cross flow filter such that the cross flow would be a viable filter device for use in all hot gas cleaning applications. Throughout the course of this program, Westinghouse in conjunction with the Coors Ceramics Company successfully modified the filter design enhancing its operational capabilities through:

- Scaleup of the filter module from a 6x6x2 inch filter to the current 12x12x4 inch geometry.
- The use of mid-rib bonds between adjacent gas channel plates, as opposed to the original rib-to-plate design.
- The incorporation of a radiused flange, as well as radiused gas channel corners.
- The incorporation of a smooth flat surface finish to remove stress risers generated by plate off-sets that resulted during assembly and firing.
- The use of weights during firing to enhance mid-rib bond formation and strength, as well as eliminate surface channel gaps.
- Elimination of the use of pore forming materials which agglomerated during processing and produced large pores and/or pore channels which approached a critical flaw size of ~500 μm .

The following sections provide a review of the cross flow filter development from concept through what is now considered to be a commercial stacked-plate product. Iterations in the manufacturing process that were made principally at Coors Ceramics, and subsequent testing performed by Westinghouse to qualify the mechanical durability of the cross flow filter elements are discussed.

2.1 PROOF-OF-CONCEPT TESTING⁽³⁾

Westinghouse received two 6x6x2 inch cross flow filter elements from Coors in the spring of 1985. Testing was initiated in Westinghouse's high temperature, high pressure (HTHP) pressurized fluidized-bed combustion (PFBC) simulator test facilities (Figure 2.2) using one of the filter elements (i.e., WCXF1).

Figure 2.2 — Westinghouse High Temperature Pressurized Fluidized-Bed Combustion Test Facility

The initial filtration properties of the WCXF1 were excellent, with an initial pressure drop across the filter of 1.0 in-wg with a gas face velocity of 10 ft/min. During the second day of test operation at temperatures of 1440°F, incomplete combustion of the natural gas feed resulted, and the filter was inadvertently exposed to an excessive forward flow pressure drop. After cool-down of the Westinghouse HTHP PFBC test facility, an ~1/8 inch thick layer of hard soot coated the surface of the filter element (Figure 2.3). Two large holes in the soot coating were evident which resulted during over-pressurization, causing the filter wall to fracture along two of the central outlet or clean gas channels. The system thermal expansion skirt was crushed inwardly by the pressure force across the tubesheet.

The cross flow filter was washed and brushed to remove the soot, and dried in an oven overnight. After Fiberfrax and a ceramic cement were used to fill the two damaged channels, the cross flow was re-installed in the Westinghouse HTHP PFBC test facility. On May 31, 1985, the system was brought to operating temperatures of 1000°F, and dust feed was initiated at a particle load of ~7,500 ppm. Seven pulse cleaning cycles were delivered to the filter. The overall particle collection efficiency of the element was observed to have decreased from 99.99% to 99.83% on June 3, 1985, and continued to decrease on June 4, 1985 to 99.15%. After slow cooling of the Westinghouse HTHP PFBC vessel, the filter was observed to have delaminated (Figure 2.4), showing a dust leak path only at the delamination site.

The filter was returned to Coors for their evaluation. After examination of the filter, Coors recommended that a higher temperature be used to fire the filter body, and similarly to infiltrate the back wall of the filter body to improve bonding and increase resistance to delamination. Additional longer term approaches which were discussed included modification of the mechanical design of the rib-to-plate bond which served to form the channels between adjacent plates in the filter body.

Similarly during this time frame, DOE received a 6x6x2 inch cross flow filter element for testing in the METC gasifier. The cross flow filter was installed, and on August 9, 1985, the METC hot gas filtration system was heated to temperatures of 950°F. The filter performed with an overall particle collection efficiency of >99.9% during the first 42 hours of test operation. On August 14, 1985, the particle collection efficiency of the filter dropped to 98.45%, and the test facility was slow cooled. Post-test inspection of the filter element indicated that the inner face closest to the clean gas plenum had fractured along the flange surface of the filter element. As a result, the entire flange had broken off, and had allowed some of the gasket material under that section to be torn and blown out during pulse cleaning. The flange was ground to 0.25 inch and returned to Coors so that the back face and flange could be coated with a slip of the parent material, and then refired at a high

Figure 2.3 — Soot Coated Cross Flow after High Temperature, High Pressure Testing
at Westinghouse

Figure 2.4 — Delamination of the Cross Flow Filter during High Temperature, High Pressure Testing at Westinghouse

temperature to improve delamination resistance. The flange was suspected to have been originally cracked during mounting of the filter, and subsequently broke after 50 hours of test operation and 65 pulse cleaning cycles.

Fabrication of two additional 6x6x2 inch filters was initiated at Coors. Flat plates were machined and prepared for firing. Since in the first HTHP PFBC test conducted at Westinghouse, debonding had occurred along all of the cross flow ribs of a clean gas channel, Coors proposed to refire and rebond the delaminated sections. One of the newly machined elements, and the element which was being repaired were first infiltrated with a slip of the parent material along the walls and front flange. Both filters were placed in the kiln and fired together. The PFBC dust which remained along the Westinghouse HTHP-tested filter fluxed the body at the high firing temperature, causing it to slump. Due to the close proximity of the Westinghouse HTHP-tested filter and the newly manufactured filter, the new unit was pushed into a curved shape.

The second filter which was being prepared was then infiltrated with slip and placed in the furnace for high fire. This element was successfully fired at 1530°C without slumping. After successfully demonstrating that the cross flow filter element could be fired at high temperature with the back and flanges infiltrated with slip, Westinghouse returned the second, unused filter element from the first pair that was shipped in the spring of 1985. Coors proceeded to dip the back and front flanges of the filter element in the slip, and then refired the unit at 1530°C. Once again firing of the 6x6x2 inch cross flow filter was successful.

Since the original raw materials used to fabricate the cross flow filter elements at Coors had been depleted, additional material was prepared. In August 1985, cross flow filter plates were pressed and machined. Two filter elements were assembled and fired. Subsequently the filters were dipped in a slip coat, and then subjected to a final high fire.

During firing, two problems were encountered. First the binder that had been used to control porosity had not been ground fine enough which resulted in many sizable voids being distributed throughout the body. Second, with the high firing temperature and the dual firing cycle, one of the filter bodies slumped, and the other developed a ripple.

Prior to shipment of the rippled cross flow filter element to Westinghouse on September 7, 1985, the back side of the flange was ground smooth and parallel to the front face. Similarly several areas along the side plates of the filter element were patched with ceramic adhesive.

METC also received one of the newly fabricated cross flow filters. The filter was initially tested at ambient temperature to determine its as-manufactured pressure drop characteristics. The clean pressure drop was 16 in-wg at a gas face velocity of 11.7 ft/min. This was somewhat higher

than the pressure drop of the previous Coors filters, which implied that porosity and/or pore size were reduced as a result of exposure to the high firing temperature and soak time.

Testing was initiated at 7:45 a.m. at the METC gasifier on September 10, 1985, with the filter vessel being heated in a nitrogen flow. At 400°F, the pressure drop across the filter was ~2.8 in-wg at 1.8 ft/min. Difficulty was encountered when the methane-fired combustor temperature was attempted to be increased. At 5:30 p.m., the filter temperature had increased to 920°F at a flow of 2,280 scfh. At midnight process gas was brought on-line at a flow of 1,050 scfh at 250 psig. At temperatures of 927°F, the pressure drop across the filter under these conditions was ~4 in-wg.

During the next 10.5 hours, the pressure drop across the filter element rose from 4 to 14.3 in-wg as dust was fed to the system at concentrations of 875-1,900 ppm at face velocities of 2.75 ft/min. During this time, the particle collection efficiencies remained relatively constant at 99.5-99.8%.

On September 11, 1985, the ignitor on the gas combustor failed, forcing the system to be shut down for several days. On September 16, 1985, the filter was brought back on-line with nitrogen, and subsequently exposed to process gas on September 17, 1985. The pressure drop rose gradually from 6 in-wg to 10 in-wg with a typical dust inlet concentration of 450 ppm. Operation of the filter continued smoothly until 50 hours had been achieved. The system was then switched to operate on coal gas. During the next 2-3 hours the filter operated without incident, when problems arose in the sampling line, and the unit was shut down and slow cooled.

Post-test inspection of the filter element indicated that the cross flow was in relatively good condition. After removing the accumulated dust from the surface of the filter, delamination had occurred as the result of a crack crossing the back face of the filter element.

A second cross flow filter was installed in the METC gasifier test facility, and testing was initiated on November 6, 1985. The filter was initially heated with nitrogen at 2,200 scfh at 150 psig. Problems were encountered with the gas combustor ignitor which had corroded, and cracks developed in the ceramic casting of the combustor. Both problems were repaired and the filter was subjected to fuel gas.

As in September, the initial pressure drop across the filter element was somewhat higher than the previous filter elements. This was considered to be an indication of reduced porosity and/or pore size resulting from the high firing temperature and soak time during manufacture. Particle collection efficiencies of 99.19% were measured with a dust load of 717 ppm.

As the operating temperature of the filter reached 1200°F with face velocities of 1,725 scfh and inlet dust concentrations of 800-1,750 ppm, particle collection efficiencies were reduced to 98%.

On November 7, 1985, the filter system was shut down due to problems encountered with the gasifier.

The unit was restarted on November 17, 1985 with hot nitrogen. At temperatures of 910°F, pressures of 150 psig, the pressure drop across the filter element was 35 in-wg with a gas face velocity of 1,750 scfh. Product gas was introduced into the filter vessel without dust. Shortly thereafter a leak was discovered in the steam heating lines inside the dust feeder vessel. After repair of the leak, dust fed concentrations of 367-280 ppm were delivered to the filter as opposed to 2,000 ppm. During the next seven hours of operation, fairly short intervals between pulse cleaning resulted, indicating poor cleaning of the filter. Subsequently the outlet sample line had failed, and the test facility was down for repair. In addition, the steam heating line inside the dust vessel had failed again, injecting large quantities of steam into the system.

After repairs had been completed, the system was brought back on-line on November 18, 1985. Problems were again encountered with the dust feeder, and an outlet loading of 22 ppm was recorded. A decision had been made to terminate operation after 50 hours of testing had been completed. After cool-down, the filter was found to be encrusted with a relatively hard, flaky coating of char in contrast to fine powdery dust deposit which typically resulted during a normal operating test run. All of the dirty channels in the 6x6x2 inch cross flow were plugged.

After the deposit had been scrapped from the filter, testing was reinitiated on November 20, 1985. Nitrogen was utilized to preheat the vessel, prior to injecting char. After achieving operating temperatures of 991°F and 150 psig, the gas face velocity through the element was 1,750 scfh. With a dust load of 2,000 ppm, particle collection efficiencies of 98-99.6% were established. Testing continued until shutdown of the filter system was initiated on November 22, 1985. Post-test inspection of the filter indicated that the element was in excellent condition, remarkably free of any adhering dust deposits. After vacuuming, neither cracks nor delaminations were evident.

2.2 NEW CONCEPT CONSIDERATIONS

Because of its configuration (i.e., thin walls; low pressure drop across the as-manufactured filter), the cross flow retains its ability to sustain a high pressure drop during high temperature filtration. As demonstrated in the proof-of-concept testing, the cross flow filters manufactured at Coors had nearly absolute barrier filtration capabilities.

A major technical difficulty which was, however, encountered in the early cross flow filter design was delamination along alternating gas channel layers in the multi-channeled structure. Failure along the layers which formed the channel network was considered to have occurred during pulse jet cleaning of the unit at process operating conditions.

A mechanical analysis of the hydraulic and thermal stresses imposed on the filter during pulse cleaning was initiated in 1986. A finite element code was utilized to produce stress contours that located and quantified regions of high stress within the cross flow filter configuration.⁽⁴⁾ The information generated through the use of the finite element analyses was then used to generate a list of alternate designs and possibly fabrication techniques which would minimize and/or eliminate problem areas in the cross flow filter element.

In the initial finite element analysis, the cross flow filter was characterized as a two-dimensional plane. Static analyses of the unit cell were conducted under pressure loading with the assumption that the material was isotropic with material properties similar to those shown below:

Young's Modulus (E) = 12.5×10^6 psi

Poisson's Ratio (ν) = 0.22

Thermal Conductivity (k) = 12.5 Btu/hr/°F/ft²/in

Coefficient of Expansion (α) = 2.5×10^{-6} in/in/°F

Modulus of Rupture (MOR) = 2,000 psi.

The conditions which were established for the finite element analyses included zero normal displacement along the rib wall and zero rotation (from symmetry) at the mid span of the filter surface.

The radius of curvature in the originally manufactured cross flow filter unit cell was estimated to be 0.0015 inches. Typically the internal pressure within the cross flow channel was estimated to be between 3 and 7 psi during pulse cleaning. With a 10 psi pressure loading, the maximum principal stress was shown to occur at the inner surface of the gas channel corner, and was estimated to be 900 psi. This stress was at a maximum where the tip of the rib bonds (i.e., sinters or fuses) to the adjacent plate. These analyses did not include any thermally induced stress.

Because of the enormous internal heat transfer surface provided by the porous ceramic filter matrix, it was assumed that the gas exited at a temperature which was in equilibrium with the porous solid. The rate at which heat was acquired by the pulse gas as it passed through the clean filter channels was equivalent to the rate that temperature was lost by the porous ceramic filter matrix. A

pulse duration of 100 milliseconds was assumed in the finite element model. Both the gas channel surface and a section of the ceramic matrix below the gas channel surface were expected to experience a temperature drop due to gas cooling. The surface was driven to the lowest temperature (i.e., $\sim 23^{\circ}\text{F}$ drop to 1577°F from an assumed 1600°F). The convection surface dropped to a temperature of 1585°F near the midpoint of the rib. The region where transpiration and convection meet was the area where the highest temperature gradient existed, and consequently was the highest thermally stressed area. This region was also expected to be the highest stressed region due to pressure loading. These combined stresses occur at the rib tip-to-plate joint which was expected to be the weakest location within the filter assembly.

Stress intensities of ~ 900 psi were projected to occur in the sharp corners formed by the rib tip-to-plate joint when the element was pulsed during cleaning. Further stresses occurred due to cooling of the element by the relatively cold cleaning pulse. Thermally induced stress levels of $\sim 1,000$ psi were estimated again at the rib tip-to-plate joint. The combined pneumatic and thermal stresses essentially add, resulting in a cumulative stress of $\sim 2,000$ psi which was approximately the modulus of rupture of the as-manufactured Coors alumina/mullite filter matrix.

With a combined pneumatic and thermal stress of $\sim 2,000$ psi, the useful operating life of the ceramic filter with a corner radius of 0.0015 at the rib tip-to-plate joint appeared to be limited. Since the maximum principal stress was inversely proportional to the square root of the corner radius, increasing the corner radius from 0.0015 inches to 0.020 inches (i.e., factor of 13) reduced the maximum principal stress at the corner radius from 985 psi to 268 psi (factor of 3.6). In this manner the combined pneumatic and thermal stress induced during pulse cycling was reduced to below the modulus of rupture for the as-manufactured filter matrix.

Further refinement of the initial finite element analyses include:

- Construction and assessment of the stress locations within a three-dimensional body as opposed to the two-dimensional unit cell.
- Identification of the actual filter surface and wall temperatures during steady state and pulse cycling. Utilization of these temperatures in the finite element model analyses.

In addition to the recommendations that resulted from finite element modeling efforts that were conducted at Westinghouse, Coors recommended that the following manufacturing improvements be considered and incorporated into production of the 1986 and 1987 cross flow filter elements:

- Production of a body with more uniform green density and springback via improved die filling techniques. Non-uniform springback appeared to produce thicker ends on the pressed plates (i.e., flange and closed top section). Consequently on firing the stacked plates, when weight was applied to the top of the stack, it was not equally applied to the center section of the plate, and as a result bonding along the center section of the filter element was marginal. Another consequence of non-uniform fill was a differential green density in the filter plate(s) which could lead to non-uniform shrinkage and/or non-uniform strength.
- Optimization of the firing cycle in order to maximize rib tip-to-plate bond strength. Once a uniform green density was established via improved die filling techniques, plates were to be bisque fired (i.e., partially sintered), and stacked in pairs for high fire. Various high firing temperatures were considered for evaluation. The bond strength between adjacent plates within the filter element would be measured by burst testing individual filter channels (i.e., pressurized water filled bladder) in order to establish an optimum firing cycle.
- Reduction of the particle size of the slip coat in order to ensure particle/plate infiltration. A slip coating would be applied along the edges of the bisque plates, and along the top of each rib. Similarly the slip would be applied along the outside of the filter channel seams, enhancing bonding of the filter plates.
- Optimization of the slip coating technique(s). Blakite, an alternate alumina bond material which forms a cement-like bond, was initially considered as a potential slip coat material. Efforts were directed to determining whether Blakite cement could be used to form fillets which increased the radius of the rib tip-to-plate joint, and possibly increase the bond strength in the contact area.
- Consideration was given to evaluating mechanical reinforcement techniques (spines) as C-clamp tee and tail, tail bar, tail and tee and dove tailing, for enhanced mechanical integrity of the cross flow filter body.
- Consideration was also given to casting the filter plates with a radius at both ends. This concept was not implemented.
- Incorporating a thicker central rib during production of full-scale, 12x12x4 inch, alumina/mullite (i.e., P-100A) cross flow filter elements. The thicker central rib was expected to minimize the sway-back appearance that frequently resulted during firing when the center of the element sagged to a greater extent than either the flange or top closed end section of the filter element. Similarly the flat plates used in the construction of the filter element would be machined to near perfect flatness before high firing the assembly of plates in order to optimize rib tip-to-plate contact.

The results of the efforts in each of these areas follow.

In efforts to improve bonding between the rib tips and flat plates, the firing temperature of the filter element, as well as the stacked-plate weighting techniques were investigated. Utilizing a higher firing temperature resulted in better bonding between the rib tips and flat plates, and provided

a stronger filter body matrix. The higher firing temperature, however, caused slumping of the filter element.

Consideration was also given to weighting the filter with a higher load applied along the flange and top closed end section of the filter element, and simultaneously with a reduced load along the central gas channel section of the filter element. Firing under these conditions resulted in poor bonding along the gas channel seams which were located between the flange and top closed end section of the filter body.

Efforts were redirected to establishing the optimum load at appropriate firing temperatures which would be needed to mitigate slumping of the filter element. A minimum weight of 84 g/in² was identified in order to obtain bonding within the filter body. Although higher weight loadings up to 126 g/in² were not detrimental to the structural integrity of the filter element, significant improvement in the bond strength was not apparent.

Initial analyses at Coors indicated that when various alumina particle sizes were suspended in a liquid slip, penetration into the ceramic substrate was dependent on both particle size and particle size distribution in the suspension. Efforts were then focused on identifying the extent of bonding along the rib tip-to-plate joint. Although scatter resulted in the data that were generated when a pressurized water filled bladder was used to determine rib tip-to-plate bond strength, the general trend indicated that the slip coated rib bonds were somewhat stronger than the non-coated rib bonds. The strength of non-coated rib bonds was nearly equivalent to the strength of the Blakite-coated rib bonds. As a result, slip coats of the parent matrix would be considered and/or utilized during further production of the Coors cross flow filter elements.

Combining the information generated for the appropriate firing temperature(s), applied weight, and slip coating, full-scale, 12x12x4 inch cross flow filters were manufactured in the following manner:

Number of Firing Cycles	Coating	Firing Level	MOR, psi*
1x	—	High **	1940
1x	—	Low	1800
2x	—	High	2170
3x	—	High	2330
2x	1	High	2480

* 3-Point Bend Flexural Strength

** Temperatures >1430°C.

One filter element which had been fired twice at temperatures of 1430°C was selected for testing in Westinghouse's HTHP PFBC simulator. After ~50 hours of testing, the filter element failed dramatically. Post-test inspection indicated that cracks occurred through the base of the ribs on the dirty gas channels, which migrated through the filter wall to the clean gas channel in a fashion similar to thermally induced stress failures experienced in heat exchangers. The fracture continued from plate to plate, until the crack reached a plate with very little rib tip-to-plate bond strength. The modulus of rupture for the filter channel walls was determined to be ~1,500 psi which was sufficiently lower than the anticipated 2,170 psi strength for simultaneously fired bend bars. Similarly, the filter channel wall modulus of rupture was significantly lower than the 2,500-3,000 psi strength of the standard alumina/mullite filter matrix. Failure of the cross flow filter element during HTHP PFBC testing resulted not only from insufficient strength of the filter matrix, but also from poor bonding between the rib tips and flat plates. A review of the filter firing log indicated that the filter element had been fired at 1430°C rather than at 1485°C in order to minimize slumping of the body. The lower firing temperature was considered to have directly impacted the as-manufactured bulk material strength, as well as rib tip-to-plate bond strength.

Testing of the cross flow filter element that was fired twice at temperatures of 1460°C in Westinghouse's HTHP PFBC simulator was, however, successful. The filter was subjected to ~250 hours of operation including 30 cold start/shutdown cycles and more than 1,000 pulse cleaning cycles. Improved cross flow filter delamination resistance was considered to have been imparted by the high temperature firing cycle, and proper application of the slip coating along the filter element.

Additional design and processing changes were considered at Coors. These included:

- Bisque firing of the stacked-plate array at the lowest temperature possible in order to maintain high reactivity for bond formation at the rib tip-to-plate joint, yet high enough to prevent slumping or deformation of the filter element during high firing.
- The final high firing temperature would be sufficiently high to generate good strength properties, yet not produce excessive deformation and/or loss of permeability.

In order to remove the binder system that was used to form the filter plates and impart sufficient strength to permit assembly of the entire filter body, each plate would be subjected to a partial or bisque firing. In the bisque fire, the raw materials or fine powders would partially react and establish the initial porosity characteristics of the filter matrix. During the bisque fire, the major portion of densification and/or shrinkage occurred, which could be controlled by the firing temperature and soak time at temperature.

In order to produce what was considered to be the monolithic filter assembly from the individually bisque fired plates, a stacking and firing procedure followed. In the subsequent higher temperature firing cycle, material densification would be completed, and plate-to-rib bonding would occur. Control of the firing temperature and cycle conditions determined the ultimate strength of the base material and the rib tip-to-plate bond strength. If warranted, an additional firing of the assembly would be conducted to modify material properties of the filter matrix, or permit a slip coating to be fired onto existing surfaces of the filter element. The combined bisque and high firing cycles were designed to densify and strengthen the ceramic matrix in the final filter assembly.

In view of the finite element analyses, further design modifications which were developed at Westinghouse and Coors resulted in mid-rib bond/paired plate cross flow filter concept. Filter plates were fabricated with half-height ribs for the long clean gas channels along one side of the plate, and half-height ribs for the short dirty gas channels along the opposite side of the plate (Figure 2.1). In this fashion, bonding was expected to occur along the entire length of the rib tip, rather than at point contact along the rib tip-to-plate joint. In the mid-rib bond concept, bonding was shifted to a low stress region along the center of the rib, as opposed to a high stress region along the rib tip-to-plate corner of the gas channel.

In the mid-rib bond/paired plate concept all filter plates were constructed identically. A thick center rib was added to prevent slumping of the filter element during firing. Each rib was radiused to reduce stress along the length of the gas channel. Consideration was also given to improving the strength of the mid-rib bond by forming a higher strength, non-porous surface with a penetrating slurry of the parent matrix.

During production at Coors, the filter plates were initially stacked to form the clean gas channel between paired plates. The paired plates were then bisque fired to establish the critical clean gas channel bond in the cross flow filter element.^a The paired plates were then stacked into the final filter assembly, weighted, and subjected to a final high fire.

Additional design and manufacturing modifications evolved after the initial manufacture and testing of the cross flow filter elements in the Westinghouse HTHP PFBC simulator, as well as at the Kellogg Rust-Westinghouse (KRW) PFBC simulator, New York University PFBC, and Texaco entrained-bed gasification test facilities. A chronological summary of the design modifications

^a If delamination occurred along the clean gas channels, fines would be detected in the effluent gas stream. Conversely, if delamination occurred along the dirty gas channels, bonding within the flange or top closed end section of the filter element would prevent fines from being released and detected in the effluent gas stream. Pairing and bisque firing the plates to initially form the clean gas channels, would not only enhance bonding, but also minimize delamination and release of fines.

which were made during the development of the cross flow filter is provided in Table 2.1. A discussion of the impact that these changes had on filter durability and performance is presented in the following section.

2.3 ORIGINAL KRW AND NYU FILTER ELEMENTS

Initially eight 12x12x4 inch cross flow filters were produced at Coors for testing at the Kellogg Rust-Westinghouse (KRW) PFBC simulator test facility, and an additional eighteen cross flow filters were manufactured for testing at the New York University (NYU) PFBC test facility. During production of the P-100A alumina/mullite filter elements,

- The space between the short dirty channels was changed to accommodate the larger center rib which was incorporated into the body to provide added stability to the filter element.
- The ribs were designed to have radiused machined corners (i.e., 100-110 mil radius).
- The top of the ribs were slip coated with material milled to 1.7 μm to enhanced penetration of the slip into the filter matrix.
- The ribs were dipped in a 4.8 μm slip to cause a curved fillet at the rib tip-to-plate bottom boundary.
- The back filter wall was slip coated with a 4.8 μm slip to provide a clamping effect.
- The bisque fired plates were machined flat to ensure uniform plate-to-plate contact during stacking and subsequent firing.
- The filters were subjected to a uniform bisque and high firing sequence.
- The flange surface was machined completely flat and uniform after firing.

During grinding, one of the filter elements delaminated. Similarly during the second high firing, slumping resulted, and the filters were rejected for testing at KRW and NYU. As a result of delamination and slumping, four mid-rib bond/paired plate filter production variations were undertaken. These included:

- Stacking the filter plates in the green state, followed by high firing.
- Utilizing a slip or glazing compound along each rib prior to firing.

Table 2.1

Cross Flow Filter Development

Filter Elements	Standard 1986 Filters	Original NYU Filters	Original KRW Filters	Test Filters
Production Date	1986	Early 1986	September 1987	October 1987
Material Designation	P-100, MODK5	Lot 1; P-100, MODK5 Lot 2; P-100, MODK5 Lot 3; P-100, MODK5	P-100, MODK5	P-100, MODK5
Number Of Elements Fabricated		18	8	4
Geometry				
Filter Wall Thickness, inch	0.065	0.065	0.065	0.1
Rib Design	Rectangular	Rectangular	Rectangular	Mid-Rib Bond
Rib Height, inch	0.125	0.125		
Rib Thickness, inch	0.187	0.187		
Number Of Ribs/inch	2	2		
Filter Surface Area, ft ²	10.4	10.4		
Overall Dimensions, inch	12 x 12 x 4	12 x 12 x 4		
Fabrication				
Firing Conditions				
Bisque Fire, °C	800	1440, 1440, 1440	Bottom 2/3 Plates Were	NA; 1400; 1400; 1400
1st High Fire, °C	1465	1430, 1460, 1440	Bisqued Fired At High	1470; 1470; 1470; 1470
2nd High Fire, °C	-	1430, 1460, 1440	Temperature; Top 1/3 Plates	---; ---; ---; 1500
3rd High Fire, °C	-	---, ---, 1440	Were Bisqued At Low	---; ---; ---; ---
Soak Time, Hours	2-4	2 2 4	Temperature To Promote	4 4 4 4
			A Better Bond	
Weighting, lbs	10-12	3-8, 3-8, 3-8	Low Weight	8 8 8 8
Bonding Agents	No Glazing Or Coating	None; Coated; Coated	Coated	Not Coated; Glazed/Coated
Pore Former	Latex	Latex	Latex	Latex
Properties				
Density, g/cc	1.43	1.54, 1.54, 1.54		
MOR, psi	3950	1400- 700- 700- 1500 1500 1500		
Pore Size, microns	67	62, 62, 62		
Porosity, %	52	48, 48, 48		
Thermal Conductivity	1.25			
Thermal Expansion, in/in°F	2.5 x 10 ⁻⁶			
Burst Pressure, psi		100-175; Top 200-300; Middle 300-500; Bottom		500; 250; 150-350; 410
Permeability (Unused) ΔP @ 10 ft/min - STP		1 in-wg		
Filter Concept	Rectangular Channels Rib Tip-To-Plate Joint Sharp Corner Radius (1.5 mils)	Rectangular Channels Radiused Ribs Rib Tip-To-Plate Joint	Short Rectangular Channels With A Wide Center Rib For Stability; Increased Rib Radius To 100-110 mills	Mid-Rib Design Initiated The Use Of An Extra Plate Layer For Burst Testing
Comment	Relatively Low MOR Low Plate-To-Plate Strength	Relatively Low MOR Low Plate-To-Plate Strength	Relatively Low MOR Low Plate-To-Plate Strength	First Mid-Rib Element Design Low Or No Plate-To-Plate Bond Strength

Table 2.1 (continued)

Cross Flow Filter Development

Filter Elements	KRW Filters	NYU Filters	Texaco Filters Lot 1	Texaco Filters Lot 2
Production Date	November 1987; April 1988	July 1988	February 1989	October 1989
Material Designation	P-100, MODK5	P-100, MODK5	P-100, MODK5	P-100, MODK5
Number Of Elements Fabricated	8 8	23	15	4
Geometry				
Filter Wall Thickness, inch	0.1 0.1	0.1	0.1	0.187
Rib Design	Mid-Rib Bond	Mid-Rib Bond	Mid-Rib Bond	Mid-Rib Bond
Rib Height, inch	0.125 0.125	0.25	0.375	0.375
Rib Thickness, inch	0.187 0.187	0.125	0.25	0.25
Number Of Ribs/inch	2 2	2	1	1
Filter Surface Area, ft ²	7 7	7	8.7	8.4
Overall Dimensions, inch	12 x 12 x 4	12 x 12 x 4	12 x 12 x 4	12 x 12 x 4
Fabrication				
Firing Conditions				
Bisque Fire, °C	1400 1400	1400	1400	-
1st High Fire, °C	1465 1485	1485	1480	(d)
2nd High Fire, °C	1475 -	-	-	-
3rd High Fire, °C	- -	-	-	-
Soak Time, Hours	4 4	4	4	(d)
Weighting, lbs	8.5-9.5 38	25-45	33	(d)
Bonding Agents	Coated Not Coated	Not Coated	Binder Used To Improve Permeability Latex	Walnut Shells
Pore Former	Latex	Latex	Latex	Walnut Shells
Properties				
Density, g/cc			2.00±0.02	
True		3.087		
Bulk		2.26±0.02		
MOR, psi	(a)		2574-4004 (ANL) 2806±242 (W-STC)	
Parallel To Channel		8995±738		
Perpendicular To Channel		8425±1196		
Thick Closed End Section			3810±200 (W-STC)	
Pore Size, microns			55.7	
Porosity, %	(a)		45	
Open		18.8±1.7	23±2.5	
Closed	8.1±1.3		11.9±2.3	
Thermal Conductivity				
Thermal Expansion, in/in°F				
Burst Pressure, psi	50-360 500	>400	335 (b)	
Seam Rupture, psi			381-544 (c)	
Fracture Toughness, MPa·m ^{1/2}			0.54-0.63 (ANL)	
Permeability (Unused) ΔP @ 10 ft/min - STP	20 in-wg 6-100 in-wg	25-50 in-wg	1.0 in-wg	0.56 in-wg @ 26 scfm
Filter Concept	Mid-Rib Bond Design	Mid-Rib Bond Design	Mid-Rib Bond Design Open Channel Design	Mid-Rib Bond Design Open Channel Design
Comment	Significantly Improved Plate-To-Plate Bond Strength Filters Made In April Were Used In The KRW Test Program	X-Ray Analysis Indicated The Presence Of >95% Mullite; Corundum As A Minor Phase 12 Filters Were Used At NYU	X-Ray Analysis Indicated The Presence Of Mullite (70%); Anorthite (17%); Corundum (12); And An Amorphous Phase Radiused Flange	Radiused flange

- (a) Testing Indicated Wide Variation In Gas Permeability For The Eight Filters. Material Properties Were Expected To Vary, And Therefore MOR And Porosity Data Were Not Generated.
- (b) Pressure >335 psi Caused Punctures To Occur In The Filter Wall.
- (c) Special Test Devised To Pull Plates Apart At The Seams.
- (d) Not Reported.

Table 2.1 (continued)

Cross Flow Filter Development

Filter Elements	W-HTHP PFBC W-Recirculating Loop	Texaco Filters Lot 3	Foster Wheeler Filters				W-HTHP Thermal Transient Testing
Production Date	October 1989	Apr-90	Sep-90	Oct-90	Nov-90	Dec-90	1992
Material Designation	P-100A	P-100A	P-100A	P-100A	P-100A	P-100A	P-100A-1
Number Of Elements Fabricated	4	14	12	8	9	2	
Geometry							
Filter Wall Thickness, inch	0.187	0.187	0.187	0.187	0.187	0.187	0.187
Rib Design	Mid-Rib Bond	Mid-Rib		Mid-Rib Bond			Mid-Rib Bond
Rib Height, inch	0.375	0.375	0.375	0.375	0.375	0.375	0.375
Rib Thickness, inch	0.25	0.25	0.25	0.25	0.25	0.25	0.25
Number Of Ribs/inch	1	1	1	1	1	1	1
Filter Surface Area, ft ²	8.4	8.4	8.4	8.4	8.4	8.4	8.4
Overall Dimensions, inch	12x12x4	12x12x4	12x12x4	12x12x4	12x12x4	12x12x4	12x12x4
Fabrication							
Firing Conditions							
Bisque Fire, °C	(a)	(a)	(a)	(a)	(a)	(a)	(a)
1st High Fire, °C	(a)	(a)	(a)	(a)	(a)	(a)	(a)
2nd High Fire, °C	—	—	—	—	—	—	—
3rd High Fire, °C	—	—	—	—	—	—	—
Soak Time, Hours	(a)	(a)	(a)	(a)	(a)	(a)	(a)
Weighting, lbs	(a)	(a)	(a)	(a)	(a)	(a)	(a)
Bonding Agents	None	None	None	None	None	None	None
Pore Former	Walnut Shells	Walnut Shells	Walnut Shells				None
Properties							
Density, g/cc	1.64	1.151					
MOR, ksi	3.17		2.99-3.07	2.99-3.07	2.99-3.07	2.99-3.07	
Pore Size, microns	48-58	40-60					
Porosity, %	39.1	44					
Thermal Conductivity							
Thermal Expansion, in/in°F							
Burst Pressure, psi	436±9.1	389±33			390±21		
Permeability (Unused) ΔP @ 10 ft/min - STP							
Filter Concept	Mid-Rib Bond Design Open Channel Design	Mid-Rib Bond Design Open Channel Design	Mid-Rib Bond Design Open Channel Design				Mid-Rib Bond Design Open Channel Design
Comment	Radiused Flange	Radiused Flange	Radiused Flange				Raw Materials Were Milled To Appropriate Size; Filters Were Subjected To Cooling/Heating Cycles For Enhanced Strength. Surfaces Were Faced Flanges Were Radiused Pore Former Materials Were Not Used; Shrinkage Was Controlled.

(a) Not Reported; Proprietary.

- Utilizing a standard bisque firing of the filter plates; assembly of the stacked-plate array; high fire at 1470°C for 4 hours with a significantly high loading weight.
- Utilizing a standard bisque firing of the filter plates; assembly of the stacked-plate array; high fire at 1470°C for 4 hours; subsequently refire at 1500°C to increase bonding and body strength.

One of the filter elements which had been stacked and high fired (i.e., bisque firing omitted) experienced ~10% shrinkage from the green state, and developed several cracks. Bonding between the ribs was, however, stronger than obtained by any of the alternate firing or assembly procedures. When burst strength tested, a 500 psi bond strength resulted in the filter element.

For the filter which was fabricated via the standard firing procedure (i.e., bisque firing; assembly; high fire at 1470°C for 4 hrs) with an 8 pound load evenly distributed over the filter element, a burst strength of 250 psi resulted along the rib bonds. In contrast when a glazing compound was applied to the rib bonds, a burst strength of 150-350 psi resulted. When fired at 1500°C, very strong bonds developed within the filter, however, significant deformation and slumping of the element also occurred.

On December 2, 1987, one of the new mid-rib bond cross flow filter elements was installed in Westinghouse's HTHP PFBC test facility. The initial pressure drop across the filter element was 4-5 in-wg at a filter face velocity of 3 ft/min. The element was heated to temperatures of 600°F prior to ignition of the gas combustor. The system was brought to temperatures of 1400°F with system pressures of 150 psig. With a filter face velocity of 2.8 ft/min, a baseline pressure drop of 4 in-wg resulted. Grimethorpe ash was fed at a rate of 550 g/hr, which resulted in a dust inlet loading of 4,000 ppm. During testing on December 3, 1987, eleven pulse cleaning cycles were delivered to the filter element. The particle collection efficiency was 99.89%, prior to shut down (i.e., 0.1 g/scf). The unit was restarted on December 4, 1987. Isokinetic gas sampling at the filter outlet indicated dust levels of 1.75-2.83 g/scf, and HTHP PFBC testing was terminated.

Post-test inspection of the filter element indicated that delamination occurred along the middle of the filter element (Figure 2.5). Substantial bonding occurred, however, along the ribs that were positioned within the flange and top closed end section of the filter element, but little, if any bonding resulted along the mid-section of the filter element. The remaining cross flow filter elements were heated in a laboratory furnace for 3 hours at 300°F (Figure 2.6). Delamination of the elements resulted.

Failure of the HTHP PFBC-tested filter was considered to have occurred due to insufficient material strength (i.e., MOR of 1,500 psi), and poor bonding along the ribs. Delamination had

Figure 2.5 — Delamination along the Mid-Rib Bonds of a Full-Scale Cross Flow Filter after High Temperature, High Pressure Testing in Westinghouse's PFBC Simulator Test Facility

Figure 2.6 — Delamination of the Mid-Rib Bond Cross Flow Filter Elements after Static Furnace Heating at Temperatures of 300°F

apparently been initiated along the dirty gas channel ribs which continued through to the adjacent clean gas channel.

Burst test strength varied throughout the filter element which was presumably due to the weight carried by the each bonding layer during firing. Analysis of the mid-rib bonds indicated that bonding was not continuous, but appeared as "tack welds" at intervals along the mid-rib seam. The "tack welds" corresponded to where there were intersections between ribs running in one direction and ribs running perpendicularly along the opposite side of the filter plates.

Based on the lack of bonding which resulted along the mid-rib bonds, further effort was focused on identifying possible alternate bonding techniques and weighting procedures during high firing. Test plates were fabricated and stacked into paired units, prior to firing at 1455°C with a 10 pound weight. Little or no bonding was evident along paired plates which did not have a slip coat or glaze applied between their surfaces. When an LG7 or LG11 glaze was applied between the plates and weighted with a 10 pound load during firing at 1455°C, bonding resulted, particularly when the LG11 glaze was utilized.

When mid-rib paired plates were fabricated with the LG11 glaze, the burst strength for the bonds ranged between 75 and 150 psi. When a twenty pound weight was applied to the bisque fired plates, burst strengths of 50-500 psi resulted. When the glaze was applied to both mid-rib paired plates, and the unit was weighted with thirty-eight pounds and fired at 1485°C, the glazed module had burst strengths of 50-150 psi. Analyses indicated that the light glaze wicked discontinuously into the ribs, resulting in discontinuous bonds. The heavy glaze partially wicked into the ribs, and the balance devitrified into a powder on the rib surface, producing virtually no bonding.

In parallel with these efforts, an unglazed module was weighted with thirty-eight pounds and fired at 1485°C. The resulting burst strength of the module was 500 psi. As a result of these efforts, the glaze approach was abandoned, and further fabrication of the filter elements would include exposure to higher temperature using higher weights to fuse the ribbed structure.

During the early months of 1988, efforts continued to improve mid-rib bonding in the cross flow filter element, as well as to recover the filtration area that was reduced through the addition of the thick central stabilizing rib. Using the existing filter plate configuration, three of the seven clean gas channel ribs, and eight of the sixteen dirty gas channel ribs were removed. Also a radius was incorporated along the inside corner of the flange. Consideration was given to shrink-fitting a compression brace around the filter element during actual test operation. An alternate round rib configuration was evaluated which would incorporate the use of identical plate weights, while avoiding potential rib misalignment problems.

Similarly effort was directed to providing a chamfer along the base of each rib. When fired, cracks resulted along the edge of the chamfer. Changing to a fillet at the base of a wider rib permitted additional structural support and bonding along the rib, and improved the ability to support the stack weight during sintering, minimizing distortion and slumping.

2.4 FIELD TESTING

2.4.1 KRW PFBC Simulator Testing^(5,6)

Eight mid-rib bond cross flow filter elements were produced at Coors in April 1988 for use at the KRW PFBC test facility. The dimensions of the mid-rib bond included a rib height of 0.125 inches, and a rib thickness of 0.187 inches. As a result of this configuration, two gas channels were constructed per inch throughout the 12x12x4 inch cross flow filter body. These elements underwent an initial bisque firing at Coors, followed by high firing with a thirty-eight pound weight applied to each element. A slip coating was not applied as a bonding agent.

A multi-element cross flow filter test unit was designed, fabricated, and operated in a previously existing high temperature, high pressure vessel at the KRW fluidized-bed combustor (FBC) simulator facility in Madison, PA (Figure 2.7). In the KRW cross flow filter test vessel, eight 12x12x4 inch, mid-rib bond filter elements were housed in a four filter module array (two elements per module; Figure 2.8). The four modules were mounted to a tubesheet assembly that was supported on an expansion cone arrangement, which was in turn clamped and held between the pressure vessel flange by a gasketed ring assembly. Four filter pulse tubes were passed through the ring assembly into the pressure housing, and were centered over the venturi section of each filter module. The purpose of the venturi was to enhance the eductor effect during pulse cleaning so as to maximize the amount of hot, clean side gas that was mixed with the pulse gas during the momentary jet cleaning pulse. This was expected to minimize both the amount of the high pressure pulse gas required, and mitigate the thermal shock that the cross flow filter elements experienced on cleaning. In addition, the venturi section provided a means for measurement of flow from each of the four modules, thereby allowing rapid detection of a failure or plugging of any of the modules.

During testing at KRW, the cross flow filters were exposed to temperatures ranging between 1550 and 1625°F, pressures of 78 to 115 psia, and operated with face velocities of 4.3-6.1 ft/min. In 1987 testing was conducted continuously over a period of 160 hours, and included 54 pulse cleaning cycles. Inlet dust loadings of reentrained PFBC fly ash ranged from 1,000 to 2,000 ppm. An overall

Figure 2.7 — KRW Fluidized-Bed Combustion Simulator Test Facility in Madison, Pennsylvania

Figure 2.8 — Cross Flow Filter Array Utilized at the KRW Test Facility

baseline pressure drop increase from 17 to 23 in-wg resulted during testing, indicating minimal loss of filter permeability, which suggested effective cleaning and baseline pressure drop stability.

Following testing, the system was disassembled and the filters were inspected. The majority of the threaded ceramic rod braces which were used to apply a compression loading to each filter element had broken. Post-test examination indicated that six of the filter elements were structurally intact, and did show evidence of dust on the clean gas side of the filters. The remaining two filters experienced delamination which permitted dust to pass through to the clean gas plenum. Based on the fracture path characteristics, evidence suggested that delamination may have been initiated in the filter flange region (Figure 2.9). With respect to the delamination plane, limited bonding resulted along many mid-rib bonds, particularly in the center of the cross flow filter element (Figure 2.10).

Although two cross flow filters had delaminated during testing at KRW, excellent filter system performance was achieved, as demonstrated by the outlet dust loadings of 2.2 to 5.2 ppm. Three of the intact cross flow filter elements which had been tested at KRW were utilized for further testing in the NYU PFBC test facility.

2.4.2 NEW YORK University PFBC Testing⁽⁷⁾

In an attempt to improve bonding along the mid-ribs, as well as to increase the "open" or filtration area of the filter element, the rib height was increased from 0.125 to 0.25 inches, and the rib thickness was decreased from 0.187 to 0.125 inches. Twenty-three filter elements were manufactured and supplied by Coors in July 1988 for testing at the New York University PFBC test facility. Once again, these elements were full-scale, 12x12x4 inch, mid-rib bond filters which were fabricated via a bisque fire, followed by a high fire with weights of 25-45 pound applied to each filter element. A slip coat was not used as a bonding agent along the mid-rib bonds or channel seams.

Twelve of the twenty-three filter elements, and three cross flow filters which had previously been tested at KRW were installed in Westinghouse's particulate filtration system at the New York University Antonia Ferry Laboratory in Westbury, NY, in 1988 (Figure 2.11). At NYU the filters were mounted in groups of three to form a five module filter array (Figure 2.12). The modules were housed in an existing 7.5 ft diameter refractory lined vessel.

While testing at NYU, each filter module was cleaned on-line by a reverse pulse gas jet. The ability of the cross flow filter to operate stably in the PFBC environment and collect ash particles was evaluated in two ~50-hour continuous tests. The particle filtration performance of the system was determined using isokinetic and cascade impactor sampling techniques.

KRW - Module 3, Element 5, Top

Figure 2.9 — Failed Cross Flow Filter after Testing at KRW

Figure 2.10 — Limited Bonding along the Mid-Rib Bonds in the KRW-Tested Cross Flow Filter Elements

Figure 2.11 — Pressurized Fluidized-Bed Combustion Test Facility in Westbury, New York

Figure 2.12 — Cross Flow Filter Array Utilized at the NYU Pressurized Fluidized-Bed Combustion Test Facility

During the initial 50 hour test period at temperatures of <1500°F, pressures of 105 psig, and nominal face velocities of 5.2 ft/min, a stable baseline operating pressure drop of 35 in-wg was achieved after reverse pulse jet cleaning. Inlet PFBC dust loadings of 305-1,056 ppm were reduced to outlet dust loadings of 2.9-8.9 ppm. During testing, a slight dust leak was detected. Post-test inspection of the cross flow filters using a borescope confirmed that one of the original fifteen elements had experienced a partial flange crack on the clean gas side. All of the other filters appeared to be in excellent condition, showing no evidence of cracking or dust penetration.

During the second 50 hours of testing, the filters were exposed to temperatures of 1300°F, pressures of 105 psig, and nominal face velocities of 10 ft/min. After three pulse solenoid valve failures, a hot gas leak in one of the facility knife valves, and a facility computer system malfunction which resulted in an inlet gas temperature excursion to 1880°F, dust loadings were observed to increase from 9.9 to 103 ppm, and testing was terminated. After disassembly, ten of the filters were shown to be undamaged, five had delamination cracks (Figure 2.13), and nine had dust seal leaks. It was evident that the majority of dust penetrating the system occurred through the dust seal leaks rather than through the delamination cracks.

Fractographic analysis was performed on several of the exposed filters in an attempt to identify probable crack initiation points and propagation paths, and structural factors which may have contributed to fracture propagation (Appendix A). From these analyses, the filter design was modified to include a radiused flange. In addition, the unrestrained filter gasket was redesigned in order to survive a wide range of possible operating conditions. Both of these efforts were undertaken to improve the durability of the flange, and to provide an adequate seal between the filter element and the clean gas plenum passage.

The three cross flow filter elements which had previously been tested at the KRW test facility remained intact after 100 hours of HTHP PFBC testing at NYU. Similarly post-test inspection also indicated that the split collars that were used to hold the compression loaded ceramic rods around the filter elements also remained intact.

2.4.3 Texaco Entrained-Bed Gasification Testing⁽⁸⁾

In 1988, Westinghouse began a 24-month program in cooperation with Texaco to demonstrate the performance of the ceramic cross flow filter elements in an entrained-bed coal gasification environment. At Montebello, California, the Texaco Integrated Gasification Combined Cycle (IGCC) process utilized two advanced hot gas desulfurization schemes (Figure 2.14).

Figure 2.13 — Failed Cross Flow Filters after Testing at NYU

Figure 2.14 — Texaco Entrained-Bed Gasification Test Facility

Prior to February 1989, Coors Ceramics in conjunction with Westinghouse redesigned the cross flow to further increase the overall surface area of the filter element for use at Texaco. In order to achieve this, the rib height was increased from 0.25 to 0.375 inches. To support the increased distance between the ribs, the thickness of the mid-rib was increased from 0.125 to 0.25 inches. In this manner, one gas channel per inch resulted throughout the cross flow structure, and the overall effective filtration surface area was increased from an estimated 7 to 8.7 ft². The cross flow filters produced in February 1989 were designed and manufactured with a radiused flange.

In an attempt to improve permeability in the cross flow filter matrix, Coors modified the binder burn-out phase which was used during the initial processing of the raw materials. Sufficient plates were isostatically pressed to fabricate fifteen filter elements. The cross flow filters were manufactured via a dual firing method whereby paired plates were first bisque fired, then machined to form the cross flow channels, and stacked into the filter array structure. The filter was then weighted and high fired in an attempt to complete bonding between the paired plates. Testing was developed at Westinghouse to identify the tensile strength and degree of bonding that resulted in the as-manufactured cross flow filter elements. As shown in Appendix B, the strength of the mid-rib bonds ranged between 364-954 psi.

One filter element was tested in Westinghouse's HTHP PFBC simulator at temperatures of 1500°F, pressures of 150 psig, with face velocities of 3 fpm. Post-test inspection of the element after 50 pulse cleaning cycles indicated that the filter element remained intact.

At Texaco, hot dust laden gas passed through a radiant cooling section and cyclone, and then entered the Westinghouse filter vessel through a radiant nozzle. Initially four ceramic cross flow filter elements were housed in the filter vessel (Figure 2.15). The filter elements were supported on two metal plenum pipes suspended from an uncooled metal tubesheet which was located inside a refractory lined pressure vessel.

A 48-hour commissioning test was conducted at Texaco in April 1989. The gasifier was operated at 350 psig pressure in an oxygen-blown mode with a Pittsburgh No. 8 seam coal, in the absence of *in situ* sulfur sorbents. The cross flow filters were successfully operated at temperatures of 790-1020°F, pressures of 330 psig, with face velocities of 0.9-3.3 fpm. During testing, the filters were periodically cleaned for a total of 270 pulses, and essentially exhibited consistent pressure drop recovery after each pulse. Inlet mass loadings varied between 1,930 and 9,110 ppm, while the outlet mass loading varied between 2.1 and 6.2 ppm. A borescope inspection of the clean side surfaces of the filter elements inside the plenum showed no evidence of particle penetration or filter delamination.

Figure 2.15 — Cross Flow Filter Array Utilized at Texaco in Montebello, California

Four additional filter test campaigns were conducted in conjunction with Texaco's air-blown gasification test program which utilized a Pittsburgh seam coal, and various modes of desulfurization. Inlet dust loadings during this phase of testing ranged from 250 to 22,500 ppm, while outlet mass loadings of 5 to 176 ppm were achieved.

After 29 hours of operation in July 1989, inspection of the filter elements indicated that one of the four cross flow filters had developed a hairline crack along the mid-rib bonds that formed a dirty gas channel (i.e., Test Campaign No. 2). This element was replaced with an alternate as-manufactured filter element. Testing continued for an additional 90 hours, prior to inspection which indicated that three of the four filter elements were intact and did not show evidence of cracks or delamination (Test Campaign No. 5). One filter had sheared off at the flange-to-filter body surface, and appeared to have been unseated by the reverse pressure generated during pulse cycling (Figure 2.16). Inspection of this filter prior to installation at Texaco indicated that the as-manufactured filter element had a thinner flange section, and also had an abnormally sharp edge along the flange-to-body contour. Despite the damage to the flange, the filter body showed no evidence of cracking or delamination.

Post-test inspection of the filter array indicated that 25 to 75 percent erosion of the gasket materials had occurred, which accounted for the downstream dust loadings. Although none of the filter elements showed any signs of in-service delamination, two of the nine filters that were tested experienced longitudinal cracks which separated the filter element body from the clamped flange section. In order to alleviate stress along the flange section, an improved high temperature mount assembly and a rugged, reinforced alumina fiber blanket gasket (Appendix C) were developed. The modified mounting assembly consisted of a creep resistant bolting material, and a flexible clamp designed to control and distribute the seating forces along the flange surface.

The original cross flow filters that were tested at Texaco were replaced with four additional cross flow filters which were supplied by Coors in October 1989. These elements had the same overall dimensions as the previously tested filters, and included the mid-rib bond design and radiused flange. While the overall filtering surface area was maintained at 8.4 ft², the number of short dirty gas channels was increased from 158 to 198, and the number of clean gas channels was reduced from 4 to 3. These changes essentially reduced the size of the openings on the short dirty side channels such that the reverse pulse gas would flow at a higher velocity to remove the dust cake.

In addition to these changes, the cross flow filters were manufactured via a single-step firing process, and the latex pore former material which had traditionally been used throughout the development of the cross flow filter program was changed to walnut shells. This resulted in order to

Figure 2.16 — Failed Cross Flow Filters during Initial Testing at the Texaco Entrained-Bed Gasification Test Facility

avoid potential environmental issues with burnout of the latex material, and to identify a lower cost pore former material.

As part of the Test Campaign No. 6, the four cross flow filter elements were tested under air-blown gasification conditions with *in situ* desulfurization for 62 hours during November 1989. Inlet dust loadings to the filters ranged between 650 and 12,350 ppm, with a gas flow rate of 50-94 acfm. Outlet dust loadings during Test Campaign No. 6 ranged between 18.9 and 176 ppm.

Post-test inspection of the filter array indicated that a cross flow filter delaminated across the clean gas channel, mid-rib bond. The filter separated completely during removal from the holder mount (Figure 2.17). High magnification optical microscopy analysis indicated the presence of large void formations within the filter matrix (Figure 2.18).

Delamination was also evident in a second cross flow filter. Failure of this element was evident along a section of the dirty gas channels which then migrated through to the adjacent clean gas channel, and finally through the flange section of the filter element (Figure 2.19). The actual direction of the crack propagation path could not be discerned. Although the filter was intact when initially removed from the filter holder mount, it was easily separated, indicating limited bonding between the filter plates. Again large voids were evident within the filter matrix.

A noticeable change had occurred in the morphology of the filter matrix as the result of changing the pore former material that was used to fabricate the filter elements. The original matrix appeared to be substantially denser, containing 40-100 μm pores. With the use of the modified pore former material, large voids and channels (i.e., $>500 \mu\text{m}$) resulted in the filter matrix. Although the large voids or channel inclusions provided increased permeability through the filter element, these inclusions not only weakened the filter matrix, but also served as critical flaws or "cracks" within the material. Similarly, areas which contained substantial voids were expected to provide limited contact for bonding along the adjoining ribs and/or plate seams.

Prior to reinitiating testing at Texaco, the modified filter element mounting assembly was incorporated into the Westinghouse cross flow filter element system prior to testing in July 1990, and also the filter array was expanded to operate with eight filter elements (Figure 2.20).

Fourteen cross flow filter elements were fabricated at Coors in April 1990, eight of which were installed in the Westinghouse particulate removal system at the Texaco entrained-bed gasification test facility. These filters had been fabricated with the alternate pore former material (i.e., walnut shells), but had been subjected to a dual firing. The manufacturing process which was used to fabricate the April 1990 filter elements was identical to that used to produce four cross flow filter elements in October 1989.

Figure 2.17 — Failed Cross Flow Filters after Testing at the Texaco Entrained-Bed Gasification Test Facility In November 1989

Figure 2.18 — Large Void Formations in the Alumina/Mullite Filter Matrix Manufactured in October 1989 for Use at the Texaco Entrained-Bed Gasification Test Facility

Figure 2.19 — Failed Cross Flow Filters after Testing at the Texaco Entrained-Bed Gasification Test Facility in November 1989

Figure 2.20 — Cross Flow Filter Array Utilized at the Texaco Entrained-Bed Gasification Test Facility in July 1990

Initially the eight cross flow filter elements which were installed at Texaco experienced a four hour test in July 1990 at operating temperatures of 1118°F, system pressures of 350 psig, and a gas flow of 31-77 acfm. Following this test, an oxygen-blown test was conducted in January 1991, without the use of sulfur sorbents. Testing continued for a period of 33 hours at temperatures of 1200°F, pressures of 350 psig, and gas flows of 60 acfm. The inlet dust loading ranged between 1,170 and 1,934 ppm. At termination of the January 1991 test, inspection of the filter elements indicated that three cross flow filters suffered delamination. These filters were located near or at the bottom level of the filter array.

The three failed filters and fourth filter which were positioned near the bottom of the filter array were replaced with elements that had been fabricated at Coors during either April or November 1990. These filters were manufactured with the alternate pore former material, and had been subjected to dual high firing.

Testing continued during Test Campaign No. 7, exposing the cross flow filter elements to an oxygen-blown gasification environment which included the use of sulfur sorbents. Testing was conducted for a period of 50 hours at temperatures of 1571°F, at system pressures of 350 psig, gas flows of 33-96 acfm, and inlet dust loadings of 1,633 ppm. Post-test inspection of the filter array indicated that the cross flow filter elements suffered severe delamination and horizontal cracking (Figure 2.21).

In summary, the cross flow filter that were used in the Westinghouse filtration system at the Texaco entrained-bed gasification test facility were manufactured at Coors Ceramics in February 1989, October 1989, and April 1990. Although the channel size and overall dimensions of the filter element had been optimized to achieve a maximum filtration surface area, and the flange had been radiused, changes were made to both the pore former material and firing cycles used to produce the various filter production lots (i.e., February 1989: latex spheres, dual firing; October 1989: walnut shells, single firing; April 1990: walnut shells, dual firing). Critical issues which were identified during operation at Texaco included delamination possibly as a result of low strength mid-rib bonds and/or channel seams, failure along the filter flange, and the inclusion of critical flaws (i.e., >500 μm) which inherently resulted from the use of walnut shells as the pore former material. In an effort to alleviate failure along the flange of the cross flow filter element, the Westinghouse holder mount was redesigned, and the gasket configuration was modified.

Figure 2.21 — Failed Cross Flow Filters after Completion of Test Campaign No. 7 at the Texaco Entrained-Bed Gasification Test Facility in Montebello, California

2.4.4 W HTHP PFBC Long-Term Durability Testing⁽⁹⁾

Two dedicated HTHP filter test facilities were constructed at Westinghouse during the mid 1980's (Figure 2.22). The HTHP test facilities were intended to provide simulated HTHP gas environments for evaluating the performance of the cross flow filter using ash materials from PFBC and coal gasification facilities. The PFBC simulator facility was designed for a gas flow up to 1,500 lb/hr, combusting natural gas to provide the thermal input. A gravimetric dust feeder and a pneumatic transport line were used to reentrain the ash. The second test system was a closed or "recirculating" loop that was electrically heated and designed to provide a HTHP reducing or inert gas environment, permitting the feeding of char/ash material. In this facility, up to 1500 lb/hr of gas flow was recirculated using a specially designed HTHP eductor. Approximately 10 percent of the gas was used as the motive flow for the eductor.

Both test loops had on-line ash collection and removal capability that permitted round-the-clock operation over extended test periods (i.e., 100 hours or more). Both facilities were operated over a wide range of flow, pressure and temperature conditions. Similarly both test loops were instrumented to acquire filter operating and system performance data via a computer-based data acquisition system.

In October 1989, Coors manufactured four mid-rib bond cross flow filters which were dimensionally similar to those produced for use in the initial hot gas filtration testing at Texaco. The rib height was maintained at 0.375 inches, and the rib thickness was maintained at 0.25 inches. One gas channel per inch resulted throughout the cross flow structure. The cross flows produced in October 1989 included a radiused flange.

The October 1989 cross flow filters were manufactured via a dual firing process, and included the use of walnut shells as the pore former material. Two of these filter elements were tested in the Westinghouse "recirculating" loop and successfully achieved 1,919 hours of operation. Both filter elements demonstrated high particulate removal efficiencies of the reentrained gasifier fines, as well as stable pressure drop characteristics.

One of the 1,919 hour "recirculating" loop filters was selected for continued testing, and a second as-manufactured October 1989 filter were subjected to testing in the Westinghouse high temperature, high pressure (HTHP) pressurized fluidized-bed combustion (PFBC) test facility. Approximately 1,300 hours of operating life was achieved during HTHP PFBC testing of the two cross flow filter elements. During HTHP PFBC testing, both cross flow filter elements were operated at filter face velocities of 6 and 10 ft/min, and were subjected to two different PFBC ashes, six cold restarts, and over 2,068 pulse cleaning cycles. The filter outlet dust loading was continuously monitored via isokinetic sampling. Over 300 individual samples were taken, which generally showed

Figure 2.22 — Westinghouse High Temperature, High Pressure Test Facilities

outlet dust loadings below 1 ppm, when inlet dust loadings of 1,000 ppm were delivered to the filter elements. After initial filter conditioning (approximately 50 cleaning cycles), a stable operating baseline pressure drop was established at ~18 in-wg at a face velocity of 10 ft/min.

Testing was terminated when both cross flow filter elements developed cracks along the flange area of the filter body (Figure 2.23). Neither filter experienced delamination. Post-test inspection indicated that ash had become wedged between the clamping mount and surface of the filter flange, applying a load that was sufficient to induce fracture along the flange. In conjunction with Westinghouse's program at the Texaco gasification test facility at Montebello, California, the flange clamping mount was redesigned to provide a more uniform mechanical loading across the entire surface of the flange.

2.4.5 Foster Wheeler Second Generation PFBC Testing⁽¹⁰⁾

Development of Second Generation PFBC depends on the parallel development of highly efficient, highly reliable, high temperature particulate removal devices. In the Westinghouse/Foster Wheeler/DOE development program, the use of ceramic cross flow filters were evaluated to commercially clean gases containing particulates generated from both the carbonizer and the circulating pressurized fluidized-bed (CPFB) units.

At Foster Wheeler, the Westinghouse particulate filtration system housed two filter modules, with each filter module supporting up to five filter elements (Figure 2.24). The two modules were suspended from a metal tubesheet. Particle-laden gases entered the pressure vessel through a tangential inlet, and a sheet metal shroud was used to distribute the gas to the top of the filter module bundle. The metal shroud also served to prevent direct impingement of the entering gas on the filter modules. The pressure vessel was equipped with internal electric heaters which were utilized during start-up to minimize heat loss. The heaters were shielded from the hot, particulate-laden gas by a metal liner.

The cross flow filters which were manufactured for use at Foster Wheeler were fabricated at Coors between September and December 1990. The mid-rib dimensions of the filter element were similar to those used to fabricate the Texaco and Westinghouse high temperature, high pressure-tested filter elements. The filters were initially bisque fired and subsequently high fired to provide mid-rib and/or channel seam bond strength. Similarly walnut shells were used as the pore former material.

During operation in September 1992, the mid-rib bond cross flow filter experienced ~150 hours of testing in the reducing gas environment that was generated in the Foster Wheeler bubbling

Figure 2.23 — Failed Cross Flow Filters after High Temperature, High Pressure Testing in the Westinghouse Pressurized Fluidized-Bed Combustion Test Facility

Figure 2.24 — Cross Flow Filter Array Utilized at the Foster Wheeler Second Generation PFBC Test Facility in Livingston, New Jersey

carbonizer. The filters were operated at maximum temperatures of 1500°F, at a nominal face velocity of 1.3 ft/min. Dust loadings entering the filter vessel ranged from 5,000 to 35,000 ppmw.

The cross flow filters exhibited a low pressure drop during testing, as well as excellent pulse cleaning of the dust cake. There was no indication of dust reentrainment during process operations. Unfortunately several of the cross flow filter gaskets failed during testing which permitted dust to pass through to the clean gas passage and into the off-gas effluent stream. As a result of the gasket failure, the filters were not adequately sealed in the holder mounts. On one filter element, the clamping bar was displaced, and ultimately fracture along the flange of the cross flow filter occurred. In addition, one cross flow filter delaminated. The major design modifications that resulted from testing at Foster Wheeler included the use of the original "window frame" gaskets which had successfully been utilized at Texaco, and proper assembly procedures to ensure adequate sealing and dimensional tolerances of the cross flow filter prior to test initiation.

2.5 ADVANCED PROOF-OF-CONCEPT THERMAL TRANSIENT TESTING

In an effort to demonstrate whether the redesigned filter holder mount and gasket configuration mitigated delamination and/or failure of the Coors stacked-plate cross flow filter configuration, several filter elements which were manufactured from same production lots as those tested at Texaco and Foster Wheeler, and at Westinghouse were subjected to proof-of-concept thermal transient testing. Two filter elements which had been manufactured in April 1990, and two which had been fabricated in September 1990 were simultaneously subjected to steady state HTHP PFBC operating conditions at temperatures of 1550°F and system pressures of 165 psia in Westinghouse's high temperature, high pressure, fluidized-bed combustion simulator. Although the April 1990 cross flow filters had previously acquired ~300 hours of HTHP testing at Westinghouse and appeared to be intact, dust was detected in the off-gas stream after all four filters were subjected to 38 hours of steady state testing, two mild and one severe thermal transient^b and three pulse cleaning cycles. Testing was terminated and the system was slow cooled. Post-test inspection of the filter array indicated that all four filter elements contained partial or complete delamination cracks, or complete horizontal cracks across the surface of the filter element (Figure 2.25).

After a step-wise (Runge-Kutta method) dynamic mass and energy balance was performed to project the temperature versus time profile of the gas in the filter vessel during the simulated turbine

^b A thermal transient or thermal shock was accomplished by turning off the fuel flow to the combustor, dropping the system pressure for 165 psia to 15 psia, and increasing the flow of cold gas to 1,100 lb/hr.

Figure 2.25 — Failure of the Cross Flow Filter Elements after Extremely Severe Thermal Transient Testing

trip transient, it was concluded that the four cross flow filters had been subjected to more severe conditions than those projected to occur in the field (i.e., American Electric Power PFBC Demonstration plant). In an attempt to subject the cross flow filters to a more representative set of test conditions, four newly manufactured cross flow filters were installed in the Westinghouse HTHP test facility. The four filters had originally been manufactured in October 1990, and were subjected to steady state operating temperatures of 1550°F, system pressures of 165 psia, and gas flow velocities of 900 lb/hr. In addition, the four cross flow filters were subjected to thermal transient conditions which simulated a turbine trip event. The thermal transient conditions were accomplished by:

- Turning off the fuel flow to the combustor
- Immediately reducing the gas flow to 400 lb/hr
- Ramping pressure from 165 psia to 110 psia over the first 100 sec
- At $t = 100$ sec, reducing flow to 300 lb/hr
- Ramping pressure from 110 to 55 psia over the next 100 sec
- At $t = 200$ sec, reducing flow to 100 lb/hr
- Ramping pressure to 20 psia over the next 400 sec.

After 320 hours of testing, including 10 simulated turbine trip transients, ~11 ppm of the Grimethorpe dust was detected in the outlet gas loading. Inspection of the filters after cool-down indicated that three of the four cross flow filters remained intact, and only one filter developed both a partial and complete delamination crack (Figure 2.26).

Once again, after review of the actual gas temperature versus time profile that resulted in the filter vessel during testing, it was concluded that the cross flow filters were subjected to more severe turbine trip transient conditions in the HTHP facility than would be expected to occur during field testing.

A third test was performed in the HTHP test facility to simulate the filter heat-up cycle that was expected during field testing. The three intact cross flow filters from the previous transient test series, and one new filter from the October 1990 fabrication lot were subjected to 1,100 lb/hr gas flow, 69 psia pressure, and temperatures of 950°F. Ninety simulated heat-up transients were conducted which consisted of ramping the combustor temperature by 1050°F in 150 sec. After 191 hours of testing, including 90 heat-up cycles, a dust leak of ~14 ppm was detected. Inspection of the filters

Figure 2.26 — Delamination of a Cross Flow Filter after Moderately Severe Thermal Transient Testing

after cool-down indicated that again, the original three cross flow filters which had survived the turbine trip transient series remained intact during the course of heat-up testing, and only one filter developed a partial delamination crack.

All filters that were used during this segment of thermal transient testing were fabricated using the same type of pore formers, and were considered to have experienced the same filter plate stacking assembly and firing temperature profile. Although three different production lots were utilized to manufacture the various cross flow filters, the strength of the April 1990 filter matrix was identified to be somewhat higher than either the September or October 1990 production lots (i.e., 3.204 ksi vs 2.99-3.07 ksi, respectively). An obvious question which was raised was whether the 1990 fabricated cross flow filters had sufficient bulk material, as well as mid-rib bond strength.

2.6 MATERIALS SELECTION AND EVALUATION

During 1991 and 1992, efforts were focused on improving the manufactured hot strength of the Coors alumina/mullite filter matrix. Enhanced strength was perceived to be an essential characteristic for filter viability, particularly during operation of the filter elements under load. Process modifications that were considered and implemented at Coors to improve the manufactured strength of the filter elements included:

- Production of the filter elements in the absence of pore former materials.
- Specification of appropriate powder milling times and cooling rates to produce a strengthened crystalline matrix.

One of the principal advantages that resulted when pore former materials were removed from the fabrication process was that the extent of shrinkage in the ceramic body was negligible when the plates or elements were high fired. Minimizing the extent of shrinkage in either the paired cross flow filter plates during bisque firing, or in the entire stack of paired plates during final high firing optimized alignment and enhanced contact (i.e., prevented slippage) along adjoining mid-rib bonds and gas channel seams. Similarly by eliminating the use of pore former materials during filter production, the potential for pore former agglomeration and subsequent critical flaw formation was reduced, and a smaller and more uniform pore structure resulted in the alumina/mullite filter matrix.

In addition to the removal of the pore former materials, Coors optimized the hot strength characteristics of the alumina/mullite material through initial powder processing modifications (i.e., milling rates; particle size; etc.), as well as variations in the cooling rates from high fire

Table 2.2
Characterization of the P-100A Alumina/Mullite Process Formulations

P-100A	Milling Time, min	Percent Porosity	Cooling Rate, °C/hr	Hot Strength, psi (870°C)	Flow Resistance, in-wg/fpm
1	A	39	Slow	4270 ± 380	0.28
2	A	44	Slow	3391 ± 142	0.18
3	A	39	Fast	2364 ± 60	ND
4	A	44	Fast	1775 ± 86	ND
5	B	36	Slow	4142 ± 283	0.84
6	B	41	Slow	3215 ± 155	0.56
7	B	36	Fast	3589 ± 129	ND
8	B	41	Fast	2960 ± 217	ND
9	C	34	Slow	4114 ± 310	1.21
10	C	39	Slow	3545 ± 421	0.84
11	C	34	Fast	3722 ± 109	ND
12	C	39	Fast	3103 ± 113	ND

ND: Not Determined

conditions. As shown in Table 2.2, the resulting hot strength (i.e., 870°C) of the alumina/mullite matrix ranged between 1,775 and 4,270 psi. To ensure high gas flow permeability during filter operation, air flow resistance measurements were performed for select material formulations.

Variation in the filter element cooling rate after final high fire directly influenced the quantity of the residual amorphous or glass phase within the alumina/mullite filter matrix. At 870°C process operating temperatures (i.e., pressurized fluidized-bed combustion applications), the amorphous phase tends to soften, and a substantially reduced filter matrix hot strength results. When the alumina/mullite filter matrix was subjected to slow cooling after final high fire, the amorphous phase that was present within the ceramic body crystallizes, forming the higher hot strength anorthite- and mullite-enriched material (Table 2.3).

As a result of high hot strength, low gas flow resistance, and phase compositions shown in Tables 2.2 and 2.3, the P-100A-1 alumina/mullite formulation was selected and subsequently used to produce both full-scale cross flow filters, as well as and 1.5 m candle filters.

2.7 MECHANICALLY IMPROVED CROSS FLOW FILTER PROOF-OF-CONCEPT TESTING

In order to reduce the large pores and channels which developed during production of the cross flow filter matrix, Coors reevaluated their manufacturing process and identified that by varying the milling time of the raw materials:

- The bulk strength of the matrix could be increased
- A pore size of 40-60 μm could be achieved
- The matrix could be manufactured without the use of pore formers.

Prior to manufacturing cross flow filters utilizing these processing changes, paired plates were subjected to various weights during high firing. Material property and non-destructive (NDE) analyses were conducted on the resulting paired plates. These results are presented in Section 3 and Appendix D. In addition, filter disc samples were fabricated utilizing various milling times to grind the raw materials, prior to subjecting the disc samples to various firing schedules. The resulting discs were characterized based on room temperature permeability, as well as room temperature and process temperature (i.e., 870°C), 4-point bend, 1/4-point flexural strength testing (Section 2.6). Based on the NDE and material property characterization results, an appropriate milling time, applied load, and firing schedule were established for production of additional cross flow filters.

Table 2.3
Phase Compositions of the Alumina/Mullite Filter Materials

	% Mullite	% Anorthite	% Alumina	% Amorphous	% Other*
P-100A	59.6	5.1	4.8	30.5	—
P-100A-1	67.8	29.5	2.7	—	Present
P-100A-10	72.3	24.2	3.5	—	Present

* Cordierite (Very Minor)

Fabrication of additional filter elements was completed in 1992 using the modified milling and firing procedure developed at Coors Ceramics for the P-100A-1 alumina/mullite filter matrix. The targeted applications for these elements were initial screening or proof-of-concept testing in Westinghouse's high temperature, pressurized fluidized-bed combustion simulator in order to demonstrate concept feasibility, and possible use at field test sites (i.e., Foster Wheeler).

Efforts at Westinghouse during January 1993 focused on evaluating the mechanical durability of the 1992 manufactured cross flow filter elements. Four cross flow filters were installed in Westinghouse's HTHP PFBC test facility. These included one filter which had been fabricated in 1990, and three additional filters that had been fabricated during 1992. All filters were dead weighted during mounting of the filters in their respective filter holder.

All four cross flow filters experienced ~50 hours of steady state conditioning (i.e., 1550°F, 100 psig, ~ 3 fpm face velocity). A mixture of the "red Grimethorpe" ash with 30% lime was used during steady state operations. A dust load of 1,000 ppm was delivered to the four filter elements. During the 50 hours of steady state operation, 10 pulse cleaning cycles were used to clean the filters. Three outlet gas samples were taken during the course of steady state operation which indicated negligible release of fines and/or debris into the outlet gas stream (i.e., 0.46, 1.92, and 0.39 ppm). All four cross flow filters during this time period were considered to have performed well, and remained intact. The pressure drop after each pulse cleaning event returned to near base-line conditions, indicating that "adequate" cleaning had occurred using the "red Grimethorpe" ash with 30% lime.

During the second phase of testing, the inlet ash was changed to a more typical Grimethorpe ash, since supplies of the "red Grimethorpe" ash with 30% lime were exhausted. In this segment of testing, all four filters were initially subjected to a series of five simulated turbine trips. In order to simulate a turbine trip, the filter vessel temperature was required to drop from 1550 to 1370°F in less than 200 seconds, and then to 1300°F in another 400 seconds. The thermal transient conditions were accomplished by:

- Turning off the combustor flame
- Reducing the air flow from 900 to 400 lb/hr
- Ramping the pressure from 165 to 110 psia in 100 seconds
- At t = 100 seconds, reducing the flow to 300 lb/hr
- Ramping the pressure from 110 to 55 psia in next 100 seconds
- At t = 200 seconds, reducing the flow to 100 lb/hr
- Ramping the pressure to 20 psia over next 400 seconds.

Gas sampling was not performed after the series of five turbine trips.

Following the turbine trip sequence, all four filters were subjected to 45 start-up cycles. Based on previous thermal transient analyses, the desired transient (69 psia and 1,100 lb/hr flow) was accomplished by raising the filter vessel inlet temperature from 950°F to 1250°F in 150 seconds, which would result in the vessel gas temperature rising to 1050°F in the same period. This sequence was performed throughout the 45 start-up simulation cycles. Outlet dust gas sampling was conducted after completion of the 45 start-up cycles. An outlet dust load of 10.2 ppm was detected indicating that a failure had occurred either in the filter elements or gasket seals.

All four cross flow filters were then subjected to 500 pulse cleaning cycles over a period of 48 hours. Dust was fed after completion of the 500 pulse cleaning cycles, and an outlet dust loading of 114 ppm was detected, indicating further failure of the filter elements or gasketing materials had occurred. Testing was terminated after ~110 hours of HTHP steady state and process transient exposure.

Examination of the four cross flow filters after steady state and transient exposure indicated that two of the 1992 filter elements suffered severe failure (Figure 2.27). Debonding along the mid-rib dirty gas channel bond resulted in each of these filters. Debonding in each of these filters terminated at approximately the central thick rib section of the filter elements, leaving both filters virtually intact along their flange base. Minor hairline cracks were evident along dirty gas channel mid-rib bonds in the two alternate filters.

During this segment of testing, an alternate gasket concept was utilized to seal one of the cross flow filter elements. Post-test inspection of the gaskets indicated excellent performance of the original gasket concept, but failure of the alternate gasket configuration. The alternate gasket concept had not only dislodged itself from under the filter holder swivel mount, but had encountered substantial fraying and deterioration of the mat thickness. Failure of the gasket was considered to be primarily responsible for detection of dust in the effluent gas stream.

During the subsequent 100 hours of HTHP testing, three cross flow filters which had been fabricated during 1992, and one which had been fabricated prior to 1992 were utilized to determine whether steady state, as well as accelerated pulse cycling produced significant thermal stress to degrade either the bulk material strength of the cross flow filter or the mid-rib bond strength. The impact of thermal fatigue during "cold" pulse cycling was considered to possibly induce hairline crack formations or catastrophic failure in the filter elements during HTHP process operation. If this occurs, then utilization of a "hot" pulse would be expected to minimize filter failure.

Figure 2.27 — Failure of the 1992 Manufactured Cross Flow Filter Elements during Thermal Transient Testing

In an attempt to demonstrate the impact of both "cold" and "hot" pulse cycling on the stability of the cross flow filter elements, a passive bed was designed which was positioned below the filter elements which served to heat the pulse gas prior to passage through the clean gas channels. Heat transfer calculations indicated that after passage through the passive bed, the temperature of the pulse gas was heated by $\sim 900^{\circ}\text{F}$, with a minimal pressure drop of ~ 1 in-wg across the bed. The passive bed was positioned below two of the 1992 manufactured cross flow filter elements, as well as below the filter that had been fabricated prior to 1992.

High temperature, high pressure testing of the filter array was reinitiated at Westinghouse in March 1993. Essentially all four cross flow filters experienced fifty hours of steady state operation at 1500°F to 1560°F and 100 psig. A Grimethorpe dust load of 1,100 ppm was delivered to the filters, and six 400 psig pulse cleaning cycles occurred during this segment of testing. Off-gas sampling was conducted during steady state testing which indicated that all four filter elements and gaskets were intact.

The filters then experienced 400 accelerated pulse cleaning cycles over a period of ~ 15 hours (i.e., 400 psig; one pulse every 1.5 minutes). Dust was fed after every 25 pulses for a period of five minutes with the off-gas being sampled in an attempt to detect if a leak occurred through the filter system (i.e., filter and/or gasket seal failure).

An additional 200 pulse cleaning cycles were then delivered to the filter array using 600 psig pulse gas over a period of ~ 8 hours. The thermal duty of the 400 or 600 psig pulse gas ranged from 11 to 26 Btu/ft². As before, dust was fed to the system for a period of five minutes with the off-gas being sampled in an attempt to detect a leak through the filter system after every 25 pulses. The somewhat higher off-gas dust concentration in comparison to steady state conditions, represented metallic-scale that was removed and collected during the relatively short dust sampling periods.

The HTHP test facility was operated at 1500°F to 1550°F and 100 psig, steady state conditions for a period of 17 hours. A Grimethorpe dust load of 1,100 ppm was delivered to the filters, and two pulse cleaning cycles at 400 psig occurred during this segment of testing. Off-gas sampling was conducted during steady state operation, which indicated that all four filter elements and gaskets remained intact.

Three turbine trips were then conducted in which the vessel temperature decreased from 1520°F to 1420°F in a period of 600 seconds. This corresponded to a decrease in the vessel inlet temperature of 1520°F to 420°F in a similar period of time. Off-gas dust sampling was performed after all three turbine trips were completed.

Four hours of steady state operation at 1500°F to 1550°F and 100 psig followed. Off-gas sampling was conducted for a one hour time period during this segment of testing. Dust penetration was not detected. Ten start-up simulations were then performed in which the vessel inlet temperature increased from 950°F to 1250°F in 150 seconds, with off-gas dust sampling taken after completion of the ten start-up cycles.

To conclude the 100 hours of HTHP testing, the filters experienced an additional 125 pulse cleaning cycles using 600 psig pulse gas. Off-gas sampling was performed after three additional pulse cycles, and no dust penetration was measured.

Post-test inspection of the cross flow filter elements indicated that all four filters remained intact, and that none had suffered delamination or hairline crack formations. All four cross flow filters in this second 100 hour test segment survived a more severe, accelerated pulse cycling sequence, in comparison to the initial 100 hours of HTHP testing. This indicated the relatively high thermal fatigue resistance of the P-100A-1 cross flow filter matrix, in comparison to the delamination and/or fracture response of the alumina/mullite cross flow filter matrix to extremely severe thermal shock transients.

2.8 SUMMARY

Throughout the course of development, the Coors stacked-plate cross flow filter underwent numerous material and processing modifications, as well as design improvements. The bulk material which initially contained mullite, anorthite, alumina, and an amorphous phase (i.e., P-100A) was replaced with a mullite-enriched, anorthite, and alumina matrix, with a negligible amorphous content which ultimately resulted in a high strength, as-manufactured material (i.e., P-100A-1). This was achieved by varying the milling time for grinding the raw materials, removal of the pore former material, and establishing the appropriate heating and cooling cycles that were used during manufacturing of the filter element.

During scaleup of the cross flow from a 6x6x2 inch element to a 12x12x4 inch structure, two separate plates were bonded together forming either a clean gas or dirty gas channel. Unfortunately during production of the early full-scale cross flow filter elements, bond strength throughout the body was not adequate, and delamination resulted during high temperature, high pressure testing. In order to overcome delamination, the filter was redesigned, eliminating the thin rib tip-to-plate bond, and replacing it with a thicker mid-rib bond. Simultaneously, the gas channels were radiused to eliminate stress risers, and a radius was incorporated along the length of the flange.

The number of channels was changed to provide the highest possible surface area throughout the filter element, while maintaining manufacturability and integrity of the "open" channel structure. A thick rib was also incorporated across the mid-section of the filter body to enhance stability of the body, and to improve rib bonding strength.

During the early development of the cross flow, slip coatings were used in an attempt to enhance bonding along the cross flow filter seams and/or ribs. Similarly, the filters were initially subjected to a single high firing cycle as a means to provide high strength in the resulting body. Both slip coating and single high firing approaches were replaced with an initial bisque fire to bond together select channels, and to provide strength in the unit, prior to final stacking and high firing the entire plate structure without the application of a slip coating. Appropriate weights were also added during the bisque and/or high fire in order to enhance bonding along the cross flow filter seams and/or ribs.

A smooth surface finish grinding was added to the manufacturing protocol for production of the cross flow filter, so that an element could be fabricated within specified dimensional tolerances for use in a specially designed holder mount configuration.

Although the process and design modifications significantly improved the bulk matrix and bond strength, the stacked-plate alumina/mullite cross flow filter configuration inherently contained "cracks" along the seams and/or ribs which could induce delamination, and ultimately reduce the operating life of the filter element. As a result, monolithic structures as the chemical vapor infiltration or injection molding processing techniques were viewed as attractive approaches to producing a monolithic filter element that would mitigate delamination during field operation.⁽¹¹⁾

As the cross flow filter developed and was tested in the field, several design advancements were made in the area of how to successfully mount and seal the elements during process operation. This included the development and manufacture of appropriate gasket materials and configurations, as well as the development and utilization of a rocker arm to uniformly apply a compression load along the length of the flange.

The technology which had been developed with respect to manufacturing full-scale cross flow filters with improved material properties was readily transferred and incorporated into the production of both the candle filter and the advanced sheet filter concepts (Section 5). Both the candle and sheet filters were considered to have relatively simple geometries which lend themselves to ease of manufacturing, and which would not experience the delamination issues previously encountered by the stacked-plate cross flow filter configuration.

The advancements which were made in the Coors filter product line have clearly been demonstrated during the successful 1,705 hour P-100A-1 candle filter test campaign in the Westinghouse Advanced Particulate Filtration system at the American Electric Power pressurized fluidized-bed combustion demonstration plant in Brilliant, Ohio. As an oxide-based matrix, the P-100A-1 alumina/mullite filter is expected to exhibit long-term chemical and material stability, as well as durability during high temperature operation in both combustion and gasification environments.

3. NON-DESTRUCTIVE EVALUATION

Westinghouse assessed the existing nondestructive evaluation (NDE) techniques as potential methods for detecting critical flaws within the cross flow filter body. To date viable, cost effective methods for detecting critical flaws within the full-scale alumina/mullite filter body are not readily available. As an alternate approach, Westinghouse focused efforts on developing NDE techniques as possible inspection methods for evaluating the extent of bonding along the mid-rib bonds and gas channel seams which result during the various fabrication stages of the cross flow filter element. As discussed in Section 2, mitigating delamination is a critical issue to successfully achieving long-term, service life of the Coors stacked-plate cross flow filter element.

3.1 OBJECTIVES

Efforts at Westinghouse were focused on demonstrating whether the existing nondestructive evaluation (NDE) techniques could be applied during filter production as a method of process control, or alternately as a post-manufacturing filter inspection tool. These efforts included:

- Development of detection methods for characterizing the nature and extent of bonding along mid-rib bonds and gas channel seams in a series of paired cross flow filter plates that were manufactured at Coors using a controlled set of process variables (i.e., original alumina/mullite matrix which included the use of pore former materials; application of weight along the paired plate surface; etc.).
- Application of a full range of ultrasonic techniques (UT) that included the use of both shear and longitudinal waves. The UT data were collected using dry couplant contact methods and full immersion methods. These methods were performed at various high and low frequencies to assess optimal sensitivity of the porous ceramic materials. The majority of testing was performed in a thru-transmission mode.
- Evaluation of radiography techniques in an attempt to correlate the resulting film optical density information with mid-rib bond and gas channel seam formation.
- Evaluation of high resolution X-ray tomography and acoustic microscopy techniques on small sections of the alumina/mullite cross flow filter material.
- Investigating the utility of vacuum infiltration and resonant frequency as potential full cross flow filter body inspection techniques.

- Assessment of the use of γ -radiography, candling, and gas leak detection as localized inspection techniques along the cross flow filter body.
- Utilizing ultrasonic time-of-flight (TOF) measurements to characterize changes in the density and porosity of the alumina/mullite matrix that result during cross flow filter production.

3.2 NDE CHARACTERIZATION RESULTS

A series of paired cross flow filter plates was produced at Coors Ceramics using the original P-100A alumina/mullite formulation. Each plate which contained pore forming materials was pressed and machined to tolerances that accounted for shrinkage during bisque and final high firing. Mid-rib bonds and gas channel seams were used to form the clean gas channels in the paired plates. Loads of 0, 10, 20, and 30 pounds were evenly distributed along the flat external surface of each paired plate. Duplicate paired plate sets were produced to demonstrate reproducibility of the manufacturing process.

Two additional paired plates were manufactured which contained bonded clean gas channels, and which had dirty gas channel mid-ribs extending along both external surfaces. The dirty gas channel mid-ribs were subjected to a 30 pound load during firing. Similarly two additional paired plates were manufactured which contained bonded dirty gas channels, and which had clean gas channel mid-ribs extending along both external surfaces. In this case the clean gas channel mid-ribs were subjected to a 30 pound load during firing.

Inspection at Westinghouse indicated that each paired set of cross flow filter plates exhibited two directions of bending or distortion — in-plane of the filter, and normal to the plane of the filter (Appendix D). The in-plane distortions varied from 0 to 0.27 inch, while the normal distortion varied from 0 to 0.15 inch. The compound effect of bending tended to twist the plates.

Each set of plates was also inspected for visible cracks or nicks, misalignment or mismatch of ribs, thickness variations caused by uneven load, and "smiles" or "rippling" caused the mid-rib bonds or gas channel seams.^c Thickness and time-of-flight (TOF) measurements were taken at sixteen different locations along the paired cross flow filter plates. Similarly each set of paired plates was x-rayed and subjected to ultrasonic immersion testing.

^c A "smile" refers to what appears to be an opening or gap along the channel seam, between adjacent ribs.

3.2.1 Radiography

X-ray characterization of the twelve cross flow filter paired plates indicated that six contained ~0.125-0.25 inch cracks in the flange radius, and that four contained similar cracks along the edge of the plates at approximately six inches above the flange. Radiographic film optical densities were measured at twenty-five locations along each radiograph. The optical density measurements were correlated with the ultrasonic amplitude plots that are described in the following section.

3.2.2 Ultrasonic Immersion Tests

Ultrasonic immersion testing was conducted on each of the twelve paired plate assemblies. The ultrasonic tests utilized a pair of 1.5 MHz, 1 inch diameter broad band transducers in thru-transmission mode. Ultrasonic testing was conducted using the following equipment in an attempt to compare the results in terms of the pulser/receivers used during testing:

- Krautkramer USIP-12 UT instrument.
- An Ultran high energy/low frequency burst pulse system. The system consisted of Ultran's BP9400A Burst Pulser and the BR-640A Broadband Receiver. The Ultran high power pulsed ultrasonic system was made to penetrate high loss materials as ceramic "composite" structures. The Ultran system was used in conjunction with Ultran's λ -series and other dry coupled transducers for this application. Other low frequency transducers manufactured by Panametrics were also shown to be compatible with the Ultran system.
- The Matec MBS-8000 system was used to acquire data on three of the twelve paired cross flow filter plates. This system was configured with the spike pulser board.

The signals that were generated during ultrasonic testing were viewed on a separate oscilloscope, as well as stored in the form of digitized A-scans obtained from a PC-based Sonotek device. Data acquisition was done continuously on the surface of the filters with the Sonotek digital device which accurately stored amplitude and time-of-flight (TOF) data.

Using the USIP-12, Ultran, and Matec MBS-8000 equipment, two separate scans were taken on the cross flow filter plates — one for amplitude and one for TOF measurement. Testing was conducted in an automated scanning tank. Amplitude, TOF, Radio Frequency Unrectified (RF) and fast fourier transform (FFT) data from all three systems were stored for comparison. The results of the amplitude and TOF scans indicated that considerable variation existed within the paired plates, but tended to correlate with the radiographic densities.

RF data and the corresponding FFT data were acquired and stored from eight locations along the top closed end section and flange of each set of cross flow filter paired plates (Figure 3.1). This

Figure 3.1 — Locations Where the RF and FFT Data Were Obtained on the Paired Cross Flow Filter Plates

permitted analysis and correlation between velocity and transmitted frequencies or frequency attenuation, as well as a comparison of the efficiencies for the three ultrasonic systems.

The results of these tests demonstrated the ability of ultrasonic inspection to reflect qualitative bond variation from plate to plate. Figures 3.2 through 3.5 illustrate the amplitude and TOF scans that were generated for paired cross flow filter plates that experienced either a 0 or 30 pound load during firing. Comparison of the scan data also indicated that the Ultran burst pulser generated the highest resolution in terms of differentiating between mid-rib bonds, gas channel seams, and possible crack locations.

The FFT and RF waveforms that were generated at select locations were compared in an attempt to identify bond quality characteristics. Figure 3.2 illustrates the spectral content of the transmitted energy from the incident broad band 1.5 Mhz signals that were obtained at the various positions along the cross flow filter plates that were fired with a 0 pound load. Based on the difference in shapes between the FFTs, the various positions were arbitrarily classified as "good bonds" and "bad bonds". The rib position signals were more difficult to analyze because the transducer diameter was larger than the rib width. The shift towards lower frequency at the top closed end section of the paired plates (i.e., position No. 8) was explained by absorption of higher frequency in the lamination between plates. These data were also correlated with the significant reduction in amplitude at position No. 8, and to the corresponding increase in TOF. A reduction in the bonding characteristics (i.e., lower bond strength; debonding; etc.) was proposed in this region.

Figures 3.4 and 3.5 illustrate the spectral content of the transmitted energy from the incident broad band 1.5 Mhz signals that were generated at the various positions of the paired cross flow filter plates that experienced the 30 pound load during firing. Once again based on the difference in shapes of the FFTs, the positions were classified as "good bonds", "medium bonds", and "bad bonds". As before, the rib position signals were more difficult to analyze because the transducer diameter was larger than the rib width. With the exception of the ribs at position No. 2, No. 3, and No. 4, the difference between the FFT shapes generated as "good bonds" and as "medium bonds" was not very significant.

A dry couplant technique was used as an independent verification of bond "quality", measuring amplitude, TOF, and the shape of the FFT. The TOF per inch (TOF/in) data were obtained in thru-transmission using a 2 Mhz shear transducer at the positions identified in Figure 3.1. As shown in Figure 3.5, the TOF/in data for the 30 pound paired plates was consistently lower (i.e., a better bond) than the TOF/in generated for the 0 pound paired plates. A significant reduction in the TOF/in data resulted along the top closed end section of the 30 pound load, paired plates, indicative of "good"

Figure 3.2 — FFT of the 1.5 Mhz Thru-Transmission RF Data Generated for the 0 Pound Paired Plates During Immersion Testing

Figure 3.3 — FFT of the 1.5 Mhz Thru-Transmission RF Data Generated for the 30 Pound Paired Plates during Immersion Testing (Five Positions)

Figure 3.4 — FFT of the 1.5 Mhz Thru-Transmission RF Data Generated for the 30 Pound Paired Plates during Immersion Testing (Three Positions)

Figure 3.5 — 2 Mhz Shear Dry Coupled Thru-Transmission TOF/in Data Generated for Both the 0 Pound and 30 Pound Paired Plates

bonding in this area. In comparison, the corresponding TOF/in data for the 0 pound load paired plates at position No. 8 was relatively high, which was consistent with the proposed "poor" bonding along this area. These data were supported by the data obtained at position No. 1 in the 0 pound load cross flow filter plates (Figure 3.5) which showed the same TOF/in height, while the immersion test data (Figure 3.6) showed a strong relative amplitude, which was consistent with areas of "good" bonds elsewhere in the plate. Additional supporting data related to "poor" bond quality was present in the corresponding FFT spectral information (Figures 3.6 and 3.7). There was an absorption of higher frequencies resulting in the relative prominence of the low frequency components along the top closed end section of the paired plates at position No. 8 in Figure 3.7.

3.2.3 Alternate NDE Approaches

Vacuum infiltration of the porous alumina/mullite ceramic cross flow filter matrix was attempted at Westinghouse using small magnetic particles. Colloidal iron particles suspended in a liquid medium were dispersed throughout the filter matrix. After drying of the carrier solution, mapping of the magnetic particles was accomplished using eddy current techniques. The resulting eddy current maps correlated with the near surface density distribution and surface flaws. Further effort would be required to demonstrate the particle infiltration and detection capabilities throughout the entire filter structure, as well as removal of the infiltrated iron particles prior to use of the filter in process applications.

Resonant frequency techniques (i.e., Grindo-Sonics) were also evaluated for use as an NDE approach to evaluate the bonding and material characteristics of the cross flow filter. Based on the complex geometry of the cross flow filter, meaningful information similar to that generated for the simpler candle filter geometry may possibly be obtained if a statistically large cross flow filter resonant frequency database were established.

3.3 SUMMARY

Further efforts are required to develop rapid, cost-effective, nondestructive evaluation techniques which can effectively be utilized as either an inspection tool throughout the entire filter element manufacturing process cycle, or to identify the remaining service life of field-tested filter elements.

Ultrasonic techniques, however, can provide a basis for characterizing the quality of the mid-rib or gas channel seam bonding in paired cross flow filter plates, particularly if the amplitude, TOF, and FFT are simultaneously used to make the assessment. The correct choice of transducers and pulser

Figure 3.6 — FFT of the 2 Mhz Shear Dry Coupled Thru-Transmission RF Data Generated for the 0 Pound Paired Plates (Five Positions)

Figure 3.7 — FFT of the 2 Mhz Shear Dry Coupled Thru-Transmission RF Data Generated for the 0 Pound Paired Plated (Three Positions).

receivers is very important. The high energy burst pulsers (i.e., Ultran) are optimal for coupling sufficient energy into the part. Appropriately damped transducers that excite the energy, especially for shear waves in porous ceramics, are also needed. The shear wave transducers from Ultran appeared to work satisfactorily.

Amplitude and time-of-flight measurements which were conducted on each paired cross flow filter plate confirmed that a minimum weight of 20 to 30 pounds was necessary to achieve "good" bonding along the mid-rib bonds and gas channel seams. The ultrasonic data confirmed that bonding was enhanced along the top closed end section of the cross flow filter, along the filter flange, as well as along the tack weld positions at the intersection of the gas channel seam ribs. When a 30 pound load was applied, the ultrasonic data indicated that uniform bonding along the mid-rib bonds was not achieved for the current cross flow filter configuration. To date the ultrasonic data have not been used to determine the exact length of the tack weld bonds, nor the strength thereof, both of which are critical to prevent crack propagation and seam delamination during in-service filter use.

4. CROSS FLOW FILTER MATERIAL EVALUATION

Characterization of the composition and physical properties of the alumina/mullite filter matrix during development of the cross flow filter was undertaken at Westinghouse in 1990. Effort focused on developing the bulk strength of the material through four-point bend, 1/4-point flexure testing, density, porosity, and x-ray diffraction analyses. As shown in Table 4.1, the alumina/mullite filter matrix that was used to manufacture the New York University cross flow filter elements had a significantly higher strength and a corresponding lower percent open porosity in relation to the alternate filter production lots.

The alumina/mullite filter matrix is principally composed of mullite ($3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$) and corundum (i.e., Al_2O_3). Secondary or minor phases of anorthite ($\text{CaAl}_2\text{Si}_2\text{O}_8$) and an amorphous or glass phase are detected within the filters fabricated for Texaco, as well as in filters which experienced the New York University PFBC environment. In parallel with these efforts, the morphology and composition of the alumina/mullite filter matrix were determined utilizing scanning electron microscopy and energy dispersive x-ray analyses (SEM/EDAX). As shown in Figure 4.1, the alumina/mullite matrix consists of mullite rods or needles that are embedded within an amorphous phase that contains the corundum and anorthite phases.

Similar physical property and compositional analyses were conducted at the Argonne National Laboratories (ANL). On September 25, 1989, two cross flow filters were submitted to ANL for materials characterization which included determination of the

- Theoretical density (i.e., crushed sample density)
- Elastic modulus evaluated by velocity of sound measurements
- Thermal conductivity measurement by laser flash techniques
- Thermal expansion measured between room temperature and 1600°F using standard dilatometry
- Flexural strength measured by four-point bend testing
- Fracture toughness measured by single-edge notch beam technique

of the alumina/mullite cross flow filter matrix. One of the cross flow filters was an as-manufactured filter element which had been produced in February 1989, and the second filter which had been

Table 4.1
Chemical and Physical Properties of the Alumina/Mullite Filter Matrix

Filter Designation	WCXF2	WCXF4	NYU		Texaco
Date Fabricated	1985	1985	<-----7/1988----->		2/1989
Status	Exposed to METC Process Gas	Exposed to METC Process Gas	Unused	Exposed	Unused
Geometry, inches	6x6x2	6x6x2	12x12x4	12x12x4	12x12x4
Configuration	<-----Rectangular----->		<-----Mid-Ribbed----->		Mid-Ribbed
<u>Physical Properties</u>					
MOR, psi*					
Parallel to Channel	3725 ± 224	2864 ± 116	9970 ± 728	5203 ± 1006	NA
Perpendicular to Channel	3274 ± 275	3026 ± 339	9074 ± 1192	5869 ± 1178	3974 ± 197
Closed Filter End	NA	NA	NA	NA	3416 ± 264
Density, g/cm ³					
True	3.087 ⁽¹⁾	3.087 ⁽¹⁾	3.087 ⁽¹⁾	3.087 ⁽¹⁾	3.087 ⁽¹⁾
Bulk	1.96 ± 0.03	2.11 ± 0.09 ⁽²⁾ 1.98 ± 0.02 ⁽³⁾	2.26±0.02	2.19 ± 0.10 ⁽²⁾ 2.21 ± 0.09 ⁽³⁾	2.00 ± 0.02
% Dense	63.4 ± 1.0	68.4 ± 2.6 ⁽²⁾ 64.3 ± 1.0 ⁽³⁾	73.1 ± 0.5	71.0 ± 3.2 ⁽²⁾ 71.5 ± 3.0 ⁽³⁾	64.9 ± 0.9
Porosity					
% Open	28.4 ± 4.2	23.3 ± 5.6 ⁽²⁾ 13.4 ± 2.8 ⁽³⁾	18.8 ± 1.7	21.2 ± 5.4 ⁽²⁾ 21.3 ± 5.9 ⁽³⁾	23.2 ± 2.5
% Closed	8.2 ± 3.8	8.3 ± 3.1 ⁽²⁾ 22.3 ± 2.1 ⁽³⁾	8.1 ± 1.3	7.1 ± 2.8 ⁽²⁾ 7.1 ± 4.6 ⁽³⁾	11.9 ± 2.3
<u>XRD Characterization</u>					
Principal Phase	Mullite	Mullite	Mullite	Mullite	Mullite
Secondary/Minor Phase(s)	Corundum	Corundum	Corundum	Corundum Anorthite Amorphous	Corundum Anorthite Amorphous

Number of Samples Tested
NA: Not Analyzed
(1) Assumed Value
(2) Parallel to Dirty Gas Channel
(3) Perpendicular to Dirty Gas Channel

Table 4.1 (Continued)

Chemical and Physical Properties of the Alumina/Mullite Filter Matrix

Filter Designation	Texaco		<u>W</u> -STC HTHP PFBC	
Date Fabricated	2/1989		2/1989	
Status	Texaco Entrained-Bed Gasifier		<u>W</u> HTHP Facility	
Geometry, inches Configuration	12x12x4 Mid-Ribbed		12x12x4 Mid-Ribbed	
<u>Physical Properties</u>				
MOR, psi (Weibull, m) Top (Closed)	—	—	3529 ± 222 (19.2)	2666 ± 168 (19.1)
Web (Core)	3234 ± 334 (14)	3089 ± 334 (9)	3148 ± 368 (8.2)	2377 ± 354 (7.0)
Flange (Bottom)	—	—	NA	2354 ± 169 (16.2)
Top + Flange	3930 ± 290 (16)	4099 ± 276 (18)	—	—
MOR, psi RT, 870°C			3040 ± 302, 2424 ± 280	
Density, g/cm ³ True			3.087	
Bulk			1.705 - 1.707	
% Dense			55.2 - 55.3	
Porosity				
% Open			41.3 - 41.5	
% Closed			3.3 - 3.4	
Total Porosity			44.7 - 44.8	

NA: Not Analyzed

Figure 4.1 — Morphology of the Alumina/Mullite Cross Flow Filter Matrix

manufactured from the same production lot, experienced ~77 hours of process operation in the Texaco entrained-bed gasification environment. Both filters were fabricated using the initial pore forming material, and had been subjected to bisque and high firing. The Texaco-exposed filter had developed a hairline crack along a dirty gas channel mid-rib bond during field operation.

As part of the effort conducted at ANL, a critical flaw size for the alumina/mullite filter matrix was calculated based on fracture mechanics. Static and dynamic finite element analyses were performed to identify the impact of pulse cleaning on the stability of the cross flow configuration. A report entitled "Materials Qualification Technology for Ceramic Cross Flow Filters" was prepared and published by ANL, and is presented in Appendix E. Highlights from this effort follow:

- The cross flow filters contain mullite as the major phase, with minor phases of anorthite and albite, and have approximately 41% porosity. The pore size varies from submicron to 112 μm , with an average size of 43 μm . When the filters were exposed to a gasifier environment, the pore size distribution broadened.
- Thermal diffusivity of the filter material ranged from 0.005 to 0.009 cm^2/sec in the temperature range of 25-1000°C. These values were approximately one-half of dense mullite. The reduced value was due to the high porosity of the filter material.
- The flexural strength of the flange sections of the as-manufactured and Texaco-exposed filters was 27.1 ± 2.0 MPa and 28.2 ± 1.9 MPa, respectively, whereas the strength of the web sections was 22.3 ± 2.3 and 21.3 ± 2.3 MPa, respectively. The higher strength values of the flange section may have been due to smaller critical flaw sizes in the flange section, which may be related to microstructural differences between the materials in the flange and the web sections.
- The Weibull modulus (m) of the strength data for the material in the web section was reduced from 14 to 9 after exposure to the gasification environment. This implied that material strength reliability was reduced.
- Fracture toughness values for the flange material from the as-manufactured and Texaco-exposed filter were 0.62 ± 0.08 $\text{MPa}\cdot\text{m}^{1/2}$ and 0.77 ± 0.07 $\text{MPa}\cdot\text{m}^{1/2}$, respectively. The corresponding values for the web section were 0.44 ± 0.07 $\text{MPa}\cdot\text{m}^{1/2}$ and 0.49 ± 0.13 $\text{MPa}\cdot\text{m}^{1/2}$, respectively. The higher fracture toughness values of the flange material may have been related to microstructural differences. Fracture toughness seemed to be unaffected by the use of the filter in a gasifier environment.
- The elastic modulus of the material in flange sections of the as-manufactured and Texaco-exposed filters was 16.1 ± 0.3 GPa and 16.5 ± 0.3 GPa, respectively, whereas an elastic modulus of the web section material for the two filters was 22.7 ± 0.4 GPa and 20 ± 0.3 GPa, respectively. The results indicated that the elastic modulus of the flange section was lower than that of the web section (i.e., the web section was stiffer than the flange). This may have been due to a slightly higher density of the web than in the flange.

- The critical flaw sizes calculated by fracture mechanics, using the measured mechanical properties, were 216-465 μm for the as-fabricated filter. The corresponding values for the filter exposed to the gasification environment were 330-584 μm , suggesting an increase in the critical flaw size of the filter material in the gasification environment due to thermal and/or mechanical fatigue.
- Thermal shock experiments indicated a continuous drop in the strength of the filter material with increasing of thermal shock severity. This observation suggested the presence of large critical flaws in the filter material, in agreement with the microscopic observations and critical flaw size fracture mechanics calculations.
- Microstructural investigations revealed needle-shaped mullite crystals on the pore surfaces of the filter. The morphology of the mullite crystals, however, appeared to have changed after exposure in the gasification environment.
- Static and dynamic finite element stress analyses for the pulse-cleaning cycle of the filter were conducted with a micro-model (considering the details of the gas channels) and macro-model (ignoring the details of the gas channels). Fracture mechanics analyses of a micro-model with preexisting cracks of various lengths at the channel corners were also performed.
- Stresses due to a pressure drop of 20 psi across the filter plates in the internal channels were quite low and should not cause failure. The maximum permissible temperature drop in the filter plate corresponding to the measured MOR using a value of 10 GPa for elastic modulus varied from 600 to 1000°F. The permissible temperature drop was 300 to 500°F if the measured value (20 GPa) of elastic modulus was used.
- Fracture mechanics analyses of the internal channels showed that, for a pressure drop of 20 psi across the plate, the filter plate could sustain a crack length of one-half the thickness of the filter plate without experiencing unstable fracture. If the temperature drop across the filter plate were >600°F, the maximum permissible crack length for the internal channels would be <0.015 in. Permissible crack lengths could be significantly larger (i.e., >0.04 in.), if the temperature drop across the plate were <400°F.
- Stress analyses of the two-dimensional macro-model showed that the maximum tensile stresses in the fillet junction between the filter and the flange were much larger for a 20-psi pressure loading than for a static 10-g acceleration loading. Further, the maximum stresses increased by a factor of ~2 when the clamping condition was changed from uniform to a line contact at the free end of the flange. A similar change in the clamping condition increased the stresses under the clamps drastically (i.e., factor of ~7). Because such narrow contact regions had been detected at Westinghouse, cracking under the clamp may have been caused by these stresses. However, the maximum tensile stress (1,400 psi) in the fillet was still not sufficiently high to cause fracture.
- Comparison of the natural frequencies of the two- and three-dimensional models showed that for the same mode shapes, the computed natural frequencies were within 10% of each other. However, the three-dimensional model predicted two additional modes involving out-of-plane bending and twisting with frequencies intermediate between the first two

modes of the two-dimensional model. The fundamental- and second-mode frequencies of the two-dimensional model decreased from 329 and 1175 Hz to 216 and 649 Hz, respectively, as the clamping condition was changed from uniform to a line contact at the free end of the flange. These reduced frequencies agreed fairly well with the frequencies (200-220 Hz and 590-620 Hz) measured at Westinghouse. The other measured frequency of 350-360 Hz could correspond to either the second or the third mode of the three-dimensional model under a similar change in clamping condition. Near coincidences of the measured (Westinghouse) and calculated frequencies provided further evidence for the narrow contact between the clamp and the flange. However, the effects of nonuniform clamping in the longitudinal direction (i.e., z-direction) of the flange should be determined.

- Transient-dynamic analyses (with 1% critical damping) of the two-dimensional model of the filter showed that tensile stresses as high as 1,000-1,200 psi could be generated in the fillet within a few cycles if the flange was shaken at near-natural frequencies of the filter in the horizontal (x) direction. Together with pressure-induced stresses, the tensile stresses approached the fracture strength of the material. Further, if the transient-dynamic cycles persisted for more than 1 sec during each pulse-cleaning, they represented a large number of cycles at an appreciable stress amplitude at the fillet that could potentially cause subcritical crack growth due to fatigue. However, because these stresses depend on the damping of the system, a better definition of damping should be obtained.

The analyses performed at ANL suggested that in order to minimize the maximum stress in the filter, a linear contact between the clamp and the flange at the flange free end should be avoided. A similar linear contact at the end of the fillet did not increase the maximum tensile stresses in the filter appreciably, although the maximum compressive stress under the clamp could remain high.

A comparison of the calculated values of maximum operating stress and stress intensity factors with the measured room-temperature mechanical properties indicated that thermal loadings, pressure pulse loadings during cleanup, or 10-g acceleration loadings (to simulate dynamic effects during pulse-cleaning) were not high enough to cause immediate cracking of the filters during first-time loading. Together with the observation that the filters operated for ~77 hours before cracking, the latter finding indicated that the observed failure was very likely caused by long-term fatigue and/or degradation of materials properties at elevated temperatures.

The life of the alumina/mullite ceramic cross flow filter may depend on the fatigue properties of the porous matrix including material degradation during bench-scale or field testing. Alternately, life may also depend on the severity of the maximum stresses that the filter is subjected to during operation. The maximum operating stresses might be enhanced if the filter is supported in a way for which it was not designed. For example, as indicated above, examination of cross flow ceramic filters after failure at Texaco indicated evidence of the clamps being cocked during testing, so that the

reaction forces on the flange were concentrated on a narrow linear region near the free end of the flange. In addition, nonuniformity of clamping was also observed in the longitudinal direction of the flange. These departures from "normal" design criteria will adversely affect the life of the ceramic filter.

5. ALTERNATE FILTER CONCEPT DEVELOPMENT

5.1 POROUS CERAMIC CROSS FLOW FILTERS

In order to provide a more "ruggedized" cross flow filter element, production of a monolithic unit was recommended. As a monolith, delamination issues that resulted from debonding of the sintered stacked seams and/or plates would be eliminated.

Efforts were focused at the DuPont Lanxide Corporation on developing a prototype cross flow filter utilizing their chemical vapor infiltration (CVI) techniques to deposit silicon carbide (SiC) along a Nicalon™ preform (Figure 5.1). High temperature, high pressure testing of the cross flow prototype in Westinghouse's pressurized fluidized-bed combustion simulator test facility in 1993 indicated that the DuPont SiC-SiC composite filter demonstrated excellent particle collection efficiencies at process operating temperatures of 1300-1500°F (i.e., <5 ppm dust were detected in the clean effluent gas stream).⁽¹¹⁾ During the twenty-six hours of steady state operation, the baseline pressure drop increased from 2.5 in-wg to ~7.0 in-wg.

Although excellent filtration performance was demonstrated within the early hours of operating life of the DuPont prototype cross flow filter, the incorporation of an integral flange-body configuration is needed prior to further performance qualification testing. In addition, the stability of the nonoxide filter matrix needs to be demonstrated during long-term operation in high temperature, combustion or gasification environments.

Similarly Blasch Precision Ceramics demonstrated successful fabrication of a monolithic segment of the cross flow filter element in 1992-1993 (Figure 5.2). Blasch's injection molding technique appeared to provide a relatively cost effective solution to the previous stacked-plate cross flow filter design. The injection molded matrix could conceivably be manufactured from a broad range of ceramic materials (i.e., oxides, nonoxides, or mixed oxide-nonoxides). During 1994 and 1995 efforts at Blasch were focused on increasing gas flow permeability through the injection molded matrix, as well as maintaining strength. Scaleup to a permeable 12x12x4 inch cross flow filter element has been considered.

Figure 5.1 — DuPont SiC-SiC Prototype Cross Flow Filter Element

Figure 5.2 — Blasch Injection Molded Cross Flow Prototype Filter Section

5.2 POROUS CERAMIC CANDLE FILTERS

Coors Ceramics utilized many of the improvements developed for the cross flow filter to successfully manufacture 1.0 m and 1.5 m candle filter elements in 1990-1991 (Figure 5.3). Initially the candle filters were manufactured utilizing pore forming materials. The pore former materials were subsequently eliminated in 1991-1992 after Coors demonstrated production of the cross flow filter matrix without the inclusion of these materials.

Coors produced ~200 candle filters for installation in the Westinghouse Advanced Particulate Filtration system which was operated in the pressurized circulating fluidized-bed combustion (PCFBC) system at the Ahlstrom test facility in Karhula, Finland. At Karhula, the alumina/mullite candle filters were operated for a period of 716 hours at ~900°C in the PCFBC environment. Both Westinghouse, as well as the Argonne National Laboratories characterized several of the filter elements which had been removed after testing. A discussion of the material characterization results is presented in Section 6.

In the initial production lots (i.e., 1990-1993), the Coors alumina/mullite candle filters were constructed with a blunt end cap which contained internal corners. Based on the stress analyses conducted by Adapco, the internal corners were expected to act as stress risers and crack initiators during process operation of the candle filter element.⁽¹²⁾ As a result of the Adapco analyses the candle filter end cap was redesigned to contain a full radius (Figure 5.4), and subsequently filters were manufactured and installed in the Westinghouse Advanced Particulate Filtration system at the American Electric Power pressurized fluidized-bed combustion (PFBC) Tidd demonstration plant in Brilliant, Ohio.

5.3 POROUS CERAMIC SHEET FILTER

As an alternate means of producing a "ruggedized" monolithic filter element, Westinghouse in conjunction with Coors Ceramics developed the sheet filter element (Figure 5.5). As a monolith which lends itself easily to production, the sheet filter concept provides many attractive advantages when compared to either the existing cross flow or candle filter concepts. These advantages include:

- The sheet filter is a relatively robust filter concept which is less fragile and susceptible to breaking during shipment, installation, and during potential ash bridging and process thermal transients.
- The sheet element is relatively compact and light weight, and can be easily installed within a particulate removal system.

Figure 5.3 — Coors Alumina/Mullite Candle Filter

Figure 5.4 — Redesigned Alumina/Mullite Candle Filter End Cap

Figure 5.5 — Coors Alumina/Mullite Sheet Filter

- As a monolith, fabricating time for manufacture of the sheet filter and cost are reduced relative to the more labor intensive production of the stacked-plate cross flow filter configuration.
- As a monolith, the potential issue of delamination in the sheet filter is eliminated.
- A relatively compact array of sheet filters can be installed within a filter vessel, providing an overall increase in the total filtration surface area in comparison to either the cross flow or candle configurations. Section 8 provides a more in-depth discussion of the sheet filter array packaging which has been considered for implementation by Westinghouse, pending successful qualification testing of the sheet filter concept under simulated process gas operating conditions.

6. CANDLE FILTER MATERIAL EVALUATION

During 1991 and 1992, modifications to the manufacturing process for the alumina/mullite cross flow filters were made at Coors Ceramics. These included:

- Production of the filter element without the use of pore former materials.
- Specification of appropriate powder milling times and cooling rates to produce a strengthened crystalline matrix.

Similarly during this time period, Coors focused their efforts on manufacturing a high strength alumina/mullite filter matrix. Not only were cross flow filters fabricated using the manufacturing process modifications, but the technology was transitioned to additionally manufacturing 1.0 m and 1.5 m alumina/mullite candle filters.

In 1992, ~200, 1.5 m candle filters were successfully manufactured at Coors for installation in the Westinghouse Advanced Particulate Filtration system in the pressurized circulating fluidized-bed combustion (PCFBC) environment at the Ahlstrom test facility in Karhula, Finland. Of these, 128 candle filters experienced 716 hours of operation in the ~900°C PCFBC environment. At Westinghouse, post-test characterization of the PCFBC-exposed alumina/mullite candle filters typically included room temperature and process temperature (i.e., 900°C) C-ring compressive and tensile strength testing, burst strength and fracture toughness analyses, x-ray diffraction (XRD) analyses, and scanning electron microscopy/energy dispersive x-ray analyses (SEM/EDAX).⁽¹³⁾ A discussion of the results of these analyses follows.

Figure 6.1 illustrates the residual bulk strength of the alumina/mullite filter matrix as a function of process operating time in the PCFBC environment. An initial loss of the bulk strength of the alumina/mullite filter matrix was experienced within the first 50 to 100 hours of operation. The bulk strength of the matrix then stabilized, and remained relatively constant throughout the remainder of the 616 to 666 hours of test operation.

Typically a greater loss of bulk material strength was experienced along the inner wall surface of the PCFBC-exposed alumina/mullite filter matrix (i.e., tensile testing: 46-49% residual inner wall strength; compressive testing: 71-83% residual outer wall strength; Table 6.1). Strength reduction was principally attributed to thermal fatigue of the alumina/mullite filter matrix as a result of the inner wall surface being contacted with the cold pulse cleaning gas.

(a) Candle Installed During Last Three Test Segments (No Fuel Event)
 (b) Fractured Filter Element

Figure 6.1 — Residual Bulk Material Strength of the Alumina/Mullite Filter Matrix after Exposure to the PCFBC Process Gas in the W-APF System

Table 6.1

Percent Strength Retention in PCFBC-Exposed Alumina/Mullite Candle Filters

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

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

During the 716 hours of PCFBC test operation in Karhula, Finland, the W-APF vessel experienced a fuel ignition event, isolated ash bridging, and a shift in the vessel shroud position — all of which were considered to have contributed to failure of thirty-six candle filter elements throughout the entire test period. As shown in Figure 6.1 and Table 6.2, the fractured candles and/or ash hopper candle sections exhibited a lower residual bulk strength, as well as reduced fracture toughness and/or elastic modulus in comparison to candles that remained intact throughout the course PCFBC testing.

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

Characterization of the alumina/mullite filter matrix utilizing SEM/EDAX analytical techniques indicated that during the initial several hundred hours of test operation, surface crystallization of the amorphous phase occurred in the PCFBC-exposed filter matrix. Mullitization (i.e., rod-like grain growth) was evident along the inner surface pore cavity walls. Nucleation or grain growth within the amorphous phase of the matrix leading to the formation of mullite, appeared to be enhanced as a result of repetitive exposure to cold pulse gas cycling conditions.

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

As shown in Table 6.3, the as-fabricated amorphous phase in the alumina/mullite matrix was initially converted into alumina and cristobalite. Further reactions included depletion of the anorthite phase, as both the mullite (i.e., the principal phase) and cristobalite (i.e., a secondary phase) concentrations increase. After 488 hours of PCFBC exposure, the glass phase in the alumina/mullite matrix appeared to have been completely converted into a crystalline phase.

After 716 hours of PCFBC exposure, tridymite which is a mineralized form of cristobalite, was not detected within the alumina/mullite filter matrix. This implied that virtually little or no gas phase sodium had been sorbed into the alumina/mullite filter matrix during the 900°C PCFBC test

Table 6.2

Fracture Toughness and Elastic Modulus of the Alumina/Mullite Candle Filters Tested at Karhula

Candle	Location	PCFBC Process Test Runs	Time Hrs	Density, g/cc	Strength, MPa	Fracture Toughness, MPa·m ^{1/2}	Elastic Modulus, GPa
<u>1992 Production Lot</u>							
C-35	—	—	—	1.69±0.05	—	0.62±0.03	21.6±1.4
AC-0	—	—	—	1.72 ±0.01	23.9±1.2	0.71±0.003	25.5±1.8
AC-Section (a)		1 - 2	<143	1.67±0.02	—	0.43±0.01	12.9 ±2.2
AC-79	T19	1 - 2	143	1.73±0.06	18.8±1.3	0.51±0.20	17.7±2.4
AC-121RB	T22			1.69±0.04	15.2±0.9	0.50±0.01	15.3±2.7
AC-150RA	B38	1-4	488	1.69±0.02	13.6±1.8	0.41±0.01	—
AC-128RA	T3	3 - 5 (b)	573	1.70±0.01	16.4±2.3	0.61±0.01	22.3±7.1
AC-204	M2	1 - 5	716	1.73±0.11	13.0±3.3	0.61±0.01	19.2±4.5
AC-274	B22	1 - 5	716	1.71±0.02	19.8±0.9	0.60±0.01	26.8±3.9
AC-80 (c)	B19	1 - 5	716	1.69±0.01	18.4±1.6	0.70±0.01	27.5±6.5
<u>1993 Production Lot</u>							
BC-337	—	—	—	1.69±0.02	21.7±1.0	0.70±0.004	24.5±0.9

(a) Fractured Section of Candle Removed from the Ash Hopper.

(b) AC-128RA Was Installed and Operated during the Last Three Test Run Segments.

Note: This Candle Did Not Experience the Initial Fuel Event as All of the Other Candle Filters.

(c) Fractured Candle.

Table 6.3

Phase Composition of the Alumina/Mullite Candle Filters Tested at Karhula

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

* May Be as High as 10%

** >2.1%

M: Major

s: Significant

m: Minor

t: Trace

(a) AC-128RA Was Installed and Operated during the Last Three Test Runs at Karhula. This Candle Did Not Experience the Initial Full Event as All of the Other Candle Filters.

campaign. Cristobalite or tridymite were expected to crystallize from the silica glass phase that is rejected during production of the anorthite in the alumina/mullite matrix.

In addition to the efforts conducted at Westinghouse, several of the PCFBC-exposed candles were sent to the Argonne National Laboratory (ANL) for material characterization. From ANL's effort, a report entitled "Mechanical Characterization and Stress Analyses of Ceramic Filters" was prepared for publication, and is presented in Appendix F. Highlights from this effort follow:

- The as-manufactured filters were generally observed to have higher mechanical properties than filters exposed to the PCFBC environment. The strength, fracture toughness, and elastic modulus of as-manufactured filters were ~22-24 MPa, ~0.7 MPa·m^{1/2}, and ~25 GPa, respectively. These values decreased to ~13-19 MPa, ~0.4-0.6 MPa·m^{1/2} and ~15-2 GPa, respectively, for the filters which experienced ~130-475 hour of PCFBC operation. Degradation in mechanical properties of the exposed filters was believed to be due to the bulk damage induced during hot gas filtration testing. Alternately the mechanical properties of the filters which had been exposed for longer time (~716 hours) in the PCFBC environment were slightly higher than those of exposed filters for shorter times (~130-475 hours). This slight increase in mechanical properties was possibly related to filter-to-filter statistical variation in flaw population and resulting strength distribution.
- In general, the Weibull modulus (m) of strength data for the as-manufactured filters were larger than those for the exposed filters. The general trend of a decrease in the values of "m" for exposed filters as compared with those for as-manufactured filters suggested a larger scatter in strength data, indicating a wider range of flaw population in the exposed filters. This may have resulted from the growth of pre-existing cracks (flaws) due to the filters being subjected to mechanical and/or thermal stresses during PCFBC operation.
- Critical flaw sizes of filter materials were estimated to assess the microstructural damage during exposure of the filters to the service environment. The calculated value of critical flaw size was 528±44 µm for the as-manufactured filter, and 680±252 µm for the filters exposed to the PCFBC environment. The higher of critical flaw size value for the exposed filters was probably due to an increase in flaw size as a result of thermal and mechanical fatigue of the filters during exposure to the combustion environment.
- Phase identification of the filter materials by x-ray analysis indicated that the filters contained aluminum silicate (mullite, 3Al₂O₃·2SiO₂) as a major phase, and anorthite (CaO·Al₂O₃·2SiO₂), SiO₂, and Al₂O₃ as minor phases.
- Microstructural investigations indicated that the as-manufactured and exposed filters had a porous microstructure with randomly oriented grains. Pore sizes ranged from a few microns to several hundred microns. Typically, the grains were ~10 µm. In addition, localized regions of agglomerated rod-shaped phases were observed on the fracture surface of both as-manufactured and exposed filters. These localized regions of rod-shaped phases were identified to be Si and Al-rich mullite phases. Some of rods-shaped mullite and Si and Al-rich mullite phases were larger than the average grain in the mullite matrix. Presence of these phases may have an important implication for the failure of filters.

- Strength measurements generated by ANL agreed with Westinghouse's data which indicated that the bending strength of the alumina/mullite filter material decreased by approximately 33% after 100 hours of service exposure.
- Stress analysis calculations were also performed at ANL which indicated that microcracking along the interior wall of the filter element could result both circumferentially, as well as axially during process operation. The actual depth of the crack would depend on the filter material, and the effect of microcracking on stress redistribution.

7. MEMBRANE COATING DEVELOPMENT

The original Coors Ceramics alumina/mullite cross flow and candle filters were manufactured without the use of an external membrane. Although pore surface blinding had not been experienced in the field, penetration of fines to depths of 1-3 pores had been encountered. In order to mitigate infiltration of fines within the Coors alumina/mullite filter matrix, development of a membrane coating along the outer surface of the filter elements was undertaken.

Prior to fabricating membrane coated 1.0-1.5 m candle filters, efforts were directed at Coors to manufacture disc samples consisting of the original P-100A-1 alumina/mullite filter matrix, as well as finer and coarser grained matrices (i.e., P-100B and P-100I, respectively). All matrices were formulated without the use of pore former materials.

Room temperature gas flow resistance measurements through 0.25 inch thick discs of each material indicated that the resistance of the standard P-100A-1 filter matrix was ~1 in-wg/fpm (Table 7.1). This value served as an acceptable gas flow resistance value for 0.25 inch thick ceramic filter discs, prior to scaleup to a full filter body. The coarse support structure, P-100I, had a lower gas flow resistance (i.e., 0.12 in-wg/fpm), indicating a high permeability and porosity within the matrix. In contrast the dense P-100B matrix had a higher gas flow resistance, and therefore lower gas permeability and porosity, both of which are beneficial attributes for a membrane.

As a result of the encouraging disc gas flow resistance measurements, full body candle filters were produced. These elements were manufactured using the P-100I body as the coarse support structure. The P-100B matrix was then co-pressed along the outer surface of the P-100I candle filter, (i.e., P-100B/I body). As shown in Table 7.2, the gas flow resistance through the P-100A-1 filter element was nearly comparable to the P-100A-1 disc gas flow resistance measurements (i.e., 2.79 vs 3.49 in-wg/fpm-in, respectively). In contrast the coarse P-100I candle, and membrane coated P-100B/I candle had significantly higher gas flow resistances in comparison to similarly manufacture discs. In an attempt to resolve the apparent discrepancy between the disc and filter gas flow resistance measurements, scanning electron microscopy analyses were conducted to identify the resulting morphology of each filter matrix. Similarly a second set of filters was manufactured in the event that possible production issues may have been responsible for the apparent gas flow resistance inconsistency.

Table 7.1
Room Temperature Gas Flow Resistance Through
Alumina/Mullite Filter Disc Samples

Disc Matrix*	Gas Flow Resistance Measurements	
	in-wg/fpm	in-wg/fpm-in
P-100A-1 (Standard)	0.87	3.49
P-100B (Dense Membrane)	3.44	13.77
P-100I (Coarse Support)	0.196	0.78
P-100B on P-100I**	0.56	2.24

* 0.25 inch Disc.

** P-100B/P-100I.

Table 7.2
Room Temperature Gas Flow Resistance Through
1.5 m Candle Filters

Candle Filter Matrix*	Gas Flow Resistance Measurements	
	in-wg/fpm	in-wg/fpm-in
P-100A-1 (Standard)	1.11	2.79
P-100I (Coarse Support)	2.70	6.79
P-100B on P-100A-1**	3.30	8.47
P-100B on P-100I**	28.90	73.4

* 10 mm Wall Thickness.

** P-100B/P-100A-1; P-100B/P-100I.

In the original P-100I and P-100B formulations, laboratory grinders were used to size the raw materials. Since these materials had been depleted, production batch grinders were used to size the P-100B and P-100I raw materials for production of additional 1.5 m candles. The standard isostatic pressing procedures utilized to produce the P-100A-1 alumina/mullite candles were used to manufacture the P-100I and P-100B/I candles. As shown in Figure 7.1, the isostatically pressed P-100I and P-100B/I candle filters had higher gas flow resistances in comparison to the originally pressed disc gas flow resistances. When the microstructure of the materials was characterized, it became readily apparent that the grains (i.e., agglomerates) used to produce the P-100I alumina/mullite matrix were "crushed" during isostatic pressing of the full-scale filter element (Figures 7.2 through 7.9). Crushing of the agglomerates, reduced the porosity of the filter matrix, particularly along the outer and inner walls of the candle filter, resulting in an increased gas flow resistance through the filter elements which contained the P-100I body.

Although Coors successfully demonstrated that a fine grain membrane could be applied along the outer surface of either the original P-100A-1 or coarse P-100I structure, efforts are needed to refine the appropriate isostatic pressure used to form the body, prior to high firing. In this manner, a permeable, membrane coated alumina/mullite filter is expected to be manufactured.

Figure 7.1 — Room Temperature Gas Flow Resistance through the Various Coors Alumina/Mullite Candle Filter Matrices

- P-100A-1: Standard Alumina/Mullite Filter
- P-100I: Coarse Support Alumina/Mullite Filter Body
- P-100B/A: Membrane Layer on the Standard Body
- P-100B/I: Membrane Layer on the Coarse Support Body

Figure 7.2 — Fresh Fractured P-100A-1 Alumina/Mullite Filter Matrix

Figure 7.3 — Higher Magnification Micrograph of the P-100A-1 Alumina/Mullite Filter Matrix

Figure 7.4 - Fresh Fractured P-100B Alumina/Mullite Filter Matrix

Figure 7.5 – Higher Magnification Micrograph of the P-100B Alumina/Mullite Filter Matrix

Figure 7.6 – Fresh Fractured P-100I Alumina/Mullite Filter Matrix

Figure 7.7 — Higher Magnification of the P-100I Alumina/Mullite Filter Matrix

Figure 7.8 – Morphology of the Cross-Sectioned Wall of the P-100I Alumina/Mullite Candle Filter (OD Surface)

Figure 7.9 — Higher Magnification of the P-1001 Alumina/Mullite Filter Matrix

8. ADVANCED PARTICULATE FILTER CONCEPT

Coors Ceramics in conjunction with Westinghouse developed an alternate porous ceramic filter concept for use in advanced coal-fired applications. As shown in Figure 8.1, the sheet filter was successfully manufactured at Coors in 1994 as either a 6x6x1 inch or a 12x12x1 inch filter element. The sheet filter concept maintains the potential packaging benefits offered by the cross flow filter, while simultaneously providing improvements over the cross flow with respect to ease of manufacturing.

As part of an initial evaluation conducted at Westinghouse, the sheet filter was identified to have several attractive advantages not only with respect to manufacturing, but also to packaging within a specified vessel dimension.⁽¹⁴⁾ The light weight compact sheet filter element was considered to be relatively easy to install, as well as to change-out during scheduled maintenance. The sheet filter was considered to be less fragile and therefore less likely to break in comparison to the long, thin candle filters, particularly during shipment, installation, and in the event of ash bridging during process operation. Similarly as a monolith, the sheet filter would not be susceptible to delamination which had previously been encountered in the stacked-plate cross flow filter design.

8.1 SHEET FILTER ARRAY CONFIGURATIONS

The 12x12x1 inch sheet filter has an effective filtration surface area of 2 ft² in comparison to ~3 ft² for a 1.5 m long, 60 mm OD candle filter. Maintaining the following Westinghouse Advanced Particulate Filtration (W-APF) vessel dimensions that were used at the American Electric Power Tidd demonstration plant in Brilliant, Ohio, an initial packaging of the sheet filters was attempted using three cluster arrays, similar to the candle filter cluster configuration that was used at Tidd in 1992 (Figures 8.2 and 8.3):

- Volume inside the shroud = 1,655,214 in³
- Height of the shroud = 21.5 ft
- Inside shroud diameter = 90.38 in.

Utilizing a 1 inch clearance between the sheet filters along a given column, and maintaining 6 inches as the closest distance between any adjacent filter, 684 sheet filters could be packaged within the filter vessel, resulting in an effective filtration surface area of 1,368 ft². Within the W-APF at

Figure 8.1 — Porous Alumina/Mullite Sheet Filters

Figure 8.2 — Star Sheet Filter Cluster Design

Figure 8.3 — Orientation of a Filter Layer Along the Star Sheet Array Cluster Concept

Tidd, 384 candle filter were utilized in three cluster arrays which provided an effective filtration surface area of $1,152 \text{ ft}^2$. An estimated void or open space of 741 ft^3 resulted when the three candle filter clusters were installed in the W-APF Tidd vessel. A void space of 837 ft^3 was estimated for the three star sheet cluster arrays. Unlike the candle cluster concept, the star sheet array eliminated the use of the shed between the individual plenums. In this manner, build-up of ash along the sheds would not occur.

Maintaining the 1 inch distance between sheet filters along a common column, an alternate cross or "x" configuration was developed for packaging the $12 \times 12 \times 1$ inch sheet filters (Figures 8.4 and 8.5). In this manner, 760, $12 \times 12 \times 1$ inch sheet filters could be installed in the W-APF Tidd vessel. The effective filtration surface area of the cross sheet filter configuration was calculated to be $1,520 \text{ ft}^2$, with a void space in the vessel of 849 ft^3 . By utilizing the cross sheet cluster design versus the star sheet cluster design, an increase in the total surface area of 152 ft^2 resulted, while marginally increasing the total void space area within the vessel. With respect to system maintenance (i.e., replacement of a failed sheet), both the star sheet and cross sheet clusters were expected to be moderately easy to maintain.

Additional total available filtration surface area could be achieved if the $12 \times 12 \times 1$ inch sheet filters were arranged in a row sheet array (Figures 8.6 and 8.7). If the 1 inch distance between sheet filters was maintained along a common column, 912, $12 \times 12 \times 1$ inch filters could be housed within the W-APF Tidd vessel. The total effective filtration surface area for this design was $1,824 \text{ ft}^2$, with a void space of 818 ft^3 inside the vessel. Maintenance of this configuration was considered to be difficult, particularly if the failed sheet filter was not positioned along the periphery of the clusters.

Conceivably the 1 inch distance between adjacent sheet filters along a common column may not be adequate in terms of the filter mount design. The design for holding and sealing the individual sheet filters was expanded to a 6 inch distance between adjacent sheet filters along a common column (Figure 8.8). As an expanded row sheet array, 672 filters would be housed within the W-APF Tidd footprint. The total effective filtration surface area for this design was $1,344 \text{ ft}^2$, with a void space of 836 ft^3 in the vessel. The expanded row sheet design has more filtration surface area in comparison to that of the W-APF candle cluster design that was used at Tidd, and more effective use of the total volume within the vessel. Maintenance of the expanded row sheet design was considered to be difficult in the event of filter failure within the cluster.

A final design was considered to further utilize the void space in the W-APF Tidd footprint. As shown in Figures 8.9 and 8.10, 1,178 sheet filters could be fitted into the vessel in a staggered sheet design. The total effective filtration surface area for this design was estimated to be

Figure 8.4 — Cross Sheet Filter Cluster Design

Figure 8.5 — Orientation of a Filter Layer along the Cross Sheet Array Cluster Concept

Figure 8.6 — Row Sheet Filter Cluster Design. Minimal Distance of 1 inch Was Maintained between Sheet Filters along a Common Column

Figure 8.7 — Orientation of a Filter Layer along the Row Sheet Array Cluster Concept

Figure 8.8 — Expanded Row Sheet Filter Cluster Design. A Distance of 6 inches Was Maintained between Sheet Filters along a Common Column.

Figure 8.9 — Staggered Sheet Filter Design

Figure 8.10 — Orientation of a Filter Layer along the Staggered Sheet Array Cluster Concept

2,356 ft², with a void space of 764 ft³. Maintenance of the staggered sheet design was, however, expected to be very difficult, due not only to the compactness of the unit (i.e., removal of an internal filter, particularly one that was not along the periphery of the cluster), but also the expected complex plenum configurations needed for pulse gas cleaning.

As shown in Table 8.1, the row/column design was identified as the optimal geometry for the sheet filter concept with respect to the maximum filtration surface area, and the potential ease of filter installation. Table 8.2 provides a comparison of the number of sheet, candle, and cross flow filters that would be used for installation in the W-APF vessel, the residual void space within the vessel, the design distance between each filter element, and the distance between the lower filter elements and the ash hopper.

8.2 SHEET FILTER SYSTEM ASSEMBLY

In the sheet filter concept, clusters would be suspended from a split tubesheet. Both tubesheet sections would contain six filter column support plenums, each of which forming a filter array (i.e., twelve column support plenums per vessel; total of twelve filter arrays; Figure 8.11). A top c-channel support beam and potentially a bottom c-channel support beam were included as part of this design so as to provide additional structural rigidity to the individual sheet filter clusters. Each column support plenum contains fifty-six, 12x12x1 in sheet filter elements which provide a maximum total filtration surface area is 112 ft².

Five inch ID plenum pipes with associated flanges are positioned above each filter column support plenum. As the top flange and plenum pipe assembly are fitted through the tubesheet, retaining rings are positioned below the top flange. A dust ring would then be welded to the tubesheet and top flange to separate and seal the clean gas side of the vessel from that of the dust-laden dirty gas area of the hot gas filtration vessel.

Once the filter column support plenums were installed into the tubesheet, fail-safe/regenerators and sheet filters would be individually installed into the support plenums to form a common array and/or cluster. Each fail-safe/regenerator was designed to be used for two sheet filters concurrently, allowing ease of both assembly and maintenance of these units, as well as reducing the total number of individual parts needed during assembly.

As an alternate option, the fail-safe/regenerator could be installed along the top section of each sheet filter. In order to prevent the pulse cycle gas from being directed into only the top several rows of sheet filters, shielding would be provided along the fail-safe/regenerator boxes, deflecting the

Table 8.1
Comparison of the Sheet Filter Cluster Design Concepts with the
Current Candle Filter Cluster Design

Filter Element	Number of Filters	Spacing Between Filter Layers, in	Total Filter Surface Area, ft ²	Void Space Between Inside Liner, ft ³	Maintenance **
Candle	384*	12	1152	741	1
Star Sheet	684	1	1368	837	1
Cross Sheet	760	1	1520	849	1
Row/Column Sheet	912	1	1824	818	2
Expanded Row Sheet	672	6	1344	836	2
Staggered Sheet	1178	1	2356	764	3

* W-APF Tidd Vessel

** Filter Replacement During Maintenance: 1: Easy; 2: Difficult; 3: Very Difficult

Table 8.2

Porous Ceramic Filter Concepts

Sheet Filter Arrays

- 12x12x1 inch sheet filters
- 672 sheet filters
- 1344 ft² total sheet filter surface area
- Sheet filter and support structure volume
 - ~ 19% used by sheet filters and support structure
 - ~ 81% open void space inside shroud
- The distance between the sheet filters was maintained at 5.00 inches
- The distance between the bottom of the lowest filter to the top of the ash hopper is 12 in

Candle Filter Arrays

- 1.5 m candle filters
- 60 mm candle filter OD surface
- 3 filter clusters, containing a total of 9 candle arrays
- 384 candle filters
- 1152 ft² total candle filter surface area
- Candle and support structure volume within the W-APF system
 - 22% used by candles and support structure
 - 78% open void space inside shroud
- Closest distance between filter OD surfaces is 2.3 inches
- The distance between the lower plenum filters (i.e., end cap) and the top of the ash hopper is 5 inches

Cross Flow Filter Array

- 12 in x 12 in x 4.125 in cross flow filters
- 138 cross flow filters
- 1104 ft² total cross flow filter surface area
- Cross flow filters and support structure volume
 - 12% used by cross flows and support structure
 - 89% open void space inside shroud
- Closest distance between filter surfaces is 2.4 inches
- The distance between the bottom of the lowest cross flow filter to the top of the ash hopper is 8 inches

Figure 8.11 — Assembly of the Column Support Plenums in the Split Tubesheet for the Sheet Filter Array Configuration

pulse cycle gas down the filter column support plenum. Similarly, the fail-safe/regenerator could be divided into two sections, permitting each sheet filter to have a stand alone system.

During assembly of the sheet filter array, rear brackets are welded onto the filter column support plenum, and hexhead bolts are slotted into the rear bracket. Hexhead nuts are then screwed onto the bolts and secured into place. Bottom gaskets are placed on top of the rear brackets, middle gaskets are placed around the outer edge of the sheet filters, and top gaskets are positioned along the top of the sheet filter flange. A top bracket is then positioned over the gasketed filter flange, and bolted into place.

The back pulse system that was used in sheet filter array design was based on previous pulse cleaning experience at the W-APF vessel at AEP (Figure 8.12). On a single sheet filter array, fifty-six sheet filters with a total surface area of 112 ft² would be cleaned with the delivery of a single cleaning pulse.

The pulse cleaning cycle would be triggered by a predetermined pressure drop that results as a dust cake layer forms along the outer surface of the sheet filters, or by a timing sequence. After the back pulse system is triggered, various plenums can be pulsed sequentially or as groups until all twelve arrays are pulsed.

Manway locations are also provided in the sheet filter array concept at the top of the dome, and at four additional entry locations along the body of the vessel. The manway locations are provided as visual inspection ports to view the conditions of the sheet filters during intermittent shutdown periods, and if maintenance is warranted, the manways would serve as entrance areas into the vessel in order to perform minor maintenance (i.e., filter change-out) during routine shutdown operations.

The expected ease of maintenance has been taken into account during the design of the sheet filter array. For example, if a sheet filter element which is adjacent to the shroud fails, maintenance may possibly be conducted through the manway. In the event that maintenance and/or change-out via the manway is not an option, the plenum array in question or entire split tubesheet cluster may be lifted out of the vessel, and appropriately serviced.

8.3 SUMMARY

- The sheet filter is viewed as a potential improvement over the cross flow filter with respect to manufacturing, while the potential packaging benefits offered by the cross flow filter are maintained.
- The sheet filter design provides an increase in filter surface area over that of the candle and cross flow designs.

Figure 8.12 — Orientation of Pulse Gas Pipes in Relation to the Split Tubesheet for the Sheet Filter Array Configuration

- The weight of the sheet filter internals is expected to be greater than either the candle or the cross flow filter designs.
- Free or void space in the pressure vessel is greatest for the cross flow filter design.
- Spacing between adjacent sheet filters is greater than either the cross flow or candle filter array concepts. The increased spacing between filters is expected to significantly mitigate ash bridging between filter elements.

Several additional advantages have been identified with respect to the sheet filter design. These include the compact geometry of the sheet filter in comparison to the long (i.e, 1.5 m), thin (60 mm OD) candle filter, as well as the total effective clean surface area of the sheet filter in comparison to the cross flow filter that is plugged with ash fines. If plugging results, the 7.8-8 ft² cross flow filter surface area is nominally reduced to 2 ft² which is comparable to that of the sheet filter.

During manufacturing, installation, and handling, the compact sheet filter design is expected to be less susceptible to breaking in comparison to the thin candle filters. Similarly during process operation, the sheet filter will not experience debonding along adjacent plates which had previously been observed during operation with the alumina/mullite cross flow filter elements.

The overall design of the sheet filter array provides a direct passage of the ash released from the filter surface during pulse gas cleaning into the ash hopper. The sheet filter design minimizes ash build-up along the column support plenum clusters, as well as along adjacent filter elements. Since the dust sheds are omitted between the sheet filter arrays, ash accumulation and bridging are minimized.

9. SUMMARY AND CONCLUSIONS

- The P-100A-1 alumina/mullite material that was developed by the Coors Ceramic Company has been used to manufacture both the cross flow and candle filter elements. The P-100A-1 alumina/mullite filter has been shown to exhibit chemical and compositional material stability in high temperature, coal-based, process gas environments.
- Manufacture of the Coors P-100A filter body originally utilized pore former materials to provide adequate gas flow permeability through the filter matrix. Improved body homogeneity and control of pore size was achieved not only by eliminating the use of pore former materials, but also by appropriately sizing the initial raw materials prior to pressing individual P-100A-1 alumina/mullite flat plates. Eliminating "green body" shrinkage during firing was achieved, as well as increasing the bulk strength of the fired body.
- Process temperature strength of the alumina/mullite filter matrix was increased by "slow cooling" the filter body from high firing conditions. Slow cooling enhanced conversion of the amorphous or glass phases, leading to the formation of a higher hot strength anorthite and mullite-enriched filter material.
- The reliability of the gasketing and mounting arrangement used to contain and seal the 12x12x4 inch cross flow filter flange was substantially improved with the development of the "window frame" gasket design, and a "rocker arm" plenum attachment. The gasket design was subsequently adapted with limited modifications for use in candle filter applications.
- Although construction advantages were realized when the cross flow filter was manufactured via the mid-rib bond/paired plate design in comparison to that of the original rib tip-to-plate construction, delamination has not been entirely eliminated during operation in coal-based applications. This has been attributed to the inability to achieve uniform bonding along the plate ribs and/or seams using the stacked-plate/high firing manufacturing procedures. Various approaches were evaluated for improving plate bonding including the application of slip coatings, different weighting schemes, and direct bonding — none of which appeared to offer a long-term, reliable solution.
- Injection molding and chemical vapor infiltration (CVI) were identified as manufacturing processes that can effectively produce a monolithic cross flow filter configuration. The feasibility of both processing approaches has been demonstrated and reported. Filter scaleup, an economic assessment, and successful qualification testing of both processing approaches remain to be demonstrated.
- The porous ceramic sheet filter was identified as an alternate high temperature barrier filter concept. The sheet filter configuration retains the packaging advantages originally envisioned for the cross flow filter design. Significant additional advantages such as improved cleaning and ash removal may also be realized. The sealing and gasketing technology developed for the cross flow filter is directly applicable for use with the sheet

filter concept. Manufacturing of the porous ceramic sheet filter in a 12x12x1 inch rectangular geometry has been demonstrated using the P-100A-1 alumina/mullite matrix. Continued development of the sheet filter concept is recommended.

- Cost effective nondestructive evaluation (NDE) techniques for detecting critical flaws within the full-scale alumina/mullite cross flow filter body are currently not available. Efforts should be directed to developing NDE techniques which can effectively be utilized as either an inspection tool throughout the entire filter element manufacturing process cycle, or as a means of identifying the remaining service life of field-tested filter elements.

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APPENDIX A

FRACTOGRAPHY OF THREE CERAMIC CROSS FLOW FILTERS AFTER TESTS

Fractography of Three Ceramic Cross Flow Filters After Tests

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ABSTRACT

Fractographic examination of three cracked ceramic cross flow filter elements after high temperature operational tests revealed that the cracking was caused partly by fatigue, and partly by overload. Imperfections in machining and bonding of the filter component plates appear to have contributed to the crack initiation and propagation.

1. INTRODUCTION

The fractographic examination of three filters after tests, described in this technical memo, constitutes a contribution to the ongoing work of the Gas Cleaning and Energy Systems Section (M31) at the R&D Center in the program of evaluation of a ceramic cross flow filter of Westinghouse design. The objective of this program is to develop a basis for evaluating the potential thermal, mechanical and/or chemical degradation mechanisms that could possibly affect the integrity of the ceramic cross flow filter during operation under high temperature [538°C to 1370°C (1000°F to 2500°F)] coal combustion or gasification conditions.

A typical specimen of the Westinghouse ceramic cross flow filter element that was tested at the KRW simulator facility is shown in Figure 1. The filter is made of "Coors alumina", which actually consists of mullite, corundum and anorthite, with some oxides of magnesium, iron, potassium and sodium.*

The three filters which were fractographically examined have been identified as KRW-5, NYU-M4B and NYU-M5B.

The investigated specimens were portions of the above filters broken off at about one quarter to one third of the length of each filter element. Most of the fractures developed in operation of the filters during high temperature tests, but in some cases smaller portions of the fractures were produced in the laboratory after the tests, during opening of the original cracks. The ash deposits and discolorations were helpful in differentiating between the two types of fractures. The KRW-5 specimen broke off completely during the tests.

*Example: 52.29% Al_2O_3 ; 39.64% SiO_2 ; 4.79% CaO ; 2.80% MgO ; 0.46% Fe_2O_3 ; 0.31% K_2O ; 0.18% Na_2O , (weight percent)

Figure 1 - Examples of a Coors 12x12x4 inch flanged cross flow filter element.

The NYU-M4B and NYU-5B filter elements were still holding together after testing, but the NYU-M4B specimen required very little effort in the laboratory to break it off entirely.

Following is some background information on these filter elements, and the results of their fractographic examination.

1. EXAMINATION OF THE FILTER SPECIMENS

1.1 FILTER KRW-5

This filter was tested in the Pressurized Fluidized Bed Combustion (PFBC) simulator Module 3, filter element No. 5, top locations.

It was subjected to multiple tests, with maximum temperatures reaching 1550°; 1600°F; 1625°F and again 1550°F. The gas pressure levels were 115 psi, 78 psi, and 115 psi, while the gas velocities at the face were 4.3 ft/min, 6.1 ft/min, and 4.3 ft/min. The dust contents of the gas were in the range of 1000 to 2000 ppm. The total operating time of the filter during the tests was 158.5 hours.

Two views of the examined specimen which broke off the filter element KRW-5 are shown in Figures 2 and 3. The top surface in Figure 2 represents the fracture face. The fracture was examined with unaided eye and using a low power binocular optical microscope. The illumination with a high intensity light was directed at a low, glancing angle to the fracture plane so that the high spots and ridges would cast shadows increasing the contrast of the fracture image.

Figure 2 - Specimen KRW-5. Overall view of the examined specimen, broken off a ceramic filter element after operational tests. The top surface is the fracture plane. The fracture started at the corner at point A and propagated from right to left.

Figure 3 - Specimen KRW-5. Side view of the specimen.

Detailed study of the fracture propagation ridges revealed that the primary origin of the fracture was located at the sharp corner marked A in Figures 2 and 4. Such a corner was a strong stress raiser, particularly since the plate below this corner was protruding (see Figure 2) creating a three-dimensional discontinuity. This was aggravated by small defects in the material marked with black arrows in Figure 4.

In view of the inherent brittleness of ceramic materials, any sharp corners, either reentrant (pointing inward) or salient (pointing outward) should be avoided. Use of fillets in the reentrant corners, and rounding-off (or at least chamfering) of the salient corners is strongly recommended.

Besides the primary origin A, the fracture in Figure 2 shows also secondary origins, B, C and D, starting from sharp reentrant corners acting as stress raisers. The fracture then propagated from right to left in Figure 2, traversing the area of the longitudinal ribs. This area did not offer much resistance to the propagation of the fracture because the ribs were predominantly unbonded. The fractures in the few bonded small areas propagated locally across the ribs (e.g., locations E and F in Figure 2) but the overall direction of the fracture propagation was along the ribs, right to left, as mentioned above. At a distance of 5.5 inches from the origin, the fracture reached a transverse crack, shown in Figure 2 (location G) and, at a higher magnification, in Figure 5. However, the crack was too short to affect the progress of the main fracture. On the other hand, the long transverse crack at the location H in Figure 2, shown also in Figures 6 and 7, which extended across several plates, caused the main fracture to move to another level. Now the fracture propagated across the ribs, continuing in the general direction from right to left in Figure 2, until it reached point K where it jumped to still another level,

Figure 4 - Specimen KRW-5. The origin of the fracture at a higher magnification, marked A. The black arrows indicate what appears to be a slight defect in the material.

Figure 5 - Specimen KRW-5. A small transverse crack at the location G in Figure 2, at a higher magnification (marked with arrows).

Figure 6 - Specimen KRW-5. A major transverse crack, extending over several plates, at the location H in Figure 2. The side view of a portion of this crack will be seen in Figure 7.

Figure 7 - Specimen KRW-5. Side view of a portion of the crack from Figure 2 (marked H) and from Figure 6.

spreading sideways from this point. At the same time, part of the fracture continued at the original level at areas M, N, O, P and Q. Area K is shown in greater detail in Figure 8.

Figure 8 shows also half-circular machining marks of the grooves between the ribs. The machining appears to be somewhat inconsistent both in depth and in location along the plates, as will be discussed below.

Figure 3, which represents the side view of the examined specimen KRW-5, shows examples of three channels formed by machined grooves, R, S and T. Both channels R and S have ribs reasonably well in register, i.e., meeting center to center, and yet channel R is considerably wider than channel S. It is conceivable that the ribs in channel S were more compressed in the bonding operation than those in channel R, but it is not very likely. The channels are in the same assembly, and directly in line with each other. Furthermore, Figure 9 provides another example of grooves machined to different depths. The marked part of the rib in the lower photograph of Figure 9 could not have been compressed to a lower height because it was outside the corresponding rib of the mating plate.

The lower photograph of Figure 9 illustrates also the other problem in the machining of the grooves in the plates: inconsistency in the location of the ribs. The mismatch shown in this photograph is not just a shift in the assembly of the plates. This is well demonstrated in Figure 11, showing the side view of specimen NYU-M4B. The top horizontal row of channels shows the ribs reasonably well matching in channel U at the beginning of the row, and almost completely out of register in channel V at the end of the row.

Figure 8 - Specimen KRW-5. Detail of the fracture marked K in Figure 2. At this point the fracture temporarily moved to another plate.

Figure 9 - Upper Photograph: Specimen NYU-M4B.
Lower Photograph: Specimen KRW-5.
Examples of uneven depths of grooves machined
in the plates. The lower photograph shows also
unsatisfactory bonding of the ribs which are out
of register in the specimen KRW-5.

These machining inaccuracies illustrate some of the problems encountered in "custom-making" of filter elements, before the manufacturing process is fully standardized. The variations of the depth of the grooves affect the cross-section area available for the gas flow, while the mismatch of the ribs seriously reduces the strength of the filter elements by reducing the bonding area between the plates and by introducing sharp steps at the ribs acting as stress raisers. Thus, considerable increase in the strength of the filters may be expected with the forthcoming improvements in the machining and in the bonding of the plates.

1.2 FILTERS NYU-M4B AND NYU-M5B

These filter elements were tested on a coal-fired, pressurized, fluidized bed combustor (PFBC). The tests were performed utilizing the existing New York University (NYU) subpilot scale PFBC. The filter unit had a multielement configuration, comprising 15 elements in 5 modules of 3 elements each. Specimen NYU-M4B was in Module 4, at the bottom, and specimen NYU-M5B occupied the bottom position in Module 5. The filters were subjected to two consecutive tests with the following parameters:

Test Parameters	Test No. 1	Test No. 2
Gas Flow Rate	4900 lb/hr	100,000 lb/hr
Gas Pressure	103 psig	150 psig
Temperature	<1500°F	1300°F
Gas Velocity at the Face	52 hrs	30 hrs
Dust Concentration	305-1056 ppm	250-600 ppm

1.2.1 Filter Specimen NYU-M4B

The overall views of the filter specimen NYU-M4B are shown in Figures 10 and 11. The top surface of the specimen in Figure 10 is the fracture face.

Figure 10 - Specimen NYU-M4B. General view of the specimen. The top surface is the fracture face. The progressive cracking in operation of the filter started at the points K, Q and R.

Figure 11 - Specimen NYU-M4B. Side view of the specimen. Note the good match of the ribs at the point U, gradually changing to an almost complete mismatch at the point V.

At the right hand side of Figure 10 the fracture started from the corner marked K and propagated towards M at the other end of this wide, rectangular rib. Figure 12 shows higher magnification photographs of the fracture areas near the origin at K, and in the middle of the rib at L. A careful examination of these photographs reveals a series of curved lines ("beach marks"), which were progressive crack fronts as the fracture propagated along the rib. Unfortunately, these lines are very faint in the photographs, because a camera often cannot record clearly what the eye can see. Therefore, a schematic sketch of the orientation of these lines was drawn from visual observation, and is shown at the bottom of Figure 12. The "beach marks" indicate that the propagation of the crack in this area was progressive ("stable") and not instantaneous. Although in some instances this kind of propagation can be caused by intermittent stress corrosion cracking, in this case the most likely cause appears to have been fatigue. The cyclic stressing in this instance could have come from mechanical vibration, gas pressure pulsation, thermal cycling, repeated thermal shock, or a combination of some of these factors. Again, stress concentration at the sharp corner at K (Figure 10) undoubtedly contributed to the initiation of this fracture.

Propagating from the wide rib K-L-M (Figure 10), the fracture crossed the adjoining narrow rib, and then jumped to the next plate by means of a transverse crack N-O (Figure 10). The latter crack initiated in the N region at the point P (Figure 13). During the operation of this filter element there was also cracking at the other end of the specimen NYU-M4B, in the flanges, at the regions Q and R (Figure 10). Cracks in the flanges could be expected to have started from the sharp, reentrant corners at Q and R; however, in this case the practically unbonded ribs N-Q and O-R constituted the large internal defects which initiated the cracks in the flanges. The flange cracks also propagated in a slow "stable" manner, apparently by fatigue, leaving "beach marks"

Figure 12 - Specimen NYU-M4B. Fracture in the wide rib near the origin (point K in Figure 10), and in the middle of the rib (area L in Figure 10). Note the "beach marks" indicating progressive fatigue crack propagation. The schematic sketch at the bottom of the page shows the general orientations of the successive crack fronts. The area near M was unbonded.

Figure 13 - Specimen NYU-M4B. Detail of the transverse fracture at the point N in Figure 10. Locally, the fracture started at the point P.

or fatigue striations on the flange fractures. These striations are barely visible in Figure 14, but can be well discerned by visual observation with a suitable illumination at a low angle to the fracture plane.

The longitudinal ribs shown in the rectangle N-Q-Q-R in Figure 10 were well bonded at the flange end of the filter (the left hand side of the photograph). Unfortunately, the bonded area of each rib decreased linearly towards the other end of the specimen, so that in the N-Q region (Figure 10) the intermittent bonds existed only along the edges of the ribs. Furthermore, as mentioned before, the two outside ribs N-Q and Q-R were almost completely unbonded on their whole lengths. Consequently, with all the cracks and unbonded areas, the specimen was easily broken off in the laboratory after the tests.

The above observation identifies the third problem in manufacturing of the filter elements: incomplete bonding of the ribs, presumably resulting from nonuniform pressure distribution along and across the ribs in the bonding operation.

Figure 15 shows the fracture of the center rib at the location S in Figure 10. It may be noticed in Figure 15 that the fracture shows typical "chevron marks" pointing upwards towards the origin of the fracture. This indicates that the fracture, which apparently was produced by overload in the laboratory, propagated downwards in Figure 15, or from left to right in Figure 10. The lack of ash deposits or discoloration on this fracture confirms that it was produced in the laboratory after the tests.

The side view of the filter element specimen NYU-M4B seen in Figure 11, shows that in this element the depth of machining of the grooves was correct, providing full size channels for the gas flow, but the matching of the ribs was still inadequate at some locations. Figure 16 provides a close-up view of such a location.

Figure 14 - Specimen NYU-M4B. Fractures in the flanges at the points Q and R in Figure 10. Note the "beach marks" indicating fatigue. These fractures started not from the corners Q and R, but from the almost unbonded ribs N-Q and O-R in Figure 10. The rib crevices acted as long, sharp pre-existent cracks.

Figure 15 - Specimen NYU-M4B. Central rib fracture at the location S (Figure 10). The "chevron marks" in the fracture pointing upwards indicate that the fracture propagated downwards in this photograph, i.e., from left to right in Figure 10. This fracture was produced in the laboratory after the tests, during the opening of the operational cracks. Note also the semicircular machining marks in the groove.

Figure 16 - Specimen NYU-M4B. Detail of ribs out of register. Such mismatch not only reduces the bonding area but also introduces stress raisers in the form of sharp steps.

1.2.2 Filter Specimen NYU-M5B

The overall views of the filter specimen NYU-M5B are shown in Figures 17 and 18. The top surface of the specimen in Figure 17 is the fracture face. It may be noticed that the narrow ribs show rough fractures, indicating that the ribs were reasonably well bonded. In contrast, the wide ribs T, V and W appear smooth, except at the edges, showing that only their edges were bonded. This is illustrated at higher magnifications in Figures 19, 20 and 21 for the ribs T, V and W, respectively.

The large unbonded areas reduced the load-carrying cross section of the filter element and acted as sharp, large, pre-existent internal cracks with high stress intensity factors.

2. SUMMARY OF OBSERVATIONS AND CONCLUSIONS

- A. The fractures in the examined filter elements appear to have occurred partially by fatigue and partially by overload. The fatigue was possibly caused by mechanical vibrations, gas pressure pulsation, thermal cycling, repeated thermal shock, or a combination of some of these factors.
- B. Structural factors contributing to fracture initiation and propagation appear to have been:
 - Partial lack of bonding of the ribs at various locations.

Figure 17 - Specimen NYU-M5B. General view of the specimen. The smooth surfaces of the flange rib T and of the wide ribs V and W indicate that they were unbonded, except for their edges. The narrow ribs were well bonded.

Figure 18 - Specimen NYU-M5B. Side view of the specimen. The fracture face is at the bottom in this photograph.

Figure 19 - Specimen NYU-M5B. Detail of the flange rib T from Figure 17. Note that the rib was unbonded except for the edge. The narrow rib U from Figure 17 shows a fracture indicating a good bond.

Figure 20 - Specimen NYU-M5B. Detail of the wide center rib V from Figure 17. Note the smooth surface of the rib indicating that it was unbonded, except for the edge. The narrow rib in the photograph shows a good bond.

Figure 21 - Specimen NYU-M5B. Detail of the end rib W from Figure 17. Its smooth surface indicates that it was unbonded, except for the edges.

- Inaccuracies in machining of the individual plates of the filter elements, resulting in occasional mismatching of the ribs of the neighboring plates to be bonded together.
- sharp corners and other stress raisers.

The above problems are associated with the "custom machining" of the individual plates, and will be eliminated in the standardized process.

3. ACKNOWLEDGEMENT

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- M. A. Alvin and J. E. Lane - background information on the filter specimens.
- J. McKenzie - overall photographs of the filters.
- R. House - typing and preparation for printing of the report.

APPENDIX B

EVALUATION OF THE BONDING STRENGTH AND CHARACTERISTICS OF THE COORS CROSS FLOW MID-RIB JOINT

EVALUATION OF THE BONDING STRENGTH AND CHARACTERISTICS OF THE COORS CROSS FLOW FILTER MIDRIB JOINT

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ABSTRACT

The tensile strength and degree of bonding were evaluated for the midrib joints in a Coors cross flow filter. Observations of the joints before and after testing indicate that joint misalignment and inadequate load transfer during filter fabrication results in incomplete bonding of the midrib joints. The actual bonded joint areas ranged from 55-68% and from 35-46% of the total midrib joint bond area for joints from the clean and dirty gas channel portions of the filters, respectively. The tensile strength of joints from the clean and dirty gas channels ranged from 638-891 psi and 364-954 psi, respectively.

1. INTRODUCTION

Fractographic examinations of failed cross flow filters indicate that poor bonding between the plates making up the filter contributes to crack initiation and propagation and eventual failure of the filters. To date, an adequate method has not been developed for evaluating the bonding characteristics of the joints between mating plates.

The Coors cross flow filters are composed of extruded plates bonded together at the center of the dirty and clean gas channel walls, i.e., the midrib joints. The channels are arranged in alternating layers with the dirty and clean gas channel directions mutually perpendicular to each other. Figure 1.1 shows the above mentioned features of the filter.

During fabrication, the extruded plates are stacked together and weighted down. The weights are intended to ensure rib-to-rib contact along adjoining plates so that the ribs, and thus the plates, bond to each other during firing. However, the geometry of the extruded plates prevents complete load transfer along the entire rib joint. Load transfer only occurs at the intersections of the mutually perpendicular clean and dirty gas channel ribs.

Because there is this potential for limited load transfer, incomplete bonding may occur at periodic locations along the various midrib joints which bond the filter together. These unbonded regions could act as cracks in the filter. A stress state which places these cracks in tension may cause crack propagation and, eventually, failure of the filter.

A test method has been developed to examine the degree of bonding and the tensile strength of the midrib joint. This test method and the results and conclusions of the test are presented in the remainder of this report.

Figure 1.1. Portion of a cross flow filter without the mounting flange. Note the orthogonal relationship between the dirty and clean gas channels and their respective midribs.

2. EXPERIMENTAL PROCEDURE

Test specimens were obtained from the top (12 x 4 x 3 in.) section of a Coors cross flow filter manufactured in the Jan. to Feb. 1989 timeframe. This filter was originally intended for evaluation in the Texaco gasifier. Five midrib joint test specimens were machined from the clean and dirty gas channels, respectively, for a total of ten samples. The two sets of samples are intended to determine if there are any differences between joints from the clean and dirty gas channels.

The filter section also contained aligned and misaligned midrib joints as shown in Figure 2.1. All the clean gas channel test samples had misaligned midrib joints. The dirty gas channel test samples had both aligned and misaligned joints. The degree of joint misalignment was measured for all test samples in terms of the percentage of the unbonded rib width portion (or mismatched portion of the rib width) to the total rib width.

The test sample shape resembled a squat I-beam as shown in Figure 2.2(a). The overall sample dimensions were 3.7 x 1.8 in. and 1.6 x 1.4 in. for the dirty and clean gas channel test specimens, respectively. The test specimen length dimension also represents the length of the midrib joint. The length of the clean gas channel joint was limited by the size of the filter portion to 1.6 in. In a full size filter, these channels are approximately 10 to 11 in. long. The length of the dirty gas channel joint was not limited, as their normal 4.0 in. length was available for testing.

The top and bottom surfaces of each sample were bonded to (3.75 x 2.0 x 0.75 in.) aluminum blocks with Hysol No. 608 Quickset Optically Clear Epoxy, as shown in Fig. 2.2(b). The large overall sample width, as compared to the midrib width, maximized the adhesive bonding area, thereby, increasing the load carrying capacity of the adhesive. This

Figure 2.1. Examples of misaligned and aligned mid-rib joints in a Coors cross-flow filter.

bonding arrangement was to ensure that failure occurred at the midrib joint and not at another location in the test sample configuration. The center of each aluminum block had a $3/4 \times 10$ mounting hole for an Instron Mechanical Test Machine. Each sample was mounted in the Instron and pulled apart at a crosshead speed of 0.050 in./min. The total available bond area and the tensile breaking load of each midrib joint were recorded.

The midrib joint fracture surfaces were observed by eye and by a low magnification optical microscope. The actual midrib joint bond area was measured from each of the fractured samples. The midrib joint tensile strength was determined from the breaking load and the actual bond area, not the total available bond area, of the midrib joint.

Figure 2.2. Drawings (not to scale) which show (a) the test sample configuration, and (b) how a test sample is mounted for tensile testing of the midrib joint.

3. RESULTS AND DISCUSSION

The midrib joint tensile strength results are shown in Table 3.1. There were not enough test samples to give meaningful statistical data; however, there does not appear to be any strength difference between the clean and dirty gas channel midribs. The sample-to-sample strength variability was less for the dirty, 638-891 psi, than for the clean, 364-954 psi, gas channel midribs.

Samples 9 and 10 had the highest degree of midrib misalignment, 50%, and the lowest actual bond area. Both these samples fractured during mounting in the Instron. These two samples were obviously weakened by midrib misalignment and a low bond area. Because of the low number of test samples, no other correlations can be made between the degree of misalignment of the other midrib samples and their respective failure stresses.

TABLE 3.1. Midrib Joint Tensile Strength Results*

Sample	Degree of Misalignment (% of rib width)	Actual Bonded Joint Area (in. ²)	Breaking Load (lbs)	Tensile Strength (psi)
1, dirty	10	0.29	207	714
2, "	5	0.35	225	646
3, "	10	0.40	255	638
4, "	15	0.33	294	891
5, "	10	N/A	276	N/A
6, clean	20	0.11	105	954
7, "	0	0.11	40	364
8, "	0	0.11	61	562
9, "	50	0.052	---	---
10, "	50	0.047	---	---

*Samples 9 and 10 fractured at the midrib joint during mounting.

However, the reduction in bond area, whether from misalignment or poor bonding during fabrication, results in a decrease of the load carrying area. This decrease places the bonded areas under higher stresses at lower applied loads than anticipated (stress = applied load / area under load) by a component design analysis which assumes aligned and completely bonded midrib joints. The sharp edges formed by misaligned joints act as stress concentrators, as do the poorly bonded regions in the joint. Because of the above conditions, these joints can fail prematurely, thereby placing more load on the other joints in the filter. The remaining joints will be overloaded and the filter will fail sooner and at lower loads than expected.

A comparison of the total available bond area and the measured actual bond area of the midrib joint for each sample is shown in Table 3.2. The actual bond area for the dirty and clean gas channel test samples ranged from 55-68% and 26-46%, respectively, of the available bond area. As noted previously, both misalignment and poor bonding during fabrication contribute to these low bond areas. The much lower, on average, bond area of the clean gas channels indicates that more cross flow filter failures may initiate between plates which form these

TABLE 3.2. Comparison of the Total Joint Area to the Actual Bonded Area

Sample	Total Joint Area (in. ²)	Actual Bonded Area	
		(in. ²)	(% of total area)
1, dirty	0.52	0.29	55.8
2, "	0.55	0.35	63.6
3, "	0.59	0.40	67.8
4, "	0.54	0.33	61.1
5, "	0.61	N/A	N/A
6, clean	0.24	0.11	45.8
7, "	0.31	0.11	35.5
8, "	0.31	0.11	35.5
9, "	0.20	0.052	26.0
10, "	0.18	0.047	26.1

channels than those that form the dirty gas channels. However, longer clean gas channel midrib samples should be tested for a more valid comparison.

Photomicrographs of sample 4, a tested dirty gas channel midrib joint, are shown in Figures 3.1 and 3.2. These pictures are representative of all the tested samples. A planar view of the fractured dirty gas channel midrib joint is shown in Fig. 3.1. The textured or rough fracture surface areas indicate where good bonding occurred in this midrib joint sample. In these areas, the fracture occurred in the midrib material, not at the joint. These good bonds occur periodically along the entire fracture surface, as do the smooth fracture surfaces which are indicative of poor bonding.

The periodic rough fractures occur at the intersection of the dirty gas channel midrib with the underlying, and unseen, clean gas channel midribs. This is clearly seen in Fig. 3.2 where the arrows denote these intersections. The alternating good and bad bonds are a result of poor load transfer along the length of the midrib joint during fabrication.

The poorly bonded areas are essentially large cracks within the cross flow filter. These cracks occur in a periodic 2D arrangement between mating plates, and within a filter they form a periodic 3D array of potential crack initiating sites. These sites weaken the filter and simplify crack propagation. They can also blunt propagating cracks; however, in the long run they weaken the filter.

Figure 3.1. A planar or cross-sectional view of the mating surfaces of a clean gas channel midrib fractured in tension.

Figure 3.2. Photomicrograph showing the relationship between the well-bonded areas of the fractured clean gas channel midrib to the location of the perpendicular dirty gas channel midribs (denoted by arrows).

4. CONCLUSIONS

1. A test method was developed and shown to be an effective procedure for evaluating the tensile strength and degree of bonding of the midrib joints in a cross flow filter.
2. The dirty and clean gas channel midrib joint strengths ranged from 638-891 psi and 364-954 psi, respectively.
3. The true bonded midrib joint areas ranged from 55-68% and 26-46% of the total midrib joint area for the clean and dirty gas channel midribs, respectively.
4. All the clean gas channel midrib samples were misaligned from 5-20% of the midrib width. The dirty gas channel midrib samples contained both aligned and misaligned samples. In the latter case, the misalignment ranged as high as 50%.
5. Observations of the joints before and after testing indicate that joint misalignment and inadequate load transfer during filter fabrication results in incomplete bonding of the midrib joints.

5. RECOMMENDATIONS

1. Sufficient midrib joint strength testing should be conducted on as-fabricated filters to acquire statistically meaningful data for use in finite element stress analyses.
2. The midrib joints of filters exposed to actual use environments should be evaluated to determine if bond strength degradation occurs after exposure.
3. This test method should be used to evaluate the effect of changes in filter fabrication procedures on the midrib joint bond strength and the degree of bonding of the midrib joint.
4. Filter fabrication procedures should be modified to prevent midrib joint misalignment and to optimize the degree of bonding of the midrib joint.
5. The desired midrib bond strength is unknown and should be determined by an appropriate design analysis, such as a finite element stress analysis.
6. The test method should be modified to test midrib joint samples at potential use temperatures in order to determine the effect of temperature on joint strength.

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APPENDIX C

SCREENING EVALUATION OF CROSS FLOW FILTER GASKET MATERIALS FOR HOT GAS CLEANING APPLICATIONS

Screening Evaluation of Cross-Flow Filter Gasket Materials for Hot Gas Cleaning Applications

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ABSTRACT

Westinghouse has developed a hot gas particulate cleaning system which can be used in pressurized fluid bed combustor (PFBC) power plants or in waste to energy incinerator plants. The particulate cleanup is accompanied by means of alumina/mullite or clay bonded silicon carbide barrier filters. These porous ceramic filters are typically supported on metal fixtures inside a refractory lined pressure vessel. Because the ceramic filter and stainless steel fixture exhibit widely differing thermal expansion at the high operating temperature (1600°F), a rugged gasket material is required to form a resilient interface between the ceramic and metal. In addition, this gasket material should form a reliable barrier to prevent the migration of dust to the clean side of the filter system.

A test program was conducted to evaluate various materials for use as gaskets in hot gas cleaning systems which use the alumina/mullite cross flow filters. Ten materials were tested and qualified for their ability to fulfill the following three requirements:

- Long-term structural integrity and resiliency at high temperature
- Dust sealing capacity
- Gas sealing capacity.

This report presents the results of this test program and ranks the materials according to the degree of meeting the above requirements. Recommendations are made to which materials merit further development for this application.

Summary

Table 1 lists the gasket materials tested in this program in order of performance for meeting the following criteria:

- Maintains structural integrity at high temperatures
- Effective dust sealing capacity
- Effective gas sealing capacity at high temperatures.

TABLE 1

RESULTS OF CERAMIC FILTER GASKET MATERIAL TEST PROGRAM
(Listed in Order of Relative Performance)

Gasket Type	High Temperature Structural Integrity ⁴	Dust Sealing Capability?	Gas Sealing Capability @ 1550°F 1(Poor) 10(Good)	Hours Tested (Hrs Pulsed @ Temperature)	No. Cycles Tested
Nextel/Interam Mat Composite	Yes	Yes	8-9	48.2	2893
Nextel/Interam Mat With Springs	Yes	Yes	9	43.6	2619
Siltemp	Yes	No @ 1600°F Yes @ 1000°F	8-10 ¹	136.8	8210
Interam/MM Mat Fiber Blanket	No ⁵	Yes	8-9	330.3	19817
Fiberfrax 550K Ceramic Paper	Yes	No	8	30.4	1826
Nextel/Interam Mat Composite	Yes	No	4-5 ²	16.0	963
Hytex With Wires	Yes	No	1	7.7	464
Interam Mat	No	No	1	2.8	166
Selco Metal Gasket	No ³	No	1	0.2	13
Hytex With Fiberfrax	Yes	No	---	0	0

1 — 1000°F Test.

2 — Bolt Configuration May Have Caused Failure. Threads Through Clamp Section Caused Bending Of Bolts Decreasing Torque On Stainless Steel Plate, Causing Leakage To Occur.

3 — Grafoil O-Ring Decomposed.

4 — Conditions Noted Upon Disassembly.

5 — Adhered To Stainless Steel Plate Upon Disassembly.

Of the materials tested, the Nextel/Interam MM Composite, the Siltemp material and the thick Interam MM Fiber Blanket meet the above criteria.

Introduction

A test program was conducted to evaluate various materials for use as high temperature gaskets in ceramic hot gas cleaning systems. These gaskets provide gas and dust tight sealing between the ceramic filter and the metal filter holder.

A test facility was designed and constructed as shown in Figure 1. For each test, the gasket material was compressed between the holder and a stainless steel plate which substituted for a ceramic cross flow filter. A "cold" (i.e., ambient temperature) check for leakage was conducted prior to hot (i.e., 1550°F) testing.

Pulse gas pressure rise inside the gasket holder was measured with a pressure transducer connected to a high-speed strip chart recorder. This allowed for the evaluation of the gasket material's gas sealing capability. The dust sealing capacity of these materials was determined visually by noting the presence of PFBC dust on the gasket sealing surface, or in cases of extreme leakage, in the furnace cavity. The high temperature structural integrity of the materials was also determined visually by noting the physical condition of the material after testing.

Conclusions

Based on the test results acquired through this test program, in terms of meeting the performance criteria of dust and gas sealing capability and high temperature structural integrity, the materials meriting further consideration for this application are the Nextel/Interam MM Composite, the Siltemp material and the thick Interam MM Fiber Blanket.

Recommendations

We recommend that the Nextel/Interam Mat Composite, the Siltemp material and the thick Interam MM Mat undergo long-term durability testing for possible use on the American Electric Power (AEP) Tidd Demonstration Plant and other hot gas filtration projects. In addition to the performance criteria used in this test program, testing should include frequent hot/cold cycling similar to what may be encountered during field operation. We also recommend that the gasket rig be modified to incorporate the improvements in the filter mount design, which were developed as part of the long-term durability test program conducted at Westinghouse. These improvement will ensure that the gasket sealing forces are uniformly distributed and maintained reasonably constant over the entire span of hot testing.

Experimental

The ceramic filter gasket test facility utilized to conduct this test program is schematically shown in Figure 1. It consists of the following major components:

Figure 1 — Schematic of the Filter Gasket Test Facility

Gasket Holder

The filter gasket holder used in this test program was positioned inside a furnace. The design and construction of the filter gasket holder and clamps were essentially the same as those used in the Westinghouse high temperature, high pressure, fluidized-bed combustion simulator test facilities, with the exception that the filter gasket holder was not attached to a plenum.

Furnace

The furnace used in this test program was a 1000°C Linberg furnace with a penetration through the back wall to allow for the passage of an 0.5 inch pulse gas tube.

Pulse Gas Reservoir

A 450 inch³ (0.26 ft³) steel cylinder was used as the pulse gas reservoir. This pulse gas reservoir was fed by a standard 1A size cylinder of nitrogen.

Pulse Solenoid Valve

The pulse solenoid valve used for pulsing was an ASCO model number 8210D2. This is an 0.5 inch normally closed valve with a 5/8 inch orifice, and an operating pressure rating of 200 psi, and a C value of 4.0.

Pulse Timer

An in-house designed and constructed pulse valve timer was used to control the length of time the pulse valve was open. Actually, the timer controlled the length of time electric power was supplied to the solenoid valve. Because of the mechanical lag time of the valve, the actual time the valve was open was obtained by analyzing the trace on the high-speed strip chart recorder.

Pulse Pressure Transducer

A Viatran 0-5 volt pressure transmitter was used to measure the pulse pressure inside the gasket holder. This was accomplished by running a small (0.125 inch) tube down the 0.5 inch pulse gas delivery tube leading to the gasket holder. The transducer was fitted to one end of the 0.125 inch tubing while the other end was positioned in the center of the filter gasket holder.

High-Speed Pulse Reorder

The output from the pressure transducer was fed to a Hewlett-Packard high-speed strip chart recorder to allow the recording and measurement of the pulse pressure rise and fall inside the

filter gasket holder. These data indicate the sealing capabilities of the particular gasket material being tested.

Pulse Counter

An electro-mechanical counter was connected to the pulse timer and used to record the total number of pulses performed during each gasket material test.

Test Procedure

The gasket material was cut or supplied in a size which would fit into the filter gasket holder. Approximately 25 grams of PFBC dust was placed on the floor of the holder and was used as a leak indicator which provided post-test identification of any leak paths across the gasket sealing surface. A stainless steel plate was then placed on top of the gasket. About 40 lbs of weight was placed on the plate to load and compress the gasket as per Westinghouse standard filter installation procedures. The side clamps were positioned on the stainless steel plate and the bolts and nuts tightened to 5 in-lbs. In one test, Inconel 750X Belleville springs were installed between the nuts and the gasket holder to provide additional resilient force at high temperature.

The holder was positioned inside the furnace and the pulse gas tubing, which enters the furnace from the rear, was connected to the compression fitting on the gasket holder. The solenoid valve was opened and the test unit was pressurized to ~30 psig. The gasket holder was checked for gross leakage.

Most of the tests were initiated by pulsing the gasket holder at either 5, 10, 15, 25, or 50 psig at ambient temperature and recording the pulse pressure rise and decay. The furnace was then powered on and brought to ~1550°F overnight. The gasket holder was then pulsed several times at the previous initial pressure while again recording and comparing the pulse pressure rise and decay. The test was then continued by repeatedly pulsing and periodically making a recording of the pulse pressure rise and decay inside the holder. Each test was continued until an obvious leak was noticed or until it was no longer necessary or appropriate to continue testing.

Test Results

Evaluation of Gas Sealing Capability

The gasket material serves three functions in ceramic filter applications. First, it should be an effective dust barrier or dust seal. Second, it should maintain structural integrity and resiliency at high temperature conditions (1600°F) over 50 to 200 startup and shutdown cycles. Thirdly, the gasket should provide a high degree of hermetic sealing in order to minimize the bypassing of pulse cleaning gas through the gasket. Whereas the first two functions are a prerequisite for a viable filter gasket, the third property would be difficult to achieve at these high temperature conditions. In this gasket test program the dust sealing behavior and the structural integrity were assessed by

means of visual observations. The hermetic sealing behavior was determined by analyzing the pulse pressure rise and decay curves during each representative pulse event.

As illustrated in Figure 2, the pulse rise and decay profiles were recorded on a high-speed recorder during representative reverse pulses. The initial slope of the curve was estimated at the instant of solenoid valve closure:

$$\left(\frac{dP}{dt} \right)_{initial}$$

Assuming that the gasket can be modeled as a lumped flow resistance, analogous to an orifice constant, we developed a flow equation to normalize the effects of pressure and temperature:

$$\dot{m} = \frac{(Mwt) \cdot V}{RT'} \frac{dP}{dt} \quad (1)$$

Compressive flow-through an orifice is given by:

$$\dot{m} = K \sqrt{\frac{dP \cdot P}{T'}} \quad (2)$$

Equating (1) and (2),

$$\frac{KR}{(Mwt) \cdot V} = \frac{1}{(\Delta P \cdot P \cdot T')^{0.5}} \frac{dP}{dt}$$

or

$$K = \left(\frac{1}{\Delta t} \right) \sqrt{\frac{P_i}{(P_i + 14.7)(T + 460)}} \quad (3)$$

where:

K' = Normalized (lumped) orifice constant

T' = Temperature, °R

T = Temperature, °F

P = Initial gauge pressure when the solenoid valve closes, psig

R = Gas constant

MWt = Molecular weight of air

$\dot{m}$ = Mass flow rate, lb/hr

ΔP = Pressure drop across the orifice, psid

V = Control volume in the test rig, ft³.

Figure 2 — Schematic of Gasket Material Test Rig and Pulse Pressure Rise and Decay Profile

Using Equation 3 we were able to estimate the value of K' for various gasket materials and thereby compare their hermetic sealing capability on a normalized basis (Table 2).

Discussion of Results

An attempt was made to rank the gasket materials tested in this program. Table 1 is a list of these materials ranked according to test performance. The three criteria used for this ranking were:

1. Did the material maintain its structural integrity after the test?
2. Did the material possess dust sealing capability?
3. Did the material possess gas sealing ability?

Since these criteria were not considered of equal weight of importance, Criteria 1 was given more weight than Criteria 2, which was given more weight than Criteria 3.

Accordingly, the Nextel/Interam MM Composite is placed at the top of the list because it exhibited both high temperature structural integrity and dust sealing capability, as well as high degree of gas sealing. The other materials fall below the Nextel/Interam MM Composite according to the above criteria.

It should be noted here that although the Interam MM Fiber Blanket has accumulated the longest test exposure and the most pulse cleaning cycles, it has not demonstrated a high degree of structural integrity at high temperature. Specifically, this material pulled apart upon post-test holder disassembly and during field testing of Westinghouse's ceramic cross flow filter system at Texaco in Montebello, California. This problem needs to be addressed for this material to be used for hot gas filter application.

It should be noted from Table 2 that the gasket material exhibiting the lowest average K value, and therefore the highest hermetic sealing capability is the Siltemp gasket. This material accumulated 3880 pulse cycles although the temperature was 1000°F, while all other testing was conducted at 1550°F. However, the Siltemp gasket was tested a second time at 1550°F with dust leakage observed after 4330 pulse cycles. This material is ranked third on Table 1 based on the combined results of the two tests. Further evaluation of this material is needed to determine if it can be used successfully at the higher temperature.

The remaining materials in this test program performed very marginally when compared to the above discussed materials, and are not considered to be viable candidates for hot gas filter applications.

TABLE 2
CERAMIC FILTER GASKET MATERIALS TESTED
(In Test Chronology Order)

Material Tested	Test Date (Start)	Initial Pulse Pressure, psig	Average K @ 1550°F	Total Number Of Pulses	Dust Leakage Noted?
Interam	9/7/89	50	0.29	166	Leakage Noted
Hytex With Wires	9/9/89	50	0.32	464	Leakage Noted
Hytex With Fiberfrax "Tadpole"	9/13/89	50	---	0	Gross Leakage Noted
Selco Metal Gasket	9/15/89	50	0.32	14 Cold 13 Hot	Leakage Noted
Nextel/Interam MM Composite	9/22/89	50	0.20	598	Leakage Noted & Bolts Bent
Nextel/Interam Composite	9/25/89	25	0.18	365	Leakage Noted
Interam MM Fiber Blanket	10/19/89	15	0.085	1450	No Leakage
Interam MM Fiber Blanket	11/2/89	5	0.048	9198	No Leakage ¹
Interam MM Fiber Blanket	11/10/89	10	0.061	9169	No Leakage
Nextel/Interam MM Composite	1/25/90	5	0.046	1438	No Leakage ¹
Nextel/Interam Mat Composite	1/30/90	10	0.060	1455	No Leakage
Siltemp Gasket	2/2/90	5	0.025	3880	No Leakage ²
Siltemp Gasket	2/9/90	10	0.085	4330	Leakage Noted
Fiberfrax 550K Ceramic Paper	3/6/90	5	0.070	1826	Leakage Noted
Nextel/Interam MM With Spring Washers	4/17/90	5	0.056	2619	No Leakage

1 — After Cool-down And Visual Inspection Of Gasket Holder, Second Test On Same Gasket Was Started.

2 — 1000°F Test.

APPENDIX D

INDEX OF CERAMIC CROSS FLOW FILTERS

NDE OF CERAMIC CROSS-FLOW FILTERS

— TOPICAL REPORT —

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ABSTRACT

Nondestructive evaluation (NDE) techniques were used to characterize the alumina/mullite based refractory, ceramic barrier cross flow filters. The characterization included density and porosity measurements as well as the integrity of the mid-rib bonds and gas channel seams. The effect of process variables like firing load and particle size distribution on inspectability was also addressed. Dry coupled and immersion through transmission ultrasonic inspection was the principal test method used in this investigation. Several other methods, which included radiography, x-ray tomography, resonant frequency, acoustic microscopy, magnetic resonance imaging, vacuum infiltration with magnetic particles, and backlighting with fiber optics, were also explored. Results show that ultrasonic tests are able to differentiate the mid-rib and flange bond quality. The bond quality was correlated to the load on firing of the paired plates of the filter. Radiography provided further confirmation of these ultrasonic results.

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1. INTRODUCTION

Westinghouse, in conjunction with the Coors Ceramics Company, has developed a ceramic cross flow filter for application in advanced fossil energy power generation systems. These filters are made by assembling several plates with cross channels into a stack and firing them under load. Evaluation of several ceramic materials in the past characterization work at Westinghouse has led to the choice of alumina/mullite for fabrication of these filters. The successive modifications of the P-100A formulation of alumina/mullite have led to excellent mechanical properties, thermal shock and corrosion resistance, and controlled pore distribution. Fracture toughness, Weibull modulus and critical flaw size were reported by Argonne National Laboratory based on their study done on one batch of P-100A alumina/mullite filters.⁽¹⁾ The critical flaw size was calculated at 500 μm for this material. The flaws were expected to be either in the P-100A matrix as a result of large interconnected pores or the result of a debonded region along the mid-rib or gas channel seam of the multi-plate filter structure.

The improvements and modifications in the design of the filter in the past year and a half were focused in three general and closely dependent categories:

1. Material Processing and choice of parameters
2. Geometrical Designs and interfaces to fixtures
3. Quality Control and Monitoring through NDE

2. PROJECT SUMMARY

The objectives of the program were to improve the reliability, uniformity and mechanical integrity of the cross-flow filter. The NDE techniques were needed to characterize the material conditions of the cross flow filter and then be able to assess its ability to withstand service. The cross flow filters are used in advanced fossil power generation applications where they are subjected to high temperatures (500 to 900° C) and pressures (5 to 20 bar) and possibility of high thermal transients during startup and operation.

Various NDE techniques to characterize and qualify the entire cross flow filter were explored. Bonds of the paired plate configuration, which forms the unit building block of these cross flow filters, were characterized in detail. The end tabs on the flanges, the mid-rib, and the plate seam bonds were given more attention during the investigations. The processing changes that led to the improvement of the strength of the filter body in general, and bonds in particular, could be detected by the ultrasonic characteristics being monitored. Ultrasonic NDE results on paired plates from this study have been applied in the manufacture of full body cross flow filters.

The scope of NDE activities included:

- o Characterize changes in density and porosity of the P-100A alumina/mullite matrix that result due to changes in process variables during cross flow filter production using the ultrasonic time-of-flight (TOF) data.
- o Application of a full range of ultrasonic techniques (UT) that include the use of both the shear waves and longitudinal waves. The UT data were collected using dry couplant contact methods and full immersion methods. These alternate methods were performed at various high and low frequencies to assess optimal sensitivity of the porous ceramic

materials. Most of the work was done in the through transmission mode.

- o Detection of mid-rib bonds and gas channel seams and assessment of bond integrity as a function of process variables in a set of paired plates manufactured with controlled process variables. These series of paired cross flow filter plates were manufactured at Coors Ceramics with the original P-100A alumina/mullite matrix formulation and pre-determined firing sequence, and the application of various weights on the paired plate surface.
- o Perform conventional radiography on paired plates that correlate film optical density to the assessment of bonds.
- o NDE development using high resolution x-ray tomography and acoustic microscopy on small sections of the P-100A alumina/mullite cross flow filter material.
- o Investigate inspection alternatives like magnetic particle vacuum infiltration and resonant frequency as potential full cross flow filter body inspection techniques.
- o Study localized inspection techniques, like γ -radiography, candling and gas leak detection to examine the finished cross flow filter product

3. BACKGROUND INFORMATION

3.1 CROSS FLOW FILTER CONFIGURATION

Advanced power generation systems require hot gas to provide motion to the turbines and subsequently generate power efficiently. Gas streams from developmental systems like the pressurized fluid bed combustors or combined cycle gasifiers are at high temperatures and pressures. The particulate control on these gas streams, before they enter the turbine, requires devices that operate as stable arresters of fine particulate without long term degradation due to temperature, pressure, chemical attack, or other fatigue mechanisms. These particulate control elements must have good reliability, low maintenance, and a high degree of uniformity as essential attributes to protect the costly hot gas turbines from particle erosion and deposition.

These particulate control elements, manufactured from an alumina/mullite based refractory formulation, have proven to be near absolute barriers to the very fine fly ash entrained in the gas streams. These filter elements are called ceramic barrier cross flow filters. They provide the hot gases a low pressure drop path through their thin walled porous channels. The construction of the cross flow filters provide for a high flow capacity, allows the entire filter to sustain high pressure drops, and transmits forces to sintered bonds without stress concentrations. The cross flow design makes the element compact and economical such that the filter surface area to volume ratio is maximized.

The ceramic cross flow filter is illustrated in Figure 1. It is made by assembling several specially shaped "trays" or "plates", described below, and sintering or firing them under load. Each plate has half (U-shaped) channels running lengthwise (12 in.) on the obverse side and half (U-shaped) channels running widthwise (4 in.) on the reverse side. Several of these plates are stacked together so that the ribs that make the half channels match to each other to make the completely enclosed channels. The total stack height is 12 in. Upon firing, these half channels sinter together to make the "mid-rib" bonds. Other flat surfaces at the edges and the center of the plates also sinter together and provide the main load bearing portions of the entire filter body.

Figure 1 — Ceramic cross flow filter assembly.

One end of the long channels is blocked and the other end is open to a flanged face that forces the gas into a plenum. The short channels are open on both sides allowing free passage of particle laden hot gases into the filter. The gas can flow through the thin walls of the short channels (also called "dirty" channels) into the long channels on the other side. During that passage the particles get arrested and the clean gas gets forced into the plenum at one end of these long channels (also called "clean" channels). The particles thus accumulate in the "dirty" channels and are periodically blown out by a "reverse pulse".

3.2 REVIEW OF EXISTING DATA

A catalog of all prior cross flow filter activity, which included changes in firing methods (single or dual), changes in stacking or pairing of filter element plates (rib-to-plate or mid-rib bonds), and in-service test location was prepared.

Several filter elements were subjected to hot gas filtration tests in the various test beds. A systematic failure analysis was performed at STC on the failed filter elements by Madeyski et al., of the Westinghouse STC Materials Reliability Department, and were available for detailed review.⁽²⁾ Portions of these filters were also used in experiments to determine bond strength and other mechanical properties. A thorough analysis of specific failure histories as described by the material characteristics, test operating conditions, post-test condition of the filter, and post-test characterization led to identifying the mid-rib and plate seam bonds as critical for filter durability and reliability.

This review of the above catalog along with the corresponding data from various analyses showed the need for standardization. The standardization was required not only in test procedures, but also in formation of the complete material and filter element profile (i.e., 4-pt bend, 1/4 flexure, Weibull distribution, percent porosity, pore size distribution, pore volume, density, burst strength, dimensional check including flatness and parallel face formation, absence of bowing or flaring, etc.).

Mechanical properties like flexural strength, fracture toughness, modulus of elasticity, and Weibull modulus of the filters were reported.⁽¹⁾ Data were obtained from

an as-manufactured filter as well as a filter that had experienced cracking after some service. The critical flaw sizes ranging from 216 μm to 583 μm were also discussed. The need to evaluate NDE techniques that can be used to specify a quality criterion in the porous structure was identified. Participants at the workshop on "Analysis of Ceramic Candle Filters" held on January 23, 1991 at DOE/METC, Morgantown, West Virginia,⁽³⁾ discussed the generally accepted industry rule of critical flaw size for porous ceramics to be five times the void size.

4. TECHNICAL DISCUSSION

4.1 COORS PROCESS

A significant portion of the efforts was devoted to the understanding of material processing variables, their impact on various properties, and the resulting ability to monitor that change via a non-destructive technique, particularly ultrasonic measurements.

The P-100A composition (P = Porcelain, 100 = 100 μm pellet size) is made from a water slurry suspension of clay ($\sim 5 \mu\text{m}$), alumina, silica, binder (40-50 nm), walnut flour ($\sim 40 \mu\text{m}$), and deflocculent. The binder is an organic material which provides pressing properties and machining capabilities. The particles are spray dried to form small spherical pellets of 100-150 μm size. The pellets are then pressed in a 40 Ton press to a certain green density. The binder and walnut flour burn off at 300-500°C and the particles start "necking" down and adhere to each other at 800-900°C. The high fire is done with a load on the stack of plates.

Table 1 is a description of the process steps in a typical manufacturing sequence and the related QA activities during production. Several NDE techniques were surveyed, with some being applied on selected samples and others were investigated in detail on a controlled sample set.

Attributes that are considered in the end product reliability include material strength, low pressure drop characteristics, high flow capacity, thermal, chemical, and mechanical stability, and long term durability. These are essential to the cross flow filter to act as near absolute barriers to particulate emissions. The method of evaluation of the NDE tests as well as manufacturing process variables, taking into account their interdependence, required the application of experimental analysis like the Taguchi matrix.

TABLE 1
COORS PROCESS FLOW CHART

<u>Department</u>	<u>Operation</u>	<u>QA Activity</u>	<u>Comment</u>
1. Materials Engineering	Raw materials preparation P-100A Al ₂ O ₃ , SOi ₂ shells, Binder	<ul style="list-style-type: none"> • Particle size • Chemical purity 	Raw material specification may be tailored to filter - uncertain
2. CMP - Ceramic Material Preparation	<ul style="list-style-type: none"> • Make slurry from P-100A formulation 		
3. Materials Engineering	<ul style="list-style-type: none"> • Pull sample and check formulation 	Chemical analysis pH Viscosity	
4. CMP - Ceramic Materials Preparation	<ul style="list-style-type: none"> • Spraying drying to form particle (spheres) of powder mixture. • Achieve filter properties that can be pressed. 	<ul style="list-style-type: none"> • Hourly samples to check: Particle size distribution at particular moisture level. 	P-100A difficult material to spray dry
5. Materials Engineering	<ul style="list-style-type: none"> • Characterize powder by forming 1" disks that are fired at filter temperature. Disks are made at different pressures to establish press conditions for manufacturing plates. 	<ul style="list-style-type: none"> • Pore size (Hg) • Porosity • Shrinkage • Fired density 	<ul style="list-style-type: none"> • Disk shrinkage is not representative of plate.

TABLE 1 (continued)

<u>Department</u>	<u>Operation</u>	<u>QA Activity</u>	<u>Comment</u>
6. Shrinkage Control	<ul style="list-style-type: none"> • Analysis shrinkage data (disk data) • Shrinkage = f(pressure) 		
7. Pressing	<ul style="list-style-type: none"> • Manufacturing filter plates (green structure) • Pressed plates are put into overnight dryer to drive off moisture (control moisture) new step 	Weight Thickness X-ray - checks uniformity at corners	<ul style="list-style-type: none"> • Press operation and inspection are iterated until desired uniformity thickness achieved. • Spring back control(?)
8. Milling	<ul style="list-style-type: none"> • Machine grooves in to green plates (CNC-mill) (Note ultimate goal is to press channels.) 	Dimensional	<ul style="list-style-type: none"> • Inspection made only on about 5% to make sure machine setting has not drifted.
9. Stacking for Bisque (1400°C)	<ul style="list-style-type: none"> • Pairing • Spray adhesive (aerosol glue); Now using a wax - hold plates • Fire - paired plates 	<ul style="list-style-type: none"> • Visual • Some measurement for shrinkage 	<ul style="list-style-type: none"> • Dirty side to dirty side - helps alignment • Clean side to clean side - tried • Have gotten 30 to 90% throw-away (before drying was implemented - recent batch no rejects)

TABLE 1 (continued)

<u>Department</u>	<u>Operation</u>	<u>QA Activity</u>	<u>Comment</u>
10. Sorting	<ul style="list-style-type: none"> • Sort paired plates • Wax and stack • Bead seam P-100 (No Walnut) (Attempt to eliminate smiles) • Buffer plates added 		<ul style="list-style-type: none"> • Plates are paired and stacked in accordance to milling sequence.
11. Loading to Kiln and Firing	<ul style="list-style-type: none"> • Stacked filter placed into new fixture (Bung) • Placed on rail car and pushed into tunnel (hydraulic) • 3-day heating cycle • Complete firing 50° C/hr • Recover buffer plates 	Visual dimensional	<ul style="list-style-type: none"> • New fixture controls weight dropping. Wax and fixture keep plates from shifting. Fixture also protects filter from direct flame impingement <p>MOR not done routinely. Can do MOR at elevated temp.</p>
12. Grinding	<ul style="list-style-type: none"> • Grind surfaces as marked on prints • Dry in oven overnight 	<ul style="list-style-type: none"> • Dimensional checks • Bladder test (usually only on one filter reject.) 	<ul style="list-style-type: none"> • Grinding solution is water based. • Filter is washed after grinding. • No longer testing each filter with bladder.
13. Shipping	<ul style="list-style-type: none"> • Packaging and shipping 		

4.2 NDE TECHNIQUES OTHER THAN ULTRASONICS

It was evident that the ultrasonic technique would be an indicator of the bulk condition for the full filter and not individual bonds if the direction of transmission was perpendicular to the plane of the bonding surfaces. The amplitude or time-of-flight through several layers of bonds would provide an average value of the condition being tested.

The bulk condition in ceramics can be assessed with techniques other than ultrasonics. Some of them are simple, like determining the fundamental resonant frequency using impulse excitation (e.g., Grindosonic equipment), and some are more sophisticated, like Magnetic Resonance Imaging and Computed X-ray Tomography.

Several NDE methods were considered to inspect the individual bonds between ribs in a full filter. The channels nearest the edges would provide access for a localized sensor in order to inspect the outer edges of the filter. The techniques investigated in the category of local bond inspection were γ -radiography, back lighting with fiber optic delivered light, gas leak detection using thermal conductivity or chromatography cells, and vacuum infiltration of small magnetic particles for inspection.

4.2.1 γ -Radiography

Several small pieces of cross flow filter were available with mid-rib bonds that were exposed by cutting away the adjacent channels. These samples were tested to determine x-ray equivalence. Acceptable film radiographs were obtained with 75 kV, 5 mA, 3 minute exposure at 60 in. on M-film. Similar radiographic density, on M-film, with a 25 millicurie Americium 241 source were obtained with different time and distance variables. Unfortunately, the size of the source at 5/16 in. diameter x 3/16 in. long was not suitable for channel insertion. Also, the source activity of 25 millicurie was too low and, at source to film distances of 2 in., exposure times between 80 minutes to 120 minutes were required in the experiment. The source should to be at 0.5 to 1.0 curie level in order to decrease exposure times.

The Americium 241 is a 59.5 keV energy source and has an exposure rate of 80 mR/hr at 1 in. distance. This level of keV is judged to be in the correct range to obtain

desirable radiographic defect sensitivity, unsharpness characteristics and x-ray equivalence for this type of ceramic material.

Americium 241, which is a low energy gamma source, is available in different activity levels. It is also appropriately encapsulated to be used in the laboratory. A special order would be needed to obtain the exact size of the source needle and radiographic film and also proper packaging of both to allow insertion in the channels from either sides of the rib to be inspected. γ -Radiography was therefore not pursued beyond the control measurement attempts described above.

4.2.2 Back Lighting with Fiber Optics

A Diaguide pure silica industrial fiberscope made by Mitsubishi Cable Industries was used to examine bonds in the cross flow filter. The smallest flexible fiberscope size is 1.5 mm diameter and 2 meters in length, but it allows only a front view. The fiberscope that would allow a side view is a semi rigid model with a diameter of 1.3 mm. The exploratory experiments were conducted with the light source on one side and the fiberscope camera on the other side of the bond. The experiments showed that the bond material concentrated the light and appeared to be much brighter than the surrounding matrix. Further, this phenomenon occurred whether the light source was held directly on the bond line or illuminated through the thickness of one plate layer. There is a potential to use this property to an inspection advantage but it needs to be evaluated further to understand its effect as a function of bond quality.

4.2.3 Gas Leak Detection

Ideas to introduce a tracer gas on one side of a channel mid-rib bond and to detect the level of the gas concentration from the other side were explored. A constant differential relay to make flow constant on both sides of a thermal conductivity or a gas chromatographic cell was investigated. A concept to introduce gas locally at the bond inside the channel and have a sniffer boot on the other side to detect the trace gas was developed. A Gow-Mac leak detector was acquired to use for the first preliminary effort.

This effort was not pursued any further because a "standard" mid-rib bond had to be established first in order to develop the test further.

4.2.4 Vacuum Infiltration of Magnetic Particles

This method is used to improve the nondestructive inspectability of hard to test materials such as highly porous ceramics by distributing sensor particles into the filter body after manufacture. The infiltration is achieved easily because the particles are in a colloidal suspension in a liquid medium called Lignosite FML, and the size of the particles is 1/5000 the pore size. After the infiltration and drying of the carrier solution, it was possible to map the distribution of the magnetic material using eddy current techniques. Figures 2 and 3 show the results from the eddy current scans of these magnetic particle infiltrated filter pieces. The work was limited to measurements on sample surface, which showed that the eddy current maps correlated with the near surface density distribution and surface flaws of the filter. Subsurface flaws and defects were not found, and would require development of tests that measure permeability as a function of biasing field strengths. The strengths of biasing fields would also be correlated to the depth of penetration below the surface of the eddy current sensing fields.

4.2.5 Resonant Frequency Technique

This technique uses the principle of pulse excitation with a mechanical impact followed by analysis of the transient vibration of the test piece. The energy from the impact is dissipated in the form of a damped vibration, which depends on the geometry of the piece, as well as the density and the elastic properties of the material. A piezoelectric probe captures the vibration and converts it into an amplified analog signal. In the case of the Grindosonic instrument, the signal is analyzed electronically in order to select the fundamental frequency from the complex vibration spectrum. The instrument reading is a relative measure of the test object's rigidity. Young's modulus can then be calculated, which can be correlated to material or component strength.

Candle filters were used to try this technique because they are of a simpler geometry than the cross flow filter and would have a repeatable flexural, longitudinal, or

Figure 2 – Magnetic particle infiltrated filter pieces.

Figure 3 – Color scan of magnetic particle infiltrated filter pieces.

torsional vibration pattern. By selecting the appropriate support points, the candle was also excited in the first harmonic mode. The results were sensitive to several test variables like operator, tube rotation, support contact points, etc. Filters made by three different manufacturers, Schumacher, Refractron, and Coors, were tested. The test was easily able to separate the three materials but within the candle set made of the same material it was not possible to arrive at any conclusion with regard to level of quality or damage. This technique requires a statistically large database to formulate a separation criterion and three samples per material category was not sufficient. Efforts to induce a meaningful vibration pattern in a cross flow filter (WRTX-65) were not successful.

Investigators at the DOE/METC workshop⁽³⁾ reported reservations with tests that use the Grindosonic equipment for resonant frequency analysis of candle filters in particular, and porous ceramics in general. It was reported that Grindosonic does not give repeatable (consistent) Young's modulus results.

4.2.6 Magnetic Resonance Imaging

- The feasibility of using this technique for ceramics was investigated by obtaining information and data from Mellon Institute in Pittsburgh, PA, and Argonne National Laboratories (ANL) in Argonne, IL. The ANL machine is a 100 MHz, 12 Tesla machine capable of performing 3-D back projection with a 40 μm voxel resolution. The best results are obtained by monitoring the proton or hydrogen spin relaxation of spatial frequencies in a steady state gradient. Thus, presence of hydrocarbons like benzene or even water is desirable in this test. This characterization technique is limited by the size of the magnet chamber and can be used only on small sections or segments of the cross flow filter.

Preliminary tests were conducted at the Mellon Institute on two 7 cm long pieces of porous clay bonded silicon carbide material containing small cracks. These pieces were extracted from two failed silicon carbide candles. One candle was cracked in the cylindrical outer wall in the lower coarse grained part of the candle, and the other was cracked below the top flange surface in the fine grained part of the candle.

The use of silicon frequency to scan for material uniformity was discarded in favor of looking for porosity distribution via pores filled with water. The material was impregnated with deionized water and its presence was monitored. The water was forced into the filter pores and interconnecting channels by drawing a vacuum on a closed chamber.

The MRI technique views a 0.5 cm slice and provides a FFT (Fast Fourier Transform) image within a couple of minutes. At a maximum available theoretical resolution of 300 $\mu\text{m}/\text{pixel}$, the image did not show satisfactory contrast to discern whether the known crack could be identified and followed. Increasing the scan time to 10 minutes showed no improvement in the resolution of the images. An example of the resulting image from these scans is shown in Figure 4. The bright areas are the water filled pores of the silicon carbide matrix.

This technique was not able to resolve small visible cracks. It may be used on small coupon segments formed in material development programs, life test efforts or segments removed from field tested filters to provide information on pore size distribution.

4.2.7 Computed X-ray Tomography

The x-ray microtomography machine at ANL was used to apply this technique on a small piece of a cross flow filter plate. The intent was to map the porosity distribution in the sample. The specimen rotates on a stage as the data are acquired. The maximum sample dimension is 7.5 cm.

The x-ray tomography facility employs a 3-dimensional cone beam x-ray CT system. A 160 kV microfocus x-ray tube is used along with a tri-field image intensifier and 512 x 512 CCD camera with stages that manipulate the sample in the x, y, and θ directions, an IBM PC compatible manipulator control and data acquisition computer. Software packages by Feldkamp or Radon for 3-D image reconstruction reside in a VAX 8700 computer. Image reconstruction is carried out using a 3-dimensional back projection algorithm incorporating a VAX/SUN computer system. The process is very computer intensive and the data were downloaded into at least three different computers.

Figure 4 — Magnetic resonance image of a candle filter cross section.

The x-ray source was set at 38 kV and 0.43 mA. The image resolution was limited by the 72 μm detector pixel size as well as the 227 μm slice thickness. The final image resolution on reconstruction was 144 μm voxel size.

The computed tomography results on a sample which was also subjected to ultrasonic microscopy, described later in Section 4.3.3, showed areas of density and/or porosity variations. Figure 5 shows the tomographic image, and the darker regions in the tomographic image were interpreted as concentration of pores, but there was no complementary testing to confirm this.

In the computed tomography, the specimen was positioned such that the porosity variations were averaged through a plane that is perpendicular to that used in the ultrasonic microscopy test. Thus, the data from these two tests could not be directly compared to determine pore resolution.

4.3 ULTRASONIC TESTS

4.3.1 James V-Meter Test

The James V-meter, which is similar to the Pundit type instrument used by Dr. Oakey at CRE on the Grimethorpe candles, was used to find differences in four selected candle filters (some were new and some were used, with at least one being a chipped filter with cracks). A preliminary assessment of the time-of-flight technique was done on these four different candle filters at the Chicago facilities of James Instruments.

The James V-meter is a 100 V spike pulse mode pulser with a receiver that simply detects the first arrival that exceeds 250 μV . The instrument can resolve a 5 μsec difference. Two transducers, 1 in. diameter, 150 kHz longitudinal wave PZT crystals, covered with thin slices of dry rubber couplant, were used in through transmission mode end-to-end across the length of the candle.

The differences in the TOF of all four candles were considered to be sufficiently significant and were attributed to changes in the candle body. Table 2 gives the results from TOF using the James V-meter. Candle filters were used for these tests but conclusions regarding the usefulness of TOF is expected to be applicable to cross flow

Figure 5 — Computed x-ray tomographic image of a cross flow filter cross section.

filter plates. It was concluded that TOF is not the preferred method to distinguish small, but perhaps critical flaws.

TABLE 2

Test No.	Candle ID	Candle Length	Frequency	Average Time-of-Flight	Average Time-of-flight per 1.5 meter length
1	S-46 (new)	59.625"	150 kHz	310.97 μ sec	310.97 μ sec
2	S-45 (old)	39.875"	150 kHz	180.85 μ sec	270.42 μ sec
3	S-43 (old)	38.75"	150 kHz	169.47 μ sec	260.77 μ sec
4	S-33 (old)	39.69"	150 kHz	165.15 μ sec	254.5 μ sec

4.3.2 Exploratory Tests with Partial Filters/ End Plates

This section describes the early experiments conducted to determine the ability of the cross flow filter material to support sound energy and the applicability of the various equipment that are readily available in the laboratory or on the market. Readily available filter sections that were parts of other on-going studies were used at this stage and no special sample requirements were imposed.

The high frequency ultrasonic energy used is expected to behave in a dispersive manner. The spectral analysis and wave shape gets distorted in passing through the filter and the higher frequency components get attenuated.

4.3.2.1 Dry Coupled Contact UT

The filters or end plate assemblies that were available could be examined in the pulse echo or transmission mode using some transducers on hand. The through transmission approach was chosen for the initial experiments.

Preliminary experiments on three filters (WRTX-65, WRTX-3, and WRTX-69) were conducted with a Krautkramer USL-33 ultrasonic instrument attached to two 2 in. diameter rubber tipped 500 kHz broadband transducers (KB-Aerotech, KBA-450A). These transducers were equipped with "hockey-puck" style hard rubber faces as dry

contact. These "facing" attachments could be screwed on to the front of the transducers after applying a small amount of gel to the inside mating surfaces. The experiments demonstrated that the passage of ultrasonic energy from end face to end face across 12 in. of bonded plates causes considerable scatter and distortion. The very broad (not sharp) signals received at the other end could be used to estimate TOF but there was no confidence that the energy had traveled in a straight line. The average TOF for that experiment was 3.25 μ sec/inch. The WRTX-65 and WRTX-69 filters were made with walnut pore formers and WRTX-3 was made with latex pore former. There was no appreciable differences in TOF values due to this variation. The repeatability and resolution were poor. This instrument was not considered further in our work.

Several other series of tests were performed on four different endplate assemblies of crossflow filter sections, identified as WRTX-75T (#2 Top), WRTX-81 (#6 Top), WRTX-62 TOP, and WRTX-62 BOTTOM. This endplate configuration consisted of a section of four full length plates with the flange, top end, side edges, and groove faces bonded together. These assemblies were not made up with the same plate designs or by stacking plates in the same sequence.

In one set of tests, the transducers used were 180 kHz, 100 kHz and 50 kHz longitudinal waves type, manufactured by Panametrics. The tests required a pair of transducers at each frequency in the transmit and receive modes for a through transmission test on the tabs of the flanges. These transducers were used with a Ultragel II couplant, which tended to migrate into the pores. Panametrics Model 5058PR pulser receiver instrument was used to excite the transducer and receive the signals. The ringdown of the signal from the three filters at 50 kHz and 100 kHz is shown in Figure 6.

These tests showed that even in a four plate assembly there can be significant variation in the wave shape introduced by the bonds or material. The lower frequency data appear to offer better discrimination, and we can conclude that WRTX-75 and WRTX-81 are similar filters and the WRTX-62 is different in its processing. Further, these tests indicated the need for a step or wedge "standard" that is well characterized in terms of its density (porosity) that would be used to normalize all data for bond evaluation.

50 kHz

WTRX-62

100 kHz

50 kHz

WTRX-75

100 kHz

50 kHz

WTRX-81

100 kHz

Figure 6 – Ultrasonic signal ringdown of ceramic cross flow filters at 50 kHz and 100 kHz.

In the second set of tests, the transducers used were a 3/8 in. diameter Ultrasonics WD50-1 (longitudinal waves) transducer acting as the transmitter and an Ultrasonics SD50-1 (Shear waves) transducer acting as the receiver. These are dry coupled, 1 MHz broad band ultrasonic transducers. They were held in a clamping device across the thickness of the end plates with a constant force of 32-35 pounds.

The test data, collected from eighteen discrete locations on the endplate assembly, as shown in Figure 7, consisted of signal amplitude, ultrasonic TOF (therefore velocity), peak output frequency, and output bandwidth. The locations included four points on left tab of the flange, four points on the right tab of the flange, and ten points on the top end of each of the endplate assemblies.

Scatter plots were produced using the data tabulated with Lotus spread sheet and showed some interesting correlations between bond position and test parameters. Two typical plots are shown in Figures 8 and 9, with Figure 8 being velocity as a function of position on the top end, and Figure 9 being velocity as a function of position on the left tab. It appears that the bond is definitely different at points closer to the edges of the filter and translates into lower velocity (or higher TOF).

The data from WRTX-62 Top filter endplate indicate that there is a weak correlation between the velocity and transmitted frequencies, as shown in Figure 10. The higher frequencies that are being transmitted through also show a higher phase velocity.

A third set of experiments was done on the WRTX-65 full filter and WRTX-62 top water logged endplate with dry coupled transducers (at Ultrasonics) using an Ultrasonics longitudinal, 1/2 in. diameter, 1.5 MHz Lambda transducer (LRD50-1.5) and Ultrasonics equipment. The high attenuation and cutoff in high frequency, and the transmitted frequency range between 800 kHz - 900 kHz, is seen in Figure 11. The TOF measured was 6.13 μ sec/inch for the WRTX-65 filter (air pores) and 6.85 μ sec/inch for the WRTX-62 top end plate (water logged pores).

These experiments were also intended to evaluate the use of Ultrasonics Burst Pulser BP-9400 and a Ultrasonics Broad Band receiver BR-640A.

Figure 7 – Ultrasonic contact positions on ceramic endplate assembly.

Figure 8 — Velocity data at top end position on WRTX-62 and WRTX-81 cross flow filters.

Figure 9 – Velocity data at left tab position on WRTX-81, WRTX-75, and WRTX-62 cross flow filters.

WRTX-62 #2 (Top)

(1MHz BB Dry Coupled)

Figure 10 - Frequency vs. velocity data on WRTX-62 cross flow filter.

Figure 11 - Dry coupled transducer data on WRTX-62 top filter end plate.

Filter Position L2

Filter Position L3

RF and spectral analysis of signal from 1 cm clear fused quartz

RF and spectral analysis of signal from filter position L1

4.3.2.2 Immersion Ultrasonic Tests

The same four end plate assemblies described in Section 4.3.2.1 above were used for preliminary experiments in immersion testing. These endplate assemblies of crossflow filter sections, identified as WRTX-75T (#2 Top), WRTX-81 (#6 Top), WRTX-62 TOP, and WRTX-62 BOTTOM, consisted of a section of four full length plates with the flange, top end, side edges, and groove faces bonded together. All these assemblies were not made up with the same plate designs nor by stacking plates in the same sequence.

Each assembly was scanned in an automated scanning tank, in the immersion, through transmission ultrasonic test mode using a pair of 0.5 in. diameter, 2.25 MHz transducers (Lithium sulfate crystal, style BB from Automation/Sperry SIL-57A 2796). Figure 12 shows a schematic of the filter immersed in the tank with the scanner and the relative transducer positions. The KBI USIP-12 UT instrument was used to excite the transducer and receive the signal, and the digital data acquisition was performed continuously with a Sonotek based digital system that can store amplitude and TOF data accurately. The color C-scans of TOF and amplitude were plotted and they showed that the amplitude was higher and the TOF lower in the top end and flange tab in each case. The water filled clean channels in the filter end plates were clearly discernible, but the bond at the intersection of the clean/dirty channel was not clear. This test established the feasibility of immersion UT and pointed to the need for better transducers.

In order to explore other instruments and transducers, three other systems were evaluated. The MATEC system was brought to our laboratory, a filter piece was sent to Iowa State University to use their UT system, and the Ultrasonics system was evaluated at the Ultrasonics laboratories in State College, PA.

The Matec MBS 8000 high power pulsed ultrasonic system was demonstrated for use with the cross flow filters by the vendor on July 22, 1991. These systems are made to penetrate high loss materials like ceramic "composite" structures. The PC-based software tools developed with the instrument appeared to be valuable in production quality control. The system appeared to be excellent but costly. An extended trial period was offered by the vendor, which was used for the paired plate studies as described later in Section 4.4.

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Figure 12 – Immersion test setup of Coors paired plate assembly.

The UT system at Iowa State University Center of NDE consists of a Panametrics 5052PR instrument connected to a 5052GPD-2 gated peak detector and a 9400A LeCroy oscilloscope. Dr. David Hsu inspected a 5.5" x 4" cross flow filter piece that was made from a stack of 4 plates, and is shown in Figure 13. This experiment showed the complexity involved due to the geometry of multi-layered channels. The back reflection method to gauge the quality of the joint was very difficult because the echoes from the plate wall and the mid-rib joints are not sufficiently resolved from each other along the full length of the ribs. The velocity of sound in the plate wall was determined by measuring the time difference at a mid-rib bond location and at a web location. The average value of the velocity was 4.23 μ sec/inch and was fairly close in both the sections. This is a smaller TOF (or higher velocity) than expected for the typical porous filter, and we assume that this piece had less porosity.

The top end of the WRTX-62T (1.288 in. thick) endplate assembly was scanned in an immersion tank at Ultrasonics using a 0.75 in. diameter, 1.5 MHz transmitting transducer and a 1 in. diameter, 1 MHz receiving transducer focused at 4.0 in., and Ultrasonics' pulser/receiver equipment. Figure 14 is the amplitude C-scan plot from that test which shows that there is a significant variation in material from the center section to the edges. The TOF data were gathered at the ten discrete locations on the top end shown in Figure 7 when the WRTX62T was fully immersed in water using a 0.37 in. diameter, 2.2 MHz immersion transducer (WS-37) and also when it was removed from water (pores still water logged) and inspected using a 0.5 in. diameter, longitudinal wave, 1.5 MHz Lambda transducers with delay lines. The comparison of TOF values at each location from both scenarios is shown in Table 3. The exercise was undertaken to prove that the dry contact is efficient and to monitor any systematic change in through thickness TOF. The material also acts as a high frequency filter since the transmitted frequencies in both cases are lower than 1.5 MHz. The average TOF from the table is 6.9 μ sec/inch.

Figure 13 – 5.5" x 4" piece of cross flow filter inspected by Dr. David Hsu.

Figure 14 – Color C-scan of WRTX-62 top end immersion test performed at Ultrason Laboratories.

TABLE 3

Location	Immersion Data		TOF	Contact data		TOF
	Transmitted Frequency	TOF + Water Path		Transmitted Frequency	TOF + Delay	
A	1.05 MHz	27.13 μ sec	9.3 μ sec	909 kHz	23.99 μ sec	9.15 μ sec
B	1.05 MHz	27.25 μ sec	9.42 μ sec	870 kHz	23.60 μ sec	8.76 μ sec
C	1.05 MHz	27.03 μ sec	9.2 μ sec	870 kHz	23.85 μ sec	9.01 μ sec
D	1.05 MHz	26.65 μ sec	8.82 μ sec	1.17 Mhz	23.45 μ sec	8.61 μ sec
E	1.05 MHz	26.81 μ sec	8.98 μ sec	860 kHz	23.75 μ sec	8.91 μ sec
F	1.11 MHz	26.54 μ sec	8.71 μ sec	1.17 Mhz	23.45 μ sec	8.61 μ sec
G	1.11 MHz	26.98 μ sec	9.15 μ sec	870 kHz	23.95 μ sec	9.11 μ sec
H	1.11 MHz	26.78 μ sec	8.95 μ sec	1.11 Mhz	23.60 μ sec	8.76 μ sec
I	1.11 MHz	26.89 μ sec	9.06 μ sec	952 kHz	23.95 μ sec	8.91 μ sec
J	1.09 MHz	26.65 μ sec	8.82 μ sec	910 kHz	23.35 μ sec	8.51 μ sec

Coors Ceramics continued to develop their process to improve the formulation as well as manufacturing of the filters and fabricated some full scale filters (in July 1991) using three alternate processing methods. The NDE samples associated with these filters were simultaneously produced as end plates and/or spare plates fabricated under similar processing conditions. These were called "JULY91" samples in the NDE database. These newer filters had "detachable" end plates for testing. These plates are stacked with a parting layer of fiberfax material.

The ultrasonic immersion tests were conducted on the "JULY91" paired plate assembly, in the automated scanning tank with the same pair of 2.25 MHz, 0.5 in. diameter, narrow band transducers in through transmission mode. The data acquisition was also done continuously on the surface of the filters with a Sonotek based digital system that can store amplitude and TOF data accurately.

The setup was done in a 22 in. diameter, round plastic immersion tank with 10 in. of water level, as shown in Figure 12. The filter is supported in a stand such that

its horizontal edge is parallel to the scan travel path. The scan distance is set at 14 in. and the vertical index is set at 1/8 in. on the scanner controls. The UT instrument is set up in the dual transducer mode. The Sonotek system is set up to accept the RF echo and the trigger from the instrument and then to digitize these signals. The software interface gate of the Sonotek is set on the main bang (at 33 μ sec) and the datagate at the first ceramic signal (163 μ sec). In subsequent scans for paired plates, a software modification allowed us to use the interface gate at "0". The water path was 4.875 in. These "JULY91" filter sections were 0.401 in. thick.

The electronic files were named "COORSX" where X described the number of trial performed. The color plots for the amplitude and time-of-flight are shown in Figures 15 and 16.

The amplitude scan shows a discontinuity in the color scheme of the bond at the top left corner. A crack was also visible in the sample in that area. This may be due to a lack of bond or change in the porosity or density of the material. The results of the amplitude scan and time-of-flight scan showed either higher density or intimate contact in the flange area and top section compared to the tests on the early end plate assemblies. The rib-to-rib bonds showed some unevenness along their length. These results showed that the new process had indeed resulted in a higher quality bond.

These encouraging process modifications and the use of NDE to track them led to future plans for further quantification of successive process changes by standardizing a test on the "JULY91" specimens and comparing them with other specimens that would be provided later by COORS. Plans were made to manufacture a controlled set of six paired plates which would include a nominal dirty side bonded pair, a nominal clean side bonded pair, and four clean channel only pairs with varying loads. Details of these are provided later in Section 4.4.1. It was also decided to use the setup described above as a "standard" to enable us to compare data.

coors filter standard thru trans LEVEL logic

Figure 15 – Amplitude color C-scan of Coors paired plate.

Figure 16 – TOF color C-scan of Coors paired plate.

4.3.3 Tests With An Ultrasonic Microscope: (ANL)

The Ultrasonic Microscope at Argonne National Laboratory was used initially to decide the inspectability of this material. A representative bulk sample which did not contain any bonded regions was inspected. The final objective of the investigators at ANL was to arrive at a correlation between bond strength (i.e., load during fabrication) and a parameter associated with attenuation and/or velocity of sound. A figure of merit would be arrived at based on velocity at a particular frequency or at two or more frequencies. Another figure of merit would be arrived at based on amplitude of through transmitted or reflected waves, referenced to a standard or taken in ratio.

The ultrasonic microscope uses a 50 MHz focused transducer with a Panametrics UT instrument. The scanning system consists of a controller and two stepper motors (Anaheim Automation, Inc.), a Daedal two axis stage with 5 cm of travel in each direction, and an IBM PC AT compatible with programs written in Microsoft C language. A MetraByte DAS 8 A/D board is used for data acquisition and control. Images are generated by "C-VUE" software.

- Four ultrasonic scans with low frequency transducers, and one with a high frequency transducer on the bulk sample provided by Westinghouse, showed the capability of imaging density and/or porosity variations. The near surface and surface scans produced in the backwall reflection mode with both 5 MHz and 50 MHz transducers were capable of detecting the machining patterns.

These techniques were not pursued further in order to retain focus on paired plate and full body filter investigations.

4.4 ULTRASONIC STUDY OF PAIRED PLATE

4.4.1 Description of Set of Plates

An NDE study was conducted on the bonds of paired plates. This NDE activity served the important and fundamental need of understanding and detecting the bond itself. A series of twelve paired cross flow filter plates was produced by Coors Ceramics using the original P-100-A alumina/mullite formulation (with the pore former). Each "paired set" is made of two plates. Both of these plates contain either the half part of

clean gas channels on one flat surface, or both of them on their two flat surfaces. The plates are bonded together to form the complete channels. The production sequence for each plate included the manufacture of green form, bisque firing, machining of the grooves, and finish or high firing in the standard production kiln. Each plate was pressed and machined to tolerances that accounted for shrinkage during bisque and final high firing. Loads of 0, 10, 20 or 30 pounds were evenly distributed along the top external surface of the paired plate "assembly". Figure 17 shows three different plate sets that were produced to demonstrate reproducibility of the manufacturing process.

Two duplicate sets of these paired plates had clean gas channel mid-ribs extending along both external surfaces and dirty gas channels formed in-between the plates. The 30 pound distributed load placed on the assembly during firing was supported by the ribs on the external surfaces.

Two duplicate sets of these paired plates had dirty gas channel mid-ribs extending along both external surfaces and clean gas channels formed in-between the plates. The 30 pound distributed load placed on the "assembly" during firing was supported by the ribs on the external surfaces.

Four duplicate sets of these paired plates had flat external surfaces and clean gas channels formed in-between the plates.

A total of twelve paired sets were produced and there were six unique variations with the ability to verify the repeatability of the process. Each of them was assigned a unique name listed in Table 4 which is descriptive of its manufacture.

All the assemblies exhibited two directions of bending or distortion, in plane of the filter and normal to the plane of the filter. The in-plane distortions varied from 0 in. to 0.27 in. The normal distortion varied from 0 in. to 0.150 in. The compound effect of both of these tended to twist the plates. The various anomalies which were recorded for each paired plate assembly included:

- a. Visible cracks and nicks, shown in Figure 18
- b. Misalignment or mismatch of ribs, shown in Figure 19
- c. Thickness variation caused by uneven load
- d. "Smiles" and "rippling" caused by cross channels

Figure 17 — Three variations of paired plate assemblies.

Figure 18 – Visible crack in 10-POUND-01 paired plate.

Figure 19 – Misalignment of ribs in 10-POUND-02 paired plate.

TABLE 4

No.	Filter ID #	Description
1	NDE-CLEAN-01	Clean channel mid-rib bonds/ dirty ribs outside
2	NDE-CLEAN-02	Clean channel mid-rib bonds/ dirty ribs outside
3	NDE-DIRTY-01	Dirty channel mid-rib bonds/ clean ribs outside
4	NDE-DIRTY-01	Dirty channel mid-rib bonds/ clean ribs outside
5	NDE-0 POUND-01	Clean chnl. mid-rib bonds, no ribs outside, no load
6	NDE-0 POUND-02	Clean chnl. mid-rib bonds, no ribs outside, no load
7	NDE-10 POUND-01	Clean chnl. mid-rib bonds, no ribs outside, 10 lb load
8	NDE-10 POUND-02	Clean chnl. mid-rib bonds, no ribs outside, 10 lb load
9	NDE-20 POUND-01	Clean chnl. mid-rib bonds, no ribs outside, 20 lb load
10	NDE-20 POUND-02	Clean chnl. mid-rib bonds, no ribs outside, 20 lb load
11	NDE-30 POUND-01	Clean chnl. mid-rib bonds, no ribs outside, 30 lb load
12	NDE-30 POUND-02	Clean chnl. mid-rib bonds, no ribs outside, 30 lb load

Each assembly was measured for thickness in 16 different spots. These data were later used to correct the time-of-flight (TOF) for thickness variation. Figure 20 is a typical data sheet prepared for the NDE-0 POUND-01 paired plate to record these data which would be later used to analyze ultrasonic data.

A conventional film radiography of the paired plates using a Siefert x-ray system was performed. The system includes a Siefert Isovolt 320, with 320 V maximum voltage and 13 mA maximum tube current. The maximum available power of 4200 W is more than sufficient for penetrating ceramic materials. The unit is controlled by a Z-80 microprocessor. Two positive images at different exposure times were developed from each negative radiograph. Figure 21 is the radiograph of the NDE-CLEAN-02 paired plate. The darker regions at the top end, rib crossovers and flange tabs are due to material thickness differences. A corner crack in the flange area is marked.

"X" FACE

COMMENTS:

FRONT-CHANNELS ALIGNED

REAR-CHANNELS OFFSET .040"-.050"

Figure 20 – Visual exam and thickness measurement data sheet for 0-POUND-01 paired plate.

Figure 21 – Positive print radiograph of NDE-CLEAN-02 paired plate.

Figure 22 is the radiograph of the NDE-0 POUND-01 paired plate. A prominent corner crack in the flange area, which is also visible to the naked eye, is marked.

All the assemblies were radiographically tested. The test parameters were 45 kV, 10 mA, 7 minutes exposure on M5 film at SFD of 60" using a Seifert machine.

Analysis of the radiographs of the twelve cross-flow filter paired plates indicated that six contained 0.125 in. - 0.25 in. cracks in the flange radius, and that four contained similar cracks along the edge of the plates at approximately six inches above the flange. Radiographic film optical densities were measured at 25 locations along each radiograph. The optical densities correlated with the ultrasonic amplitude plots are described later in Section 4.4.2.1.

Each assembly was measured for thickness in 16 different spots along the edges. Five readings each on the flange and top end were taken. Three readings each were taken at the long edges. These provided data on how uneven the "slumping" would be for a given pair fired under a specified load.

4.4.2 Ultrasonic Immersion Tests on Paired Plates

Ultrasonic immersion tests were conducted on each of the twelve paired plate assemblies. All the filter paired plates were pre-soaked in water for a minimum of 12 hours before scanning. The test fixtures for immersion tests consisted of a X-Y manipulator to which the transducers were attached. The two transducers, one transmitting and another receiving, moved together continuously in the length direction of the plate and indexed in the width direction. The entire system moves under water. Figure 23 shows the paired plate and transducer immersed in water with the signal from these transducers on the oscilloscope screen. The movement is encoded and information sent to a data acquisition and processing computer.

Figure 22 - Positive print radiograph of 10-POUND-01 paired plate.

Figure 23 – Ultrasonic immersion test on 20-POUND-02 paired plate.

The ultrasonic test employed a pair of 1.5 MHz, 1 in. diameter, broad band transducers in through transmission mode focused at 4 in. in water (L100-1.5-P4, Serial No. 143301 Transmit Transducer and Serial No. 143302 Receive Transducer). Repeated tests were performed with three different instruments in an effort to compare different types of pulser/receivers. The instruments used were:

1. Krautkramer USIP-12 UT instrument. This is a standard ultrasonic laboratory/field instrument containing a spike pulser and a medium to narrow band receiver that has been found acceptable in several high performance test applications.
2. Ultran high energy/low frequency burst pulse system. The system consisted of Ultran's BP9400A Burst Pulser and the BR-640A Broadband Receiver. The Ultran high power pulsed ultrasonic system is made to penetrate high loss materials as ceramic "composite" structures.
3. The Matec MBS-8000 system was used to acquire data on three of the representative filter assemblies from the full set of 12 paired plates. This system was configured with the spike pulser board. The PC based software tools developed with the instrument appeared to be valuable for production quality control.

The signals that were generated during testing were viewed on a separate Tektronix 465 oscilloscope (in the case of USIP-12 it is integral to the chassis, as well in the form of digitized A-scan obtained from the PC-based Sonotek device). Data acquisition was done continuously on the surface of the filters with the Sonotek digital device that can store amplitude and time-of-flight (TOF) data accurately.

Using each of the three instruments, two separate scans were taken on each paired plate assembly, one for amplitude and one for time-of-flight measurement. The tests were conducted in the automated scanning tank described in Section 4.3.2.2 and the distance between the two transducers was 7.5 in. (started with 8 in. for tests with USIP-12), to allow for 14 in. scan in the round tank. The filter is centered between the transducers, and the top of filter tab is even with the filter holder. Amplitude, TOF,

Radio Frequency Unrectified (RF), and Fast Fourier Transform (FFT) data have been stored for comparisons of data gathered using the three systems. The RF data and the corresponding FFT data were stored from eight locations in the top end and the flanged end of each filter. The locations are identified in Figure 24. This allowed us to analyze and correlate between the velocity and transmitted frequencies or frequency attenuation, and compare the efficiency of the three UT systems. The RF and FFT data were transferred to a Quattro Pro database program to be able to plot them.

Ultran's BP9400A Burst Pulser was set at a pulse width of 335 nanoseconds (half the transducer wavelength), at a 3000 rep rate (kept low to reduce duty cycle and prevent transducer damage). The pulses per burst were also limited to 1. Ultran's BR-640A Broadband receiver gain was sufficient at 8 db to drive a signal through the plates. The widest receiver filter range was selected; i.e., 1 MHz low pass and 10 kHz high pass, and the input impedance was selected at 50 ohms. The signals were viewed on an oscilloscope as well as the digitized A-scan from the Sonotek. The scan parameters and the Sonotek digitizer/gate setup are both shown in the Figure 25. These are the input screens of the PC, with the top screen being the mechanical setup values of the manipulator as input and the bottom screen being the electronic setup values for the signal digitization. The acquisition program was modified such that the data (D) gate was able to trigger when the interface (I) was at 0 μ sec and 0 μ sec length. This change was done because the excellent shielding and separation of transmitter and receiver of the Ultran system prevents existence of the "leakage" main bang signal that is always present at 0 time in other systems. All the electronic files were assigned names such that information of filter identity, TOF or amplitude run, and RF data file or FFT data file at the exact location, are encoded in the name. Further, all files collected from the Ultran instrument start with FX in their names.

All the Matec MBS-8000 instrument settings are done in software with menu driven inputs. The pulser was set at a pulse width of 1000 nanoseconds and at a 1000 rep rate. The receiver gain was set at 38.5 db. The receiver filter range was selected at "1". The signals were viewed on an oscilloscope as well as on the digitized A-scan

Figure 24 — Ultrasonic contact and mechanical measurement locations on paired plate.

Scan inc : 0.032 in Index inc : 0.125 in
 Scan min : 0.864 in Index min : 0.888 in
 Scan max : 12.864 in Index max : 50.888 in
 Scan axis : X Index axis : Z1
 Scan num : 400 Index num : 400
 Data Set :

interface tof = 0 0.00 us, amp = 0
 data tof = baa 119.44 us, amp = 128

Sample Rate : 25 Mhz Trigger Type : External - I Gate Start : 0.00 us
 Sample Len : 650.40 us Trigger Level: 0 I Gate Length: 0.00 us
 Trigger Delay: 0.00 us I Gate Level : 255
 Input Range : 1.0 Volts Comp Sense: Greater Than D Gate Start : 115.48 us
 Signal type : RF Comp Value: No Abs. Value D Gate Length: 10.20 us
 Video Filter: 7 Comp Logic: Level D Gate Level : 11
 Disp Delay : 112.64 us

interface tof = 0 0.00 us, amp = 0
 data tof = b5c 116.40 us, amp = 128

Figure 25 — Mechanical and electronic setup values using Sonotek data acquisition.

from the Sonotek. The scan parameters and the Sonotek digitizer/gate setup are similar to those described previously for Ultram. All the electronic files were assigned encoded names as described for Ultram above except all Matec instrument collected files start with MX in their names.

The USIP-12 pulser was set on automatic rep rate. The receiver gain was set at 40 db to drive a signal through the plates. The receiver filter range was selected at "1 MHz BB" after experimenting with all available settings (i.e., 1 MHz BB, 1 MHz NB, 2.25 MHz BB, and 2.25 MHz NB during the NDE-30 Pound -02 tests). The signals were viewed on the USIP-12 oscilloscope as well as on the digitized A-scan from the Sonotek. The scan parameters and the Sonotek digitizer/gate setup are similar to those described for Ultram, above. All the electronic files were assigned encoded names as described for Ultram above except all USIP-12 instrument collected files start with XF in their names.

4.4.2.1 Results of UT Immersion Tests on Paired Plates

The results of the amplitude scan and time-of-flight (TOF) scan showed considerable variations from filter to filter, and each output (only Ultram equipment acquired data are presented) for all the twelve filters is shown in Figures 26 to 49. The changes in both the amplitude and TOF for each of these plates tend to correlate with the radiographic densities. Similar sets of data acquired by Matec and USIP-12 are available but not presented in this report.

The ultrasonic scans are able to detect the rib mismatch and cracks that are at least 3/32 in. wide and through wall. Cracks that were narrower or were only 50% through wall were missed. The cracks were detected in two paired plates; the "NDE 10# 01" shown in Figure 30, and "NDE 20# 02" shown in Figure 36.

The results showed the ability of ultrasonic inspection to reflect the qualitative bond variation from plate to plate. Figures 26 and 27 and Figures 40 and 41 show the color plots of amplitude scan and time-of-flight (TOF) scan of two selected plates. One of these plates is a 0 lb load and the other is a 30 lb load during firing. Comparison of the scan data has also shown that the Ultram burst pulser gave the best results in terms of resolution of ribs, seams, and possible crack locations.

The Fast Fourier Transform (FFT) of the Radio Frequency Unrectified (RF) waveforms at selected locations were compared to identify some characteristic that may be related to bond quality. Figure 50 shows the the spectral content of the transmitted energy from the incident broad band 1.5 MHz signals taken at the various positions of the 0 lb filter. Based on the difference in shapes between the FFTs, the positions have been arbitrarily classified as "good bonds" and "bad bonds". The rib position signals are more difficult to analyze because of the large transducer diameter compared to the rib width. The shift towards lower frequency in the lower top closed section (position 8) is explained by absorption of higher frequency in the lamination between plates. These data also correlate with the significant reduction in amplitude at position 8 in Figure 26 and a corresponding increase in time-of-flight in Figure 27. There is a complete lack of bond postulated in that region. Figure 52 shows the spectral content of the transmitted energy from the incident broad band 1.5 MHz signals taken at the various positions of the 30 lb filter. Figure 53 shows no significant reduction in amplitude for these same corresponding positions. Based on data from Figures 40 and 53 (amplitude) and Figures 41 and 52 (time-of-flight), it can be concluded that the bonds are acceptable at these locations.

4.4.3 Dry Couplant Tests On Paired Plates

The dry couplant technique has provided the independent verification of bond "quality" to be tracked by amplitude, time-of-flight (TOF), and the shape of the FFT. The TOF per inch (TOF/in) obtained in through transmission using a 2 MHz shear transducer, at the same points as those discussed above (in Section 4.4.2.1) on the 0 pound and 30 pound paired plate, are shown in Figure 54. The TOF/in in the 30 pound plate is consistently lower, i.e., a better bond compared to the 0 pound plate. The more significant result is the very marked reduction of TOF/in at positions 6, 7, and 8, which is the top well bonded region, for the 30 pound plate. In comparison, the corresponding 0 pound data for position 8 exhibits a relatively high TOF/in. This observation is consistent with postulating a "poor" bond at position 8, but these data alone are not sufficient in all cases to arrive at this conclusion. This is based on the observation of data at position 1 of the 0

pound plate in Figure 54 which shows the same height for TOF/in; but in the immersion test data of Figure 52 showed a strong relative amplitude, consistent with areas of good bonds elsewhere in the plate. The more credible data related to poor quality bond are present in the corresponding FFT spectral information of Figures 55 and 56. There is absorption of higher frequencies resulting in the relative prominence of the low frequency components of the lower top closed position (position 8) of Figure 55.

The ultrasonic techniques are providing a good basis to make the judgment on bond quality, particularly if the amplitude, TOF, and FFT are used simultaneously to make the assessment.

COORS 10/3/91 NDE CERAMIC FILTER STANDARD

Figure 26 – Amplitude color C-scan of 0-POUND-01 paired plate.

COORS 10/3/91 NDE CERAMIC FILTER STANDARD

Figure 27 – TOF color C-scan of 0-POUND-01 paired plate.

COORS 10/3/91 NDE CERAMIC FILTER STANDARD

Figure 28 – Amplitude color C-scan of 0-POUND-02 paired plate.

Figure 29 - TOF color C-scan of 0-POUND-02 paired plate.

COORS 18/3/91 NDE CERAMIC FILTER STANDARD

COORS 10/3/91 NDE CERAMIC FILTER STANDARD

Figure 30 – Amplitude color C-scan of 10-POUND-01 paired plate.

Figure 31 - TOF color C-scan of 10-POUND-01 paired plate.

COORS 18/3/91 NDE CERAMIC FILTER STANDARD

COORS 10/3/91 NDE CERAMIC FILTER STANDARD

Figure 32 – Amplitude color C-scan of 10-POUND-02 paired plate.

COORS 10/3/91 NDE CERAMIC FILTER STANDARD

Figure 33 – TOF color C-scan of 10-POUND-02 paired plate.

COORS 10/3/91 NDE CERAMIC FILTER STANDARD

Figure 34 - Amplitude color C-scan of 20-POUND-01 paired plate.

COORS 10/3/91 NDE CERAMIC FILTER STANDARD

Figure 35 – TOF color C-scan of 20-POUND-01 paired plate.

COORS 10/3/91 NDE CERAMIC FILTER STANDARD

Figure 36 – Amplitude color C-scan of 20-POUND-02 paired plate.

COORS 10/3/91 NDE CERAMIC FILTER STANDARD

Figure 37 – TOF color C-scan of 20-POUND-02 paired plate.

COORS 10/3/91 NDE CERAMIC FILTER STANDARD

Figure 38 — Amplitude color C-scan of 30-POUND-01 paired plate.

COORS 10/3/91 NDE CERAMIC FILTER STANDARD

Figure 39 – TOF color C-scan of 30-POUND-01 paired plate.

COORS 10/3/91 NDE CERAMIC FILTER STANDARD

Figure 40 – Amplitude color C-scan of 30-POUND-02 paired plate.

COORS 10/3/91 NDE CERAMIC FILTER STANDARD

Figure 41 – TOF color C-scan of 30-POUND-02 paired plate.

COORS 10/3/91 NDE CERAMIC FILTER STANDARD

Figure 42 -- Amplitude color C-scan of NDE-CLEAN-01 paired plate.

Figure 43 — TOF color C-scan of NDE-CLEAN-01 paired plate.

COORS 10/3/91 NDE CERAMIC FILTER STANDARD

Figure 44 – Amplitude color C-scan of NDE-CLEAN-02 paired plate.

Figure 45 - TOF color C-scan of NDE-CLEAN-02 paired plate.

COORS 10/3/91 NDE CERAMIC FILTER STANDARD

Figure 46 – Amplitude color C-scan of NDE-DIRTY-01 paired plate.

COORS 10/3/91 NDE CERAMIC FILTER STANDARD

Figure 47 – TOF color C-scan of NDE-DIRTY-01 paired plate.

COORS 10/3/91 NDE CERAMIC FILTER STANDARD

Figure 48 – Amplitude color C-scan of NDE-DIRTY-02 paired plate.

COORS 10/3/91 NDE CERAMIC FILTER STANDARD

Figure 49 – TOF color C-scan of NDE-DIRTY-02 paired plate.

Figure 50 — Spectral analysis of transmitted energy in 0-POUND-01 paired plate.

Figure 51 – Amplitude analysis of transmitted energy in 0-POUND-01 paired plate.

**FFT, 30 lb. #2, Paired Plate
1.5 Mhz, Immersion, Ultran**

Figure 52 – Spectral analysis of transmitted energy in 30-POUND-02 paired plate.

RF, 30 lb. #2, Paired Plate
1.5 Mhz, Immersion, Ultran

Figure 53 — Amplitude analysis of transmitted energy in 30-POUND-02 paired plate.

**Contact 2 Mhz Shear TOF/in
Paired Plate Bonds**

Figure 54 — TOF analysis of typical 0-POUND and 30-POUND paired plate assemblies at different locations.

Figure 55 – Spectral analysis of transmitted energy in 0-POUND-01 paired plate using dry contact method.

Figure 56 – Spectral analysis of transmitted energy in 30-POUND-02 paired plate using dry contact method.

5. TIME-OF-FLIGHT STUDIES ON PRODUCTION LOTS

Studies were done to monitor time-of-flight changes as a function of known differences in material processing of the P-100A alumina/mullite used by Coors to make either the cross-flow or candle filters. Processing changes included the presence or absence of pore forming material, raw material milling time, porosity, initial room temperature, and hot strength and cooling rates.

The studies were conducted on different production batches of candle filters. The candle filters were measured for full length TOF and through thickness TOF. The discs were measured for through thickness TOF. Table 5 presents the TOF data for candle filters from different production batches.

TABLE 5
ULTRASONIC TOF IN COORS CANDLE FILTER

LOT	Characteristic descriptors	Full Length TOF $\mu\text{sec/in}$	Through Thickness TOF $\mu\text{sec/in}$
1	Pore Former,	6.7	6.0
2	No Pore Former, P-100A-10	5.0	4.5
3	No Pore Former, P-100A	5.6	5.0
4	No Pore Former, P-100A	5.9	5.9
5	No Pore Former, P-100A	5.7	5.1

6. CONCLUSIONS

The ultrasonic tests on paired plates confirmed that the 30 pound load during high firing was optimal for mid-rib and seam channel bonds, while minimizing slumping and "smile" formation between the bonding surfaces.

The current manufacturing technique causes the mid-rib bonds to form localized tack welds rather than a continuous uniform bond due to the point contact transmission of the load. This may be structurally sufficient as long as the rest of the "partially bonded" surfaces provide adequate seal against leakage of dirty hot gases into clean channels.

The paired plate studies also evaluated three ultrasonic instrument systems and found that the Ultrason system gave the best results. The image resolution for rib edges as well as amplitude variation for the bonds within the filter were sharper.

7. RECOMMENDATIONS

NDE efforts should be directed at creating a set of ceramic P-100A alumina/mullite filter material "standards" of well characterized uniform material properties. These would have several pieces of different density with porosity and permeability constant, several pieces of different porosity with density and permeability constant, and several pieces of different permeability with density and porosity constant. These would help to find a method to normalize all ultrasonic data. These, coupled with a series of other controlled step thickness standards, will make the identification of bond quality more definite. Ultrasonic data can thus be collected again on these paired plates, after these standardization based corrections are recognized. Finally, this set of paired plates can be destructively examined to validate the conclusions of this work.

In order to confirm the NDE results, further destructive testing (i.e., plate "pull-apart" strength; bond flexural strength) is recommended. A correlation of strength to one of the UT parameters or a composite index of the parameters can be investigated.

More detailed and extensive work on outer edge bond inspection using γ -Ray or light as a source for inspection is recommended as the next step from paired plate configuration. This will provide the ability to inspect for local bond quality in a full filter made from a stack of plates (not only 2 plates). A small effort was devoted to investigating these techniques, as a part of this program. That effort has shown encouraging results and established them as candidates worth considering for detailed work.

Finally, a quick assembly line method is needed to inspect the entire filter with all its mid-rib as well as edge bonds in one step such that the filter can be passed or diverted for further local inspections described in the paragraphs above. Monitoring the acoustic energy during the formation of bubbles is a technique that will be able to distinguish between the air/gas leakage from pores and air/gas leakage that results from through path due to lack of bond; i.e., critical flaws, poorly bonded "smiles", mid-rib bonds or gas channel seams, and delaminations).

8. ACKNOWLEDGMENTS

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APPENDIX E

MATERIALS QUALIFICATION TECHNOLOGY FOR CERAMIC CROSS FLOW FILTERS

Distribution
Category:
Coal Gasification
(UC-109)

ARGONNE NATIONAL LABORATORY
9700 South Cass Avenue, Argonne, Illinois 60439

ANL/FE-91/1

**MATERIALS QUALIFICATION TECHNOLOGY FOR
CERAMIC CROSS-FLOW FILTERS**

by

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Materials and Components Technology Division

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MATERIALS QUALIFICATION TECHNOLOGY FOR CERAMIC CROSS-FLOW FILTERS

by

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ABSTRACT

The physical, mechanical, and microstructural properties of ceramic cross-flow filters in as-fabricated condition (Filter A) and after exposure to a gasifier environment (Filter B) were studied. The filters contain mullite as the major phase and are approximately 41% porous. The pore size varies from submicron to 112 μm , and this distribution broadens upon exposure to a gasifier environment.

Stress and fracture mechanics analyses were completed and the property data, together with stress and fracture mechanics analyses, were used to determine probable cause(s) of failure.

The flexural strength of the flange sections of Filters A and B is 27.1 ± 2.0 MPa and 28.2 ± 1.9 MPa, respectively, whereas the strength of the web sections is 22.3 ± 2.3 and 21.3 ± 2.3 MPa, respectively. The higher strength of the flange section may be related to the microstructural differences between the materials in the flange and the web sections. The Weibull modulus (m) data for the material in the web section is reduced from 14 to 9 after exposure to a gasifier environment. This suggests a larger scatter in strength data and hence a decrease in structural reliability after exposure to the gasifier environment. The fracture toughness of the flange material from Filters A and B is 0.62 ± 0.08 MPa $\sqrt{\text{m}}$ and 0.77 ± 0.07 MPa $\sqrt{\text{m}}$, respectively. The corresponding values for the web section are 0.44 ± 0.07 MPa $\sqrt{\text{m}}$ and 0.49 ± 0.13 MPa $\sqrt{\text{m}}$. The fracture toughness seems to be unaffected by the gasifier environment.

The critical flaw sizes calculated by fracture mechanics using the measured mechanical properties were 216–465 μm for Filter A and 332–584 μm for Filter B, suggesting an increase in the critical flaw size of the filter material in a gasifier environment due to thermal and/or mechanical fatigue. The thermal-shock experiments indicate a continuous drop in the strength of the filter material with increasing severity of thermal shock. This observation suggests the presence of large critical flaws, in agreement with the microscopic observations and fracture mechanics calculations of critical flaw sizes.

The thermal diffusivity of the filter material varies from 0.005–0.009 cm^2/s in the temperature range of 25–1000°C. These values are approximately one-half those of dense mullite and are due to the high porosity of the filter material.

Static and dynamic finite-element stresses on the filter were analyzed during a pulse-cleaning cycle, using a micro model (considering the details of the gas channels) and a macro model (ignoring the details of the gas channels). Fracture mechanics analyses of the micro model with pre-existing cracks of various lengths at the channel corners were also carried out. Near coincidences of the calculated and measured (Westinghouse) frequencies during pulse-cleaning provided strong evidence for narrow contact between the clamp and the free end of the flange. The analyses suggested that, to minimize the maximum stresses in the filter, such a linear contact between the clamp and the flange should be avoided. The pressure-induced stresses during pulse-cleaning were small. However, based on a transient thermal analysis, the transient thermal stresses in the filter could be high.

1 INTRODUCTION

This project supports the development of cross-flow ceramic filters for cleanup of hot, dirty gases. It develops the materials qualification technology needed to ensure satisfactory performance of filter modules acquired for installation in the hot, dirty gas stream and/or to identify those materials that are unlikely to provide satisfactory performance. This is an experimental and analytical program.

Hot, dirty gas exiting from a gasifier or an advanced combustor contains sufficient particulates to warrant it undesirable for direct utilization in gas turbines and heat engines. Today's hot, dirty gas cleanup systems, such as cyclones, can remove only the fraction containing larger size particulates. Smaller particulates can be removed only by cooling and filtering the gas.

The U.S. Department of Energy is currently supporting a program to develop a cross-flow ceramic filter technology for combined-cycle power generation with coal gasification. Ceramic filters should remove essentially all of the fines from a hot, dirty gas stream. Development and testing of filter materials and the configuration have been carried out at many commercial institutions. The candidate materials for the filter include alumina silicate (Coors), cordierite (GTE), silicon-nitride-based materials (Garrett), and cordierite silicon nitride (Acurex Corp.). System testing has been done at Westinghouse, among many other laboratories. The testing of the cross-flow filter units in a pressurized fluidized bed coal combustor has shown very promising results, but on various occasions several different kinds of failures have occurred, as summarized below.¹⁻⁴

- Cracks in the filter unit.
- Break in the mounting flange.
- Delamination of a filter element.
- Break across a filtering flat.
- Breakage of a 2 x 6-in. piece from the back corner of the filter.

Initial data indicated that the failures may have occurred because of both system design and materials-related problems. Furthermore, for commercial application, the filters must have reproducible properties, a goal that has not yet been achieved.

Design methodology for brittle materials requires an approach different from that for ductile materials. Recent advances in the design of brittle materials have evolved from work on high-temperature ceramic gas turbines, which began under Advanced Research Program Agency funding in the early 1970s and continued with DOE Conservation funding under the program "Brittle Materials Design, High-Temperature Gas Turbine." Heat transfer and stress analyses of the components were performed with finite-element codes. In addition, mechanical properties of the candidate materials were evaluated. In view of the large scatter in strength of brittle materials resulting from a wide distribution of flaw populations, a statistical analysis was used to correlate mechanical properties (strength, fracture toughness) with the probability of the existence of critical flaws in the material. Given that there is no unique value of strength that can be ascribed to a material at any given stress level, there is always a finite probability of failure, which is statistically dependent on flaw population and strength distribution. As a result, design of brittle materials depends upon arbitrarily choosing an acceptable failure rate or "factor of safety" and ensuring that the finite-element analysis shows no local element with a higher stress

than that which correlates with the chosen statistical probability of failure. Finally, fabricated components should be used to verify the properties and design of material by proof testing, nondestructive evaluation, and/or destructive testing.

Improvement in component reliability will depend on the reliability of material properties. The reliability of a component is increased with an improvement in mechanical properties (strength and fracture toughness) and a decrease in statistical scatter of the mechanical-properties data. The improvement in mechanical properties and reduction in statistical scatter will permit designs with higher local stress levels. For this reason, a data base of the physical and mechanical properties of the filters under study was developed and supported by microstructural characterization.

Test procedures were developed to characterize the mechanical and microstructural properties of the materials. The results of mechanical and microstructural characterization provided a data base of mechanical properties (strength, toughness, elastic modulus, and thermal-shock resistance), as well as information on critical flaw size. A statistical analysis of the mechanical-properties data established the Weibull modulus, which provided information on the reproducibility of the materials.

Stress and fracture mechanics analyses established the fracture criterion for the filter system. These analyses were carried out in two steps. First, the results from the finite-element analysis of the filter ("uncracked") were used to define the maximum average tensile stress over a sufficiently large volume so that it could be compared to the tensile strength of the bulk ceramic. Next, in the regions of stress concentrations or geometric discontinuities, where large stress gradients are present, flaws were introduced to determine the stress intensity factors that could then be compared with the fracture toughness of the material. Such an analysis enabled the determination of the largest flaw that the filter could tolerate in the regions of stress concentration or geometric discontinuities. Both approaches were used to evaluate the potential for filter fracture based on known tensile strength, fracture toughness, and flaw population in the ceramic material. Conversely, similar analyses were performed to determine the minimum properties required for reliable filter performance.

2 WESTINGHOUSE CERAMIC CROSS-FLOW FILTERS

2.1 CONFIGURATION OF WESTINGHOUSE CROSS-FLOW FILTERS

The ceramic cross-flow filters that are the subject of this report were tested by Westinghouse Electric Corporation and fabricated by Coors Ceramics, Ltd. The technology basis was focused on filters from a lot processed in February 1989, primarily for use in the Texaco gasifier. The filters were made of mullite, an alumino-silicate material, and are considered to be the most compact experimental filters of particulates in coal gasification technology.

Figure 1 is a schematic of these filters, reproduced from Ref. 5. Each filter consists of multiple filter cells made of ceramic tiles that are bonded together. The tiles are configured to form rectangular cells in such a manner that the direction in which the gas enters is perpendicular to the direction in which it leaves. Alternate upstream and downstream cells are blocked, so that the dirty gas is forced to flow through the porous ceramic tile walls. The particulates are trapped in the pores at this stage and can be back-flushed with a pulse of air.

Fig. 1. Schematic of Westinghouse ceramic cross-flow filter

Each cell is $\approx 2.5 \times 2.5 \times 0.5$ cm; each tile is ≈ 0.5 cm thick. Thus, two tiles bonded side by side make a wall ≈ 1 cm thick. The array of cells is supported on a thick ceramic flange made of the same material.

This study was conducted on two filters supplied by Westinghouse Electric Corporation. Filter A, an as-fabricated filter, was new and unused. Filter B had been tested in a gasifier environment. Study with these two filters allowed an evaluation of the effect of a gasifier environment on the material properties of the filter.

2.2 PHYSICAL PROPERTIES

High-purity mullite⁶ ($3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$) has a theoretical density of 3.23 g/cm^3 . The bulk density of the porous filter material, however, is affected by porosity and the presence of impurities. To estimate the bulk density of the filter material, the mass and physical dimensions (hence, the volume of a rectangular specimen cut from the filter tile) were measured and the density was determined.

To estimate the porosity of the filter material and the pore size distribution, the specimens were subjected to mercury intrusion porosimetry. This not only provided the total open porosity and the pore size distribution, but also gave an independent estimate of the bulk density of the material. The results of these studies are presented in Table 1.

Table 1. Physical properties of the as-fabricated filters

Physical Property	Measured Value
Bulk density by direct measurement	1.9 g/cm ³
Bulk density by mercury intrusion	1.6 g/cm ³
Median pore diameter	42.4 μ m

Based on the direct measurement of the density shown in Table 1 and the theoretical density of 3.23 g/cm³ of dense mullite,⁶ one can estimate the porosity of the filter material, which yields 41% as the total porosity. The porosity obtained from mercury intrusion porosimetry data is 40.7%. These two values are very close. The slight difference may be due to the closed pores in the material, which are not accessible to the mercury.

Figure 2 is a typical pore size distribution of the Filter A (unused) material obtained by mercury intrusion porosimetry. The pore size varies from submicron to approximately 112 μ m.

To see the effect of the gasifier environment on the filter material, pore size distribution was also measured for Filter B (which had been tested in a gasifier environment). The results are presented in Fig. 3. Although the range of pore size ($\approx$ 0.9–93 μ m) in this used filter is not much different from that in the as-received filter, the pore size distribution has considerably changed in the used filter after being exposed to the gasifier environment.

Based on the data for Figs. 2 and 3, the cumulative pore volume is plotted in Fig. 4 as a function of pore size. Overall, the cumulative pore volume appears to have increased in the used filter after being exposed to the gasifier environment. This is believed to be due to the increase in pore size as a result of damage caused by erosion/corrosion in the gasifier environment. The smaller cumulative pore volume in the filter subjected to the gasifier environment for small pores (<15 μ m) is consistent with the fact that the damage due to erosion/corrosion in the gasifier environment has enlarged these pores and reduced the number and volume of small pores. In the higher pore size regime (>45 μ m), the cumulative pore volume, as measured by mercury intrusion, in the used filter is slightly lower than that of the as-received filter. This is probably due to the accumulation of debris in the pores, giving an apparently lower porosity.

2.3 CHEMICAL COMPOSITION

The composition of the filter material was determined by powder X-ray diffraction analysis. The major phase was mullite (3Al₂O₃·2SiO₂); a minor phase was alumina (Al₂O₃). Very minor phases of anorthite (CaAl₂Si₂O₈) and albite (NaAlSi₃O₈) were also found.

Fig. 2. Pore size distribution of a typical specimen of as-fabricated Filter A

Fig. 3. Pore size distribution of filter tested in gasifier environment (Filter B)

Fig. 4. Cumulative pore volumes of the filters in as-received and exposed conditions

2.4 THERMAL PROPERTIES

To calculate the stresses and strains due to thermal fluctuations in the filter, it is essential to know the value of the thermal diffusivity. For this reason, thermal diffusivity of the as-fabricated material was measured as a function of temperature by a laser flash technique at Virginia Polytechnic and State University. The results are shown in Fig. 5.

The thermal diffusivity decreases with temperature. At higher temperatures, the diffusivity of Sample 1 is slightly higher than that of Sample 2. This is probably due to a small variation in the porosity of the material. Considering these fluctuations, one may conclude that the thermal diffusivity of the filter material varies between 0.005 and 0.009 $\text{cm}^2/\text{sec.}$ in the temperature range of 25 to 1000° C. The thermal diffusivity reported in the literature⁷ for dense mullite at room temperature is 0.013 $\text{cm}^2/\text{sec.}$, which is more than twice the value we obtained for the filter material. This difference may be due to the high porosity of the filter material.

3 MECHANICAL PROPERTIES OF FILTER MATERIAL

Improvement in component reliability will depend on the reliability of materials properties, and, in the present context, on the reliability of the mechanical properties (strength and fracture toughness) of the ceramic filter material. Improvement in mechanical properties and reduction in statistical scatter will permit better designs with higher local stress levels. For this reason, a detailed investigation of the mechanical properties of the filters was carried out.

The mechanical properties we measured were flexural strength, fracture toughness, and modulus of elasticity. In addition, Weibull modulus of strength data were evaluated to assess the reproducibility of the data. To assess the effect of a gasifier environment on these properties, measurements were also carried out on the used filter. For mechanical testing, rectangular bar specimens were machined from the filters and subjected to a

Fig. 5. Diffusivity of as-fabricated filter material

standard surface finish. For each filter, specimens were obtained from the flange section and the web section to evaluate spatial variation in material properties.

3.1 FLEXURAL STRENGTH

Flexural strength was measured on rectangular bar specimens (5 x 0.6 x 0.3 cm) in a four-point bending mode with an Instron machine. In the strength test, an outer span of 4.45 cm, inner span of 0.9525 cm, and a crosshead speed of 0.05 cm/min were used. From the strength data, we constructed a stress-vs.-failure probability curve and calculated the Weibull moduli, as discussed in the next section. The average strength values for the two filters are presented in Table 2.

The flexural strength of the flange section is consistently higher than that of the web section. This may be due to smaller critical flaw size for the flange section, which may be related to microstructural differences between the materials in the flange and the web sections.

3.2 WEIBULL STRENGTH DISTRIBUTION

A statistical analysis of the flexural-strength data was carried out to obtain Weibull strength distribution. Weibull modulus is a measure of the statistical distribution of the flexural strength. Higher values are reflected in smaller scatter of the data. The Weibull moduli were evaluated from the slopes of the log-log plots of the stress vs. probability of failure, shown in Figs. 6-9. Table 3 gives the values of the Weibull moduli in the four cases we considered.

Two important observations can be made from the data in Table 3. First, the Weibull modulus m for the filter materials in the as-fabricated condition ranges from 14 to 16, suggesting that the material strength does not have a wide scatter. Second, the Weibull modulus for the web section changes from 14 in the as-received filter to 9 in the used filter, indicating a change in flaw population with exposure to a gasifier environment.

Table 2. Average flexural strength of the filter material (MPa)

Filter	Web Section	Flange Section
Filter A (as-fabricated)	22.3 ± 2.3	27.1 ± 2.0
Filter B (subjected to gasifier environment)	21.3 ± 2.3	28.2 ± 1.9

Fig. 6. Weibull distribution of flexural strength for the flange section of the as-fabricated filter

Fig. 7. Weibull distribution of flexural strength for the web section of the as-fabricated filter

Fig. 8. Weibull distribution of flexural strength data for the web section of the filter exposed to gasifier environment

Fig. 9. Weibull distribution of flexural strength data for the flange section of the filter exposed to gasifier environment

Table 3. Weibull moduli of the filter material

Filter	Web Section	Flange Section
Filter A (as-fabricated)	14	16
Filter B (subjected to gasifier environment)	9	18

The reduction in Weibull modulus indicates a larger scatter in the strength data and an increase in flaw size. In contrast, the Weibull modulus remains nearly the same for the flange section after exposure to the gasifier environment. This is because the hot gas primarily flows through and affects the material properties of the web section.

3.3 FRACTURE TOUGHNESS

The filter material was measured for fracture toughness by the single-edge notch beam technique. Approximately 80 specimens, 20 each machined from the web and flange sections of each filter, were tested. The typical dimension of the specimens was 5 x 0.25 x 0.21 cm.

The results presented in Table 4 indicate that the fracture toughness of the flange section for both filters is higher than that of the web section. The higher value of the flange material may be related to the microstructural difference between the web and flange materials. Unlike strength, fracture toughness is a material property that depends on the total bulk of the material and is not significantly affected by small local changes in microstructure. Therefore, small changes in pore structure due to exposure to a gasifier environment have not caused much change in toughness values.

3.4 MODULUS OF ELASTICITY

Elastic moduli were evaluated from the slopes of load deflection plots obtained during four-point bend tests of strength. Approximately 60 specimens from the web and flange sections of each of the filters were tested separately. The effect of machine compliance was taken into account by independently measuring the machine compliance with a stiff alumina specimen. The results are presented in Table 5.

The results in Table 5 show that, overall, the elastic modulus of the flange section is lower than that of the web, i.e., the web is stiffer than the flange. This may be due to a slightly higher density of the web section as compared to the flange.

3.5 CRITICAL FLAW SIZE

To develop filter materials with improved mechanical properties, it is very important that we understand failure-causing critical flaws. Exact measurement of critical flaw sizes by fractography was difficult, because of the coarse nature of the microstructure. Therefore, analytical methods were used to evaluate critical flaw sizes. The critical flaw size C_{cr} for the modulus-of-rupture (MOR) bars machined from Filters A and B was calculated from the fracture mechanics equation⁸, $K_{IC} = 1.35 \sigma_f \sqrt{C_{cr}}$, where K_{IC} is fracture toughness, and σ_f is strength of the material. Based on the measured values of K_{IC} and σ_f , critical flaw sizes were calculated. The critical flaw sizes for the flange section of the as-received filter were

Table 4. Average fracture toughness of the filter material (MPa√m)

Filter	Web Section	Flange Section
Filter A (as-fabricated)	0.44 ± 0.07	0.62 ± 0.08
Filter B (subjected to gasifier environment)	0.49 ± 0.13	0.77 ± 0.07

Table 5. Average elastic moduli of the filter material (GPa)

Filter	Web Section	Flange Section
Filter A (as-fabricated)	22.7 ± 0.4	16.1 ± 0.3
Filter B (subjected to gasifier environment)	20 ± 0.3	16.5 ± 0.3

216–465 μm and the corresponding values for the used filter were 330–584 μm. This indicates an increase in critical flaw size of the filter material in a gasification environment, probably due to thermal and/or mechanical fatigue.

3.6 THERMAL-SHOCK RESISTANCE

The filter materials are generally subjected to a thermal-shock environment during fabrication and service. Therefore, to ensure structural reliability, measurement of thermal shock resistance and its correlation with microstructure is necessary. Thermal-shock resistance was measured by quenching rectangular bar specimens (≈5 x 0.5 x 0.5 cm) at various temperatures in room-temperature water. Water was used as a quenching medium to simulate an enhanced heat transfer and thermal shock condition for experimental convenience. The heat transfer and thermal shock for filters in a gasifier environment is relatively less severe. Testing in the enhanced heat transfer environment will quickly provide an understanding of crack propagation and thermal shock failure and furnish information to develop microstructures with improved thermal shock resistance. The specimens were slowly heated to predetermined temperatures in an electrically heated furnace, held at that temperature for ≈30 min to attain thermal equilibrium, and dropped into the water bath. Four specimens were used for each test condition. Mechanical degradation of the specimens was determined by measuring their flexural strengths before and after the thermal quench. The results presented in Fig. 10 show that the flexural strength of the material decreases steadily with quenching temperature difference. The continuous drop in strength with quenching temperature drop is consistent with the presence of large critical flaws (216–465 μm), as calculated in the previous section and discussed in the unified theory of thermal fracture.⁸

This study was limited to a single material. To develop microstructures with improved thermal shock resistance, thermal shock behavior of materials with different microstructures must be studied and correlated with microstructure.

Fig. 10. Thermal shock behavior of cross-flow filter

4 MICROSTRUCTURAL INVESTIGATIONS

To understand the processes that govern the physical and mechanical properties of the filters, detailed microscopic investigations were carried out. Scanning electron microscopy (SEM), coupled with energy-dispersive X-ray (EDX) analysis, was performed on fractured surfaces of specimens obtained from Filters A and B.

Figure 11 shows a typical fractured surface from as-fabricated Filter A. The micrograph clearly shows that pores range from a few micrometers to more than 100 μm . This is visual confirmation of what has been observed by mercury intrusion porosimetry, presented in Section 2. Many of these pores are connected to each other, forming pore channels. Thus, one may consider the structure of the filter as a porous material with connected pore channels. These channels may be very tortuous in cross section.

To study the material at grain level, SEM studies were done at higher magnification. Figure 12 shows the fractured surface of the as-fabricated Filter A, displaying details of the interior of a pore. This pore is lined with needle-shaped crystals of few micrometers in length. To identify the crystals, EDX analyses were performed. Figure 13 shows the micrograph of another pore with growing needles, along with EDX outputs of specific areas identified by numbers on the micrograph. The EDX output presented in Fig. 13a from locations 1 and 2 on the needles shows strong peaks of aluminum and silicon and a small peak of calcium. This suggests that these crystals are aluminosilicates, possibly mullite.

In contrast, the EDX output from location 3 in the bulk material, as shown in Fig. 13b, indicates the presence of aluminum, silicon, calcium, sodium, and sulfur. The sulfur peak must be a contamination, because it was not found elsewhere. The sodium and calcium peaks represent minor phases, which were often seen in different regions. This is consistent with X-ray diffraction data, presented in Section 2, which identified the existence of sodium- and calcium-containing minerals, i.e., anorthite ($\text{CaAl}_2\text{Si}_2\text{O}_8$) and albite ($\text{NaAlSi}_3\text{O}_8$).

Fig. 11. SEM micrograph of fractured surface of the as-fabricated filter material, showing pores that range from a few microns to 100 μm

Figure 14 is a micrograph of the fractured surface of the filter material subjected to a gasifier environment. The structure of the material is nearly the same as that of the fractured surface of the as-fabricated filter. However, there is no evidence of crystal growth on the pore surfaces. In fact, broken ends of the crystals and the corresponding debris are noticeable in the micrographs. It is likely that these crystals were destroyed during exposure to the gasifier atmosphere due to impact of the rushing gas or simply to thermal shock. In addition, corrosion of these crystals in the gasifier environment is also a possibility. It is evident from porosimetry and microstructural evaluation that these processes contribute to microstructural changes, such as pore size and morphology, and have important implications for the performance of these filters in gasifier environments. Further studies are needed to understand the role of such erosion/corrosion processes.

5 STRESS AND FRACTURE ANALYSES OF FILTERS

In parallel with mechanical, thermal, and microstructural evaluations, stress and fracture mechanics analyses were performed and the results were correlated with the measured property values to establish fracture criteria. In all cases, the theory of linear elasticity was used for stress analysis, and linear elastic fracture mechanics was used to predict onset of fracture. Both the stress and fracture analyses were performed using the

Fig. 12. SEM micrograph of fractured surface of the as-fabricated filter, showing details of the interior of a pore

finite-element method. In a similar fashion, the analyses for determining the natural frequencies and transient vibrations of the filter were conducted using finite-element methods.

Filters are subjected to stresses from the following loadings:

- Residual stresses due to fabrication.
- Stresses during setup and clamping.
- Thermal stresses during cold start-up.
- Stresses during steady-state operation due to mismatch in thermal expansion coefficients between various parts.
- Thermal stresses during pulse-cleaning.
- Pressure-induced stresses during pulse-cleaning.
- Transient vibratory stresses during pulse-cleaning.
- Thermal stresses during cool-down.

Fig. 13. SEM micrograph of fractured surface of the as-fabricated filter (showing growing needles), and EDX outputs for (a) locations 1 and 2, and (b) location 3 on the micrograph

Fig. 14.
SEM micrograph exhibiting corroded crystal of filter material subjected to gasifier environment

Under the current program, the thermal, pressure-induced, and transient vibratory stresses that occur during pulse-cleaning were investigated.

5.1 PULSE-CLEANING CYCLE

A filter was subjected to both thermal and pressure-induced stresses during a pulse-cleaning cycle. The peak pressure loading during the pulse-cleaning cycles was estimated by Westinghouse engineers to be ≈ 20 psi. In addition, measurements during blowdown pulse-cleaning of the filter at Westinghouse indicated the presence of 10-g accelerations (with frequency components at ≈ 200 , ≈ 350 , and ≈ 600 Hz) in the platforms to which the filters were attached. Although the duration of the pressure pulse itself was on the order of 0.45 sec (with a rise time of ≈ 0.05 sec), the dynamic effects, as detected by the measured accelerations, persisted for more than 1 sec.

A thermal analysis of a pulse-cleaning cycle was conducted by Allied Signal on their (6 x 6 x 2-in. with plate thickness = 0.05 in.) geometrical model but with their material thermal properties replaced by those of the Coors mullite. This analysis indicated that the maximum temperature drop through the 0.05-in. thick plate near the flange during the transient could be as great as 600°F. Based on this analysis, the temperature drop through the 0.1-in.-thick plate of the Texaco filter would be even higher. However, this conclusion was not accepted by the Westinghouse engineers, who estimated the temperature drop through the 0.1-in.-thick plate to be much lower. Because of the uncertainty in the thermal analysis of the filter during pulse-cleaning, the stress analysis was conducted parametrically.

5.2 METHOD OF ANALYSES OF FILTERS

Stress analysis (elastic) with the finite-element code ANSYS was conducted on two different models of the filter: (a) a micro model and (b) a macro model. The micro model was used to analyze for thermal and pressure-induced stresses and fracture, considering the details of the channel geometry. First, the analyses were performed to obtain the bulk stresses in the channel walls, without considering the stress concentration effects at the channel corners (i.e., using a coarse mesh at the channel corners) and using both a three-dimensional and a two-dimensional model. These stresses were compared with the measured modulus of rupture (MOR) of the bulk material to predict the failure loads of the filter. Note that these predicted failure loads did not correspond to crack initiation at channel corners, which might occur at lower loads due to stress concentration effects. No analysis was carried out to determine the stress concentration effects at the channel corners, because the local maximum stresses computed at the corners could not be compared directly to the MOR which is a bulk property. Instead, cracks were assumed to have been initiated at the corners, and the stability of the cracks was investigated. Stress intensity factors were determined by introducing cracks of various lengths at the channel corners of a two-dimensional model. The stress intensity factors were compared with the measured fracture toughness of the material to predict the loads that caused onset of unstable fracture.

The macro model was used primarily for analyzing pressure, and vibration-induced stresses in the filter on a global scale, ignoring the details of the gas channel configurations. A full three-dimensional model of the filter was used to determine the natural vibration modes and frequencies from effective homogeneous anisotropic elastic properties of the filter flange and internals. A simplified two-dimensional model, which considered the stress concentration effects at the fillet junction between the filter and the flange, was used for analyzing pressure-induced stresses, natural vibration modes, and transient vibratory stresses.

5.3 MODELS

5.3.1 Micro Model

The micro model consisted of a typical unit cell, located in the interior of the filter, with a clean-gas-side channel and a dirty-gas-side channel attached to a single filter plate. Because of symmetry, it was necessary to analyze only half the channels (Fig. 15). The plates, 0.1 in. thick, were stacked in the y-direction, and the geometry was interchangeable and repetitive in the x and z directions. As a result, periodic boundary conditions could be used in the x, y, and z directions. The loadings we considered were a temperature rise ΔT and a pressure drop Δp across the filter plate from the clean-gas side to the dirty-air side. The pressure and temperature drops across the channel walls were assumed to be zero, because global variations in pressure and temperature were ignored in the analysis.

To simplify the analysis for the fracture mechanics calculations, a two-dimensional, generalized plane strain micro model was used. Comparisons between the three-dimensional and two-dimensional analyses for the thermal stresses in an uncracked filter are shown in Figs. 16 and 17, respectively. Similar comparisons for the pressure-induced stresses are shown in Figs. 18 and 19. Note that the maximum three-dimensional thermal stress could be estimated to within 20% by a two-dimensional analysis. On the other hand, because the pressure load is carried by biaxial bending, a two-dimensional, generalized plane strain model overestimated the three-dimensional maximum stress by $\approx 50\%$. Therefore, the two-dimensional fracture mechanics analysis provided a conservative upper bound for the stress intensity factor in the case of the pressure loading.

Each stress intensity factor was determined in two steps. First, a global analysis of the model was carried out using regular solid elements, as shown in Fig. 20. In the second step, a detailed analysis of the crack tip region (Fig. 21) was carried out with crack-tip singular elements and displacement boundary conditions obtained in the previous step. Figure 22 shows a nondimensional plot of the computed stress intensity factors as functions of the crack length a , for both thermal and pressure loadings.

5.3.2 Macro Model

Two different macro models, one two-dimensional and the other three-dimensional, were used. The two-dimensional model consisted of one of the forty $12 \times 4 \times 0.1$ in. plates attached to the $11.78 \times 5.5 \times 0.75$ in. flange with fillets of 0.25-in. radius (Fig. 23). This model was used to analyze for stresses due to gas pressure and 10-g acceleration loadings (to simulate vibratory loadings) during a clean-up cycle, and for conducting natural-frequency and transient dynamic analyses. The three-dimensional model (Fig. 24), which was used to check for the accuracy of the two-dimensional modal frequency analysis, consisted of the full $12 \times 12 \times 4$ in. filter divided into three zones: (a) the active filtering zone ($10.73 \times 3.67 \times 11.78$ in.) containing orthogonal gas channels, (b) the section of the flange containing only the clean-gas channels ($3.67 \times 11.78 \times 0.75$ in.), and (c) the remaining solid section of the flange. To simplify the analysis, the first two zones, with gas channels and their elastic properties, were replaced by equivalent solid zones of homogeneous anisotropic elastic materials with the same overall stiffnesses as those of the respective regions. The resulting effective elastic constants (using the axes shown in Fig. 23) for the two zones, together with those of the solid mullite, are given in Table 6 and are based on the following stress-strain law:

Fig. 15. Three-dimensional model of the internal channels of the Texaco filter. Because of symmetry, it was necessary to analyze only one-quarter of a typical unit cell.

Fig. 16. Stress distribution (in ksi; 1 ksi = 6.895 MPa) in the three-dimensional micro model of the filter due to a 600°F temperature drop through the filter plate

Fig. 17. Stress distribution (in ksi) in the two-dimensional micro model of the filter due to a 600°F temperature drop through the filter plate

Fig. 18. Stress distribution (in ksi) in the three-dimensional micro model of the filter due to a 20-psi pressure drop through the filter plate

Fig. 19. Stress distribution (in ksi) in the two-dimensional micro model of the filter due to a 20-psi pressure drop through the filter plate

Fig. 20. Global analysis of the two-dimensional micro model of the filter with a 0.03-in. crack due to a 10-psi pressure drop across the filter plate

Fig. 21. Near-tip finite-element model (with singular elements) of the cracked filter with a 0.03-in. crack. The displacement boundary conditions for the analysis were obtained from the global analysis shown in Fig. 19.

Fig. 22. Nondimensional stress intensity factors vs. normalized crack length due to a temperature drop ΔT and pressure drop Δp across the filter plate of the two-dimensional micro model. L and h are geometrical parameters; E and ν are Young's modulus and Poisson's ratio, respectively; and α is the thermal expansion coefficient.

Fig. 23. Two-dimensional macro model of the filter

Fig. 24. Three-dimensional macro model of the filter

Table 6. Thermomechanical properties for solid mullite and the two zones

Solid Mullite (50% dense)

Young's Moduli $E_x = E_y = E_z = 1.5 \times 10^6$ psi (10^4 MPa)

Poisson's Ratios $\nu_{xy} = \nu_{xz} = \nu_{yz} = 0.3$

Shear Moduli $G_{xy} = G_{yz} = G_{xz} = 0.6 \times 10^6$ psi (4×10^3 MPa)

Thermal Expansion Coefficients $\alpha_x = \alpha_y = \alpha_z = 2.8 \times 10^{-6}/^\circ\text{F}$
($4 \times 10^{-6}/^\circ\text{C}$)

Mass density = 1.51×10^{-4} lb-s²/in⁴ (1600 kg/m³)

Thermal Diffusivity = 0.02 ft²/h (5×10^{-7} m²/s)

Specific heat = 0.25 Btu/lb ·°F (1050 J/kg ·°C)

Thermal conductivity = 0.5 Btu/ft · h ·°F (0.84 W/m ·°C)

Zone 1 (Material with orthogonal gas channels)

$E_x = E_y = 6.2 \times 10^5$ psi (4.3×10^3 MPa) $E_z = 1.4 \times 10^5$ psi (930 MPa)

$\nu_{yz} = 0.05$ $\nu_{xy} = 0.26$ $\nu_{xz} = 0.05$

$G_{xz} = G_{yz} = 0.2 \times 10^6$ psi (1.4×10^3 MPa) $G_{xy} = 0.3 \times 10^6$ psi (1.8×10^3 MPa)

Mass density = 0.71×10^{-4} lb-s²/in⁴ (750 kg/m³)

Zone 2 (Flange material with clean gas channels)

$E_x = E_y = 1.1 \times 10^6$ psi (7.6×10^3 MPa) $E_z = 0.25 \times 10^6$ psi (1.8×10^3 MPa)

$\nu_{yz} = 0.05$ $\nu_{xy} = 0.26$ $\nu_{xz} = 0.05$

$G_{xz} = G_{yz} = 0.3 \times 10^6$ psi (2.1×10^3 MPa) $G_{xy} = 0.4 \times 10^6$ psi (2.8×10^3 MPa)

Mass density = 1.11×10^{-4} lb-s²/in⁴ (1175 kg/m³)

$$\{\sigma\} = [E] \{\epsilon\},$$

$$\text{where } \{\sigma\} = \begin{Bmatrix} \sigma_x \\ \sigma_y \\ \sigma_z \\ \sigma_{xy} \\ \sigma_{yz} \\ \sigma_{xz} \end{Bmatrix} \quad \{\epsilon\} = \begin{Bmatrix} \epsilon_x \\ \epsilon_y \\ \epsilon_z \\ \gamma_{xy} \\ \gamma_{yz} \\ \gamma_{xz} \end{Bmatrix}$$

$$\text{and } [E]^{-1} = \begin{bmatrix} 1/E_x & -\nu_{xy}/E_y & -\nu_{xz}/E_z & 0 & 0 & 0 \\ -\nu_{xy}/E_y & 1/E_y & -\nu_{yz}/E_z & 0 & 0 & 0 \\ -\nu_{xz}/E_z & -\nu_{yz}/E_z & 1/E_z & 0 & 0 & 0 \\ 0 & 0 & 0 & 1/G_{xy} & 0 & 0 \\ 0 & 0 & 0 & 0 & 1/G_{yz} & 0 \\ 0 & 0 & 0 & 0 & 0 & 1/G_{xz} \end{bmatrix}$$

5.4 RESULTS

5.4.1 Static-Stress Analysis (no cracks)

The maximum stresses computed from the finite-element analyses of the micro model were equated to the measured bulk MOR to predict the maximum permissible temperature drop and pressure drop across the filter plate, as shown in Figs. 25 and 26. Depending on the MOR, the maximum permissible temperature drop ranged from 600 to 1000°F. The range in temperature drop was reduced to 300 to 500°F if the elastic modulus was increased from 10 GPa to the measured value of 20 GPa. On the other hand, the maximum permissible pressure drop was far in excess of the expected maximum value of 20 psi during the pulse. It should be emphasized that the failure temperature and pressure drops referred to bulk failure of the filter and did not refer to crack initiation at the channel corners due to stress concentration effects.

Examination of cross-flow ceramic filters after recent cracking showed evidence of the clamps being cocked during the tests so that the clamping forces on the flange were concentrated on a narrow linear region near the free end of the flange. Also, as mentioned earlier, accelerations on the order of 10 g were measured in the platforms to which the filters were attached during pulse-cleaning. To determine the effects of a narrow clamp on the maximum stresses in the filter, elastic analyses were carried out for the two-dimensional macro model (Fig. 22) of the filter under a gas pressure of 20 psi and a static 10-g acceleration loading. Assuming that the pressure and the inertia loadings on the whole filter were equally shared by each of the forty 0.1-in.-thick plates, the equivalent pressure and acceleration loadings on each plate were 50 MPa and 16 g, respectively.

Fig. 25. Maximum permissible temperature drop across filter plate as a function of modulus of rupture (MOR). Range of measured MOR and corresponding maximum permissible temperature drop are indicated.

Fig. 26. Maximum permissible pressure drop across filter plate as a function of modulus of rupture (MOR). Range of measured MOR and corresponding maximum permissible pressure drop are indicated.

The results comparing the stresses in a uniformly clamped (i.e., y displacement equal to zero from the end of the fillet to the free end of the flange) filter with those in a filter clamped along a line at the free end of the flange are summarized in Table 7. Although, as a result of a reduction of the clamping area, the maximum tensile stresses in the fillet were increased by more than a factor of 2, they were still below the measured fracture strength (≈ 2.5 ksi) of the material. The maximum stresses under the clamps were increased by much larger factors because of the clamping area was reduced to a line. Theoretically, the maximum compressive stresses under the clamps were infinite for such a clamping condition. More important, large transverse tensile stresses, which exceeded the tensile fracture strength of the material, were also generated under the clamps. Although these stresses were highly peaked and localized, they might cause cracking under the clamps. If the line contact of the clamps were at the end of the fillets instead of at the free ends of the flange, the enhancement in the maximum tensile stresses in the fillet would be rather small ($\approx 5\%$).

Table 7. Effect of support clamping condition on the maximum stresses in the filter due to 20-psi pressure and 10-g vertical (y) loading

Stress Mode	Uniform support on flange ^a (psi)		Line support on flange ^b (psi)	
	20 psi	10 g	20 psi	10 g
Max. fillet stress	650	160	1400	350
Max. compressive stress under the clamp	-2150	-540	-14500 ^c	-3700 ^c
Max. tensile stress under the clamp	220	50	4900	1200

^aThe flange is supported uniformly from the end of the fillet to the free end of the flange.

^bThe flange is supported along a line at the free end of the flange.

^cThese stresses would be theoretically infinite if the contact were truly a line.

5.4.2 Analysis of Static-Fracture Mechanics

These analyses were conducted with the two-dimensional micro model with cracks at the channel corners. Stress intensity factors for cracks of various lengths located at the channel corners are shown in Fig. 27 for three values of temperature drop and a pressure drop of 20 psi across the thickness of the filter plate. The measured fracture toughness of the material was ≈ 0.5 ksi-in.^{1/2} (0.55 MPa-m^{1/2}). It was evident that a pressure drop of 20 psi by itself could not cause unstable fracture up to a crack length of 0.05 in. Figure 28 shows a plot of the allowable temperature drop through the thickness of the plate vs. crack length for three values of fracture toughness. If the Allied Signal thermal analysis was correct and the temperature drop through the filter plate was $\geq 600^\circ\text{F}$, then, for a fracture toughness of 0.5 ksi-in.^{1/2}, the maximum permissible crack length in the filter plate was

Fig. 27. Variation of stress intensity factor with crack length for three temperature drops and a 20-psi pressure drop across filter plate

Fig. 28. Maximum allowable temperature drop across filter plate vs. crack length for three values of fracture toughness of the material. Average value of measured toughness was $0.5 \text{ Ksi-in}^{1/2}$.

Table 8. Summary of the natural frequencies (Hz) of the filter as determined from three- and two-dimensional analyses. The fundamental-mode shapes for both analyses were the same. The second-mode shape for the two-dimensional model coincided with the 4th mode shape of the three-dimensional model. The involved out-of-plane bending and twisting, which were not included in the two-dimensional model.

Mode	3-D Model Uniform ^a	2-D Model	
		Uniform ^a	Line ^b
1st	351	329	216
2nd	515	1175	649
3rd	714	-	-
4th	1256	-	-

^aThe flange was supported uniformly from end of fillet to free end of the flange.

^bThe flange was supported along a line at free end of flange.

≤0.015 in. On the other hand, if the maximum temperature drop was ≤400°F, the filter could tolerate a crack > 0.04 in. without any gas pressure, and a crack >0.03-in. if a gas pressure of 20 psi was superimposed.

5.4.3 Analysis of Natural Frequency

Recent measurements of acceleration in the filter support platforms during the blowdown pulse-cleaning cycle at Westinghouse revealed the presence of the following frequencies: 42, 200-220, 350-360, and 590-620 Hz, with major energy inputs from the 200, and 350-Hz signals. To check whether these frequencies corresponded to any of the natural frequencies of the filter, modal-frequency analyses were performed with both the three-dimensional and two-dimensional models of the filter. Uniform clamping of the flanges was assumed for both models, but an additional case, in which the clamping was confined to a line at the free ends of the flange, was also investigated for the two-dimensional model. The natural frequencies are summarized in Table 8 and the mode shapes are given in Figs. 29-34.

For similar boundary conditions and mode shapes, the frequencies corresponding to the three and two-dimensional analyses were within 10% of each other. However, in addition to the two modes detected by the two-dimensional model, the three-dimensional model provided two additional modes involving out-of-plane bending and twisting with intermediate frequencies. Also, to constrain the clamping to a line at the free ends of the flange, the clamping conditions were changed. This resulted in significant reduction in natural frequencies. Because evidence for such narrow clamping was recently obtained at Westinghouse, the coincidences of some of the measured frequencies with the calculated

Fig. 29. Fundamental-mode shape of three-dimensional model of filter; corresponding frequency was 351 Hz

Fig. 30. Second-mode shape of three-dimensional model of filter; corresponding frequency was 515 Hz

Fig. 31. Third-mode shape of three-dimensional model of filter; corresponding frequency was 714 Hz

Fig. 32. Fourth-mode shape of three-dimensional model of filter; corresponding frequency was 1256 Hz

Fig. 33. Fundamental-mode shape of two-dimensional model of filter; actual mesh used for the analysis was much finer, and corresponding frequency was 329 Hz

Fig. 34. Second-mode shape of two-dimensional model of filter; actual mesh used for the analysis was much finer, and corresponding frequency was 1175 Hz. This mode shape was similar to fourth-mode shape of three-dimensional model.

Fig. 35. Variation of input (node 32), transverse (x) displacement, and response of node 1186 situated at top of the filter, with time. Input frequency was 329 Hz and amplitude was selected to correspond to a maximum 10-g acceleration under sinusoidal cycling. Filter flange was uniformly supported in the y direction from the end of the fillet to free end of flange.

frequencies may not be fortuitous. Note that the natural frequencies were not altered appreciably when the clamping was constrained to a line at the ends of the fillet rather than at the free ends of the flange.

5.4.4 Analysis of Transient Dynamics

As mentioned earlier, 10-g accelerations were measured at Westinghouse on the filter support platform during pulse-cleaning cycles, with major energy inputs in the frequency range of 200-220 and 350-360 Hz. Also, it was shown in the last section that the fundamental frequency of the filter decreased from 329 to 216 Hz as the support condition was varied from uniform clamping on the flange to clamping along a line at the free ends of the flange. Therefore, two two-dimensional transient-dynamic analyses were conducted on the filter by subjecting the two lateral edges (0.75 in. thick) of the flange to a number of constant-amplitude cycles at frequencies of 329 and 216 Hz, with corresponding clamping conditions on the filter flange. The amplitudes of the imposed cycles were chosen to correspond to a maximum 10-g acceleration under sinusoidal cycling. In both cases, 1% critical damping was assumed. Figure 35 shows the time variation of the input displacement (node 32) and the response of node 1186 situated at the top end of the filter; Fig. 36 shows the variation of the maximum principal stresses at the flange fillet for the cycles with a frequency of 329 Hz. Corresponding plots for the 216-Hz frequency are shown in Figs. 37

Fig. 36.

Variation of maximum principal stress (in psi) at fillet with time. Input frequency was 329 Hz and amplitude was selected to correspond to a maximum 10-g acceleration under sinusoidal cycling. Filter flange was uniformly supported in the y direction from the end of fillet to the free end of flange.

Fig. 37.

Variation of input (node 32), transverse (x) displacement, and response of node 1186 situated at top of the filter with time. Input frequency was 216 Hz and amplitude was selected to correspond to a maximum 10-g acceleration under sinusoidal cycling. Filter flange was supported in the y direction along a line in the z direction at free end of flange.

Fig. 38. Variation of maximum principal stress (in psi) at fillet with time. Input frequency was 216 Hz and amplitude was selected to correspond to a maximum 10-g acceleration under sinusoidal cycling. Filter flange was supported in the y direction along a line in the z direction at free end of flange.

and 38. In both cases, in addition to the fundamental frequency, higher harmonics were excited and maximum stresses were on the order of 1000–1200 psi. However, depending on damping and the number of imposed cycles, higher stresses might be generated.

6 SUMMARY AND CONCLUSIONS

Physical, mechanical, and microstructural evaluations were carried out on two cross-flow filters, one in as-fabricated condition (Filter A), the other after exposure to a gasifier environment (Filter B). Stress and fracture mechanics analyses were completed. Property data, in conjunction with stress and fracture mechanics analyses, were used to determine the probable cause(s) of failure. The results are summarized below.

The filters contain mullite as the major phase, with minor phases of anorthite and albite, and have approximately 41% porosity. The pore size varies from submicrometer to 112 μm , with an average size of 43 μm . When the filters are exposed to a gasifier environment, the pore size distribution broadens.

Thermal diffusivity of the filter material ranges from 0.005 to 0.009 cm²/sec in the temperature range of 25–1000°C. These values are approximately one-half those of dense mullite. The reduced value is due to the high porosity of the filter material.

The flexural strength of the flange sections of Filters A and B is 27.1 ± 2.0 MPa and 28.2 ± 1.9 MPa, respectively, whereas the strength of the web sections is 22.3 ± 2.3 and 21.3 ± 2.3 MPa, respectively. The higher strength values of the flange section may be due to smaller critical flaw sizes in the flange section, which may be related to microstructural differences between the materials in the flange and the web sections.

The Weibull modulus (m) of the strength data for the material in the web section is reduced from 14 to 9 after exposure of a filter to a gasifier environment. This implies that material strength reliability is reduced after the exposure of the filter to a gasifier environment.

Fracture toughness values of the flange material from Filters A and B are 0.62 ± 0.08 MPa $\sqrt{m}$ and 0.77 ± 0.07 MPa $\sqrt{m}$, respectively. The corresponding values for the web section are 0.44 ± 0.07 MPa $\sqrt{m}$ and 0.49 ± 0.13 MPa $\sqrt{m}$, respectively. The higher fracture toughness values of the flange material may be related to microstructural differences. Also, fracture toughness seems to be unaffected by the use of the filter in a gasifier environment.

The elastic modulus of the material in flange sections of Filters A and B is 16.1 ± 0.3 GPa and 16.5 ± 0.3 GPa, respectively, whereas that of material in the web sections of the two filters is 22.7 ± 0.4 GPa and 20 ± 0.3 GPa respectively. The results indicate that the elastic modulus of the flange section is lower than that of the web section, i.e., the web section is stiffer than the flange. This may be due to a slightly higher density of the web than in the flange.

The critical flaw sizes calculated by fracture mechanics, using the measured mechanical properties, were 216–465 μm for the as-fabricated filter. The corresponding values for the filter exposed to a gasifier environment are 330–584 μm , suggesting an increase in the critical flaw size of the filter material in a gasifier environment due to thermal and/or mechanical fatigue.

Thermal shock experiments indicate a continuous drop in the strength of the filter material with increasing severity of thermal shock. This observation suggests the presence of large critical flaws in the filter material, in agreement with the microscopic observations and fracture mechanics calculations of critical flaw sizes.

Microstructural investigations revealed needle-shaped mullite crystals on the pore surfaces of the filter. These crystals, however, were corroded/eroded after exposure to a gasifier environment. The details of these erosion processes must be studied to improve the durability of the filter material.

Static and dynamic finite-element stress analyses for the pulse-cleaning cycle of the filter were conducted with a micro model (considering the details of the gas channels) and macro model (ignoring the details of the gas channels). Fracture mechanics analyses of a micro model with preexisting cracks of various lengths at the channel corners were also carried out.

Stresses due to a pressure drop of 20 psi across the filter plates in the internal channels are quite low and should not cause failure. The maximum permissible temperature drop in the filter plate corresponding to the measured MOR using a value of 10 GPa for elastic modulus varies from 600 to 1000°F. The permissible temperature drop is 300 to

500°F if the measured value (20 GPa) of elastic modulus is used. If the thermal analysis of the pulse-cleaning cycle by Allied Signal is valid, the filter channels are at or close to the fracture limit. However, because the internal channels seldom fractured during testing, the analysis conducted by Allied Signal probably overestimated the temperature drop and should be verified in the future. Also, thermal stresses at the fillet radius between the filter and the flange should be analyzed in the future.

Fracture mechanics analyses of the internal channels showed that, for a pressure drop of 20 psi across the plate, the filter plate could sustain a crack length of one-half the thickness of the filter plate without experiencing unstable fracture. If the temperature drop across the filter plate were $\geq 600^\circ\text{F}$, the maximum permissible crack length for the internal channels would be ≤ 0.015 in. The permissible crack lengths could be significantly larger (>0.04 in.) if the temperature drop across the plate were $\leq 400^\circ\text{F}$.

Stress analyses of the two-dimensional macro model showed that the maximum tensile stresses in the fillet junction between the filter and the flange are much larger for a 20-psi pressure loading than for a static 10-g acceleration loading. Further, the maximum stresses increase by a factor of ≈ 2 when the clamping condition was changed from uniform to a line contact at the free end of the flange. A similar change in the clamping condition increased the stresses under the clamps drastically (factor of ≈ 7). Because such narrow contact regions have been detected recently at Westinghouse, the cracking under the clamp may have been caused by these stresses. However, the maximum tensile stress (1400 psi) in the fillet was still not sufficiently high to cause fracture.

Comparison of the natural frequencies of the two- and three-dimensional models showed that, for the same mode shapes, the computed natural frequencies are within 10% of each other. However, the three-dimensional model predicted two additional modes involving out-of-plane bending and twisting with frequencies intermediate between the first two modes of the two-dimensional model. The fundamental- and second-mode frequencies of the two-dimensional model decrease from 329 and 1175 Hz to 216 and 649 Hz, respectively, as the clamping condition is changed from uniform to a line contact at the free end of the flange. These reduced frequencies agree fairly well with the frequencies (200-220 Hz and 590-620 Hz) measured at Westinghouse. The other measured frequency of 350-360 Hz could correspond to either the second or the third mode of the three-dimensional model under a similar change in clamping condition. Near coincidences of the measured (Westinghouse) and calculated frequencies provided further evidence for the narrow contact between the clamp and the flange. However, the effects of nonuniform clamping in the longitudinal direction (i.e., z direction) of the flange must be determined in the future.

Transient-dynamic analyses (with 1% critical damping) of the two-dimensional model of the filter showed that tensile stresses as high as 1000-1200 psi could be generated in the fillet within a few cycles if the filter flange was shaken at near-natural frequencies of the filter in the horizontal (x) direction. Together with pressure-induced stresses, the tensile stresses approach the fracture strength of the material. Further, if the transient-dynamic cycles persisted for more than 1 sec during each pulse-cleaning, they represented a large number of cycles at appreciable stress amplitude at the fillet that could potentially cause subcritical crack growth due to fatigue, which must be evaluated in the future. However, because these stresses depend on the damping of the system, we must obtain a better definition of damping.

The analyses carried out under the present program suggest that, to minimize the maximum stresses in the filter, a linear contact between the clamp and the flange at the

flange free end should be avoided. A similar linear contact at the end of the fillet did not increase the maximum tensile stresses in the filter appreciably, although the maximum compressive stress under the clamp could have still remained high.

A comparison of the calculated values of maximum operating stresses and stress intensity factors with the measured room-temperature mechanical properties indicates that thermal loadings, pressure pulse loadings during cleanup, or 10-g acceleration loadings (to simulate dynamic effects during pulse-cleaning) are not high enough to cause immediate cracking of the filters during first-time loading. Together with the observation that the filters operate for a fair number of hours before cracking, the latter finding indicates that the observed failure is very likely caused by long-term fatigue and/or degradation of materials properties at elevated temperatures.

On one hand, the life of the ceramic filter will depend on the fatigue properties of the ceramic material, including any degradations due to usage. On the other hand, it will also depend on the severity of the maximum stresses to which the filter is subjected during operation. These maximum operating stresses might be enhanced if the filter is supported in a way for which it was not designed. For example, as indicated above, examination of cross-flow ceramic filters after recent cracking have shown evidence of the clamps being cocked during the tests, so that the reaction forces on the flange were concentrated on a narrow linear region near the free end of the flange. In addition, nonuniformity of clamping has also been observed in the longitudinal direction of the flange. These departures from "normal" operations will adversely affect the life of a ceramic filter.

The above observations suggest a need for further materials evaluations and stress analyses under both normal and nonnormal operating conditions.

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APPENDIX F

MECHANICAL CHARACTERIZATION AND STRESS ANALYSIS OF CERAMIC FILTERS

Mechanical Characterization and Stress Analysis of Ceramic Filters

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ABSTRACT

The mechanical/microstructural properties evaluations and stress analysis for the ceramic candle filters provided by Westinghouse Electric Corporation have been carried out to investigate the structural reliability and probable causes of filter failure. The filters primarily consisted of mullite (aluminum silicate) and were evaluated in both as-received and exposed to an oxidizing, pressurized circulating fluidized-bed combustion gas environment. Specifically, the geometric density and mechanical properties (strength, fracture toughness, and elastic modulus) were evaluated and a Weibull strength distribution was constructed to analyze the strength data, which provides information on strength variation for ceramic filters. Microstructural evaluation was also performed to evaluate grain size and morphology of the filter materials. In parallel with mechanical and microstructural evaluation, stress analysis was performed to estimate and evaluate the thermal stresses during pulse cleaning cycles. Analysis was also carried out for a disk flow-through test which simulated typical process operation (i.e., steam and gas phase alkali; pulse cycle conditions).

1. INTRODUCTION

This project has initiated further development of the materials qualification technology which is needed to ensure satisfactory performance of porous ceramic filter elements that are currently being considered for use in either oxidizing or reducing gas, advanced coal-fired applications. The basis for this effort is to identify those materials that are unlikely to provide satisfactory performance during actual field operation.

In order to provide long-term viability of the porous ceramic filter elements for use in utility or industrial applications, it is not only essential to improve but also demonstrate component reliability. Enhanced reliability of a component can be obtained by both improving mechanical properties as well as reducing the scatter of mechanical property data of the manufactured part. In view of the fact that brittle porous ceramic filter materials may have a wide distribution in the flaw density and flaw size within the material and therefore significant scatter in the strength value of the matrix, a single value of the mechanical properties is difficult to obtain. As a result, statistical analysis can provide a correlation between the mechanical properties of the matrix and the probability of the existence of critical flaws within the material. In addition, stress analysis have assisted in the development of the fracture criterion for the filter elements which are subjected to thermal stresses during field operation. Thermal stress results during pulse cleaning, as well as process thermal transition.

In this study, mechanical properties characterization, microstructural evaluation, and stress analyses were performed for field-tested porous ceramic candle filters in order to investigate possible filter failure modes. Specifically, the geometric density and mechanical properties (strength,

fracture toughness, and elastic modulus) of various filter elements were evaluated. In order to analyze the strength data, a statistical analysis known as the Weibull strength distribution was used. The Weibull strength distribution estimated the Weibull modulus which provides information on the strength variation for a given ceramic filter element. Microstructural evaluation was performed to evaluate grain size and morphology of the filter materials. In parallel with mechanical and microstructural evaluation, stress analyses were performed to estimate and evaluate the thermal stresses during pulse cleaning cycles. Similar analyses were also conducted for a disk geometry which experienced flow-through testing simulating the potentially corrosive oxidizing gas environment of a pressurized-fluidized-bed combustion gas system, as well as pulse cycling.

2. CONFIGURATION AND IDENTIFICATION OF CANDLE FILTERS

2.1. CONFIGURATION OF CANDLE FILTERS

Alumina/mullite candle filters for evaluation were obtained from Westinghouse Electric Corporation. The Alumina/mullite matrix consisted of mullite as a major phase and anorthite, alumina, and some minor glassy phases. The schematic of the filter element geometry is shown in Fig. 1. The filters are tube-shaped with an inside diameter (ID) of ≈ 4 cm, an outside diameter (OD) of ≈ 5.9 cm, and typically a total length of ≈ 1.5 m. The hot dirty gas flows through the outer wall of the filter, and exits through the open end as a clean gas. Dust particles are collected along the outer surface of the filter elements. As fines collect, the pressure drop through the element increases, and pulse of gas is delivered through the ID which permeates

through the porous ceramic wall, "pushing" the ash off of the candle OD surface.

2.2. IDENTIFICATION AND TEST CONDITIONS OF CANDLE FILTERS

To assess the effect of the high temperature process environment on the mechanical properties of the porous ceramic filter matrix, both as-manufactured and field tested candles were obtained from the Westinghouse Electric Corporation. The filters which were used in this study are identified in Table 1. Filters AC, BC337, and C35 were as-manufactured elements which had not been subjected to field testing and the section of Filter C35 which was characterized had undergone burst strength testing. The remaining filters had been exposed to an oxidizing environment in a pressurized circulating fluidized-bed combustor (PCFBC) test facility operated by Ahlstrom and located in Karhula, Finland. Filters AC79 (T19), AC128RA (T3), AC121RB (T22), and AC150RA (B38) were exposed to service conditions at $\approx 900^{\circ}\text{C}$. Each candle experienced thermal fatigue, and catastrophically failed after exposure periods of ≈ 130 , 227, 345, and 475 h, respectively. In addition, Filters AC204 (M2), AC274 (B22), and AC80 (B19) were tested for ≈ 716 h, while Filter AC204 (M2) was located in the middle plenum, and Filters AC274 (B22) and AC80 (B19) were located in the bottom plenum in the test vessel.

3. MECHANICAL PROPERTIES OF CANDLE FILTER MATERIALS.

For good structural reliability and improved design margins, improvement in mechanical properties and reduction in statistical scatter is needed for the filter materials. To this end, a detailed evaluation of the mechanical properties (strength, elastic modulus, and fracture toughness) of the candle filters before and after exposure to the process gas environment was carried out.

3.1. MECHANICAL PROPERTIES MEASUREMENTS

Mechanical properties evaluation included measurements of strength, fracture toughness, and elastic modulus. Strength was measured by C-ring compression test. C-ring specimens (ID $\approx$ 4 cm, OD $\approx$ 5.9 cm, thickness $\approx$ 1.5 cm) were machined from each filters and were fractured in compression mode at a cross-head speed of 0.13 cm/min. Depending on the availability of materials, approximately 7-25 rings were tested from each filter element. Schematic of C-ring specimen is shown in Fig. 2. From the fracture load, stress can be calculated by the following equation [1].

$$\sigma = \frac{P \cdot R (r - r_a)}{A \cdot r (R - r_a)} \cos \theta$$

where, P : fracture load

$$R = \frac{(r_o - r_i)}{\ln \left(\frac{r_o}{r_i} \right)}$$

$$r_a = \frac{r_o + r_i}{2}$$

$$A = (r_o - r_i) W$$

r_o is outer diameter, r_i is inner diameter, and W is width of C-ring specimen. As seen in above equation, the maximum stress occurs at $r = r_o$ and $\theta = 0^\circ$, which was taken to be the strength of the material.

Strength data was used to construct a Weibull strength distribution plots (failure probability distribution). Subsequently, Weibull modulus, m , was calculated from the slope of the failure probability line on logarithmic plots. For the fracture toughness and elastic modulus measurements, rectangular bar specimens ($\approx 0.6 \times 0.5 \times 5$ cm) were machined from each filter along the longitudinal direction and these specimens were ground on a SiC wheel with grit size of $20 \mu\text{m}$ to provide a standard surface finish. Fracture toughness was measured by the single-edge notch beam technique with a support span of 4 cm, a loading span of 2 cm, and a cross-head speed of 0.13 cm/min [2]. Notch width was approximately ≈ 0.03 cm. Elastic modulus was evaluated from the slope of the load-displacement plots obtained for the rectangular bar specimens. True specimen displacement was determined by subtracting system displacement from the total displacement. Approximately 5-6 specimens for each filter were tested for fracture toughness and elastic modulus measurements.

3.2. MECHANICAL PROPERTIES OF FILTER MATERIALS

The values of measured mechanical properties of as-manufactured filters as well as of the filters exposed to combustion environment as a function of exposure time are shown in Table 2. The table indicates that, in general, the mechanical properties (strength, fracture toughness, and elastic modulus) of the as-manufactured filters (AC, BC337, and C35) are superior relative to those of filters which were exposed to the combustion environment (AC79 (T19), AC128RA (T3), AC121RB (T22), AC150RA (B38), AC204 (M2), and XK

except for filters AC274 (B22) and AC80 (B19). For example, the strength, fracture toughness, and elastic modulus of as-manufactured filters was ≈ 22 - 24 MPa, ≈ 0.7 MPa $\sqrt{m}$, and ≈ 25 GPa, respectively. These values decreased to ≈ 13 - 19 MPa, ≈ 0.4 - 0.6 MPa $\sqrt{m}$, and ≈ 15 - 22 GPa, respectively, for the exposed filters. This degradation in mechanical properties of the exposed filters is believed to be due to the bulk damage induced during hot gas filtration testing.

A comparison of the mechanical properties of filters exposed for longer time (≈ 716 hours) with those of filters exposed for shorter times (≈ 130 - 475 hours) indicates a slightly increasing trend in mechanical properties after longer exposure time, i.e., at ≈ 716 hours. It is interesting to note that elastic modulus of these two filters (AC274 (B22) and AC80 (B19)) exposed for ≈ 716 hours was observed to be higher than that of as-manufactured filter AC. The reason for this observed increase after longer exposure time to combustion environment is not clear. As discussed in the following section 3.3, this could be related to filter-to-filter statistical variation in flaw population and resulting strength distribution.

3.3. WEIBULL STRENGTH DISTRIBUTION OF CANDLE FILTERS.

A statistical analysis of the flexural strength data was used to obtain a Weibull strength distribution. A Weibull plot of the failure probability (F) distribution for these specimens was constructed on a logarithmic scale as shown in Figure 3. The Weibull modulus, m , was calculated from the slope of the failure probability line. The Weibull plots and m values of as-manufactured filters (AC and BC337) and of filters (AC79 (T19), AC128RA (T3), AC121RB (T22), AC150RA (B38), AC274 (B22), and AC80 (B19)) exposed to combustion environment are shown in Figs. 3a and 3b, respectively.

The values of m were 22.4 and 23.2 for the as-manufactured filters AC and BC337, respectively (Fig. 3a). In comparison, the values of m for exposed filters (AC79 (T19), AC128RA (T3), AC121RB (T22), AC150RA (B38), AC274 (B22), and AC80 (B19)) varied from 8.5 to 25.7. In general, the m values for as-manufactured filters were larger than those for the exposed filters, except for filter AC274 (B22). As shown in Table 2, Filter AC274 (B22) also showed unexpectedly high strength as compare with other exposed filters. This could be related to filter-to-filter statistical variation in flaw population and resulting strength distribution. The general trend of a decrease in the values of m for exposed filters as compared with those for as-manufactured filters suggests a larger scatter in strength data, indicating a wider range of flaw population in the exposed filters. This could have resulted from the growth of pre-existing cracks (flaws) due to filters being subjected to mechanical and/or thermal stresses during exposure to combustion environment.

3.4. CRITICAL FLAW SIZE

Critical flaw sizes of filter materials were estimated to assess the microstructural change during exposure of filters to service environment. In view of the coarse and porous microstructure of candle filters, it was difficult to accurately locate critical flaws by fractographic technique. Therefore, the critical flaw size of each filters was estimated from the fracture mechanics equation [3],

$$K_{IC} = 1.35 \sigma_f \sqrt{c}$$

where K_{IC} is the fracture toughness, σ_f is the strength, and c is the critical flaw size. Measured values of fracture toughness and strength were used to calculate critical flaw sizes. The calculated value of critical flaw size was $528 \pm 44 \mu\text{m}$ for the as-manufactured filters (AC and BC337) and $680 \pm 252 \mu\text{m}$

for the filters exposed to combustion environment (AC79 (T19), AC121RB (T22), AC150RA (B38), AC128RA (T3), AC204 (M2), AC274 (B22), and AC80 (B19)). The higher value of critical flaw size for the exposed filters is probably due to increase in flaw size as a result of thermal and mechanical fatigue of the filters during exposure to the combustion environment.

4. MICROSTRUCTURE AND PROPERTIES OF FILTER MATERIALS

4.1. DENSITY

The density of the porous filter materials was measured by geometric method. Specifically, the mass (m) and physical dimensions (hence, the volume (v) of rectangular specimens cut from the filter element) were measured and density (ρ) was determined by $\rho = m/v$. The average measured density of the as-manufactured and exposed filter was 1.70 ± 0.03 g/cc. It was observed that there was no significant difference in densities of as-manufactured and exposed filters.

4.2. PHASE COMPOSITION AND X-RAY ANALYSIS

The phase composition of the filter materials was determined by X-ray analysis. The X-ray diffraction pattern of the as-manufactured filter (AC) is shown in Fig. 4. It can be observed that the major phase is aluminum silicate (mullite, $3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$) and minor phases are anorthite ($\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$), SiO_2 , and Al_2O_3 .

In addition, a few areas of the Filter AC150RA (B38) were observed to have been partially discolored ("yellow spots") on the outer surface and also along the cross-section of the filter. X-ray diffraction analysis for the area containing yellow spots indicates that the major phase is a mixture of mullite and anorthite, and the minor phases are SiO_2 and Al_2O_3 , as shown in Fig. 5.

These results were also confirmed by energy dispersive X-ray spectroscopy (EDX) data. From the comparison of Figs. 4 and 5, it can be observed that X-ray intensities of the anorthite phase increased significantly for the specimen containing yellow spots.

4.3. MICROSTRUCTURAL CHARACTERIZATIONS

In view of the fact that physical and mechanical properties of filter materials may be related to the microstructural changes, a microstructural evaluation of as-manufactured and exposed filters was performed. Specifically, scanning electron microscopy (SEM) of the fracture surface of the filters was carried out. Fig. 6 shows the micrograph of as-manufactured filter (Filter AC) at low magnification (X100). The micrograph clearly indicates a porous microstructure. The pores are interconnected and form pore channels. Pore sizes range from a few micrometers to several hundreds of micrometers. Similar microstructures were also observed for the other as-manufactured and exposed filters. Micrographs in Fig. 7 show the grain microstructures of as-manufactured filter (Filter AC) and filters exposed to combustion environment for ≈ 345 hours (Filter AC121RB (T22)) at a higher magnification (X1500). Typically, the grains were approximately $10\text{ }\mu\text{m}$ and there was little difference in microstructure between as-manufactured and exposed filters.

Fig. 8 shows microstructure of filter exposed to combustion environment for ≈ 716 hours (Filter AC204 (M2)). It can be observed that the typical grains were approximately $10\text{ }\mu\text{m}$ and there was no evidence of grain growth for the exposed filters as compared to microstructure of as-manufactured filter. The figure also shows the rod-shaped phases protruding along the pore cavity surface. EDX analysis indicated that the rod-shaped phases were

aluminosilicates, possibly mullite phases. In addition, localized regions of agglomerated rod-shaped phases were observed from the fracture surface of the Filters AC204 (M2) as shown in Fig. 9 (at magnification of 5000X). These localized regions of rod-shaped phases were identified to be Si-rich and Al-rich mullite phases by EDX analysis. Similar microstructure and phase compositions were also observed for the other as-manufactured and exposed filters. Some of rods-shaped mullite and Si-rich and Al-rich mullite phases were larger than the average grain in the mullite matrix. Depending upon their mechanical properties, these rod-shaped microstructures may impede crack propagation and hence may have an important effect on the failure of filters.

5. STRESS ANALYSIS OF CANDLE FILTERS

Stress analysis was conducted for the ceramic candle filter subjected to thermal stresses during pulse cleaning cycles. Analysis was also carried out on a disk which experienced flow-through testing which simulated exposure to a potentially corrosive oxidizing gas environment, as well as pulse cycling condition.

5.1. TEMPERATURE PROFILE (PULSE CLEANING CYCLE)

According to calculations made by D. Bachovchin [4], the plenum gas temperature can drop by as much as 774°C during the cleaning cycle. During the cleaning cycle transient, the temperature distribution in the candle is determined by the relative rapidity of heat transfer in the axial and

the thickness directions. Two approaches were taken to analyze the thermal shock problem of the candle filter during the pulse cleaning cycle.

In the first series of analyses it was assumed that initially, the whole filter was at 900°C and the stress everywhere was zero. First (case 1), a linear temperature distribution was assumed through the thickness of the filter wall varying from 800°C at the ID to 900°C at the OD throughout the entire length of the candle. This temperature distribution corresponded to a rapid mixing of the gas inside the filter together with a very efficient heat transfer in the porous wall. Second (case 2), the same linear temperature variation through the thickness of the filter wall was assumed to exist from the plenum end to an axial distance (which was treated as a variable) from the plenum end beyond which the filter wall was assumed to remain at a uniform temperature of 900°C. This case corresponds to a more rapid heat transfer in the thickness direction than in the axial direction. Because of the absence of any temperature gradient in the axial direction, case 1 loading gave a lower estimate of the maximum stresses in the filter than loading due to case 2.

In the second series of analyses, one-dimensional transient temperature profiles calculated by Westinghouse for the Coors candle filter was used. Basically for the temperature profile analysis, it was assumed that the gas temperature inside the filter equilibrated much faster than any conduction heat transfer could take place through the wall of the filter. The radial temperature distributions for two hypothetical cases were provided by Westinghouse. The pulse gas temperature was reduced in the first case (case 3, theoretical) from 900°C to 50°C in 0.2 s and in the other (case 4, Karhula experiment) from 890°C to 620°C in 0.2 s. These temperature data were applied to a finite element model to calculate stresses in the filter, assuming that there was no temperature gradient in the axial direction.

5.2. STRESSES IN COORS CANDLE FILTERS

Cases 1 and 2: The filter geometry considered for analysis is shown in Fig. 10. Figure 11 shows that, for case 1 with a uniform temperature drop of 100°C through the thickness, the maximum stress (2600 psi) is meridional (axial) and occurs at an axial distance of 1" from the plenum end of the filter. On the other hand, for case 2 in which a temperature shock front enters the plenum end of the filter and travels a distance into the filter, the peak stress is hoop and occurs at the shock front. The maximum stress distributions in the axial direction for shock front penetrations of 1" and 10" from the plenum end are shown in Figs. 12-13, respectively. The variations of the maximum meridional (axial) and hoop stresses at the front with the penetration distance of the shock front from the plenum end are shown in Fig. 14. Note that a high peak meridional (axial) stress (2900 psi) occurs when the shock has penetrated to ~2" from the plenum end. However, after the shock front moves into the interior of the filter, the maximum stress becomes hoop. Thus, if the failure occurs early during the cleaning cycle when the shock front has penetrated a relatively short distance into the filter, a transverse crack could occur at a distance of 2" from the plenum end. On the other hand, if the filter survives the early peak meridional (axial) stress and the shock moves into the interior of the filter, the peak stress becomes hoop which could cause cracking in the axial direction. Although initiation of an axial crack may tend to relieve the high hoop stresses, it could subsequently propagate in the transverse direction because of the presence of a high tensile meridional (axial) stress near the shock front. In all cases, crack initiation is predicted to occur at the ID surface where the maximum tensile stresses are present.

Case 3 (Theoretical): The axial distributions of the maximum hoop and axial stresses at the ID surface are shown in Fig. 15. The ID surface of the tube is subjected to a high biaxial state of stress with maximum hoop and axial stresses of 20,500 psi and 20,250 psi, respectively, at the inlet at time $t=0.2$ s. These stresses increase respectively to 24,500 psi and 24,000 psi at 0.4 s and 27,000 psi and 26,500 psi at 0.6 s, both occurring at the inlet. However, the stress distribution at any time t does not vary significantly in the axial direction. The ID surface hoop stresses increase and the axial stresses decrease somewhat near the junction with the end cap. The radial penetration of the hoop stress field (shown in Fig. 16) from a maximum at the ID surface to a level of 3000 psi (representing the fracture strength) range from 0.03 in at 0.2 s to 0.04 in at 0.6 s.

Case 4 (PCFBC Model): In this case, stresses are lower because the thermal shock is less severe. As in Case 3, the ID surface of the tube is subjected to a biaxial state of stress with maximum hoop and axial stresses of 7800 psi and 7700 psi, respectively, at the inlet at time $t=0.2$ s (Fig. 17). These stresses increase respectively to 8800 psi and 8700 psi at 0.4 s and 9450 psi and 9250 psi at 0.6 s, both occurring at the inlet. The radial penetration of the hoop stress field (shown in Fig. 18) from a maximum at the ID surface to a level of 3000 psi (representing the fracture strength) range from 0.02 in at 0.2 s to 0.03 in at 0.6 s. Again, because of the less severity of the shock the radial penetration of the stress field is less than in Case 3.

5.3. SIMULATION BY DISK FLOW-THROUGH TESTS

Westinghouse conducted several simulated flow-through tests for the filter using a holder/disk arrangement in which a 2.5" dia, 0.25" thick disk was clamped inside a holder with 6 bolts around the periphery. For the

present purpose, a uniform and linear temperature drop through the thickness of the disk from 900°C to 800°C (The test was conducted at 870°C, and pulsed with cold air) was considered. Depending on the clamping force, coefficient of friction and the clearance between the bolts and the hole diameter, various boundary conditions could be imposed on the disk. If the bolts clamped the disk completely such that there were no radial displacements and rotations at the edge, the maximum stress in the disk is 4000 psi. If the rotations were zero but the disk was free to deform in the radial direction, the maximum stress is 2000 psi. If neither the rotations nor the displacements were constrained at the edge, i.e., the disk was free, then the stresses are zero. In all cases, the stress field is equi-biaxial, i.e., the radial stress and the hoop stress at any point are equal. Also, the maximum tensile stresses occurred at the surface that was at 800°C.

6. SUMMARY

Mechanical/microstructural evaluations and stress analyses were performed on ceramic candle filters (in both as-manufactured and exposed condition) to investigate the structural reliability and probable causes of filter failures. The results are summarized below.

- The as-manufactured filters were generally observed to have superior mechanical properties relative to filters exposed to a combustion environment. The strength, fracture toughness, and elastic modulus of as-manufactured filters was $\approx 22-24$ MPa, ≈ 0.7 MPa $\sqrt{\text{m}}$, and ≈ 25 GPa, respectively. These values decreased to $\approx 13-19$ MPa, $\approx 0.4-0.6$ MPa $\sqrt{\text{m}}$, and

$\approx 15\text{-}22$ GPa, respectively, for the filters exposed to combustion environment ($\approx 130\text{-}475$ hours). This degradation in mechanical properties of the exposed filters is believed to be due to the bulk damage induced during hot gas filtration testing. On the other hand, it was observed that the mechanical properties of exposed filters for longer time (≈ 716 hours) were slightly superior to those of exposed filters for shorter times ($\approx 130\text{-}475$ hours). This slight increase in mechanical properties is probably related to filter-to-filter statistical variation in flaw population and resulting strength distribution.

- In general, the Weibull modulus (m) of strength data for the as-manufactured filters was larger than those for the exposed filters. The general trend of a decrease in the values of m for exposed filters as compared with those for as-manufactured filters suggests a larger scatter in strength data, indicating a wider range of flaw population in the exposed filters. This could have resulted from the growth of pre-existing cracks (flaws) due to filters being subjected to mechanical and/or thermal stresses during exposure to combustion environment.

- Critical flaw sizes of filter materials were estimated to assess the microstructural damage during exposure of filters to service environment. The calculated value of critical flaw size was 528 ± 44 μm for the as-manufactured filters and 680 ± 252 μm for the filters exposed to a combustion environment. The higher value of critical flaw size for the exposed filters is probably due to increase in flaw size as a result of thermal and mechanical fatigue of the filters during exposure to the combustion environment.

- The phase identification of the filter materials by X-ray analysis indicated that the filters consist of aluminum silicate (mullite, $3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$)

as a major phase and anorthite ($\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$), SiO_2 , and Al_2O_3 as minor phases.

- Microstructural investigations indicated that the as-manufactured and exposed filters have a porous microstructure with randomly oriented grains. Pore sizes ranged from a few micrometers to several hundreds of micrometers. Typically, the grains were approximately $10 \mu\text{m}$. In addition, localized regions of agglomerated rod-shaped phases were observed on the fracture surface of both as-manufactured and exposed filters. These localized region of rod-shaped phases were identified to be Si-rich and Al-rich mullite phases. Some of rods-shaped mullite and Si-rich and Al-rich mullite phases were larger than the average grain in the mullite matrix. Presence of these phases may have an important implication for the failure of filters.

- According to Westinghouse data, the bending strength of the alumina/mullite filter material dropped by approximately 33% after 100 h service exposure. The latter strength agreed well with that measured at Argonne National Laboratory. A comparison of the fracture strength with the calculated maximum stresses (cases 1 and 2) would imply that the maximum temperature drop through the thickness of the filter during the transient cannot be much greater than 100°C and is probably less.

- The maximum stresses at the ID surface of the Coors candle filter corresponding to either theoretical analysis in case 3 (27,000 psi) or the PCFBC Model in case 4 (9500 psi) far exceeded the fracture strength of the material (≈ 3000 psi). Because of the more severe nature of the shock in the case 3 (theoretical), the penetration of the maximum stress field at time 0.6 s was 0.04 in (≈ 0.16 mm) which is greater than that of 0.03 in (≈ 0.12 mm) in the PCFBC Model case. If these temperature distributions are anywhere close

to reality, the ID surface of the filter should experience microcracking in the circumferential as well as the axial directions. The actual depth of crack penetration will depend on the fracture toughness of the material and the effects of microcracking on stress redistribution.

- The maximum stresses in the filter were intermediate between the maximum stresses in the simulated disk specimens (assuming the same temperature drop in the simulated disk specimens as in the filter) corresponding to the boundary conditions of fully clamped and clamped against rotation but not against radial displacement. Thus, it is possible for the filters to fail during the cleaning cycles but the disk specimens to survive the simulated cleaning cycle tests if two conditions are satisfied. First, the boundary conditions on the disks are such that, at most, they are prevented from rotation at the periphery but are not constrained effectively from deforming in the radial direction which could be either due to insufficient clamping force generated by the bolts or due to too low a value of the coefficient of friction between the holders and the ceramic material. Second, even if the friction forces are overcome, radial displacements can occur in the disk only if there is sufficient clearance between the bolts and the bolt holes in the disk.

References

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Table 1. Filter identification and test conditions.

Filter identification	Operating (exposure) time (h)	Filter condition	Filter location	Filter test operating experience*
AC	0	As-manu-factured	Not installed	None
BC337	0	As-manu-factured	Not installed	None
C35	0	As-manufac-tured; burst strength test	Not installed	None
AC79 (T19)	≈130	Failed candle	Top plenum	Initial test sequence; adjacent to a failed candle resulting during fuel ignition event
AC128RA (T3)	≈227	Intact candle	Top plenum	Candle installed after fuel ignition event. 227 h of operation experienced during the last segment of CPFBC testing
AC121RB (T22)	≈345	Failed candle	Top plenum	Initial test sequence; fuel ingition event
AC150RA (B38)	≈475	Failed candle	Bottom plenum	Initial test sequence; fuel ignition event
AC204 (M2)	≈716	Intact candle	Middle plenum	Entire test sequence including fuel ignition event
AC274 (B22)	≈716	Intact candle	Bottom plenum	Entire test sequence including fuel ignition event; candle was adjacent to a failed candle at test termination
AC80 (B19)	≈716	Failed candle	Bottom plenum	Entire test sequence including fuel ignition event
XK	Unknown	Failed candle		

* Normal operating temperature of 900°C

Table 2. Measured mechanical properties of filter materials as a function of exposure time to combustion environment.

Specimen	Operating (exposure) time (h)	Strength (MPa)	Fracture toughness (MPa√m)	Elastic modulus (GPa)
AC	0	23.9 ± 1.2	0.71 ± 0.003	25.5 ± 1.8
BC337	0	21.7 ± 1.0	0.70 ± 0.004	24.5 ± 0.9
C35	0	-	0.62 ± 0.03	21.6 ± 1.4
AC79 (T19)	≈ 130	18.8 ± 1.3	0.51 ± 0.20	17.7 ± 2.4
AC128RA (T3)	≈ 227	16.4 ± 2.3	0.61 ± 0.01	22.3 ± 7.1
AC121RB (T22)	≈ 345	15.2 ± 0.9	0.50 ± 0.01	15.3 ± 2.7
AC150RA (B38)	≈ 475	13.6 ± 1.8	0.41 ± 0.01	-
AC204 (M2)	≈ 716	13.0 ± 3.3	0.61 ± 0.01	19.2 ± 4.5
AC274 (B22)	≈ 716	19.8 ± 0.9	0.60 ± 0.01	26.8 ± 3.9
AC80 (B19)	≈ 716	18.4 ± 1.6	0.70 ± 0.01	27.5 ± 6.5
XK	Unknown	-	0.43 ± 0.01	12.9 ± 2.2

Fig. 1. Schematic of ceramic candle filters.

Fig.2. Schematic of C-ring specimen.

(a)

(b)

Fig. 3. Weibull failure probability (F) distribution as a function of stress.

Fig.4. X-ray diffraction pattern for As-manufactured filter AC.

Fig. 5. X-ray diffraction pattern for Filter AC150RA (B38) containing partially discolored area ("yellow spots").

Fig. 6. SEM photomicrograph of fracture surface of the as-manufactured filter (Filter AC), showing porous microstructure.

(a)

Fig. 7. SEM photomicrographs of fracture surface of (a) as-manufactured filter (Filter AC) and (b) exposed filter (Filter AC121RB (T22)) showing typical grain size and shape.

(b)

Fig. 7. Continued.

Fig. 8. SEM photomicrographs of fracture surface showing typical grain size and shape of exposed filter (Filter AC204 M(2)).

Fig. 9. SEM photomicrographs of fracture surface showing rod-shaped Si-rich and Al-rich phases in Filter AC204 M(2).

Fig. 10. Geometry of candle filter (dimensions in inches).

Fig. 11. Axial distributions of hoop and meridional (axial) stresses near the plenum end of the filter due to a linear temperature distribution from 800°C at the ID to 900°C at the OD over the entire length of the filter.

Fig. 12. Axial variations of hoop and meridional (axial) stresses at ID surface due to a linear temperature distribution of 800°C at ID to 900°C at OD existing from the plenum end to a distance of 1" from the plenum end and a uniform temperature of 900°C beyond.

Fig. 13. Axial variations of hoop and meridional (axial) stresses at ID surface due to a linear temperature distribution of 800°C at ID to 900°C at OD existing from the plenum end to a distance of 10" from the plenum end and a uniform temperature of 900°C beyond.

Fig. 14. Variations of the maximum meridional (axial) and hoop stresses at the shock front with the depth of penetration of shock from the plenum end. Behind the shock front there is a linear temperature distribution of 800°C at ID to 900°C at OD. Temperature distribution is uniform at 900°C ahead of the shock front.

Fig. 15. Variations of transient axial and hoop stresses in the Coors candle filter for loading case 3 (theoretical).

Fig. 16. Radial variation of the transient hoop stress in the Coors candle filter for loading case 3 (theoretical).

Fig. 17. Variations of transient axial and hoop stresses in the Coors candle filter for loading case 4 (PCFBC Model).

Fig. 18. Radial variation of the transient hoop stress in the Coors candle filter for loading case 4 (Karhula experiment).