#### APPENDIX I

- 1. CHARACTERIZATION OF THE ASH AND CHAR FINES USED IN SIMULATOR TESTING
- 2. CHARACTERIZATION OF ALUMINA/MULLITE FILTERS EXPOSED IN THE WESTINGHOUSE PFBC DURABILITY TEST FACILITY

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#### 1. MORPHOLOGY AND SIZE VARIATION OF PROCESS FINES

The morphology of the ash and char fines which were generated at the Exxon and Grimethorpe pressurized fluidized bed combustion (PFBC) facilities, Kellogg Rust-Westinghouse (KRW) fluid bed gasifier, and Texaco entrained bed gasifier are shown in Figures I.1.1 and I.1.2. All fines with the exception of the Exxon ash were collected by ceramic barrier filter systems. Since the Exxon ash was collected by cyclones, the material was pulverized prior to use in the <u>W</u>-HTHP long-term durability test facility. The small particle size distribution was considered to more closely represent fines that would have been carried over and collected by ceramic barrier filters.

The cenosphere formations shown in Figure I.1.1 are typical of ash formed under combustion gas environments. The 10-20  $\mu$ m cenospheres contain micron and submicron fines which frequently are released during processing. Interdispersed in the Exxon ash are irregularly shaped particles, agglomerates, and infrequently larger (>20  $\mu$ m) particles. As determined by coulter counter analysis, the 50% mass mean diameter of the pulverized Exxon fines and Grimethorpe filter catch fines is 6.1 and 5 $\mu$ m, respectively (Figure I.1.3).

In contrast with the spherical ash formations are the jagged and irregularly shaped char particles formed in the KRW fluidized bed gasifier (Figure I.1.2). Greater than 97% of the rather nondescript, mottled KRW fines are less than 20  $\mu$ m. As identified by coulter counter analysis, the 50% mass mean diameter of the KRW filter catch char is 4  $\mu$ m. Cascade impactor data for fines carried to the filter during testing at KRW were identified to have a drag equivalent diameter of 0.46  $\mu$ m. Unfortunately we are not able to identify whether the coulter counter sample material is representative of the cascade impactor material.

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Exxon



# Grimethorpe



Figure I.1.1 - Scanning Electron Micrographs of the Exxon and Grimethorpe PFBC Ash Fines

KRW



# Texaco



Figure I.1.2 - Scanning Electron Micrographs of the KRW Fluid Bed and Texaco Entrained Bed Gasifier Char Fines.



Figure I.1.3a - Ash and Char Particle Size Distribution.



Figure I.1.3b - Ash and Char Particle Size Distribution.



Figure I.1.3c - Ash and Char Particle Size Distribution.



Figure I.1.3d - Ash and Char Particle Size Distribution.

Unlike the KRW gasifier fines, the char formation which results during entrained bed gasification at Texaco is morphologically similar to the ash fines formed under PFBC conditions. Cenosphere formations and agglomerates can be identified in the Texaco fines, as well as submicron fines and particles which exceed 20  $\mu$ m in diameter. The 50% mass mean diameter of the Texaco filter catch material is 5.3  $\mu$ m. Again the mass mean diameter is based on coulter counter analysis.

## 2. CHARACTERIZATION OF ALUMINA/MULLITE FILTERS EXPOSED IN THE WESTINGHOUSE PFBC DURABILITY TEST FACILITY

Sections from an alumina/mullite filter (WRTX-1) which had been exposed for 366 hours in the Westinghouse HTHP (high temperature, high pressure) durability test facility were removed for characterization. This filter had experienced temperatures of  $815-843^{\circ}C$  ( $1500-1550^{\circ}F$ ), pressures of 50-150 psi, and 1500-4400 ppm loadings of Exxon ash, simulating pressurized fluidized bed combustion conditions (6%  $0_2$ , 73%  $N_2$ , 14%  $H_20$ , 7%  $C0_2$ ). Testing of this filter was terminated when a crack developed horizontally, near the thick mid-ribbed bond support section. An uneven crack line resulted, traversing all of the clean and dirty gas channels.

Sections were also removed from an alumina/mullite filter (WRTX-10) which had been exposed for approximately 1300 hours in the Westinghouse HTHP durability test facility. This filter had experienced temperatures of 815-870°C (1500-1600°F), pressures of 70 psi, and 1000 ppm loadings of dust fines. Exxon ash fines were used during the first 1188 hours of filtration, while Grimethorpe ash was used during the later part of the test. Testing of this filter was terminated when dust outlet loadings exceeded 149 ppm. Although the filter had not delaminated during operation, a break was evident across the entire length of the flange section.

Figures I.2.1 and I.2.2 illustrate the cross-sectional morphology of the alumina/mullite filter matrix near the dirty gas channel surface. Neither accumulation of fines within the surface pores, nor significant penetration of fines which fill or "plug" internal pores or interconnecting channels are evident in either of these filters. Since both filters had been fabricated in the same

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# Westinghouse PFBC Test Facility Filter WRTX-1 - 366 Hours

# SEM



Figure I.2.1 - Scanning Electron Micrograph Illustrating Depth of Fines Penetration Along the Dirty Gas Channel of an Alumina/ Mullite Filter Which Had Experienced 366 Hours of Operation in the Westinghouse PFBC Durability Test Facility

Dirty Gas Channel

# Westinghouse PFBC Test Facility Filter WRTX-10 - 1300 Hours

SEM



Figure I.2.2 - Scanning Electron Micrograph Illustrating Depth of Fines Penetration Along the Dirty Gas Channel of an Alumina/ Mullite Filter Which Had Experienced 1300 Hours of Operation in the Westinghouse PFBC Durability Test Facility filter lot, porosity appears to be relatively similar. Both filters appear to contain a uniform pore size, and do not contain large (i.e.,  $300-500 \ \mu\text{m}$ ) voids. The relatively high conditioned permeability of both filters (WRTX-1 = 27%; WRTX-10 = 27%) reflects the absence of fines and subsequently negligible surface pore closure after 366 or 1300 hours of exposure to simulated PFBC test conditions.

An alternate scanning electron microscope was used to more clearly identify the morphology of the alumina/mullite filter matrix after 366 and 1300 hours of exposure in the simulated PFBC environment, as well as the possible location for adherence of fines. Figure I.2.3 illustrates the cross-sectioned morphology of the 366 hour filter plate, near the dirty channel surface, while Figure I.2.4 illustrates the morphology of the 1300 hour filter plate. Intermittent in both materials are mullite rod-like formations embedded in the "amorphous" phase. Fines in both filter matrices adhere to the "amorphous" pore cavity wall. Pore plugging was not observed in either filter. Fines are identified to contain silicon, calcium, magnesium, and iron.

The actual surface of the alumina/mullite dirty gas channel from the 1300 hour exposed filter was characterized, and is shown in Figure I.2.5. Note the relative abundance of "blunted" crystals which are considered to be the ends of the mullite rods which are embedded in the "amorphous" phase. After 1300 hours in the simulated PFBC environment, the dirty channel surface "amorphous" phase is retained, while mullite appears to be surfacing.

After 1300 hours of exposure in the simulated PFBC test environment, the majority of the edges along the dirty gas filter channels, close to where the Eremco paint is applied at the seams, appear to have been removed. One last analysis was attempted along these regions to identify possible changes in the filter matrix which may have attributed to cracking or erosion of the matrix. The matrix

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Figure I.2.3a - Scanning Electron Micrographs Illustrating the Morphology of the Alumina/Mullite Filter Matrix After 366 Hours of Exposure in the Westinghouse PFBC Durability Test Facility



Figure I.2.3b - Scanning Electron Micrograph Illustrating the Morphology of the Alumina/Mullite Filter Matrix After 366 Hours of Exposure in the Westinghouse PFBC Durability Test Facility



Figure I.2.4a - Scanning Electron Micrographs Illustrating the Morphology of the Alumina/Mullite Filter Matrix After 1300 Hours of Exposure in the Westinghouse PFBC Durability Test Facility





Figure I.2.4b - Scanning Electron Micrographs Illustrating the Morphology of the Alumina/Mullite Filter Matrix After 1300 Hours of Exposure in the Westinghouse PFBC Durability Test Facility



Figure I.2.4c - Scanning Electron Micrograph Illustrating the Morphology of the Alumina/Mullite Filter Matrix After 1300 Hours of Exposure in the Westinghouse PFBC Durability Test Facility



Figure I.2.5 - Surface Morphology of the 1300 Hour PFBC Exposed Alumina/Mullite Filter Dirty Gas Channel

morphology is comparable with that of sections removed from various locations within the filter. At this time we feel that either cracking of the Aremco paint has occurred, pulling with it a portion of the underlying matrix, or that after long term exposure to dust fines, erosion at the sharp corners of the dirty channels may have occurred. Further effort will be necessary to resolve this issue.

Additional characterization of the 1300 hour exposed alumina/mullite filter will include strength testing along both the filter flange, as well as within the webbed section, similar to the work conducted by Argonne on the Texaco exposed filters. Data will be compared with as-fabricated material strength.

## APPENDIX J

### DESCRIPTION OF CROSS FLOW FILTER DURABILITY DURING PFBC SIMULATOR THERMAL TRANSIENT TESTING

#### THERMAL TRANSIENT TESTS

The existing gas-fired HTHP (high temperature, high pressure) pressurized fluidized bed combustor (PFBC) simulator filter test loop was used in this series of tests to demonstrate whether cross flow filter elements can withstand heat-up and cool-down system transients, similar to those projected for a PFBC plant. In the first thermal transient test, four cross flow filters (WRTX-48 and WRTX-53 were fabricated in April 1990, and had been used in prior durability testing; WRTX-66 and WRTX-70 were fabricated in September 1990, and were only used in this segment of testing) were subjected to 900 lb/hr gas flow, 165 psia pressure, and temperatures of 1550°F. Simulated turbine trip transients were accomplished by:

- Turning off the fuel flow to the combustor
- Dropping the pressure from 165 psia to 15 psia
- Increasing the gas flow to 1100 lb/hr.

Cross flow filters WRTX-48 and WRTX-53 acquired a total of 335 hours of testing with 472 pulse cycles, two mild and one severe transient, using Grimethorpe dust<sup>a</sup> (see Appendix B) prior to detection of a 25 ppm dust outlet loading. Alternately cross flow filters WRTX-66 and WRTX-70 acquired a total of 38 hours of testing, 3 pulse cleaning cycles, prior to detection of the dust outlet loading increase. Based on the outlet dust loadings throughout the test, all four cross flow filters were considered to be intact up to the time of the severe

a. Two different Grimethorpe dust compositions were used in the initial turbine trip transient test. The compositional differences of the dust generally include variation in the iron and sorbent content, as related to the processing conditions (i.e., coal feed and sorbent used) at Grimethorpe. In the second turbine trip test, as well as the heat-up transient test, the lower iron content Grimethorpe ash was used. The lower iron content ash is representative of the dust previously used in the long-term durability studies, where 1800 hours of operating life was demonstrated for two cross flow filter elements. These two cross flow filters were fabricated at the vendor site in February 1989. A comparison of the various Grimethorpe dust compositions, and their influence on filter cleaning will be provided in the next Quarterly Report.

transient event. After system shutdown and removal of the filters, all four filters were observed to have developed partial or complete delamination cracks. A description of each filter after testing, including the location of the various cracks or delaminations, and their relation to filter position within the HTHP vessel are provided in Section 1.

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 trip transient, we concluded that the initial four cross flow filters were subjected to more severe conditions than those projected to occur at a PFBC plant (Figure J.1). In an attempt to subject the cross flow filters to a more representative set of test conditions, four new cross flow filters were selected and installed in the Westinghouse HTHP test facility (WRTX-76, WRTX-77, WRTX-78, and WRTX-81; October 1990 fabrication lot). Test conditions again included exposure of the cross flow filters to 900 lb/hr gas flow, 165 psia, and temperatures of 1550°F. In this test turbi sients were simulated by:

- Turning off the low 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 (Appendix E), approximately 11 ppm of the Grimethorpe dust was detected in the outlet gas loading. Inspection of the filters after cool-down of the system indicated that three of the four cross flow filters remained intact, and only one filter (WRTX-81) had developed both a partial and complete delamination crack. A description of the location of these cracks within the filter, as well as the position of the filter within the HTHP vessel are provided in Section 2.



Figure J.1 - Projected Gas Temperature Versus Time Profiles in the Filter Vessel During a Turbine Trip Transient

Once again, after review of the actual gas temperature versus time profile that resulted in the filter vessel during this test, we concluded that the cross flow filters were again subjected to more severe turbine trip transient conditions in the HTHP facility than would be projected to occur for a PFBC plant.

A third test was performed in the HTHP test facility to simulate the filter heat-up cycle that is anticipated at a PFBC plant. The three intact cross flow filters from the second turbine trip transient test, and one new filter (WRTX-80; October fabrication lot) were subjected to 1100 lb/hr gas flow, 69 psia pressure, and temperatures of 950°F. The simulated heat-up transients consisted of ramping the combustor temperature 90 times, such that the inlet temperature rises to 1250°F in 150 sec (Appendix D). Under these conditions, the vessel temperature was expected to reach 1050°F, as expected at a PFBC plant (Figure J.2). After 191 hours of testing, including 90 heat-up cycles, a dust leak of approximately 14 ppm was detected. Inspection of the filters after cool-down indicated that again, the original three filters (WRTX-76, WRTX-77, and WRTX-78) which survived the second turbine trip transient series remained intact during the course of heat-up testing, and only one filter (WRTX-80), which was most recently installed, developed a partial delamination crack. A description of the location of the partial delamination within the filter, as well as the position of the filter within the HTHP vessel are provided in Section 3.

Table J.1 provides an abbreviated summary of these three system transient tests, and the status of each cross flow filter as described above.

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Figure J.2 - Projected Gas Temperature Versus Time Profile in the Filter Vessel During a Startup Transient

## TABLE J.1

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## SUMMARY OF THERMAL TRANSIENT TESTING

Test	Simulation Conditions	Filter Designation Location	Fabrication Lot Date	Comment				
1	Turbine Trip: 2 Mild; 1 Severe	WRTX-48 P1T WRTX-53 P1B	April 90 April 90	Partial Delam Partial Delam				
	Transient; 335 Hrs For WRTX-48 & WRTX-53:	WRTX-70 P2T	Sept 90	Complete Delam Hairline Crack Side Wall Crack				
	38 Hrs For WRTX-70 & WRTX-60	WRTX-66 P2B 8	Sept 90	Horizontal Crack				
2	Turbine Trip:	WRTX-77 P1T	Oct 90	Intact				
	320 Hrs; 10 Transients	WRTX-78 P1B WRTX-81 P2T	Oct 90 Oct 90	Intact 2 Complete Delam				
		WRTX-76 P2B	Oct 90	Intact				
3	Heat-Up Cycles: 191 Hrs; 90 Heat Up Transients	WRTX-77 P1T - WRTX-78 P1B WRTX-80 P2T	Oct 90 Oct 90 Oct 90	Intact Intact Partial Crack				
	· · · · · · · · · · · · · · · · · · ·	WRTX-76 P2B	Oct 90	Intact				

#### 1. SEVERE TURBINE TRIP TRANSIENT TEST

Cross Flow Filter WRTX-48, Figures J.3 and J.4

The cross flow filter designated as WRTX-48 was located in the top holder of plenum #1 during the initial simulated turbine trip transient test. After 335 hours of testing at 1550°F with 472 pulse cycles, 2 mild transients, and 1 severe transient, a dirty gas channel delamination crack occurred in WRTX-48. The delamination crack was located at the 12th dirty gas channel (Figure J.3), farthest from the plenum. The twelfth and thirteenth channels were not identically matched along the mid-rib bond, particularly at the top closed section of the filter element. The fourteenth through eighteenth dirty channels had "near perfect" alignment between mid-rib bonds.

The delamination crack which may have started at the top closed section of the filter appeared to have traveled along the mid-rib bond section of the dirty channels until it reached the thicker central bond section. At that point, the crack cut through the dirty channel wall (not through the thicker central bond section), and ran along the cleanto-clean channel seam for approximately two inches. The delamination crack appeared to have stopped at approximately three dirty channels above the flange area, initially keeping the entire filter body intact. These comments describe the delamination crack that ran along the filter face that was opposite from the baffle plate which separated the two filter plenums (Side A). Due to the thick AREMCO paint along the face of the filter which was closest to the baffle plate, several areas of the crack path could not be followed (Side B).

The delamination crack along Side A was identified to have occurred in the portion of the filter element which contained wider dirty channels. Wider dirty gas channels are considered to represent the top section of the filter plate stack, adjacent to where weights are applied during filter element production. Alternately, the delamination crack along Side B appeared to have occurred in the tighter dirty

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channels. Tighter dirty gas channels are considered to represent the section of the filter which had been at the bottom of the stack of filter plates during firing. This apparent discrepancy between which end of the filter was at the bottom or top during firing, may be the result of an uneven distribution of weight applied during filter element firing.

The location of the delamination crack along the closed top section of the filter, was noticed to be along a slight arc or "bowed" section of the filter faces. Both faces were parallel, but not perfectly flat. In relation to the bolt pattern on the filter clamp, the delamination crack appeared to be located between the mid bolt (No. 4) and the first bolt (No. 5) in the subsequent series of three bolts.

A "rusty-colored" haze was evident along the bottom (underside) of the filter where the clean gas channels were located. The haze appeared to extend along the entire four inch width of the filter (from flange edge-to-flange edge as shown in Figure J.4). The section of the filter that was closer to the plenum appeared to have a "darker" and wider hazy band in comparison to the section of the filter that was located farthest from the plenum. A clean gap was evident between these two bands. A similar hazy band coloration is evidenced on the top filter surface. All of the clean channels appeared to be free of fines.

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Figure J.4 - Dust Track Pattern Along the Clean Gas Channels in Cross Flow Filter WRTX-48

#### Cross Flow Filter WRTX-53, Figures J.5 through J.9

The cross flow filter designated as WRTX-53 was located in the bottom holder of plenum #1. After 335 hours of testing at 1550°F with 482 pulse cycles, 2 mild transients, and 1 severe transient, a dirty gas channel delamination crack which ran completely through the filter, and a partial dirty gas channel delamination crack resulted (Figure J.5).

Initially an open seam had been identified in the top closed section of the filter prior to use, and was patched with Nextel cloth and AREMCO paint. After testing, sections of the cloth were no longer attached to the filter, and debonding along the dirty gas channel midribs occurred, resulting in the formation of a partial delamination (Figure J.6). The partial delamination or crack appeared to have started along the top closed section of the filter in dirty gas channel No. 14. The crack extended through the dirty channel mid-rib bonds in the upper section of the filter. The last dirty channel above the thicker central rib was the location where the crack cut through the plate, and then ran down the clean-to-clean channel seam. The crack did not appear to travel completely through the flange section. The crack path was evident along the opposite face of the filter which had been adjacent to the baffle plate. The location of this crack appeared to be at bolt No. 5. of the clamp. The patched crack appeared to be located in the tighter dirty gas channel portion of the filter. This section was considered to have been at the bottom of the stacked plates during firing and manufacturing of the filter body. Neither the partial delamination crack nor fines were evident along the bottom (underside) of the filter flange (Figure J.7).

A complete delamination crack was evident along the 9th dirty channel (Figure J.8). Initially the mid-rib bonds in this channel were well matched and bonded. The delamination was slightly to the left of the central bolt (No. 4). Caked fines were readily evident along the delaminated dirty gas channel. The mid-ribs along this section were also covered with a "greyish" perhaps "wet" mat of fines.

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Figure J.5 - Location Of Delamination Cracks In Filter WRTX-53

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Figure J.6 - Patched Cracked Area of Cross Flow Filter WRTX-53 After Durability and Transient Testing



Figure J.7 - Absence of Dust Along the Clean Gas Channel Surface of Cross Flow Filter WRTX-53



Figure J.8 - Debonding of the Mid-Rib Bonds in Cross Flow Filter WRTX-53


Figure J.9 - Dust Track Patterns Along the Poorly Bonded Section Iennad) as Util tasi end woled egnali retif end 10

### Cross Flow Filter WRTX-66, Figures J.10 through J.13

The cross flow filter designated as WRTX-66 was located in the bottom holder of plenum #2. After 38 hours of testing at 1550°F with 3 pulse cycles, 2 mild transients, and 1 severe transient, a complete horizontal crack was observed through the upper section of the filter body (Figure J.10). The crack appeared to have originated along the side of the filter that was directly opposite from the plenum. Again this section of the filter contained the tighter dirty gas channels, indicating the side of the filter which had been at the bottom of the stack of plates during firing and production of the filter element body.

The horizontal crack ran from the bottom of the 5th dirty gas channel located at the side of the filter that was opposite of the plenum, to the top of the 5th dirty gas channel located at the side of the filter that was closest to the plenum.

Fines were not generally observed in the clean gas channels along the horizontal crack, except possibly in the last two clean channels (No. 17 and No. 18) farthest from the plenum (Figure J.11). Along the mid section of the horizontal crack, the clean channels appear to be free of fines. There was no apparent evidence of fines traveling to the bottom of the clean gas channels at the base of the flange (Figure J.12).

No reference is made to the position of the bolt holes, since the crack occurred horizontally. However, it should be noted that this filter was mounted with the redesigned clamping arrangement (Figure J.13).



Dirty Channel Column Number

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Figure J.11 - Cracked Surface Of Cross Flow Filter WRTX-66



Figure J.12 - Absence Of Dust Along the Clean Gas Channel Surface Of Cross Flow Filter WRTX-66



Figure J.13 - Redesigned Clamping Arrangement

### Cross Flow Filter WRTX-70, Figure J.14

The cross flow filter designated as WRTX-70 was located in the top holder of plenum.<sup>b</sup> After 38 hours of testing at 1550°F with 3 pulse cycles, 2 mild transients, and 1 severe transient, a hairline crack was evident along the bottom (underside) of the flange, as well as along the closed side wall plate of the filter (Figure 14). The crack appeared to have originated in the 6th clean channel from the plenum side. The crack cut through the plate, and ran through the clean-toclean mid-rib bond seam. The crack extended through the flange section and then crossed through the last plate of the dirty gas channel which was adjacent to the flange, and then into the center of the dirty gas channel. The crack path appeared to have stopped at this location. After 38 hours of testing, the clean gas channels appeared to be free of fines.

In relation to the bolt pattern, the crack appeared to have occurred at approximately the same location as bolt No. 3 of the filter clamp. As with cross flow filter WRTX-66, WRTX-70 was also mounted with the redesigned clamping arrangement (as Figure J.13).

A second minor crack formation was also evident along the closed side wall plate of this filter. The location of this crack is again at the 5th row of dirty gas channels, similar to the position of the horizontal crack which formed in cross flow filter WRTX-66. Unlike WRTX-66, the crack in WRTX-70 did not "cut" completely through the filter matrix.

Similar to the other three filter in this test series (WRTX-48, WRTX-53, and WRTX-66), the tighter dirty gas channels of filter WRTX-70 were located farthest from the plenum.

b. WRTX-70 was subsequently used at Westinghouse for optical sensor testing by VPI personnel. Sensors and bonding media are shown in the top portion of Figure J.14.



Dirty Channel Column Number





### 2. MODERATE TURBINE TRIP TRANSIENT TEST

Cross Flow Filter WRTX-81, Figures J.15 through J.17

The cross flow filter designated as WRTX-81 was located in the top holder of plenum #2. In contrast with the two previous cross flow filter fabrication lots which had 18 columns of dirty gas channels, WRTX-81 had only 17 columns of dirty gas channels. After 320 hours of transient testing, a complete delamination resulted along the mid-rib bond of dirty gas channel No. 5, as well as a partial delamination crack along dirty gas channel No. 15 (Figures J.15-J.17). After testing, the delamination crack located at dirty gas channel No. 15 was seen to be partially held together at the flange. The delamination crack was observed to diverge at the top closed section of the filter, leaving an approximate 1/8 inch gap between filter plates. Debonding occurred primarily along the mid-rib bonds. Along the top closed section, as well as along the flange area, a segment of the filter plate material was also removed (i.e., as opposed to merely debonding along the seam). A "better" bond is speculated to have been formed in these sections (i.e., top closed section and flange area) in comparison to the midribbed bonds. Fines were observed along the delaminated mid-ribbed bonds after testing.

No visible identification could be made relative to which side of the filter had been weighted during filter fabrication. What was evident, however, was that the position of delamination along dirty gas channel No. 15 corresponded to the position of bolt No. 3. The delamination crack which formed along dirty gas channel No. 15 was located between bolt No. 5 and No. 6.

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Figure J.15 - Delamination Along Dirty Gas Channel Column No. 5 Which Resulted in Cross Flow Filter WRTX-81 after Turbine Trip Transient Testing



Figure J.16 - Delamination Along Dirty Gas Channel Column No. 15 Which Resulted in Cross Flow Filter WRTX-81 After Turbine Trip Transient Testing

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Figure J.17 - Location of Delamination Cracks in Filter WRTX-81

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### 3. HEAT-UP CYCLE TRANSIENT TEST

Cross Flow Filter WRTX-80, Figure J.18

The cross flow filter designated as WRTX-80 was located in top holder of plenum #2 during the heat-up transient tests. After 191 hours of testing, including 90 heat-up cycles, a dust leak of approximately 14 ppm was detected. An initial inspection of the filter as it remained mounted in the filter vessel indicated that an obvious "dust seam' formed along the filter face. The "dust seam" appeared to run along the clean channel seam, between dirty gas channel No. 3 and No. 4. Further inspection of WRTX-80 after removal from the vessel and subsequent cleaning, indicated that a crack had formed in the filter flange, in approximately the same area as the clean plate "dust seam" (Figure J.18). The crack appeared to form within a filter plate (not on a clean or dirty gas channel seam), which then crossed over into the clean gas channel seam, near the last row of dirty gas channels. The distance that the crack traveled from the flange towards the top closed section of the filter could not be discerned due to the relatively wide "smile" formations along the plate edges of the clean gas channel seam.

The crack had initiated in the area of the filter that was mounted closest to the plenum, and was located between bolt No. 2 and No. 3. An attempt was made to try and identify which end of the filter was located at the top or bottom of the stack of filter plates during firing. We believe that the tighter gas channels, indicative of the bottom set of plates in the stack, were positioned opposite of the plenum.

When viewing the filter along the open clean gas channel surface, the crack was observed to run through the plate (not between clean or dirty gas channel seams), and then into the "outer-most" clean gas channel. The crack did not appear to travel completely across the

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clean gas channel seam that runs along the underside of the filter. The crack was, however, apparent between the opposite "outer-most" clean gas channel and the edge of the flange. Dust fines were evident along the two "outer-most" clean gas channels that contained the crack formations. COMMENT

In the thermal transient test series, Westinghouse has attempted to subject cross flow filters to conditions that may represent turbine trip and heat-up cycles similar to what may be expected in a PFBC plant. The thermal transient conditions are generally considered to be more severe than the "steady state" conditions achieved in our durability test program, where our major accomplishment was the successful demonstration of approximately 1300 hours of operational life for two cross flow filter elements (WRTX-9 and WRTX-10).

All filters that were used in the thermal transient tests were fabricated with the same type of pore former, and were considered to have experienced the same filter plate stacking assembly and firing temperature profile. WRTX-48 and WRTX-53 were produced in April 1990, while WRTX-66 and WRTX-70 were produced in September 1990. Both WRTX-80 and WRTX-81 were produced in October 1990. The April 1990 batch lot was identified to have a strength of 3.204 ksi (4-pt bend), while the September and October 1990 batch lots were identified to have strengths ranging from 2.99-3.07 ksi (4-pt bend).

In contrast with these filters are the durability test filters, WRTX-9 and WRTX-10. These filters which were fabricated in February 1989, were manufactured using a different pore former,<sup>c</sup> as well as filter plate stacking sequence during production. The February 1989 fabrication lot had an apparently higher initial material strength, as reflected by the vendor's 3-pt bend strength data of 4.06 ksi<sup>d</sup> (ANL reported a 4-pt bend strength of 3.11 ksi for a filter element from this production batch).<sup>(1)</sup>

c. The different pore formers used in fabricating the cross flow filters may have somewhat changed the porosity and pore size distribution within the ceramic matrices. This is considered as a result of possibly different initial pore former particle size distributions, and possibly the different "burn-out" patterns of the pore formers, which could lead to changes in the interconnecting pore channel geometry.

d. Caution should be exercised when attempting to compare 8-pt and 4-pt bend strength data. These values are not expected to be directly related.

The fact that the durability cross flow filters may have had an initially higher material strength, and were subjected to less severe "steady state" conditions, are perhaps criteria which directly relate to their 1300 hours of operating life in comparison to the 38 to 335 hours of filter life achieved under thermal transient test conditions. Further efforts should be directed to verifying the extent of possible variation in material strength by testing at least one as-fabricated, untested filter from each filter lot, using the same bend bar test geometry (i.e., 4-pt bend, 1/4 flexure). Similarly, data are also needed which reflect mid-rib and plate seam bond strength for both filter lots, as well as fracture toughness, porosity, pore size distribution, density, and phase composition. This information may provide insight into some of the currently considered reliability aspects of the cross flow filter from a production point of view.

The possibility also exists that the HTHP test facility may artificially induce localized stresses as a result of either system operation or design modifications. Minor modifications that were made either to the HTHP system (i.e., clamping design, blowback system, dust type) or to the individual cross flow filter elements (i.e., AREMCO painting of the seams) during the last long-term durability test and the thermal transient test series are summarized in Table J.2.

With respect to the Westinghouse HTHP filter vessel, several observations have been identified in an attempt to determine why cross flow filters delaminate or form cracks at what seems to be reoccurring locations. These include:

> • The most severe failures in the initial turbine transient test (four filters) occurred in the filters located in the bottom holders (WRTX-53: complete delamination and delamination crack; WRTX-66: complete horizontal delamination). This may be related to a thermal effect (i.e., either the pulse gas is colder at this location, and

> > J-35

# TABLE J.2

	Filter No.	Maximum Life,Hrs	No. Plenums/ No. Filters	Type of Ash Feed	Plate Seams	Flange Clamping	Blowback	Filter Plenum Positions
Durability Testing	WRTX-9 WRTX-10	~1300 ~1300	1/2	d	e	Original	Original	P2B P2T
Severe Thermal Transient Test No. 1	WRTX-48 WRTX-53 WRTX-70 WRTX-66	335 335 38 38	2/4	b b b,c b,c	e e As-Fabricated As-Fabricated	Original Original Rocker Rocker	Segmented Segmented Original Original	P1T P1B P2T P2B
Thermal Transient Test No. 2	WRTX-77 WRTX-78 WRTX-81 WRTX-76	a(320)* a(320)* 320 a(320)*	2/4	ପ ଏ ଏ ଏ	As-Fabricated As-Fabricated As-Fabricated As-Fabricated	Rocker Rocker Rocker Rocker	Original Original Original Original	P1T P1B P2T P2B
Thermal Transient Test No. 3	WRTX-77 WRTX-78 WRTX-80 WRTX-76	a(191)* a(191)* 191 a(191)*	2/4	d d d d	As-Fabricated As-Fabricated As-Fabricated As-Fabricated	Rocker Rocker Rocker Rocker	Original Original Original Original	P1T P1B P2T P2B

### SUMMARY OF W HTHP SYSTEM MODIFICATIONS

a - Intact

\* - Hours during specific test

b - Mixture of both high and low iron content Grimethorpe dust
c - Majority of operational hours with high iron content Grimethorpe dust
d - Low iron content Grimethorpe dust
e - AREMCO paint along gas channel seams

is directed to the clean gas channels farthest from the plenum, or is a combination of cold pulse gases within perhaps a hotter section of the filter vessel).

- Crack formation in the initial series of thermal transient tests appeared to result in the area of the cross flow filter farthest from the plenum. The question which arises is whether this has any relationship to either the HTHP system design (i.e., gas flow pattern, etc.), or to its operation. Perhaps the plenum provides "protection" which is not afforded to the side of the filter that is directly in the gas passage.
- In the initial thermal transient test series, the tighter channels of each filter appeared to be positioned at the location farthest from the plenum. The tighter channels represent the bottom set of plates during weighting and high firing of each filter element. This area may have experienced a higher stress under load, and therefore may be more vulnerable to failure.
- Bolt patterns may still induce crack formation, particularly in bolt positions No. 3 and No. 5, and possibly No. 4.
   Perhaps this region may have experienced the "coldest" portion of the pulse gas during blowback.

Although in the first turbine trip transient test, the relative strengths of the Plenum #2 (top: WRTX-70; bottom: WRTX-66) filters in the initial thermal transient test series were somewhat lower than the Plenum #1 (top: WRTX-48; bottom: WRTX-53) filters, these filters had not experienced the extensive number of hours of testing as the filters located in Plenum #1. Perhaps, during initial testing, the filters in Plenum #1 had experienced a reduction in strength, leaving them with a resulting strength comparable to the filters in Plenum #2. If this were so, then due to the severity of the transient, both sets of filters would be expected to perform in a similar manner, forming cracking and delaminating. Alternately the "severity" of the initial turbine trip test may have been such that even the "strongest, most reliable" ceramic cross flow filter would delaminate or form cracks.

The more moderate turbine trip transient test conducted in this series certainly demonstrated the long-term "survivability" of the cross flow filters to "non-steady state" conditions, since only one filter failed under the more representative transient test conditions, as opposed to all four filters suffering a partial or complete delamination crack under the "less realistic" severe set of transient conditions. Questions which arise are:

- If the October 1990 filter lot had an initially higher material strength, would filter life for WRTX-81 have increased?
- Was WRTX-81 "perfectly" fabricated?

Since, however, three out of the four filters survived the moderate turbine trip test, and were subsequently subjected to heat-up cycle transients and survived an additional 191 hours of testing, the question which then arises is whether there is an issue with the HTHP system which induces failure in a filter that is placed in the top holder of the second plenum. Alternate systems issues which may provide insight into cross flow reliability include:

> • Identifying whether similar blowback pulse durations were used in the durability and thermal transient test series, as well as tracking the time interval between pulsing.

• Identifying the change in temperature throughout an entire filter during blow back pulse delivery. Identifying whether each filter element experiences the same temperature profile per pulse.

These and other fabrication and systems issues will continued to be reviewed during the next quarter. A Taguchi approach is planned to assess the relevance of all possible fabrication and HTHP system contributing parameters.

### REFERENCES

 Singh, J. P., Majumdar, S., Wagh, A. S., Wenzel, T., and Peoppel, R. B., "Materials Qualification Technology For Ceramic Cross Flow Filters," Argonne National Laboratory, to be issued.

# APPENDIX K

# PROPERTY CHARACTERIZATION - ROOM TEMPERATURE

### FILTER MATERIAL PROPERTY CHARACTERIZATION

Strength data are presented for an as-fabricated, untested alumina/mullite cross flow filter, and for the filter which experienced 1300 hours of operation in the Westinghouse high temperature, high pressure (HTHP) test facility. Under simulated pressurized fluidizedbed combustion (PFBC) conditions, the 1300 hour cross flow filter experienced temperatures of 1600°F, pressures of 150 psig, and 2068 pulse cleaning cycles. During filter operation, inlet mass loadings were 1000 ppm, while outlet mass loadings of 1 ppm were achieved. Testing was terminated when the filter experienced longitudinal cracks in the flange used to attach the filter to the clean gas plenum. Both filters were produced from the same fabrication lot in February 1989. Bend bar samples were prepared from the top (closed) and web (core) of both filters. Bend bars were also prepared from the flange section of the 1300 hour filter. Since the flange section of the as-fabricated, untested filter had been removed prior to shipment by the manufacturer, it was not available for strength analysis.

### 1. MATERIAL PROPERTY CHARACTERIZATION TECHNIQUES

Room temperature strength testing was performed on the alumina/mullite filter that had been exposed for approximately 1300 hours in the Westinghouse HTHP test facility in an attempt to determine whether changes had occurred in the physical properties of the filter during long-term operation. Four point bend strength testing was conducted at room temperature using 1/4-point flexure, with upper and lower spans of 20 and 40 mm, respectively. The crosshead speed was 0.02 cm/min.

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Initially the 1300 hour cross flow filter was divided into four sections (Figure K.1). From each 6x6x4 inch web (core) section, twelve bend bars (48 in total that were 0.08 in. thick) were prepared for room temperature strength testing (4-point bend, 1/4-flexure). From the flange section of the filter, twenty-two 0.08 inch thick, and fifteen 0.12 inch thick bend bars were prepared. From the top (closed) section of the filter, twenty-six 0.08 inch thick, and twenty-three 0.12 inch thick bend bars were prepared.

In an attempt to identify whether a change in material strength has resulted after 1300 hours of filter operation, an as-received, untested filter from the same fabrication lot was sectioned for room temperature strength characterization. Thirty-three 0.08 inch thick bend bars were prepared from the top (closed) section of the filter, while thirty 0.08 inch thick bend bars were prepared from the web (core) section. Since the flange section of the as-received, untested filter had previously been removed by the manufacturer, it was not available for strength analysis. Figure K.2 identifies the location where bend bars were removed from the filters at Westinghouse.

In addition, an attempt was made to determine whether bend bar thickness had an effect on the resulting strength data. This issue was raised by ANL in a similar effort which characterized an as-fabricated alumina/mullite filter, and a filter which had operated for 77 hours in the Texaco entrained-bed gasification facility.<sup>(1)</sup> In the ANL effort, the flange material was actually a combination of samples which had been removed from the flange, as well as from the top (closed) section of the filter. ANL detected an apparent difference in strength between bend bars removed from the flange areas and the web (core) section. Even though the flange samples were thicker than the web (core) samples, the flange samples were identified to have a higher strength in comparison to the web (core) samples. The thicker the test sample is, the larger the volume of material under stress. Normally, the larger the volume of

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Dwg. 9418A81



Figure K.1 - Quadrant Sections Used at Westinghouse for Determining Material Strength of the 1300 Hour Filter



Parallel to

Dirty Gas Channels



Stack Plates (b) Flange Section Bend Bars Were Removed

Transverse to Stacked Plates



Figure K.2 - Bend Bar Locations for Material Strength Characterization at Westinghouse

sample under test, the lower the resulting strength, since the flaw population is also considered to increase, and therefore the probability of larger flaws is increased. Figure K.3 identifies the location where bend bars were removed from the filters at ANL.

### 2. STATISTICAL ANALYSES

Analysis of variance (ANOVA) techniques were used to determine if the strength differences observed between bend bar samples taken from different filter locations, or the differences that were observed between the 1300 hour tested filter and the as-fabricated, untested filter material were larger than what would be expected from chance or random variation. If the differences do not result from random variation, then there is an effect of the tested variable (i.e., either filter location or filter condition) on the filter material strength. The strength distribution for the given variable (i.e., filter location or filter condition) then is representative of the strength of the material for that variable.

The Weibull statistical fracture theory is widely used in ceramic material applications. This theory predicts that the strength of a ceramic material decreases with increasing sample size (i.e., area). The most used parameter of this theory is the Weibull modulus (m), which is an index of ceramic material strength reproducibility. The higher the m value, the more reliable (i.e., predictable) the material is, since there is a lower scatter in fracture strength.

An important consideration in determining the Weibull modulus is the sample size (i.e., number). The uncertainty in the m value decreases, as sample size increases. This uncertainty, or error, is shown in Figure K.4<sup>(2)</sup> as a function of sample size for a 90 percent confidence interval. For example, a sample size of 40 gives an approximate error of  $\pm 22\%$  for the calculated Weibull modulus.

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- (b) Flange Section Bend Bars Were Removed Transverse to Stacked Plates.
- Note: No Differentiation is Made Between Bend Bars Removed from the Top Closed Section and the Bottom Flange Area

Figure K.3 - Bend Bar Locations for Material Strength Characterization at ANL



Figure K.4 - Weibull Slope Error Versus Sample Size

### 3. RESULTS AND DISCUSSION

Table K.1 summarizes the Westinghouse room temperature 4-point bend strength and Weibull modulus results for the as-fabricated filter material and the 1300 hour HTHP PFBC exposed filter. The strength data for all 198 bend bars are presented at the back of this appendix beginning with Table K.8. The average bend bar height and width are also shown in Table K.1. The length of the bend bar under test is the same as the lower span length (1.57 inches) of the test fixture, while the actual bend bar length used in this effort was 1.8 inches. The sample size was kept as consistent as possible to minimize the effect of sample volume variation on material strength. The height of the bend bars was limited to the thickness of the web (core) section material. For consistency, the top (closed) and flange section samples were machined to approximately the same thickness as in the web (core). Two sets of bend bars were prepared from the 1300 hour filter with a larger thickness, but a smaller width. The material strength results that are shown in Table K.1, as well as conclusions generated in the ANOVA analyses are discussed in the following sections.

# 3.1 As-Fabricated Filter Material

Table K.1 indicates that there is a difference in material strength between the top (closed) and web (core) sections of the asfabricated, untested filter. The ANOVA results presented in Table K.2 indicate that the strength difference is based on where the bend bars are taken from (i.e., either the top (closed) or web (core) section), and that this difference is not a result of random variation. The strength distributions from the top (closed) and web (core) sections are therefore considered to be significantly different, and are not from the same parent distribution. This is shown more clearly by the 95 percent confidence intervals for the top (closed) and web (core) section group means in the "Group Summaries" portion of Table K.2.

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# TABLE K.1

### MATERIAL STRENGTH AND WEIBULL VALUES FOR THE AS-FABRICATED UNTESTED ALUMINA/MULLITE FILTER AND THE 1300 HOUR HTHP PFBC TESTED FILTER

As-Fabricated	1300 Ho	our Filter
<u>Top</u> * 33	<u>Тор</u> 26	<u>Top</u> ** 23
3529 <u>+</u> 222	2666 <u>+</u> 168	2653 <u>+</u> 220
19.2	19.1	14.3
0.082 <u>+</u> 0.005	0.083 <u>+</u> 0.003	0.120 <u>+</u> 0.001
$0.190 \pm 0.005$	0.200+0.002	0.160 <u>+</u> 0.005
$\frac{\text{Web}^{*}}{30}$		
3148+368	2377+354	
82	7 0	
0.073+0.008	$0.070 \pm 0.003$	
0.196+0.004	0.198+0.005	
	Flange 22	$\frac{\text{Flange}}{15}^{**}$
	2354+169	2495+258
	16.2	11.4
	0.078+0.003	0.115+0.006
	$0.197\pm0.001$	$0.160\pm0.001$
	$\frac{T_{op}^{*}}{33}$ 3529+222 19.2 0.082+0.005 0.190+0.005 $\frac{Web^{*}}{30}$ 3148+368 8.2 0.073+0.008 0.196+0.004	As-Fabricated1300 Ho $\frac{Top}{33}$ $\frac{Top}{26}$ $3529+222$ $2666+168$ $19.2$ $19.1$ $0.082+0.005$ $0.083+0.003$ $0.190\pm0.005$ $0.200\pm0.002$ $\frac{Web}{30}^*$ $\frac{Web}{48}$ $3148+368$ $2377\pm354$ $8.2$ $7.0$ $0.073\pm0.008$ $0.070\pm0.003$ $0.196\pm0.004$ $0.198\pm0.005$ $\frac{Flange}{22}$ $2354\pm169$ $16.2$ $0.078\pm0.003$ $0.197\pm0.003$

\* Top (Closed Section); Web (Core). \*\* Thicker Bend Bars.

### TABLE K.2

# ANOVA AND GROUP SUMMARIES COMPARISON OF MATERIAL STRENGTH FOR THE TOP AND WEB SECTIONS OF THE AS-FABRICATED UNTESTED FILTER

### ANALYSIS OF VARIANCE TABLE

	DF	SS	MS=SS/DF	F	VALUE	SIG	LEVEL
FACTOR ERROR TOTAL	1 61 62	2273053.111688 5499273.745455 7772326.857143	2273053.111688 90152.028614		25.21		0

The null hypothesis that the samples come from populations with equal means can be rejected (p = 0).

Group Summaries

Group N Mean Stdev Name artop 33 3528.727273 222.112341 arweb 30 3148.400000 367.685642

Group 3011.1 3607.48 Name +-----X-----+ artop -----X------

Error bars represent non-simultaneous 95 percent confidence intervals for the group means.

The resulting difference in material strength is considered to be related to the critical flaw size in the porous ceramic matrix. Critical flaws are most likely considered to result from large pores or interconnected porosity in the alumina/mullite matrix. Consequently, the strength differences between the as-fabricated untested filter top (closed) and web (core) sections are probably related to differences in the average pore size, and the pore size distribution of each section.

As shown in Table K.1, the Weibull modulus for the top (closed) section is more than twice that of the web (core) section. This indicates that there is a larger degree of scatter in the web (core) section strength data, and therefore less predictability (i.e., lower reliability) in the results. Weibull values greater than 10 are considered to be very good for ceramic materials. Weibull plots for all materials analyzed are presented in Appendix C.

### 3.2 Long-Term Durability Filter

Material strength and Weibull modulus comparisons were made for the HTHP PFBC 1300 hour filter based on sample location and thickness. These data are also presented in Table K.1. Similar to the asfabricated untested cross flow filter, the top (closed) section of the HTHP PFBC 1300 hour filter appears to be stronger than its web (core) section. The flange section of the 1300 hour filter, however, appears to have a resulting strength that is similar to that of its web (core) section. Note that the apparent lower material strength in the 1300 hour filter flange area, tends to coincide with the location of filter failure.

The ANOVA results shown in Table K.3 for top (closed), web (core), and flange sections indicates that a difference in strength results depending on where the bend bar is taken from, and that this difference is not a result of random variation. The "Group Summaries" portion of Table 3 indicates that the web (core) and flange sections have similar means and 95 percent confidence intervals, whereas, the top

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# ANOVA AND GROUP SUMMARIES COMPARISON OF MATERIAL STRENGTH FOR THE TOP, WEB, AND FLANGE SECTIONS OF THE HTHP PFBC 1300 HOUR FILTER

# ANALYSIS OF VARIANCE TABLE

	עני	SS	MS=SS/DF	ч	VALUE	SIG	LEVEL
 FACTOR ERR^R TOTAL	9 9 5	 1677111.042249 7203546.916084 8880657.958333	838555.521125 77457.493721	1	10.83	5               	0
The null equal mea	hypo ans c	thesis that the an be rejected (	samples come frc p = 0).	B	populat	ions	with

Group Summaries

Group Name Flange Top	N 262	<b>Mean</b> 2354.545455 2666.538462	Stdev 169.317602 167.816622	
Group	2273	.89		2734.3
Flange Top Web		><		XX
Error ba interva	ars rolls for	epresent non-: r the group me	simultaneous ( eans.	95 percent confiden

(closed) section mean and confidence interval differs significantly from the web (core) and flange sections. Figure K.5 graphically illustrates a comparison of the three filter sections.

From the web (core) section, an equal number of bend bars were prepared with tensile surfaces from the dirty and clean gas channels. This was done in an attempt to determine if the strength of the dirty channel surfaces was affected by direct contact with ash fines during the 1300 hours of filter operation. Most of the strength test bend bar failures were seen to be initiated at or near the outer surface, which is in tension. Table K.4 shows the ANOVA results and the "Group Summaries" data for the dirty and clean surfaces in the web (core) section. Since there are no apparent differences between the dirty (2381 • 320 psi) and clean (2373 • 392 psi) channel surfaces, these data are grouped together simply as web (core) section data.

The Weibull moduli for the top (closed), web (core), and flange sections are shown in Table K.1. The top (closed) and flange sections have high m values (19.1 and 16.2, respectively). The web (core) section has an m value of 7.0, which indicates considerably more scatter in the web (core) fracture strength values in comparison to the top (closed) and flange sections.

As previously discussed, the ANL data indicated that the thicker bend bar samples from the flange and top (closed) sections were as a group, stronger than the thinner samples from the web (core) section. ANL proposed that due to the high porosity of the alumina/mullite material, the large flaws, if present in the thin web (core) section, may lower the resulting material strength. This would not be expected if the web (core) bend bars were prepared with a thickness that was comparable to that of the thicker flange and top (closed) sections. A similar effort was undertaken at Westinghouse using the 1300 hour alumina/mullite filter.



Figure K.5 - Simultaneous Comparisons Between Groups
# ANOVA AND GROUP SUMMARIES COMPARISON OF MATERIAL STRENGTH FOR DIRTY AND CLEAN SIDE SURFACES IN THE WEB SECTION OF THE 1300 HOUR HTHP PFBC FILTER

#### ANALYSIS OF VARIANCE TABLE

	DF	SS		MS=S	S/DF		F VA	LUE	SIG	LEVE	EL
FACTOR	1	736.	333333	7	36.333	333	0	.01		0.9	4
ERROR TOTAL	46 47	5896712. 5897449.	868667 000000	1281	89.405	797					
There is	. insu	fficient	evidence	to	reject	the	null	hvp	othes	is t	ha.

incre is insufficient evidence to reject the null hypothesis that samples come from populations with equal means (p = 0.94).

Group Summaries

Group	N	Mean	Stdev	
Name clean dirty	24 24	2372.833333 2380.666667	392.052755 320.426980	
Group	2207	. 28		2538.38
Name clean dirty	+		·>< ·><	+

Error bars represent non-simultaneous 95 percent confidence intervals for the group means.

The intent of preparing the additional 4-point bend samples from the flange and top (closed) sections of the 1300 hour filter was that these samples would be 50 percent thicker than the web (core) bend bar thickness, but they would maintain the same width. These samples would therefore have a 50 percent larger volume than the original bend bar samples, and they would be expected to show a lower average 4-point bend strength as a result of the increased volume of material under test. However, the thicker bend bar samples were inadvertently prepared with a smaller width which resulted in only a slight increase in sample volume under test. As such, no difference in strength would be expected from the thicker samples when compared to the normal height samples. This conclusion was verified by the results in Table K.1. The flange and top (closed) section 4-point bend strength results were therefore not affected by test sample thickness for similar volume samples.

The Weibull w values do, however, indicate differences between the "thinner" and "thicker" flange and top (closed) section bend bars. The Weibull m value for the thicker bend bar samples are generally less than those of the thinner bend bars. These differences may be attributed to the large errors (Figure K.4) that could result when using relatively small sample sizes.

# 3.3 Comparison of the As-Fabricated and Long-Term Durability Filter Materials

Since flange data are not available for the as-fabricated, untested alumina/mullite filter material, a comparison will be made only between the top (closed) and web (core) sections of the as-fabricated and 1300 hour filter materials. This comparison will not include the thicker bend bar sample data.

There is an apparent 24 percent decrease in strength between the as-fabricated, untested alumina/mullite filter matrix and the matrix material after 1300 hours of operation in the Westinghouse HTHP PFBC

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test facility. This decrease is consistent for both the top (closed) and web (core) sections of the filters. Because the strength differences between the as-fabricated, untested and 1300 hour filter are large, an ANOVA is not required to confirm that the decrease in strength is related to 1300 hours of filter operation. However, an ANOVA was performed using the as-fabricated, untested and 1300 hour filter web (core) section data. These results along with the "Group Summaries" are shown in Table K.5.

There is no apparent change in the Weibull m values for the asfabricated, untested and 1300 hour filter top (closed) and web (core) sections. The m values for the top (closed) sections of the asfabricated, untested and 1300 hour filters are 19.2 and 19.1, respectively, and for the web (core) sections, 8.2 and 7.0, respectively. The web (core) section continues to have a much higher degree of scatter in the material strength values.

In contrast with the Westinghouse as-fabricated, untested and the 1300 hour filter web (core) Weibull modulus data are the ANL data. The ANL data shown a definite change in the flaw population of the 77 hour Texaco tested web (core) material in comparison with the original material (14 versus 9, respectively). Further efforts are needed to generate a substantially larger material's properties data base, prior to concluding whether the variation between the Westinghouse and ANL Weibull modulus data reflect filter material variation within a given fabrication lot, or simply procedural and evaluation techniques.

If we assume that initially the strength of the 1300 hour filter flange was equivalent to that of its top (closed) section (i.e., 3529 psi) as in the ANL effort, then an apparent 33 percent decrease (i.e., 3529 to 2354 psi) in the flange material strength is expected to have occurred during the 1300 hours of filter operation. In the current filter failure mechanism, a reduction in material strength (maximum

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# ANOVA AND GROUP SUMMARIES COMPARISON OF MATERIAL STRENGTH FOR THE AS-FABRICATED UNTESTED AND 1300 HOUR FILTER WEB SECTIONS

#### ANALYSIS OF VARIANCE TABLE

FACTOR	1	10992807.184615	10992807.184615	85.09
ERROR	76	9818038.200000	129184.713158	
TOTAL	77	20810845.384615		

SIG LEVEL

Group N Mean Stdev

	-	-	-	-		-	-	-	-	-	-	-	-	-	-	-	-
F	A	C	T	0	R												0
E	R	R	0	R													
T	0	Т	A	L													

The null hypothesis that the samples come from populations with equal means can be rejected (p = 0).

Group Summaries

Name				
Web	48	2376.75	354.228229	
arweb	30	3148.40	367.685642	
Group	2273	. 89		3285.7
Name	+	~~~~~~		+
Web		><		
arweb				><

Error bars represent non-simultaneous 95 percent confidence intervals for the group means.

limit to be determined) coincides with failure in the ceramic component. Therefore over time, the entire filter body may lose strength, and when the maximum change in material strength results or a lower tolerance limit is achieved, failure occurs.

3.4 Summary

Table K.6 summarizes the room temperature strength data generated at Westinghouse for the as-fabricated, untested and 1300 hour filter materials. These data indicate that:

- As expected, for a constant volume of material, bend bar thickness does not influence the resulting room temperature strength.
- The top (closed) section from both the as-fabricated, untested and 1300 hour filters appears to be stronger than the web (core) section.
- Material strength in the web (core) and flange sections of the 1300 hour filter appears to be nearly equivalent.
- An apparent 24 percent loss in material strength results after 1300 hours of exposure to PFBC ash at 1600°F (870°C), 150 psig, and 2068 pulse cleaning cycles.

The loss of material strength data reflects only an apparent change in the properties of the filter plates, or within the thicker top or flange sections. These data do not reflect any change in bond strength which may have resulted along the mid-ribbed bond.

The apparent loss of material strength after 1300 hours of exposure in the HTHP PFBC test rig is considered to have resulted from the thermal cycling (and/or vibration) which the filter experienced

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#### ROOM TEMPERATURE STRENGTH DATA FOR THE 1300 HOUR WESTINGHOUSE PFBC HTHP FILTER MATERIALS

#### -Alumina/Mullite -

Material	Location in Filter	MOR, psi	Bend Bar Thickness, in.	Weibull
As-Received	Top <sup>(a)</sup>	3529+222(33)*	0.082+0.005	19.2
1300 Hr. Filter	Тор	2666+168(26)	0.083+0.003	19.1
1300 Hr. Filter	Тор	2653 <u>+</u> 220(23)	$0.120 \pm 0.001$	14.3
As-Received 1300 Hr. Filter	Web <sup>(b)</sup> Web	3148 <u>+</u> 368 (30) 2377 <u>+</u> 354 (48)	0.073 <u>+</u> 0.008 0.070 <u>+</u> 0.003	8.2 7.0
1300 Hr. Filter 1300 Hr. Filter	Flange Flange	2354 <u>+</u> 169(22) 2495 <u>+</u> 258(15)	0.078 <u>+</u> 0.003 0.115 <u>+</u> 0.006	16.2 11.4

\* () Indicate The Number Of Bend Bars Used In Determining Bend Strength.

(a) Top (Closed) Section Of The Filter.(b) Web Or Core Section Of The Filter Body.

during testing. In the HTHP simulator test rig, the gas phase  $(73\% N_2, 14\% H_20, 7\% CO_2, and 6\% O_2;$  no gas phase sulfur or alkali species) and the Grimethorpe or Exxon fly ash particulates are not considered to react with the alumina/mullite matrix. What is considered at this time to have occurred, is a phase change which results in the surfacing of the mullite rods. <sup>(3)</sup> Additional efforts are needed to identify whether the extended time at filter operating temperature and/or thermal cycling induces further mullitization within the alumina/mullite filter matrix.

Comparison of the Westinghouse data presented in Table K.6 can be made with the ANL data shown in Table K.7. Note that both the asfabricated, untested filter materials that were tested at Westinghouse and ANL, as well as the 1300 hour and Texaco exposed filters were fabricated from the same lot in February 1989.

Generally the flange and/or top closed sections have a higher Weibull value (m) in comparison with the web (core) section. This suggests that the material strength in the flange and/or top (closed) sections is relatively homogeneous, and not as widely scattered or variable as in the web (core) section.

Secondly, for the ANL data, the Weibull value for the web (core) section changes from 14 to 9 indicating a change in the flaw population when the filter material is exposed to the Texaco gasifier environment. For the Westinghouse 1300 hour simulated PFBC filter, no apparent change in the Weibull value is evident. This may imply that the Westinghouse simulated PFBC HTHP test facility which contains ash and no gas phase sulfur or alkali, is not as chemically reactive with the filter matrix, as possibly an actual process gas environment. Alternately, the Weibull variation may be attributed to variation between filters within a given fabrication lot.

#### ROOM TEMPERATURE STRENGTH DATA FOR THE TEXACO EXPOSED FILTER MATERIALS

#### -Alumina/Mullite -

Material <sup>*</sup>	Location in Filter	MOR, <sup>**</sup> psi	Weibull	
As-Received Texaco Exposed	Web <sup>(a)</sup> Web	3234+334 3089+334	14 9	
As-Received Texaco Exposed	Flange <sup>(b)</sup> Flange	- 3930+290 4099 <u>+</u> 276	16 18	

\* Bend Bars: 5 cm x 0.6 cm x 0.3 cm; 60 Samples/Area.

\*\* 4-Point Bend Mode: Outer Span = 4.45 cm; Inner Span = 0,925 cm; Cross Head Speed = 0.05 cm/min.

(a) Web or Core Section of the Filter Body.

(b) Flange Consists Of Both Top (Closed) Section Of The Filter And Actual Flange Area. In contrast with the Westinghouse data, ANL indicates that for the Texaco gasifier filter, the flexural strength of the flange is consistently higher than in the web (core) section. Note that the flange data reported by ANL is a combination of both the flange and top (closed) sections of the filter. Perhaps the flange and top (closed) sections are initially stronger than the web (core) section due to better sintering between flat plates during manufacturing, and that with time (i.e., 167 hours at Texaco versus 1300 hours in the Westinghouse HTHP rig), the flange area is weakened due to vibration and/or thermal cycling during pulse cleaning, such that the strength in the flange section approaches (i.e., is equivalent or possibly even less than) the strength of the web (core) section. Further effort is obviously needed to more fully understand the effect that operating time, gas phase chemistry, and temperature have on changes which may be occurring in the alumina/mullite material properties during actual filter operation.

#### REFERENCES

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- Alvin, M. A., D. M. Bachovchin, J. E. Lane, and T. E. Lippert, Thermal/Chemical Degradation of Ceramic Cross Flow Filter Materials, Westinghouse Electric Corporation, DOE Contract No. DE-AC21-88MC25034, June 1990 Monthly Report.

## FOUR-POINT BEND STRENGTH DATA

# TABLE K.8 TOP AND WEB SECTIONS OF THE AS-FABRICATED UNTESTED ALUMINA/MULLITE FILTER MATERIAL

Specimen	RT Stress	Height	Width	Filter
ID	(psi)	(in)	(in)	Location
T-1	3119	0.076	0.195	top
T-2	3512	0.077	0.194	top
<b>T-3</b>	3417	0.086	0.187	top
<b>T-4</b>	3403	0.085	0.182	top
<b>T-5</b>	3820	0.076	0.195	top
<b>T-6</b>	3822	0.084	0.185	top
T-7	3388	0.086	0.188	top
T-8	3558	0.078	0.195	top
T-9	3559	0.076	0.196	top
T-10	3728	0.078	0.195	top
<b>T-11</b>	3477	0.087	0.181	top
T-12	3347	0.087	0.185	top
T-13	3468	0.086	0.187	top
T-14	3873	0.076	0.195	top
T-15	3575	0.086	0.189	top
T-16	3707	0.077	0.196	top
T-17	3503	0.078	0.196	top
T-18	3072	0.077	0.196	top
T-19	3471	0.077	0.196	top
T-20	3631	0.088	0.189	top
T-21	3872	0.076	0.195	top
<b>T-22</b>	3799	0.076	0.195	top
<b>T-23</b>	3314	0.085	0.187	top
T-24	3112	0.091	0.183	top
T-25	3451	0.086	0.187	top
<b>T-26</b>	3499	0.086	0.188	top
T-27	3622	0.087	0.187	top
<b>T-28</b>	3840	0.077	0.196	top
T-29	3553	0.077	0.196	top
<b>T-30</b>	3175	0.085	0.186	top
T-31	3811	0.087	0.183	top
T- <b>32</b>	3454	0.076	0.196	top
T-33	3496	0.085	0.181	top
W-1	2713	0.082	0.201	web
W-2	3332	0.072	0.193	web
W-3	3010	0.073	0.192	web
W-4	3217	0.082	0.197	web
W-5	3366	0.073	0.193	wah
W-6	2998	0.081	0.198	wah
W-7	3438	0.072	0.103	web
-		0.014	V. 100	web

# TABLE K.8 (continued)

3132	0.072	0.193	web
3019	0.071	0.194	web
3456	0.071	0.192	web
3454	0.083	0.195	web
3003	0.084	0.198	web
3195	0.08	0.201	web
2994	0.074	0.192	web
3040	0.083	0.201	web
3251	0.073	0.193	web
3503	0.079	0.194	web
3014	0.073	0.188	web
3286	0.075	0.196	web
3053	0.063	0.197	web
3636	0.08	0.196	web
3438	0.081	0.192	web
1708	0.078	0.196	web
2501	0.06	0.201	web
3364	0.059	0.202	web
3297	0.06	0.202	web
3216	0.06	0.203	web
3246	0.059	0.2	web
3024	0.063	0.197	web
3548	0.078	0.195	web
	3132 3019 3456 3454 3003 3195 2994 3040 3251 3503 3014 3286 3053 3636 3438 1708 2501 3364 3297 3216 3246 3024 3548	3132 $0.072$ $3019$ $0.071$ $3456$ $0.071$ $3454$ $0.083$ $3003$ $0.084$ $3195$ $0.08$ $2994$ $0.074$ $3040$ $0.083$ $3251$ $0.073$ $3503$ $0.079$ $3014$ $0.073$ $3286$ $0.075$ $3053$ $0.063$ $3636$ $0.08$ $3438$ $0.081$ $1708$ $0.078$ $2501$ $0.06$ $3246$ $0.059$ $3024$ $0.063$ $3548$ $0.078$	\$132 0.072 0.193   \$019 0.071 0.194   \$456 0.071 0.192   \$454 0.083 0.195   \$003 0.084 0.198   \$195 0.08 0.201   2994 0.074 0.192   \$040 0.083 0.201   \$251 0.073 0.193   \$503 0.079 0.194   \$014 0.073 0.188   \$286 0.075 0.196   \$053 0.063 0.197   \$636 0.08 0.196   \$438 0.081 0.192   \$1708 0.078 0.196   \$2501 0.06 0.201   \$364 0.059 0.202   \$297 0.06 0.202   \$297 0.06 0.203   \$246 0.059 0.2   \$2024 0.063 0.197   \$548 0.078 0.195

# WEB SECTION OF THE 1300 HOUR HTHP PFBC TESTED FILTER

Specimen	<b>RT</b> Stress	Height	Width	Filter
ĪD	(psi)	(in)	(in)	Location
C1A1	2378	0.071	0.202	clean
C1A2	2536	0.07	0.201	clean
C1B1	1510	0.07	0.198	clean
C1B2	1522	0.07	0.201	clean
C1C1	2608	0.071	0.201	clean
C1C2	2780	0.071	0.201	clean
C2A1	2687	0.066	0.194	clean
C2A2	2164	0.069	. <b>0.194</b>	clean
C2B1	2466	0.066	0.193	clean
C2B2	2783	0.066	0.192	clean
C2C1	2705	0.07	0.197	clean
C2C2	2778	0.071	0.196	clean
C3A1	2668	0.07	0.192	clean
C3A2	2768	0.07	0.192	clean
C3B1	2187	0.072	0.203	clean
C3B2	2411	0.072	0.203	clean
C3C1	2394	0.068	0.195	clean
C3C2	2113	0.067	0.196	clean
C4A1	2410	0.073	0.202	clean
C4A2	2349	0.072	0.201	clean
C4B1	2444	0.068	0.201	clean
C4B2	2572	0.07	0.2	clean
C4C1	2245	0.073	0.203	clean
C4C2	1470	0.071	0.203	clean
D1A1	2423	0.077	0.192	dirty
D1A2	2693	0.076	0.193	dirty
D1B1	2700	0.072	0.2	dirty
D1B2	2642	0.072	0.199	dirty
D1C1	2465	0.075	0.19	dirty
D1C2	2668	0.075	0.194	dirty
D2A1	2640	0.066	0.192	dirty
D2A2	2493	0.072	0.199	dirty
D2B1	2332	0.072	0.198	dirty
D2B2	1327	0.075	0.197	dirty
D2C1	2452	0.071	0.195	dirty
D2C2	2458	0.065	0.100	dinty
D3A1	2877	0.078	0.208	dinty
DSA2	2467	0.072	0.200	disto
D\$B1	1964	0.071	0.204	dirty
D3B2	2523	0.079	0.40%	dirty
D3C1	2112	0.012	0.200	dirty d:
DSC2	2246	0.000	0.103	dirty
~~~~	24XV	0.001	0.101	airty

# TABLE K.9 (continued)

D4A1	2591	0.069	0.2	dirty
D4A2	2420	0.067	0.2	dirty
D4B1	2255	0.066	0.204	dirty
D4B2	1766	0.067	0.201	dirty
D4C1	2597	0.066	0.203	dirty
D4C2	2525	0.067	0.199	dirty

## FLANGE SECTION OF THE 1300 HOUR HTHP PFBC TESTED FILTER

Specimen	<b>RT</b> Stress	Height	Width	Filter
ID	(psi)	(in)	(in)	Location
1FL-1	2355	0.081	0 198	Flange
1FL-2	2515	0.082	0.198	Flange
1FL-3	2273	0.081	0.198	Flange
1FL-4	2301	0.081	0.197	Flange
1FL-5	2273	0.081	0.198	Flange
1FL-6	2515	0.08	0.197	Flange
1FL-7	2549	0.08	0.198	Flange
1FL-8	2444	0.082	0.198	Flange
2FL-1	2215	0.076	0.197	Flange
2FL-2	2596	0.074	0.198	Flange
2FL-3	2222	0.077	0.196	Flange
2FL-4	2509	0.077	0.195	Flange
2FL-5	1862	0.074	0.197	Flange
2FL-6	2291	0.075	0.195	Flange
2FL-7	2445	0.077	0.197	Flange
2FL-8	2406	0.076	0.198	Flange
2FL-9	2301	0.075	0.196	Flange
2FL-10	2355	0.076	0.196	Flange
2FL-11	2453	0.078	0.198	Flange
2FL-12	2523	0.074	0.194	Flange
2FL-13	2304	0.076	0.198	Flange
2FL-14	2093	0.076	0.196	Flange

# TOP SECTION OF THE 1300 HOUR HTHP PFBC TESTED FILTER

Specimen	RT Stress	Height	Width	Filter
ID	(psi)	(in)	(in)	Location
3T-1	2693	0.086	0.202	Top
3T-2	2826	0.086	0.202	Top
3T-3	2810	0.085	0.203	Top
3T-4	2634	0.086	0.201	Тор
3T-5	2865	0.086	0.203	Тор
ST-6	2610	0.087	0.202	Тор
3T-7	2865	0.086	0.202	Тор
ST-8	2960	0.086	0.204	Top
3T-9	2810	0.086	0.203	Тор
<b>3T-10</b>	2826	0.085	0.201	Top
ST-11	2647	0.085	0.201	Top
3T-12	2952	0.086	0.203	Top
4T-1	2480	0.079	0.198	Top
4T-2	2401	0.08	0.198	Тор
4T-3	2591	0.082	0.201	Top
4T-4	2646	0.083	0.2	Тор
4T-5	2791	0.082	0.2	Тор
4T-6	<b>2</b> 50 <b>3</b>	0.082	0.2	Top
4T-7	2705	0.083	0.198	Тор
4 <b>T-</b> 8	2518	0.081	0.199	Тор
4T-9	2728	0.082	0.198	Тор
<b>4T-10</b>	2611	0.076	0.198	Тор
4T-11	2494	0.08	0.198	Тор
4T-12	2527	0.081	0.198	Top
4T-13	2463	0.083	0.198	Тор
4 <b>T-14</b>	2374	0.079	0.198	Тор

#### FLANGE SECTION OF THE 1300 HOUR HTHP PFBC TESTED FILTER - THICKER BEND BAR DATA -

Specimen	RT Stress	Height	Width	Filter
IJ	(Psi)	(111)	(11)	Location
T1FL-1	2154	0.122	0.161	ThFlange
<b>T1FL-2</b>	1919	0.121	0.16	ThFlange
T1FL-3	2712	0.124	0.161	ThFlange
T1FL-4	2742	0.122	0.16	ThFlange
T1FL-5	2426	0.124	0.161	ThFlange
T1FL-6	2464	0.124	0.161	ThFlange
T2FL-1	2728	0.11	0.159	ThFlange
T2FL-2	2941	0.11	0.161	ThFlange
T2FL-3	2306	0.11	0.159	ThFlange
T2FL-4	2343	0.111	0.16	ThFlange
T2FL-5	2575	0.109	0.158	ThFlange
T2FL-6	2435	0.11	0.159	ThFlange
<b>T2FL-7</b>	2420	0.109	0.159	ThFlange
<b>T2FL-8</b>	2675	0.111	0.159	ThFlange
T2FL-9	2591	0.111	0.158	ThFlange

# TOP SECTION OF THE 1300 HOUR HTHP PFBC TESTED FILTER - THICKER BEND BAR DATA -

Specimen	<b>RT</b> Stress	Height	Width	Filter
D	(psi)	(in)	(in)	Location
T3T-1	2381	0.121	0.156	ThTop
T3T-2	3035	0.12	0.157	ThTop
T3T-3	2695	0.121	0.154	ThTop
T3T-4	2944	0.121	0.153	ThTop
T3T-5	3012	0.121	0.153	ThTop
T3T-6	2976	0.121	0.156	ThTop
T3T-7	2917	0.12	0.153	ThTop
T3T-8	2878	0.12	0.157	ThTop
T3T-9	2402	0.121	0.153	ThTop
T3T-10	2764	0.121	0.157	ThTop
T4T-1	2649	0.121	0.164	ThTop
T4T-2	2605	0.12	0.163	ThTop
T4T-3	2526	0.121	0.165	ThTop
T4T-4	2381	0.119	0.162	ThTop
T4T-5	2709	0.12	0.166	ThTop
T4T-6	2666	0.12	0.164	ThTop
T4T-7	2479	0.121	0.162	ThTop
T4T-8	2550	0.12	0.163	ThTop
T4T-9	2507	0.118	0.167	ThTop
<b>T4T-10</b>	2565	0.12	0.164	ThTop
T4T-11	2414	0.12	0.166	ThTop
T4T-12	2628	0.12	0.165	ThTop
T4T-13	2331	0.12	0.168	ThTop









Figure K.7 - Weibull Probability of Failure For the Web Section of the As-Fabricated, Untested Alumina/Mullite Filter Material



Figure K.8 - Weibull Probability of Failure For the Top Section of the 1300 Hour HTHP PFBC Filter







Figure K.10 - Weibull Probability of Failure For the Flange Section of the 1300 Hour HTHP PFBC Filter



Figure K.11 - Weibull Probability of Failure For Thicker Bend Bar Samples Prepared From the Top Section of the 1300 Hour HTHP PFBC Filter





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# APPENDIX L

# HOT STRENGTH CHARACTERIZATION OF THE 1300 HOUR, HTHP TESTED CERAMIC CROSS FLOW FILTER

# HOT STRENGTH CHARACTERIZATION OF THE 1,300 HOURS HIGH TEMPERATURE, HIGH PRESSURE TESTED CERAMIC CROSS FLOW FILTER

In order to develop the alkali kinetics and material life model for the alumina/mullite filter material in advanced coal fired processes, data have been generated at 870°C for material that has been exposed to:

- Gas phase alkali at 870°C for 100-1560 hours
- Gas phase alkali at 870°C for 100-1560 hours with pulse cycling
- 870°C for 1500 hours without gas phase alkali or pulse cycling.

As a result of this effort, we have demonstrated that the P-100A alumina/mullite matrix undergoes a conversion of the amorphous phase that is present in the material to anorthite at temperatures of 870°C in the presence of gas phase alkali. An increase in the hot strength of the material occurs as a result of anorthite formation (Figure L.1). As the period of exposure time to gas phase alkali increases, anorthite is converted into tridymite, leading to a reduction in the hot strength of the material.

When the alkali exposed alumina/mullite matrix is subjected to thermal cycling, simulating pulse cleaning, a somewhat reduced hot strength of the matrix results. We currently suspect that this results primarily from microcrack formations within the structure. Although we have not attempted to differentiate between the various phases or phase concentration through the thickness of the alkali exposed disc, the



Figure L.1 - Influence of In-Service Parameters on the Hot Strength of the Alumina/Mullite Filter Matrix as a Function of Time

possibility exists that an additional or alternate phase may have formed along the pulsed surface, or that the concentration of tridymite is higher along the pulsed surface versus the alkali exposed surface.

A shown in Figure L.1, the alkali/steam/air environment tends to accelerate the increase in hot strength of the alumina/mullite matrix over that of the matrix that was simply subjected to long-term, high temperature exposure. Again we suspect that the amorphous phase is being converted into anorthite, however, as a slower rate.

In addition data have been generated for material removed from an alumina/mullite cross flow filter that operated for 1,300 hours at 850°C, filtering pressurized fluidized-bed combustion (PFBC) ash fines in one of Westinghouse's high temperature, high pressure (HTHP) test facilities. If alkali were present in the HTHP system, it was generated via the coal processed fines that were filtered during HTHP testing. No attempt was made to further introduce alkali into the system which would represent possible gas phase concentrations at actual process conditions.

Characterization of the 1,300 hour tested filter material consisted of room temperature strength analysis of the flange, web, and top closed sections of the filter, as well as x-ray diffraction analyses. As shown in Table L.1, post-test analysis room temperature flexural strength analysis of the matrix indicates that a 25% loss of strength had occurred uniformly throughout the entire cross flow filter element body. X-ray diffraction analyses of the ~1300 hour HTHP filter matrix identified the conversion of ~50% of the amorphous phase contained within the matrix to that of the crystallized anorthite phase.

As shown in Table L.2 hot strength data generated for the 1,300 hour HTHP tested filter indicate nearly comparable strengths along the clean and dirty gas channels (i.e., 3278+359 and 3362+521 psi, respectively). Note that the resulting hot strength of the filter

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#### TABLE L.1

## ROOM TEMPERATURE STRENGTH DATA FOR THE 1,300 HOUR WESTINGHOUSE PFBC HTHP TESTED FILTER MATERIAL

#### -Alumina/Mullite -

Material	Location in Filter	MOR, psi	Bend Bar Thickness, in.	Weibull
As-Received	Top*	3529+222(33)**	0.082+0.005	19.2
1300 Hr. Filter	Top	2666+168(26)	0.083+0.003	19.1
1300 Hr. Filter	Тор	2653 <u>+</u> 220 (23)	0.120-0.001	14.3
As-Received	Web	3148+368 (30)	0.073+0.008	8.2
1300 Hr. Filter	Web	2377 <u>+</u> 354 (48)	0.070+0.003	7.0
1300 Hr. Filter	Flange	2354+169(22)	0.078+0.003	16.2
1300 Hr. Filter	Flange	2495 <u>+</u> 258 (15)	$0.115 \pm 0.008$	11.4

\* Closed Section At The Top Of the Filter

\*\* () Indicate The Number Of Bend Bars Used In Determining Bend Strength

Chart Speed: 5 cm/min. Upper Span: 20 mm Lower Span: 40 mm 4 Pt; 1/4 Flexure

#### TABLE L.2

### HOT STRENGTH DATA FOR THE 1,300 HOUR WESTINGHOUSE PFBC HTHP TESTED FILTER MATERIAL

#### -Alumina/Mullite -

Test Sample No.	Temp., °C	Cross Head Speed, cm/min	Width, in.	Height, in.	Loads, 1bs.	Strength, psi
Clean Gas Ch	annels				<u></u>	
1	870	0.02	0.249	0.064	2.97	3429
2	870	0.02	0.248	·0.063	2.68	3206
3	870	0.02	0.255	0.066	3.54	3753
4	870	0.02	0.248	0.067	3.53	3734
5	870	0.02	0.250	0.069	2.93	2899
6	870	0.02	0.249	0.069	3.30	3278
7	870	0.02	0.250	0.070	3.47	3335
8	870	0.02	0.249	0.066	3.35	3637
9	870	0.02	0.249	0.062	2.80	3445
10	870	0.02	0.256	0.067	2.84	2910
11	270	0.02	0.249	0.070	3.25	3137
12	870	0.02	0.249	0.066	2.37	2573
Dirty Gas Cl	annels					
1	870	0.02	0.249	0.071	3.38	3171
2	870	0.02	0.249	0.071	2.83	2655
3	870	0.02	0.249	0.068	3.11	3181
4	870	0.02	0.249	0.074	3.46	2988
5	870	0.02	0.250	0.077	5.02	3988
6	870	0.02	0.249	0.073	4.20	3727
7	870	0.02	0.250	0.074	5.20	4473
8	870	0.02	0.250	0.074	4.65	4000
9	870	0.02	0.248	0.061	2.50	3190
10	870	0.02	0.249	0.062	2.64	3248
11	870	0.02	0.250	0.067	2.88	3022
12	870	0.02	0.250	0.066	2.85	3082
13	870	0.02	0.249	0.060	2.27	2982
A]	<u>Pahni cated</u>	Average Vet	Strongth			

As-Fabricated Average Hot Strength:

Clean Gas Channel Average Hot Strength:	3278 <u>+</u> 359 (12)
Dirty Gas Channel Average Hot Strength:	3362 <u>+</u> 521 (13)
Average Hot Strength (All Channels):	3321 <u>+</u> 443 (25)

Chart Speed: 5 cm/min. Upper Span: 20 mm Lower Span: 40 mm 4 Pt; 1/4 Flexure material appears to be greater than than of its corresponding room temperature strength. To date we have not generated the hot strength characteristics of the as-fabricated matrix. During May we tentatively plan to recharacterize the room temperature and hot strength of a ceramic cross flow filter that was produced in the same fabrication lot as the 1,300 hour HTHP tested filter. Since we have demonstrated conversion of the amorphous to anorthite phase occurred during HTHP testing, we believe that the initial as-fabricated untested matrix will have a lower initial hot strength in comparison to the 1,300 hour HTHP filter hot strength, as well as a lower as-fabricated hot strength in comparison to its as-fabricated room temperature strength.

The as-fabricated amorphous content in the P-100A structure was apparently sufficient for the filter to survive the stresses induced by the filter mount and pulse gas cleaning cycles during the 1,300 hours of HTHP testing. As a result of these data, a reevaluation needs to be performed to determine the validity of the concept that is currently accepted which indicates that "all" porous ceramic filter materials experience a loss of strength during hot gas filtration. We are recommending that post-test evaluation of filters include hot strength testing at process temperatures, as well as a comparison of these data with the hot strength data generated for an as-fabricated filter from the same fabrication lot. In addition we need to determine whether the maximum stress levels at the critical stress areas in the filter (i.e., flange, channels, etc.) are the same at process operating temperature as at room temperature.

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