Fabrication of Full-Scale Fiber-Reinforced Hot-Gas Filters by Chemical Vapor Deposition

FINAL REPORT

for the period November 1, 1994 to December 31, 1995

Prepared by:
Robert G. Smith, Principal Investigator
Joseph H. Eaton
Douglas J. Pysher
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January 1996

Report Prepared by
3M Ceramic Technology Center
3M Center, Bldg. 201-4N-01
St. Paul, MN 55144
under
Subcontract No. 93X-SB482C

for

OAK RIDGE NATIONAL LABORATORY
Oak Ridge, Tennessee 37831
managed by
LOCKHEED MARTIN ENERGY SYSTEMS, INC.
for the
U.S. Department of Energy
under Contract No. DE-AC05-84OR21400

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ABSTRACT

The overall goal of this contract and its extensions has been to develop a hot gas candle filter which is light weight, has a thin wall, resists mechanical and thermal shock, and is resistive to alkali attack. A ceramic fiber reinforced, ceramic matrix composite approach has been followed to fabricate this new candle filter.

Past reports covered the first test results of two ceramic composite candle filters at the Westinghouse Science and Technology Center in March of 1993, subsequent improvements made in the filter's construction and fabrication processing, and the testing of six improved full size, 60 mm diameter by 1575 mm length, filters that met or exceeded performance requirements set for them. Completion of the 172 hours of simulated PFBC testing and thermal transients plus maintaining less than 4 ppm clean side ash concentration provided a basis for moving to the next step of testing in the Tidd PFBC Demonstration Project.

In this contract extension 3M fabricated 110 filters to be used for tests in demonstration power plant facilities and other tests that become available. The filters were tested to meet all quality assurance specifications and inventoried for Oak Ridge National Laboratory, ORNL. The filters are being shipped to various industrial, university, and national laboratory test facilities as requested by ORNL.

Ten ceramic composite filters were installed in December, 1994 in the Tidd PFBC Demonstration Project filter vessel for their test period #5. Five filters were installed in a top cluster and five in a bottom cluster. The filters were removed in May 1995 after operating for 1110 hours in a temperature range of 760°C to 843°C, with 80% of the run above 815°C. The filters operated at a face velocity of 4.8 cm/sec (9.5 ft/min) and an inlet dust loading of 18000 ppmw. Westinghouse retained two filters while the remaining eight were returned to 3M for analysis.

Characterization tests after the Tidd test showed that the filters were in very good condition and that they operated well as filters. As has been shown in past ceramic composite exposure and characterization tests, after a typical initial decrease from as fabricated property levels occurred, the filters showed stable mechanical properties that are quite capable of providing filtration in the hostile power generation system environment.

TABLE OF CONTENTS

Section	Page
ABSTRACT.....	i
1.0 INTRODUCTION.....	1
2.0 CONTRACT EXTENSION DELIVERABLES.....	3
3.0 EXPERIMENTAL PROCEDURES AND DISCUSSION.....	4
3.1 Filter Fabrication.....	4
3.2 Filter Installation and Removal at Tidd.....	4
3.3 Filter Supply for External Testing	6
3.4 Filter Characterization	9
3.4.1 Mechanical Properties Characterization	9
3.4.2 Filtration Characterization	16
3.4.3 Other Characterization Results.....	18
4.0 SUMMARY.....	22
5.0 ACKNOWLEDGMENTS.....	23
6.0 REFERENCES	23
APPENDIX I – Ceramic Composite Filter Test Methodology	25
REFERENCES FOR APPENDIX I	35
APPENDIX II – Mechanical Test Data Details	36
APPENDIX III - Distribution.....	42

LIST OF FIGURES

Figure	Page
1. An as-fabricated and as-tested 3M brand Ceramic Composite Filter, type 203.....	3
2. Three of the 3M brand Ceramic Composite Filters hanging in the Tidd Facility during removal	7
3. 3M brand Ceramic Composite Filters in boxes being examined in laboratory.....	8
4. Sectioning Diagram for 3M brand Ceramic Composite Filter 45-17-01	9
5. Sectioning Diagram for 3M brand Ceramic Composite Filter 45-13-01 ..	10
6. Sectioning Diagram for 3M brand Ceramic Composite Filter 45-17-03 ..	10
7. Sectioning Diagram for 3M brand Ceramic Composite Filter 44-11-06 ..	11
8. Load-displacement curves for maximum and minimum strength baseline and exposed tensile specimens	13
9. Load-displacement curves for maximum and minimum strength baseline and exposed O-ring compression specimens	13
10. Load-displacement curves for maximum and minimum strength baseline and exposed O-ring burst specimens.....	14
11. Pressure drop and filter efficiency for filter sample 45-13-10, section 13.....	17
12. Pressure drop and filter efficiency for filter sample 45-13-10, section 14.....	18
13. SEM of outside edge of outer mesh on exposed filter 45-17-03.....	20
14. SEM of middle of outer mesh on exposed filter 45-17-03	21
15. SEM of middle of filter layer on exposed filter 45-17-03.....	21
16. Sectioning Diagram for 60" ceramic composite filter for test samples.....	26
17. Schematic of Filtration test.....	26
18. Example of filtration test performed on 4" candle filter section.....	28
19. Tensile test specimen and fixture.....	28
20. Typical or representative load-displacement curves for tensile tests of filters.....	30
21. Schematic of O-ring diametral compression test	30
22. Typical or representative load-displacement curves for O-ring compression tests of filters.....	31
23. Schematic of burst test	32
24. Load-displacement curves for burst test on a 1" section of filter	34
25. Flange bend test specimen and fixture.....	34

LIST OF TABLES

Table	Page
1. Ceramic Composite Filter Installed at Tidd Power Plant	5
2. Ceramic Composite Filters for External Evaluation.....	8
3. Summary of Mechanical Property Tests	12
4. As Fabricated – O-Ring Compression Tests	36
5. As Fabricated – Tensile Tests.....	37
6. As Fabricated – Burst Tests.....	38
7. Tidd Plant, Exposed – O-Ring Compression Tests.....	39
8. Tidd Plant, Exposed – Tensile Filter Test	40
9. Tidd Plant, Exposed – Burst Tests	41

1.0 INTRODUCTION

The objective of this contract was to develop and fabricate a set of ceramic fiber reinforced, ceramic matrix composite hot gas candle filters for testing in a simulated Pressurized Fluidized Bed Combustion (PFBC) environment. These objectives were met and in this contract extension 3M fabricated 110 filters for tests in demonstration power plant facilities and other tests that become available. The commercialization of the ceramic composite filter is based in part on technology developed at Oak Ridge National Laboratory.¹

The initial use for these ceramic composite hot gas candle filters is to remove hot particulate material from PFBC and Integrated Gasification Combined Cycle (IGCC) power plant processes.² In these environments the filters are subjected to temperatures of 850°C and higher in operating pressures of about 1013 kPa (10 atmospheres) while filtering fine ash particulate. In use, the filters are repeatedly cleaned and therefore thermally shocked by a reverse flow pulse with room temperature air. The atmosphere can be either reducing or oxidizing depending upon the process. In addition, the PFBC and IGCC processes can have environmental conditions in the system which are corrosive to some of the ceramic materials used in various manufacturer's filters.

The use of ceramic fiber reinforced, ceramic matrix composite hot gas candle filters brings several benefits to the filtering system. These include:

- light weight,
- a balance of toughness and strength,
- good thermal mechanical properties at high temperature,
- excellent thermal shock resistance,
- thin filter walls with higher permeability, and
- capability to retrofit into existing filter holders.

During the original contract period of May 1992 to June 1993, research and development on the ceramic composite technology was completed and four full scale filters were fabricated.^{3,4,5} Two of the ceramic fiber reinforced hot gas

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candle filters were tested for 100 hours in a simulated PFBC test at Westinghouse Science and Technology Center.^{5,6} The Westinghouse test showed that a workable filter surface on a strong substrate had been developed. The test also pointed out several areas of filter construction that needed improvement. The first main area was the chemical vapor infiltration (CVI), bonding of the filter surface to the intermediate layer and substrate, which had been suspect during the filter fabrication. The second main area for improvement was reinforcing the seal flange to permit forming a tighter seal. Both ceramic composite filters survived the thermal transients as would be expected of a composite filter construction.

With these promising results, a second, longer simulated PFBC test was proposed to test six improved ceramic composite hot gas candle filters. This work was funded via a contract extension from July 1993 to October 1994. 3M improved the fabrication process and the construction of each of the filter's layers and then fabricated twelve additional filters. Six of these were successfully tested in December 1993.^{7,8,9}

These six filters completed 172 hours of testing first using the Old Grimethorpe ash, #132, and then the more difficult to filter Red Grimethorpe ash, #129. Fourteen turbine trip simulations were run over a 12 hour period to subject the filters to the thermal shocks typical of a system trip or fast shutdown. The inlet gas flow had an ash concentration of 2000 ppm. The average clean side ash concentration was 2.5 ppm with one early sample at 6.49 ppm and the remainder below 4 ppm during the 100 hours of sampling. An as-fabricated and as-tested 3M brand Ceramic Composite Filter, type 203, are shown in Figure 1 on the following page.

With these promising test results, a second contract extension was approved to fabricate ceramic composite filters and include them in one or more power plant demonstration tests such as at the Tidd PFBC Demonstration Project in Brilliant, OH.¹⁰

Figure 1. An as-fabricated and as-tested 3M brand Ceramic Composite Filter, type 203

2.0 CONTRACT EXTENSION DELIVERABLES

1. 3M shall fabricate 110 ceramic composite hot-gas filters consisting of Nextel™ 312 ceramic fibers and a silicon carbide matrix. The filters are to be 1.5 meters in length and 60 mm in diameter with a mounting flange for installation in the Tidd power plant or Karhula test facility. All finished filters are to be stored at 3M and shipped to test facilities per ORNL request.
2. 3M shall assist Westinghouse Science and Technology Center and American Electric Power personnel during the filter installation to ensure optimum filter performance. 3M personnel should be present to ensure the filters were not damaged during shipping and that the gaskets and filters are fitted into the mounting flanges properly. During removal of these filters 3M personnel should be present to assist with visual inspection and removal of filters. As much information as possible, about filter usage, shall be collected.

3. 3M should sufficiently characterize the as-fabricated and exposed filters to correlate their properties with performance in the Tidd power plant.
4. 3M shall supply as-fabricated and exposed filters to J. P. Singh of Argonne National Laboratories for mechanical property characterization and R. E. Tressler of Pennsylvania State University for evaluation in a flow through simulated combustion environment screening test.
5. 3M shall present test results at the 1995 Fossil Energy Materials Conference in Oak Ridge, TN.
6. 3M shall publish a final contract report .

3.0 EXPERIMENTAL PROCEDURES AND DISCUSSION

3.1 Filter Fabrication

One hundred and ten filters were fabricated between September 1994 and May 1995 using standard 3M brand ceramic composite filter production procedures. These filters were fabricated during regular 3M production runs of filters. Processing details and standards are not covered since this is a proprietary fabrication process. All filters were tested to meet all quality assurance specifications and inventoried for Oak Ridge National Laboratory, ORNL. The filters are being shipped to test facilities as requested by ORNL. Ten filters were shipped to and installed in the Tidd PFBC Demonstration Project filter systems. Other sample quantities have been shipped for a variety of uses such as in the Morgantown Energy Technology Center's, METC, gasifier tests, ash characterization and filter tests at the Energy and Environmental Research Center at the University of North Dakota, and filter demonstration at the Karhula test facility. The remaining filters will continue to be inventoried and shipped per ORNL requests.

3.2 Filter Installation and Removal at Tidd

Twenty-four ceramic composite filters were shipped to American Electric Power on October 31, 1994 for use in the Tidd plant. The plant is owned and operated by

American Electric Power, AEP. Westinghouse Science and Technology Center, WSTC, is responsible for the filter system in which the ceramic composite filters were installed. Both companies are under contract to METC to operate the facility and testing program.

On December 21, 1994, two 3M employees worked with WSTC personnel to install ten filters in the filter vessel as part of the variety of filters in the vessel. WSTC had decided to install only ten each of several brands of experimental filters of the total 288 filters in the vessel for this test. In Westinghouse and AEP reports this test period is generally referred to as test period number five.¹¹ Five filters were installed in a top cluster and five in a bottom cluster. Table 1 lists the ten filters and their mounting position. All ten filters were installed with the Westinghouse gasket system and a slightly shortened mounting plug. The standard plug was shortened after laboratory tests showed the original long plug neck was at least partially responsible in combination with the ash bridging for the flange neck fracture of two of the filters in Tidd test period four. Those two filters' flange necks fractured after 1015 hours of operation. A third filter worked throughout the 1705 hours of test period four.

Table 1. Ceramic Composite Filters Installed at Tidd Power Plant

Filter #	Filter Mount #	Disposition
Top cluster, plenum C		
45-17-03	16	Returned to 3M
45-17-01	17	Returned to 3M, half to Argonne
45-18-02	18	Retained by WSTC
45-13-02	19	Returned to 3M
44-10-03	20	Returned to 3M
Bottom cluster, plenum B		
44-11-06	3	Returned to 3M
45-18-06	4	Returned to 3M
43-06-02	5	Returned to 3M
45-17-02	6	Retained by WSTC
45-13-04	7	Returned to 3M

On May 23, 1995, 3M personnel met WSTC personnel at the Tidd plant to assist in the removal of the ceramic composite filters and make any observations possible regarding their operation. The filters had operated for 1110 hours in a temperature range of 760°C to 843°C, with 80% of the run above 815°C. The filters operated at a face velocity of 4.8 cm/sec (9.5 ft/min) and an inlet dust loading of 18000 ppmw. Two filters had already been removed by the WSTC technician and were retained by Westinghouse. The mounting collars in the top cluster of the system were very difficult to remove due to the ash packed in parts of the mount. The technician had to use a pry bar and twist the filters back and forth to remove the mounts. As a result the flanges cracked on several of the filters. Photos were taken of the filters, observations noted, and the eight remaining filters packed for shipment to 3M.

The eight filters returned to 3M were spread out in the laboratory for additional visual observations and determination of sectioning plans for testing at 3M and at Argonne National Laboratory. Figures 2 and 3 show the filters as they are being removed from Tidd and being examined in the laboratory, including brushing off the ash and cutting samples from the filters. Specific observations related to the filters and their condition made during removal from the Tidd filter vessel and on return to the 3M laboratory are summarized below in Section 3.4.3.

3.3 Filter Supply for External Testing

Three filters and one filter substrate have been supplied to Argonne National Laboratory and Pennsylvania State University for testing and are summarized in Table 2. The filter substrate provides the mechanical strength of the filter over which the filter layer and outer mesh are applied. Filter number 45-17-01 exposed at Tidd was cut as shown in Figure 4 to retain the ends for testing at 3M while sending a 127 cm (50") tubular section of the filter to Argonne National Laboratory. Dr. J. P. Singh at Argonne National Laboratory is doing mechanical property characterization on an as fabricated substrate, a complete as fabricated filter, and the filter exposed at Tidd. Dr. E. R. Kupp and Professor R. E. Tressler are evaluating hot-gas filters in a flow through simulated combustion environment screening test. 3M has interacted with both groups regarding the tests and has supplied the 3M test methodology paper to help in standardizing methods on testing a ceramic composite filter. The test methodology paper is included as

Appendix I in this report. Each group will report their results through the appropriate reporting channels based on their funding support from DOE Office of Fossil Energy.

Figure 2. Three of the 3M brand Ceramic Composite Filters hanging in the Tidd Facility during removal

Figure 3. 3M brand Ceramic Composite Filters in boxes being examined in laboratory

Table 2. Ceramic Composite Filters For External Evaluation

Filter #	Construction	Condition	Sent to
45-28-02	substrate	as fabricated	J. P. Singh, Argonne Nat. Lab.
45-16-05	filter	as fabricated	J. P. Singh, Argonne Nat. Lab.
45-28-05	filter	as fabricated	J. P. Singh, Argonne Nat. Lab.
45-17-01	filter	exposed in Tidd run #5	J. P. Singh, Argonne Nat. Lab.
44-13-01	filter	as fabricated	E. R. Kupp, Pennsylvania State Univ.

Figure 4. Sectioning Diagram for 3M brand Ceramic Composite Filter 45-17-01

3.4 Filter Characterization

3.4.1 Mechanical Properties Characterization

One unexposed and two exposed filters were used for mechanical properties and filtration testing. Filters were sectioned using a diamond saw as shown in Figures 5, 6, and 7. Filter 45-13-01, which was used as a reference, was sectioned into six 12.7 cm (5 inch) tensile specimens and twelve 2.54 cm (1 inch) O-rings. Filter 45-17-03 was sectioned into seven 12.7 cm (5 inch) tensile specimens, four 2.54 cm (1 inch) O-rings, two 10.12 cm (4 inch) filtration specimens, and one 19 cm (7.5 inch) flange bend specimen while filter 44-11-06 was sectioned into two filtration specimens and forty-one 2.54 cm (1 inch) O-rings.

Three types of mechanical properties tests were performed: tensile, O-ring diametral compression, and O-ring burst. Details of the test methodologies are given in Appendix I. The O-ring specimen dimensions (OD, wall thickness, width) were measured using digital calipers. The average dimensions of the burst and O-ring compression specimens were used for computing tensile properties.

Figure 5. Sectioning Diagram for 3M brand Ceramic Composite Filter 45-13-01

Figure 6. Sectioning Diagram for 3M brand Ceramic Composite filter 45-17-03

Figure 7. Sectioning Diagram for 3M brand Ceramic Composite filter 44-11-06

The complete data tables which include the physical dimensions and mechanical property results are included in Tables 4, 5, 6, 7, 8, and 9 in Appendix II.

Representative load-displacement curves from tensile, O-ring compression, and burst tests shown in Figures 8, 9, and 10, respectively, are from specimens having minimum and maximum peak loads for both as fabricated baseline and exposed filters. Thus, they indicate the range of observed load-displacement curves.

Retained strength and stiffness of exposed and baseline filters are compared in Table 3.

As discussed below, it is difficult to determine a correct wall thickness for these braided composite materials which leads to difficulties in stating a representative strength value. In comparing the as fabricated ceramic composite filters and those exposed in the Tidd power plant, it is very useful to also compare the actual peak load values of the tests. These are shown in the bottom half of Table 3. As can be seen from Table 3 or examining the detailed data tables in Appendix II, the ceramic composite filters start out at an extremely high peak load, 1200 kg (2645 lb) in tensile testing, for these light weight composite materials. After 1110 hours of

exposure, the filters still showed a high value of 255 kg (562 lb) peak load in tensile testing. This is more than enough capability to carry the filter's own 1 kg weight plus many kilograms of ash if bridging should cause a buildup. Similar comparisons and retained peak load ability are shown for the burst and O-ring compression tests.

Strength values were determined as discussed in the section below. Stiffness values were determined from the maximum slope of the load-displacement curve. For the O-ring compression tests, the fracture energy is also given. This is simply the area under the load-displacement curve and relates to the filter's resistance to fracture. Efforts are underway to adapt standardized fracture toughness test methods to the cylindrical geometry of the ceramic composite filter.

Table 3. Summary of mechanical property tests

	Tensile Strength (MPa)	Tensile Stiffness (kg/mm)	Burst (Hoop) Strength (MPa)	Burst (Hoop) Stiffness (kg/mm)	O-Ring Strength (MPa)	O-Ring Stiffness (kg/mm)
As-Produced	20	2081	87	430	11.1	11.3
After Exposure	4	1123	28	131	6.9	5.6
Retained	20%	54%	32%	30%	62%	50%
	Tensile Peak Load (kg)	Burst (Hoop) Peak Load (kg)		O-Ring Compression Peak Load (kg)		
As-Produced	1207	277		6.8		
After Exposure	255	94		4.4		
Retained	21%	34%		64%

Figure 8. Load-displacement curves for maximum and minimum strength baseline and exposed tensile specimens

Figure 9. Load-displacement curves for maximum and minimum strength baseline and exposed O-ring compression specimens

Figure 10. Load-displacement curves for maximum and minimum strength baseline and exposed O-ring burst specimens

The tensile strengths were calculated using equation 1,

$$1. \quad \sigma_{tensile} = \frac{F}{\pi(r_o^2 - r_i^2)},$$

where F is the fracture load in Newtons, and r_o and r_i are the outer and inner tube radii, respectively. Tensile stiffness values were determined from the maximum slope of the load-displacement curves.

For stress analyses, the composite filter is treated as a thin-wall cylinder (i.e. the assumption of constant stress across the wall results in negligible error) since its internal diameter-to-thickness ratio is greater than 10.¹²

The maximum stress in an O-ring subjected to diametral compression is given by equation 2,¹³

$$2. \quad \sigma_{\text{O-Ring}} = \frac{2Fr_a (6r_a - t)}{\pi dt^2 (2r_a - t)},$$

where r_a is the average radius ($r_a = [r_o + r_i] / 2$), t is the wall thickness, and d is the specimen width.

A uniform tensile hoop stress is applied to 2.54 cm sections of the ceramic composite filter by uniaxially compressing a silicone rubber plug that has been placed inside the ring. The analysis given by Jadaan et al.¹⁴ leads to the following equation, 3., for the pressure exerted by the plug on the tube.

$$3. \quad p' = \frac{v_r q}{1 - v_r} = \frac{v_r P}{\pi r_i^2 (1 - v_r)}$$

where P is the applied load and v_r is the Poisson's ratio of the rubber plug. This equation can be simplified by assuming that the ratio of the elastic modulus of the rubber to that of the ceramic filter is zero. Thus the maximum hoop stress in the filter is given by equation 4.

$$4. \quad \sigma_{\text{hoop}} = \frac{p' r_a}{t}$$

Table 3 is a summary of the calculated retained strengths and stiffnesses.

A critical parameter in determining accurate stresses is the wall thickness, (see Equations 1 and 2 above). For the 3M brand Ceramic Composite Candle Filter, the thin-walled, high-porosity, multi-layer construction makes it difficult to decide on a representative wall thickness. Work has begun on determining an "effective" wall thickness from the true density based on submerged weight measurements. The effective wall thickness would be the thickness of a 100% dense composite tube having the same average radius and length.

Recall that the 3M brand Ceramic Composite Candle Filter is composed of 3 layers. The principal function of the substrate is mechanical support. The other

layers, the filter media, and the outer mesh are not intended to be structural. However, depending on the stress state, they may or may not contribute to the overall strength of the filter.

Efforts to develop mechanical test methodologies have utilized substrate tubes only, in order to simplify the stress analyses. Typical burst and O-ring compression strengths of substrate composites calculated using the actual measured porous wall thickness are 190 and 30 MPa, respectively. Burst and O-Ring compression strengths calculated using "effective" wall thickness are 400 and 135 MPa, respectively. Thus, the strengths reported here that were determined using the entire filter wall thickness are conservative and underestimate the "true" material strength. Regardless of the absolute value of the measured strengths, since the same procedure was used for both baseline and exposed filters, the percentages of retained strength and stiffness would still be accurate.

3.4.2 Filtration Characterization

Filtration testing is performed on a TSI Automated Filter Tester model 8110. The filtration test is performed automatically by the machine once a flow rate has been set which corresponds to a given face velocity (currently 3.0 cm/sec). Face velocity equals flow rate in cc/sec divided by surface area in cm^2 . The surface area of a 10.12 cm (4 inch) long sample is 174.4 cm^2 which is based on a 10 cm length after it is mounted and a 5.55 cm diameter. The filtration test utilizes NaCl particles with a mean diameter of 0.1 micron and a concentration of 100 mg/m^3 . Efficiency is calculated by dividing the concentration of particles exiting the filter by the input concentration.

These tests are derived from ASTM E128-89 entitled "Standard Test Method for Maximum Pore Diameter and Permeability of Rigid Porous Filters for Laboratory Use." ¹⁵

Baseline filtration tests were run as described above on Sections 13 and 14 of ceramic composite filter 45-13-01 which was sectioned according to Figure 5.

Figures 11 and 12 show the pressure drop and efficiency curves for each sample. These data curves are typical of the behavior of the 3M brand ceramic composite candle filter.

Sections 13 and 14 were also tested for maximum pore size and permeability at 5.0 cm/sec. Section 13 had a maximum pore size of 52 microns and a permeability of 118 cm/min/cm H₂O. Section 14 had a maximum pore size of 65 microns and a permeability of 108 cm/min/cm H₂O. The values for both sections, and the range of variability between them, are also typical of the 3M brand ceramic composite candle filter.

Ceramic composite filter 45-17-03 was exposed for 1110 hours at the Tidd test site and sectioned according to Figure 6. No filtration data were gathered on these sections due to the high back pressure with any given flow rate. This was caused by the accumulation of ash on the inside of the filters which blocked the open substrate inner layers and contributed to the back pressure and loss of permeability. The ash build up from the system clean side, referenced earlier, was not due to a filter leakage but rather some other problem in the filter vessel for which we have not been given the details.

Figure 11. Pressure drop and filter efficiency for filter sample 45-13-10, section 13

Figure 12. Pressure drop and filter efficiency for filter sample 45-13-10, section 14

3.4.3 Other Characterization Results

The following comments summarize observations on filters both during removal from the Tidd filter vessel and later in the laboratory before the filters were cut up for testing.

There was a fairly uniform layer of ash about 6 mm thick on filters surface. The ash on the filters in the bottom cluster was friable, whereas much of the ash around the filters in the top cluster was very hard and unfriable. This also was evidenced during removal. The bottom filters slid out of the mounts easily, but the top mounts had to be pried open to remove the filters. The technician not only used a pry bar to open the mount but forcibly moved the filter laterally to help loosen the mount. As a result, the flanges on several filters were cracked.

The top filters all showed weight increases, and balancing indicated they were bottom heavy. Filters 45-18-02 and 44-10-03 had ash filling the bottom 12.7 cm (5 inches) of the closed end of the filter. Westinghouse personnel informed us this

was not due to a failure of the 3M filters but was due to a problem elsewhere in the filter system. When 44-10-03 filter was later sectioned at 3M a "tree ring" pattern was readily observable and a small hole down the center of the ash plug in the bottom several inches of the filter revealed how the ash had come from the clean side and built up on the walls with each additional cleaning pulse. Microscopic observation of the cross section showed ash packed up on the interior as described above and a normal ash cake on the exterior. Within the filter layer there was a small amount of fine ash on the exterior and interior with the center of the filter layer being the cleanest. This would seem to confirm ash approaching the filter layer from both sides. Filter 45-18-02 had a 10 to 13 cm (4 to 5 inch) long vertical split in the side of the filter at the bottom where the plug of ash material was located. It had apparently been damaged by the temperature variations and contraction and expansion of the ash plug inside. The positive implication of this crack caused by the ash was that the filter had completely remained intact without any catastrophic failure. This lack of catastrophic failure is one of the key benefits of using the composite construction for filters. The filter mounting gaskets were clean and white at the seal points.

The bottom cluster filters showed only a normal weight increase proportional to the ash layer on the exterior. The filter interiors were clean. The filter mounting gaskets were clean and white at the seal points. Filter 44-11-06 in the bottom cluster was slightly damaged at the closed end tip. In an approximately 3 cm diameter circle, some of the outer mesh braid had been lost. The surface fibers in this area around the damage were white in color, soft and flexible. The white indicates a loss of SiC in that area. The substrate material underneath still retained its strength.

Rechecking process records it was found that the last few inches of the closed ends of this lot of filters were probably only lightly coated with SiC or off stoichiometric SiC during the final CVD step. As a result, the thin outer layer was easily oxidized or sloughed off the exterior closed end. Changes in the process have eliminated the uneven coating problems experienced in earlier small size batches. The substrate layer which provides the mechanical strength was not affected by this problem.

Some effect of the formation of the glassy SiO₂ protective surface on top of the SiC coating can be seen in the SEM photomicrograph of Figure 13 in the smoothing

Figure 13. SEM of outside edge of outer mesh on exposed filter 45-17-03

and rounding of the normal nodular like pattern of the SiC surface. That natural SiO₂ protective layer can react with some of the components of the atmosphere and ash. In such a reaction, SiO₂ is slowly removed and SiO₂ is regenerated from the remaining SiC. This iterative process removes the SiC layer. This effect, if present, has the greatest impact on the outer mesh braid and the filter layer. These areas of the filter have the thinnest CVD coatings. Several filters had especially thin coatings on the last few inches of the closed end. In contrast, the substrate or mechanical layer has about ten times the SiC thickness of the outer layers. Thus, its mechanical properties are not degraded significantly. The fibers in all parts of the filter are still completely intact and therefore not causing any property losses. Figures 14 and 15 show areas in the outer mesh and in the middle of the filter layer. Figures 13, 14, and 15 are remnants of exposed filter 45-17-03. These remnants have ash particles on the surface as well as fragments of SiC which resulted from breaking the sample off.

A reference unexposed sample and exposed samples from Tidd filter 45-17-03 were examined by x-ray diffraction to identify crystalline phases. The reference filter sample 94-143-02 section 2 and 45-39-09 section 2 show patterns for Beta SiC and for mullite as is expected with Nextel™ 312 fibers. Two exposed samples

Figure 14. SEM of middle of outer mesh on exposed filter 45-17-03

Figure 15. SEM of middle of filter layer on exposed filter 45-17-03

were examined; one as cut from the exposed filter 45-17-03 and one washed to remove as much of the ash material as possible. The x-ray diffraction results on each layer of the filter construction from both samples showed the same patterns for Beta SiC and for mullite in Nextel™ 312 ceramic fiber as the unexposed samples plus patterns for CaSO₄, Alpha Fe₂O₃, and small amounts of silica and a feldspar material. The patterns for the exposed as cut sample and the washed sample were the same but with lower intensity peaks for the ash material on the washed sample. No evidence of any changes from the Tidd exposure could be seen in the ceramic composite filter materials via x-ray diffraction.

4.0 SUMMARY

During this contract extension, 110 fiber reinforced ceramic composite filters were fabricated by 3M for tests in demonstration power plant facilities and other tests that become available. The filters passed all quality assurance specifications and were inventoried for Oak Ridge National Laboratory, ORNL. The filters have and are being shipped to various industrial, university, and national laboratory test facilities as requested by ORNL.

Ten ceramic composite filters were installed in the Tidd demonstration power plant filter vessel and operated for 1110 hours in a temperature range of 760°C to 843°C as part of test period #5. 80% of the run was above 815°C. The filters operated at a face velocity of 4.8 cm/sec (9.5 ft/min) and an inlet dust loading of 18000 ppmw.

Characterization tests after the Tidd exposure showed that the filters were in good condition, that they operated well as filters, and that no failures occurred. As has been shown by these and prior mechanical property test results, even though the composites experience an initial decrease in mechanical properties, they maintain more than adequate properties to accomplish their filtration task in the hostile power generation system environment.

5.0 ACKNOWLEDGMENTS

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APPENDIX I

CERAMIC COMPOSITE FILTER TEST METHODOLOGY

Introduction

The test methodologies presented in this document are currently used by the Ceramic Technology Center in the evaluation of 3M brand Ceramic Composite Filters. These tests evolved from the need to characterize the properties of the 3M brand Ceramic Composite Filters. Traditional (monolithic) material property tests (i.e. bend bars or dogbone test) on these sparse-matrix composites yield very inconsistent and inaccurate results. Therefore a test methodology was developed to subject filters to simulated conditions that they would experience in service. Additional test methods are currently under development and as comments are received on the utility, they will continue to evolve.

Sectioning

Standardized sectioning has been established for the 60" candle filter. Sectioning of a 60" candle filter is as shown in Figure 16. From each filter tube, 6 tensile specimens, 2 filtration specimens, 12 O-ring specimens, and 1 flange bend specimen can be obtained by sectioning the tube with a high speed diamond saw. If exposure testing will be done, the tube is sectioned as shown in Figure 16 to allow comparisons to be made between exposed and unexposed specimens. O-ring specimens should have lowest priority during sectioning.

Filtration Testing

Filtration testing is performed using a TSI Automated Filter Tester, model 8110. Four inch long filter tube sections are attached to an aluminum plate (6.5" dia, 1.5" dia. center hole) using RTV silicone. A rubber stopper (attached using RTV silicone) is used to plug the other end of the tube. A schematic of the apparatus is shown in Figure 17. Two types of tests are performed: an initial permeability (flow) and a filtration (loading) test. During the flow test, back pressure is recorded at 10 different flow rates between 10 and 100 liters/min.

60" Candle Filter

Figure 16. Sectioning Diagram for 60" ceramic composite filter for test samples

Figure 17. Schematic of Filtration test

The filtration test utilizes NaCl particles with a mean diameter of 0.1 μm at a concentration of 100 mg/m^3 . The filtration test is performed at a given flow rate of 30 liters/min (face velocity of 3.0 cm/sec for 4" filter tube section). Face velocity equals flow rate (cm^3/sec) divided by surface area (cm^2). Efficiency is calculated by dividing the concentration of particles exiting the filter by the input concentration. Typical efficiency and pressure drop curves are shown in Figure 18.

Maximum Pore Diameter and Permeability

These tests are derived from ASTM E 128-94 entitled "Standard Test Method for Maximum Pore Diameter and Permeability of Rigid Porous Filters for Laboratory Use."

Mechanical Testing

Tensile Testing

In this test, the maximum axial load bearing capacity of a filter is determined. Both as-fabricated and environmentally-exposed filters are evaluated to help establish service criteria and predict filter lifetime.

Specimens are fabricated by attaching standard pipe reducer fittings to the ends of a 5" section of tube using Scotch-Weld™ 1838-L B/A epoxy from 3M. Masking tape is wrapped around the cut end of the tube to contain the low viscosity epoxy and also over the small end of the reducer fitting to prevent it from adhering to the inside threads. The second end is fabricated by attaching a standard pipe reducer fitting into the opposite end of the 5" section of the tube using the same procedure. Fixtures are then screwed into the fittings and attached to the test frame and load cell using orthogonally-oriented pins. Such a configuration minimizes non-axial loading, since the tolerance between the reducer fitting and the tube is small. A schematic of the test specimen is shown in Figure 19. A crosshead speed of 2.0 mm/min is used.

The tensile strengths are calculated using

$$\sigma_{\text{tensile}} = \frac{F}{\pi(r_o^2 - r_i^2)},$$

Figure 18. Example of filtration test performed on 4" candle filter section

Figure 19. Tensile test specimen and fixture

where F is the fracture load (in Newtons), and r_o and r_i are the outer and inner tube radii, respectively. Modulus values are determined using the above stress formula in conjunction with the measured displacement and the gage length (5 inches).

Example load-displacement curves for 5" candle filter tubes are shown in Figure 20.

O-Ring Diametral Compression Testing

The O-ring test is used as an effective tool for monitoring process or service variables which influence composite behavior.

O-ring testing is performed by diametrically compressing a 1" section of a filter tube or a substrate, as shown schematically in Figure 21. The 3M brand Ceramic Composite Filter is treated as a thin-wall cylinder (i.e. the assumption of constant stress across the wall results in negligible error) since its internal diameter-to-thickness ratio is greater than 10 (30)¹. The equation used to calculate the maximum stress in an O-ring is that for a thin-walled cylinder and is given by²

$$\sigma_{O-Ring} = \frac{2Fr_a (6r_a - t)}{\pi dt^2 (2r_a - t)},$$

where r_a is the average radius ($= [r_o + r_i] / 2$), t is the wall thickness, and d is the specimen width. A crosshead speed of 4.0 mm/min is used and specimens are tested to 5 mm total displacement.

Representative load-displacement curves for 1" wide O-rings are shown in Figure 22.

O-Ring Burst Testing

Two types of burst tests can be performed. In the first test, an expandable rubber bladder is placed inside a 1" wide section of tube and is pressurized with hydraulic oil until the ring fails. A peak pressure is recorded.

Figure 20. Representative load-displacement curves for tensile tests of filters

Figure 21. Schematic of O-ring diametral compression test

Figure 22. Representative load-displacement curves for O-ring compression tests of filters

The equation for determining the hoop stress in an internally pressurized thick-walled tube is given by³

$$\sigma_{hoop} = p \frac{r_i^2(r_o^2 + r^2)}{r^2(r_o^2 - r_i^2)},$$

where p is the applied internal pressure and r is a position between r_o and r_i . For a thick-walled tube, the maximum stress occurs at $r = r_i$ and the hoop stress equation reduces to

$$\sigma_{hoop} = p \frac{(r_o^2 + r_i^2)}{(r_o^2 - r_i^2)}.$$

A second type of burst test can be performed on various length segments of a filter tube (typically from 1" to 6"). Test specimens are prepared by placing an elastomeric cylinder inside a section of filter. Testing involves axially compressing the elastomer with two metallic end plugs to provide a uniform tensile hoop (circumferential) stress on the tube. Stresses are determined by equating the radial strain for the rubber and the tube at their interface ($r=r_i$). A schematic of the test is shown in Figure 23.

Figure 23. Schematic of burst test

The pressure exerted by the rubber on the tube, is given by⁴

$$p' = \frac{v_r q}{1 - v_r} = \frac{v_r P}{\pi r_i^2 (1 - v_r)},$$

where v_r is Poisson's ratio of the rubber and q = the applied pressure = P/A , where P is the applied load and A is the cross-sectional area of the plug. The last equation is simplified because of the low elastic modulus of the rubber compared to that of the ceramic composite.

For a thin walled tube, the hoop stress equation is even simpler and is given by⁵

$$\sigma_{hoop} = \frac{p' r_a}{t}.$$

Substituting typical values of r_o and r_i for the 3M brand Ceramic Composite Filter, the values obtained for the maximum hoop stress by using the thick walled equation versus the thin-walled equation are $16.5P$ and $16P$, respectively. (A difference of 3%.)

A representative load-displacement curve obtained during a burst test is shown in Figure 24.

Flange Bend Testing

Flange bend testing is performed to simulate the loads a filter tube would experience due to ash bridging in service. Specimens are fabricated using a procedure similar to that described above for fabricating tensile test specimens. A standard pipe reducer fitting is attached to the end of the 7.5" flanged tube section using Scotch-Weld™ 1838-L B/A epoxy from 3M. Masking tape is wrapped around the cut end of the tube to contain the low viscosity epoxy and also over the small end of the reducer fitting to prevent it from adhering to the inside threads. The flange bend specimen is mounted in the flange fixture using the same procedure that is used for mounting filter tubes into tube sheets. The loading fixture is then screwed into the reducer fitting and oriented perpendicular to the loading rod. A schematic of a flange test sample and test fixture is shown in Figure 25. A crosshead speed of 4.0 mm/min is typically used. Since failure is somewhat arbitrary, tests are usually halted after 30 to 40 mm of displacement.

Figure 24. Load-displacement curves for burst test on a 1" section of filter

Figure 25. Flange bend test specimen and fixture

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Table 4

**Tidd II, Baseline
Filter 45-13-01**

O-Ring Compression Tests

Specimen Number	Wt. grams	Outer Diameter cm	Wall Thickness cm	Width cm	r avg cm	Peak Load kg	O-Ring Compression Strength MPa	Stiffness Kg/mm	Energy to Peak Load Kg-mm	Total Energy Kg-mm
3	14.50	5.75	0.368	2.49	2.69	6.55	10.3	12.88	4.20	18.40
4	15.03	5.75	0.348	2.57	2.70	7.71	13.1	16.11	6.26	23.44
5	14.08	5.75	0.348	2.43	2.70	7.10	12.8	9.99	4.76	19.97
8	14.58	5.72	0.348	2.48	2.69	6.54	11.4	11.02	4.20	22.08
9	14.97	5.74	0.358	2.53	2.69	6.88	11.2	10.50	4.41	23.12
10	14.98	5.74	0.358	2.50	2.69	6.00	9.9	9.67	2.81	21.69
18	15.00	5.74	0.373	2.51	2.68	7.00	10.6	12.62	3.15	17.17
19	15.24	5.82	0.381	2.56	2.72	6.62	9.5	7.94	7.70	19.29
Avg.	14.80	5.75	0.360	2.51	2.70	6.80	11.09	11.34	4.69	20.65
Std. Dev.	0.38	0.03	0.013	0.05	0.01	0.50	1.32	2.50	1.60	2.28
COV	2.56%	0.49%	3.52%	1.82%	0.42%	7.39%	11.86%	22.04%	34.21%	11.06%

Table 5

Tidd II Baseline Tensile
Preform # 45-13-01
Tensile Tests

Specimen Number	Outer Diameter cm	Average Wall Thickness cm	Width cm	r avg cm	r inner cm	Cross Sectional Area cm²	Peak Load Kg	Peak Stress MPa	Elongation at Peak Load mm	Stiffness Kg/mm
flange			8.57							
2	5.38	0.37	12.70	2.51	2.32	5.82	890	14.98	0.82	1549
7	5.38	0.37	12.70	2.51	2.32	5.82	1120	18.85	0.93	1434
12	5.38	0.37	12.70	2.51	2.32	5.82	1342	22.58	1.04	1959
15	5.38	0.37	12.70	2.51	2.32	5.82	1358	22.85	0.56	2747
17	5.38	0.37	12.70	2.51	2.32	5.82	1306	21.98	0.84	2318
20	5.38	0.37	12.70	2.51	2.32	5.82	1225	20.61	0.80	2477
Avg.							1207	20.31	0.83	2081
Std. Dev.							178	3.00	0.16	524
COV							14.79%	14.79%	19.28%	25.19%

Table 6

Tidd II, Baseline
Filter 45-13-01

Burst Tests

Burst Test Results

Specimen Number	Outer Diameter cm	Measured Wall Thickness cm	Width cm	r avg cm	r inner cm	Cross Sectional Area cm ²	Bulk Volume cm ³	Peak Applied Load Papp kg	p'	Hoop Strength MPa	Stiffness Kg/mm	Elongation at Peak Load mm	Energy to Peak Load Kg-mm
6	5.74	0.34	2.55	2.70	2.53	5.77	14.71	255.58	6.18	49.0	419.77	2.73	139.7
11	5.78	0.36	2.58	2.71	2.53	6.13	15.82	284.78	6.88	51.8	430.58	2.78	157.5
16	5.74	0.35	2.54	2.70	2.52	5.93	15.05	291.94	7.11	54.7	441.27	2.71	152.8
Avg.	5.75	0.35	2.56	2.70	2.53	5.94	15.19	277.43	6.72	51.86	430.54	2.74	150.00
Std. Dev.	0.02	0.01	0.02	0.01	0.01	0.18	0.57	19.26	0.49	2.85	10.75	0.04	9.22
COV	0.40%	2.86%	0.81%	0.28%	0.23%	3.05%	3.73%	6.94%	7.25%	5.50%	2.50%	1.32%	6.15%

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0.327

Table 7

**Tidd II, Exposed
Filter 44-11-06**

O-Ring Compression Tests

Specimen Number	Outer Diameter cm	Wall Thickness cm	Width cm	r avg cm	Peak Load kg	O-Ring Compression Strength MPa	Stiffness Kg/mm	Energy to Peak Load Kg-mm	Total Energy Kg-mm
6	5.87	0.360	2.63	2.76	4.63	7.3	6.37	1.85	6.86
8	5.80	0.394	2.51	2.70	3.61	4.9	5.19	1.31	5.64
10	5.83	0.340	2.49	2.75	4.22	7.9	7.83	1.56	6.72
12	5.80	0.367	2.45	2.72	4.06	6.6	5.13	1.77	7.59
14	5.82	0.384	2.46	2.72	3.63	5.3	4.68	2.2	7.04
16	5.79	0.350	2.43	2.72	4.08	7.3	5.28	2.1	6.18
18	5.79	0.370	2.51	2.71	3.67	5.7	5.73	2.94	6.69
20	5.81	0.375	2.49	2.72	4.09	6.2	4.55	1.86	8.01
22	5.79	0.370	2.59	2.71	4.18	6.3	6.01	3	7.97
24	5.81	0.370	2.74	2.72	4.83	6.9	6.8	2.01	8.1
28	5.85	0.385	2.54	2.73	5.37	7.7	6.93	2.97	7.72
30	5.83	0.370	2.43	2.73	4.58	7.4	5.79	2.44	6.9
32	5.83	0.355	2.54	2.74	5.3	8.9	6.98	2.17	7.69
34	5.84	0.360	2.52	2.74	5.82	9.6	6.87	3.35	8.1
36	5.85	0.365	2.45	2.74	5.38	8.9	5.66	2.69	7.57
38	5.81	0.380	2.47	2.72	4.5	6.7	5.47	1.9	7.83
40	5.82	0.360	2.38	2.73	4.73	8.2	5	2.26	7.53
42	5.84	0.383	2.43	2.73	3.85	5.8	4.04	1.79	6.56
44	5.87	0.370	2.40	2.75	3.71	6.1	4.15	1.7	7.08
46	5.87	0.385	2.42	2.74	3.29	4.9	3.19	1.87	6.76
Avg.	5.83	0.370	2.49	2.73	4.38	6.93	5.58	2.19	7.23
Std. Dev	0.03	0.013	0.09	0.01	0.70	1.32	1.16	0.55	0.69
COV	0.46%	3.60%	3.43%	0.54%	15.95%	19.05%	20.82%	25.02%	9.51%
				Retained	64.36%	62.43%	49.22%	46.67%	35.01%
				(compared to Unexposed)					

Tensile Filter Test

Specimen Number	Outer Diameter cm	Average Wall Thickness cm	Width cm	r avg cm	r inner cm	Cross Sectional Area cm²	Peak Load Kg	Peak Stress MPa	Elongation at Peak Load mm	Modulus GPa	Stiffness Kg/mm
flange			8.57								
4	5.74	0.37	12.80	2.69	2.50	6.24	241.5	3.79	0.237	7.2	1319
5	5.74	0.37	12.70	2.69	2.50	6.24	296.0	4.65	0.473	6.3	1156
7	5.74	0.37	12.80	2.69	2.50	6.24	402.4	6.32	0.448	6.7	1215
10	5.74	0.37	12.80	2.69	2.50	6.24	271.1	4.26	0.475	7.7	1410
11	5.78	0.37	12.90	2.71	2.52	6.29	180.6	2.81	0.274	4.6	832
13	5.78	0.37	12.90	2.71	2.52	6.29	184.9	2.88	0.24	5.6	1018
14	5.75	0.37	12.60	2.69	2.51	6.25	206.9	3.24	0.356	1.9	910
Avg.	5.75	0.370	12.79	2.69	2.51	6.26	254.77	4.0	0.36	5.71	1123
Std. Dev.	0.02	0.000	0.11	0.01	0.01	0.02	78.12	1.2	0.11	1.97	213
COV	0.33%	0.00%	0.84%	0.35%	0.38%	0.35%	30.66%	30.89%	30.34%	34.46%	18.94%

Table 9

Tidd II, Exposed
Filter 44-11-06

Burst Tests

Burst Test Results

Specimen Number	Outer Diameter cm	Measured Wall Thickness cm	Width cm	r avg cm	r Inner cm	Cross Sectional Area cm ²	Bulk Volume cm ³	Peak Applied Load P _{app} kg	p'	Hoop Strength MPa	Stiffness Kg/mm	Elongation at Peak Load mm	Energy to Peak Load Kg-mm
5	5.76	0.360	2.66	2.70	2.52	6.11	16.25	57.71	1.34	10.1	80.66	1.55	36.90
7	5.75	0.391	2.58	2.68	2.48	6.58	16.98	87.60	2.10	14.4	137.98	2.05	61.50
9	5.80	0.360	2.53	2.72	2.54	6.15	15.57	76.72	1.76	13.3	139.28	2.00	54.50
11	5.76	0.350	2.49	2.71	2.53	5.95	14.81	86.61	2.00	15.5	137.62	2.14	64.50
13	5.76	0.380	2.50	2.69	2.50	6.42	16.06	89.99	2.13	15.1	129.60	2.20	67.60
15	5.81	0.360	2.52	2.73	2.55	6.16	15.53	79.02	1.81	13.7	109.62	2.17	64.70
17	5.79	0.365	2.47	2.71	2.53	6.22	15.37	86.78	2.01	14.9	106.16	2.30	73.40
19	5.84	0.350	2.54	2.75	2.57	6.04	15.33	71.78	1.61	12.6	71.31	2.26	68.50
21	5.80	0.360	2.54	2.72	2.54	6.15	15.63	107.23	2.46	18.6	161.63	2.53	87.90
23	5.81	0.360	2.47	2.73	2.55	6.16	15.22	110.74	2.53	19.2	175.65	2.58	91.30
25	5.82	0.380	1.85	2.72	2.53	6.49	12.01	75.90	1.75	12.6	78.24	2.42	76.70
27	5.83	0.370	2.49	2.73	2.55	6.35	15.80	97.30	2.22	16.4	125.03	2.68	95.50
29	5.82	0.380	2.45	2.72	2.53	6.49	15.91	103.27	2.39	17.1	136.24	2.69	98.60
31	5.82	0.370	2.51	2.73	2.54	6.34	15.90	106.58	2.44	18.0	128.27	2.82	107.10
33	5.88	0.380	2.49	2.75	2.56	6.57	16.35	92.85	2.10	15.2	103.15	2.68	95.00
35	5.82	0.380	2.39	2.72	2.53	6.49	15.52	103.33	2.39	17.1	143.86	2.59	93.70
37	5.85	0.380	2.50	2.74	2.55	6.53	16.33	109.57	2.50	18.0	176.09	2.52	89.80
39	5.80	0.360	2.44	2.72	2.54	6.15	15.01	121.25	2.78	21.0	179.94	2.54	95.90
41	5.82	0.375	2.38	2.72	2.54	6.41	15.27	68.83	1.59	11.5	54.38	2.27	71.10
43	5.85	0.375	2.40	2.74	2.55	6.45	15.48	104.53	2.38	17.4	172.49	2.30	76.40
45	5.87	0.380	2.44	2.75	2.56	6.55	15.99	134.65	3.05	22.1	211.25	2.44	93.20
Avg.	5.81	0.37	2.46	2.72	2.54	6.32	15.54	93.92	2.16	15.88	131.35	2.37	79.23
Std. Dev.	0.04	0.01	0.15	0.02	0.02	0.20	0.95	18.72	0.42	3.06	40.32	0.29	17.59
COV	0.61%	3.09%	6.25%	0.64%	0.74%	3.09%	6.13%	19.93%	19.57%	19.27%	30.70%	12.45%	22.21%
Poisson's of RTV 0.32								Retained (compared to Unexposed)		30.63%	30.51%	86.43%	52.82%

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