4. CONCLUSIONS

TASK 3

1. Under the initial objectives of Task 3, product modifications were conducted and evaluated on the "baseline" PRD-66 Hot Gas Filter.

   - Filters were produced which had lower backpressure, good membrane adhesion and a stronger flange region.
   - These filters passed permeability and "particle collection efficiency" tests conducted by Westinghouse Science and Technology Center (W-STC).
   - Strength characterization of the filter material, conducted by W-STC and by DLC, deemed PRD-66 to have sufficient strength for PFBC applications.
   - The feasibility of producing a wound ("yarn only") membrane on the inside diameter of the filter was demonstrated.

2. Independent field trials of the "baseline" PRD-66 filter, at American Electric Power's Tidd Facility, suggested that inadequacies existed in the membrane and the underlying support wall. These problems would not have been corrected by the modifications under evaluation at that time. More radical changes were required and evaluated.

3. Modifications to the alumina slurry composition were effective at reducing the interlaminar voids within the wall of the filter element.

4. A new DLC lab-scale test procedure (PIT) was capable of evaluating the membrane integrity of 2" long specimens at room temperature. Once it was possible to differentiate between "good" membranes and "poor" membranes, membrane experiments could be conducted.

5. A preferred membrane construction, which combined a wound slurry-coated yarn and a larger particulate alumina, produced the best combination of good surface filtration and low backpressure.

6. The preferred membrane construction was fine-tuned, and two types were selected for continued evaluation.

   - PRD-66C - nominal 25μ pore size
   - PRD-66M - nominal 10.5 μ pore size

7. Both PRD-66C and PRD-66M Hot Gas Filters successfully passed high temperature and high pressure (HTHP) tests conducted by Westinghouse.

8. PRD-66C was evaluated in pressurized circulating fluidized bed (PCFBC) conditions in Foster Wheeler's Karhula facility.

   - Throughout the testing, no in-process failures, no delaminations, no cracking, and no "divots" occurred.

   - Examination of the cross-section of exposed filters confirmed that the elements had provided effective surface filtration.

   - Exposed filters proved to be both chemically and physically stable, as determined by evaluating strength, composition, and microstructure.

TASK 4

1. A raw materials plan was completed which found that the quality assurance provided by our suppliers was adequate for the needs of PRD-66 filter manufacturing.

2. All critical in-process instrumentation and calibration procedures were reviewed; improvements were implemented where necessary.

3. An analysis of process sensitivity, as it related to the WINDING OF THE FILTER, was conducted at the extremes of the normal process limits.

   - Product quality was stable within normal process limits except for a slight decrease in alumina pickup when the slurry viscosity was very low. The "low-viscosity limit" was raised.

   - Winding interruptions of less than fifteen minutes had no impact on product quality, unless the relative humidity of the winding environment fell below the normal process limit. This allowable "window" makes it possible to use "short" bobbins of feed yarn without risk to the quality of the filter.

4. An analysis of process sensitivity, as it related to the fabrication of the filter membrane, was conducted at the extremes of the normal process limits.

   - Slightly higher amounts of fusible binder improved the adhesion of the Type-C particulate membrane.

- The backpressure of the filter was insensitive to normal variations in amount of particulate membrane applied.
- Cracks in membrane occasionally resulted where the particulate membrane was noticeable "too thick".
- A few extremely fine cracks in the Type-M membrane were common in most PRD-66M filters, when examined in transmitted light.

5. A reasonable 70% yield was demonstrated during a process capability run of thirty filters made to the specifications required by the Westinghouse Advanced Particulate Filtration System.
    - A variety of equipment modifications were implemented throughout the "capability demo" which improved processability, including different mandrel designs and a different filter cutting technique.
    - The "length of the flange" and the "inside diameter of the flange" were the most difficult specifications to meet.
    - The equipment utilization was well below expectations due to a high level of maintenance and repair required for the prototype winders.

GENERAL

Inherent thermal shock resistance and low cost raw materials made PRD-66 a promising candidate for a hot gas filtration applications, but the support and funding provided by FETC enabled the modifications required to create a product which was far-superior to the "baseline" candidate.

5. RECOMMENDATIONS

1. Prototype winding equipment should be redesigned specifically for fabricating hot gas filters.
   - The support winder needs to be more reliable for demanding production schedules.
   - The winding of the hoop membrane should be incorporated into the support winder to improve product quality.
   - The winding of the flange should be incorporated into the same winding unit to streamline the entire filter winding process.
   - A manufacturing capability run of 50 filters should be performed using the modified winding equipment and the results compared with data generated with the old prototype winder.
2. Since ash contamination from the "clean side" can limit the useful life of a filter element, a more extensive study of the feasibility of adding an inside membrane should be pursued.
   - A simple lab-scale test needs to be developed to challenge the integrity of an ID membrane
   - Original methods for winding an ID membrane (Task 3.1) need to be reevaluated using the capabilities of the redesigned prototype winder.
   - Determine if the ID wound membrane would provide effective protection from unexpected ash contamination without causing an unacceptable increase in backpressure.
3. From the filters produced on the modified winder, specific units should be subjected to destructive testing by ACI and SRI. An evaluation of product reproducibility and NORMAL variations will be essential in evaluating the impact of exposure.
4. Additional PFBC field experience is necessary to determine their long-term potential.
5. Since the type-M membrane may be preferable for systems with a finer ash, modifications should be evaluated to eliminate the membrane cracks.

6. ACKNOWLEDGMENTS

The authors and AlliedSignal Composites Inc. gratefully acknowledge the contributions of several individuals whose support has been vital to this program.

We appreciate the technical guidance and assistance of Ted McMahon, our DOE-FETC COR, as well as his support, throughout these tasks.

Prior to the initiation of this contract, and throughout the duration of Task 3, Mary Anne Alvin of Westinghouse Science and Technology Center made significant contributions to our understanding of the field performance of our products, and provided valuable feedback on our product modifications.

During the failure analysis of the Tidd-5 filters, we were extremely fortunate to have technical assistance from Rich Dennis and Dwayne Smith of FETC, Tom Lippert and Rich Newby of Westinghouse Science and Technology Center, Tina Watne and John Holmes of UND's Energy and Environmental Research Center, and Dick Tressler of Penn State. Their knowledge, experience and creativity provided an excellent foundation for the work in this task.

We are also grateful for the assistance of Juhani Isaksson, Reijo Kuivalainen & Timo Eriksson, of Foster Wheeler Energia Oy. The exposure of our material at their Karhula R&D Center, was PRD-66C's first field trial, and their input was critical to understanding our product's performance.

7. REFERENCES

1. M.A. Alvin, R.E. Tressler, T.E. Lippert, and E.S. Diaz, "Evaluation of Ceramic Filter Material, Selection for Application," *Proceedings of the Coal-Fired Power Systems '93 - Advances in IGCC and PFBC Review Meeting.* DOE/METC-93/6131 (DE93000289) June 1993.

2. M.A. Alvin, T.E. Lippert, E.S. Diaz, and E.E. Smeltzer, "Filter Component Assessment," *Proceedings of the Advanced Coal-Fired Power Systems '95 Review Meeting - Volume I.* DOE/METC-95/1018 (DE95009732) June 1995.

3. M.A. Alvin, "Filter Component Assessment," *Monthly Status Report, December '96,* DOE DE-AC21-94MC31147 December 1996.

4. M.A. Alvin, "Advanced Ceramic Materials for Use in High Temperature Particulate Removal Systems," *Ind. Eng. Chem. Res.* 1996, 35, 3384-3398.

5. M.A. Alvin, T.E. Lippert, E.S. Diaz, E.E. Smeltzer and R.E. Tressler, "Durability of Ceramic Filters," *Proceedings of the Advanced Coal-Fired Power Systems '94 - Advances in IGCC and PFBC Review Meeting,* 545-571, DOE/METC-94/1008.

6. M.A. Alvin, T.E. Lippert, E.S. Diaz, E.E. Smeltzer, and G.J. Bruck, "Filter Component Assessment," *Proceedings of the Advanced Coal-Fired Power Systems '97 Review Meeting,* Pittsburgh, PA, July 22-24, 1997.

7. J.A. Chambers, "Develpment and Testing of PRD-66 Hot Gas Filters," *Proceedings of the Advanced Coal-Fired Power Systems '95 Review Meeting - Volume II.* DOE/METC-95/1018 (DE95009732) June 1995.

8. J.A. Chambers, "Performance of PRD-66 Hot Gas Candle Filters in the AEP/Tidd PFBC Facility," *Proceedings of the Advanced Coal-Fired Power Systems '96 Review Meeting.* July 16-18, 1996.

9. M. Davidson, E. Galloway, X. Guan, H. Hendrix, R. Read, P. Scarborough, R. Dahlin, and C. Landham, "Combustion Testing of the Westinghouse Advanced Particle Filter at the Power Systems Development Faciility (PSDF)," *Symposium on High-Temperature Particlulate Cleanup for Advanced Coal-Based Power Systems*, Birmingham, AL, April 20-23, 1998.

10. O.M. Jadaan, D.L. Shelleman, J.C. Conway Jr., J.J. Mecholsky Jr., and R.E. Tressler, "Prediction of the Strength of Ceramic Tubular Components: Part I - Analysis, " *Journal of Testing and Evaluation*, JTEVA, Vol. 19, No. .3, May 1991, pp. 181-191.

11. ASTM Designation: C 1239-93, Standard Practice for Reporting Uniaxial Strength Data and Estimating Weibull Distribution Parameters for Advanced Ceramics, Published June 1993.

12. M.A. Alvin, "Advanced Hot Gas Filter Development:", *Summary Report, March 31, 1998,* DuPont/DOE FETC Contract No. DE-AC21-94MC31214A, see Appendix 2.

Appendix 1

DLC Raw Materials Specification Form

STANDARD OPERATING PROCEDURE

Effective Date:
July 1, 1996
Rev. A EO: 4

SUBJECT: Material Specifications

PURPOSE: To document the procedure to develop and update Material Specifications for Essential Materials to be purchased

EXPLANATION OF CHANGE: ORIGINAL ISSUE

AUTHORIZED BY:*

Manufacturing Manager (DLC) Gene Mathis/s/ Date: 6/10/96

Business Manager (DLC) Gary Knox/s/ Date: 6/14/96

Technical Manager (DLC) Aspi Patel/s/ Date: 6/5/96

Purchasing Mgr (Lanxide Corp) Debbie Facciolo/s/ Date: 6/11/96

* Electronic EO signatures on file in the TPN Fileserver; paper EO signatures on file with the EO Coordinator

DU PONT LANXIDE COMPOSITES INC.
1300 MARROWS ROAD
NEWARK, DELAWARE 19714

Material Specifications

1. SCOPE

1.1 Purpose

1.1.1 This document establishes the content and administration of Material Specifications for Du Pont Lanxide Composites Inc. (DLC).

1.2 Applicability

1.2.1 This procedure applies to all goods and services that are Essential Materials for DLC products sold to customers. This SOP **does not** apply to materials bought for internally-funded experiments and conceptual development.

1.3 Terminology

1.3.1 An Essential Material is any material (including tooling) that directly impacts product quality and that cannot be changed without affecting plant performance, customer-use requirements, or product quality.

1.3.2 Quality Manual Section 3.0 (Terms and Definitions) contains definitions of other terms used in this document.

1.4 Auditing

1.4.1 The Management Representative will audit this SOP at least once a year.

2. REFERENCES

2.1 Quality Manual Sections 3.0 (Terms and Definitions) and 8.0 (Quality in Procurement),

2.2 SOP DLC-7.1, Document Control

2.3 SOP DLC-8.1, Purchase of Goods and Services

2.4 SOP DLC-11.1, Material Receiving Inspection

3. RESPONSIBILITIES

3.1 The **Project Engineer** (or equivalent responsibility) is responsible to develop a Material Specification (MS) for each new Essential Material to be bought and used to make a product sold to a customer.

3.2 The **Project Engineer** is also responsible to make sure the MS is kept up-to-date during the production life of the product. As part of the set-up for a new or revised material, the **Project Engineer** also completes a new Material Receipt Inspection Log in the TPN Fileserver (SOP DLC-11.1, Material Receiving Inspection).

3.3 The **requisitioner** of an EssentialMaterial will:

- print and attach a copy of the MS to each "Purchase Requisition/Blanket Order Release" form submitted to Lanxide Purchasing to buy the respective Essential
- attach a copy of the Material Safety Data Sheet (MSDS) to a Purchase Order whenever the MS references an MSDS (if DLC does not have an MSDS on file, the **requisitioner** requests one from the supplier)
- list such items as Certificates of Analysis or Conformance as deliverable items on the Purchase Requisition.

4. PROCEDURE

4.1 Attachment 1 is a template for the contents of each MS. The MS will be generated and kept in the "Material Specification" database on the TPN Fileserver.

4.2 Attachment 2 lists the Quality Assurance Codes which print their respective statements on a printed MS when specified in the database.

4.3 The Engineering Order (E.O.) form is the mechanism to approve new or revised MSs (ref.: SOP DLC-7.1, Document Control)

4.4 The Quality Plan for each Control Level 1 product will specify Essential Materials and will reference the MS numbers.

Attachment 1

Material Specification (MS) Content

1. Material
   Application
   Chemical Formula: (if applicable)
   MS Number and Revision No.
   DLC Part No
   DuPont MS replaced (if applicable)

2. Approved Supplier(s)
   Addresses
   Supplier's phone number
   Supplier's Part No:

3. Physical Specifications:

   Dimensions:
   Weights:
   Workmanship Standards:
   Materials:
   Material Lot Numbers:
   Drawing Numbers
   Other (Thermal specifications, Conductivity, etc.):

4. Yarn/Fabric/Prepreg Specifications

Property	Units	Aim	Lower Limit	Upper Limit

   Other Specifications

5. Chemical Specifications: (if applicable)

Property	Units	Aim	Lower Limit	Upper Limit	Test Method

   Appearance:
   Chemical Identification Method:
   Other:

6. Packaging:

   Container Type:
   Container Material:
   Container Size:
   Container Labeling:
   Other Packaging Info:

Attachment 1 (Cont.)

7. Acceptance/Rejection

Lot Size:
Inspection/Test
Inspection/Test Method
Decision Criteria ("Accept If"):

8. Safety, Health, and Environmental Information:

Hazardous Material: Yes ____ No ____
MSDS No._Rev Date: ______
Is this, or does this contain, an ozone-depleting substance: Yes_No___
DOT Reg.: (if applicable)

9. Handling, Storage, Preservation and Disposal Information:

Expiration Date, if any
Handling Requirements:
Storage Requirements:
Disposal Requirements:
Shipping Requirements:

10. Quality Assurance Requirements: ____, ____, ____, ____

(Inserts appropriate paragraph to match QA codes entered. Nothing will be printed if Code "00"" is entered—a "required entry" field))

Key Characteristics (if any - to accompany Code #15)
Other Quality Requirements

11. Pertinent Infomration

Applicable Documentation

12. Other Information: (e.g., minimum order quantity...)

13. Revision History

Revision Date:
MS Change
EO Number:
Author:

Attachment 2

Quality Assurance Codes

Code	Description
00	No Extra Quality Systems Requirements
	(None printed—the "default" required entry)
01	Certificate of Conformance
	The supplier shall submit a Certificate of Conformance with each shipment that is signed by an authorized supplier's representative and states that the materials supplied to Du Pont Lanxide Composites are in conformance with applicable requirements of the contract, drawings, and specifications and that supporting documentation is on file and will be made available to Du Pont Lanxide Composites, Du Pont Lanxide Composites' Customer, or Government representatives upon request. The Certificate of Conformance must include: Du Pont Lanxide Composites part number, purchase order number, revision level, quantity, and any exceptions to specification or purchase requisition requirements.
02	Certificate of Analysis
	The supplier shall submit a Certificate of Analysis with each supplier's material lot in each shipment that is signed by an authorized supplier's representative and states that each property value contained was the result of a valid laboratory test or analysis. The Certificate of Analysis must include: Du Pont Lanxide Composites' part number, purchase order number revision level, manufacturer's lot number, manufacturer's lot production date, analyses and test values, corresponding analysis or test method number (including reference to ASTM or equivalent standard method).
03	Receiving Inspection at Du Pont Lanxide Composites
	Items purchased under this purchase order are subject to incoming inspection and final acceptance at the Du Pont Lanxide Composites facility named on the purchase order.
04	Du Pont Lanxide Composites Inspection at the Supplier's Facility
	Du Pont Lanxide Composites source inspection is required before shipment of items from your facility. Notify Lanxide Corporation buyer (agent for Du Pont Lanxide Composites) at least three (3) working days before the scheduled date of shipment from your facility.
05	Government Inspection at the Supplier's Facility

Government inspection is required before the shipment of this item. Upon receipt of this purchase order, promptly notify the Government Representative who normally services your plant to plan appropriately for Government inspection. If not, notify the nearest Defense Supply Agency Inspection office in your area.

06 Customer Inspection at the Supplier's Facility

Inspection by Du Pont Lanxide Composites' is required before the shipment of this item. Notify Lanxide Corporation buyer (agent for Du Pont Lanxide Composites) at least five (5) working days before the scheduled date of shipment from your facility.

07 Dimensional Inspection Report

Dimensional inspection data for all drawing attributes shall be included in an Inspection Report on all items delivered under this purchase order. This report shall reference part number, revision level, serial number (if applicable) and the purchase order number. This report will be shipped with the material, else the material will be rejected by receiving inspection and may be returned at the supplier's expense.

08 Special Process Certification

The supplier shall have records of any special process(es) he is qualified/certified to perform available for review by Du Pont Lanxide Composites personnel. Examples of special processes are: cleaning, welding, plating, soldering, and non-destructive testing. The supplier shall identify any sub-tier suppliers that perform special processes and supply this information to Du Pont Lanxide Composites with each shipment.

09 Approval of Inspection Procedures

The supplier shall provide a detailed inspection procedure that describes the inspections to be performed, where they occur in the manufacturing cycle, and the equipment to be used. These procedures are subject to Du Pont Lanxide Composites' approval before starting actual work.

10 Approval of Test Procedures

The supplier shall provide a detailed test procedure that describes the tests to be performed, test methods, test equipment and environment, and the sequence of testing and test data requirements. These procedures are subject to Du Pont Lanxide Composites' approval before starting actual work.

11 Customer Witness

A representative of Du Pont Lanxide Composites' customer may witness any inspection or test without affecting Du Pont Lanxide Composites' exclusive right to give direction to the supplier or to accept or reject any procedure, test data, or item.

12 Government Witness

A Government representative may witness any inspection or test without affecting Du Pont Lanxide Composites' exclusive right to give direction to the supplier or to accept or reject any procedure, test data, or item.

13 Written Approval for Changes

The supplier shall notify Du Pont Lanxide Composites of any changes in design, fabrication methods, or processes and obtain Du Pont Lanxide Composites' written approval before making the changes.

14 Reporting of Test Data

All test data shall be reported in the correct format: either 1) "variables" format when the test method produces data on a continuous numeric scale, or 2) "attribute" format for such counted data and defects or "pass/fail". In addition to the lot average data, the sample standard deviation(s) and Sample size are to be reported for each characteristic. If multiple test replicates are run on product samples from the same lot, portion average will be used for the lot average (use as single data point) and not each individual replicate.

15 Key Characteristics

Key Characteristics (those specified in the Purchase Order or Material Specifications) of product supplied must have a minimum process capability, Cpk, of 1.0 with a 90% confidence level (this translates into Cpk of 1.30 minimum for a sample size of 20 data points to a Cpk of 1.07 for sample sizes of 250 data points). This process capability shall be substantiated by process capability calculations on the certifications supplied with the shipment.

16 Material Safety Data Sheet to be Provided

The supplier shall include a copy of the latest Material Safety Data Sheet (MSDS) with the first shipment of each item in this purchase order.

17 Proof of Statistical Control

Supplier shall provide proof of statistical control of key properties. The proof will be in the form of property histograms and control charts for the lot(s) shipped.

Appendix 2

This appendix contains a copy of the Summary Report of work performed by Westinghouse Electric Corporation, Science and Technology Center, under a subcontract of this program.

ADVANCED HOT GAS FILTER DEVELOPMENT

SUMMARY REPORT

M. A. Alvin
March 31, 1998

By

Westinghouse Electric Corporation
Science and Technology Center
1310 Beulah Road
Pittsburgh, PA 15235-5098

Under
Westinghouse Reference No. WL-1 3059-CE
DuPont/DOE FETC Contract No. DE-AC2I-94MC31214A

For

DuPont Lanxide Composites
1300 Marrow Road
P. 0. Box 6077
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ADVANCED HOT GAS FILTER DEVELOPMENT

SUMMARY REPORT

M. A Alvin
March 31, 1998

Abstract

During the past five years, the filament wound DuPont PRD-66 filter element has undergone considerable development to improve the structural integrity of the outer membrane, and to produce a nearly complete barrier vs. bulk filter element. Additional improvements have included the incorporation of a strengthened, integral flange and reinforced end cap area, and achievement of acceptable gas flow resistance through the as-manufactured filter body.

DuPont PRD-66 filters were installed and operated in the Westinghouse Advanced Particulate Filtration unit at the American Electric Power pressurized fluidized-bed combustion test facility in Brilliant, OH, in 1994 and 1995, and at the Foster Wheeler pressurized circulating fluidized-bed combustion test facility in Karhula, Finland, in 1997. Both field test operations, as well as bench-scale qualification testing conducted in Westinghouse's pressurized fluidized-bed combustion simulator test facility in Pittsburgh, PA, have identified several life limiting issues that warrant continued development prior to commercial use of the filament wound PRD-66 candle. Additional efforts remain to be focused on the development and production of a dual membrane, barrier candle filter; further strengthening of the flange; and incorporation of a chip resistant outer surface. This report provides a summary of the efforts conducted at Westinghouse which have supported the development, manufacture, and field test operation of the DuPont PRD-66 candle filters.

Introduction

Two tasks were conducted by Westinghouse in support of DuPont's DOE/FBTC program entitled "Advanced Hot Gas Filter Development" (Contract No. DE-AC21-94MC3 1214A). These included:

Task 2- Test Plan Definition
Task 3- Development, Qualification, and Testing of Hot Gas Filters.

Initially Task 3 was identified to include:

- Task 3.1 - Material Qualification
- Task 3.2- Corrosion Testing
- Task 3.3 - High Temperature, High Pressure (HTHP) Filter Testing.

Due to budget constraints incurred by DuPont, Task 3.2 was eliminated from Westinghouse's workscope. In the following sections, a summary of the results obtained at Westinghouse between February 9, 1995 and March31, 1998 for conduct of Task 2, Task 3.1, and Task 3.3 is provided.

Program Overview

On January 20, 1994, the dimensional tolerances and filtration characteristics that are required for retrofit of porous ceramic candle filters into Westinghouse's Advanced Particulate Filtration (APE) systems were provided to the DuPont Lanxide Corporation (DLC)[1]. During 1994, filter elements were fabricated by DLC, and were delivered for use in the Westinghouse APE slipstream test facility that was operated at the American Electric Power (AEP) pressurized fluidized-bed combustion (PFBC) Tidd Demonstration plant in Brilliant, Ohio. The Westinghouse APF system at AEP consisted of three filter clusters (i.e., nine filter arrays) which housed 384, 1.5 m filter elements.

Testing of three, 1.5 m, DLC PRD-66 filament wound candles in the PFBC environment was initiated in July 1994, and continued for a period of 1705 hours [1]. At the conclusion of testing in October 1994, the filter vessel was slow cooled and inspected. Post-test inspection indicated that all three filters elements remained intact.

Additional 1.5-m PRD-66 filter elements were fabricated for inclusion in Test Segment 5 at AEP (January through March 1995). Twenty-two PRD-66 candle filters were installed in the Westinghouse APF system, filling an entire top array. After 232 hours of operation, sections of the PRD-66 matrix were identified in the ash hopper discharge, implying that failure of an element or elements had occurred. Testing continued, and after 775 hours of operation, additional sections of the PRD-66 filter matrix were found in the ash hopper discharge.

At the conclusion of 1110 hours of operation in Test Segment 5, the filter vessel was slow cooled and inspected. Only two ERD-66 filter elements remained intact, four had suffered either mid-body fracture or failure at a location that was ~3/4 below the flange, and sixteen filters had fractured at the base of the flange. The outer surface of the intact and fractured filters was generally "ash free", particularly along the portion of the body that was adjacent to the plenum support pipe, and to approximately mid-way down the length of each filter element. Alternately a 1-2 mm ash deposit remained along the outer surface of the PRD-66 candles, primarily near the bottom end cap. Surface "divot-like" formations resulted in lines which ran parallel down both sides of the remaining intact and fractured filter elements. Localized "divoting" was also observed below the gasket sleeve, which was installed around the filter flange, as well as in alternate, isolated areas along the filter body.

[1] Proprietary Westinghouse filter specifications served in part fulfill Task 2- Test Plan Definition.

The mechanisms leading to divoting and mid-body failure of the FRD-66 filter elements in Test Segment 5 were considered to be primarily related to delamination areas that were present within the wall of the filament wound matrix (i.e., uneven winding and/or localized drying or positioning of the elements during manufacturing of the elements). Post-test inspection indicated that ash and sorbent fines were present within the 7 mm PRD-66 filter wall. These were expected to have resulted from penetration of submicron fines through the PRD-66 outer membrane, or were back pulsed into the matrix after failure of an alternate candle(s). PFBC ash which had been shown by Westinghouse to have a high thermal coefficient of expansion in comparison to the ceramic filter matrix, may have induced localized internal failure within the filter wall during the plant shutdown and startup cycles in Test Segment 5. Mid-body failure of the element conceivably resulted once the filter wall had sufficiently weakened or thinned after "divoting" had occurred. Failure at the base of the PRD-66 filter flange was attributed to the low load bearing capability of the filter flange to support the thermal expansion loads applied by the ash, once fines became "wedged" in between the outer surface of the filter element and the metal holder.

In Task 2, Westinghouse recommended that

- The flange be densified and/or strengthened
- Modifications be made to the membrane to prevent fines infiltration into subsurface layers. In this manner, accumulated ash fines would not lead to fracture of the filament winding pattern during system startup and cooldown (i.e., higher thermal coefficient of expansion of the ash relative to the ceramic filter matrix).
- Modifications be made to the winding pattern to prevent localized internal delamination areas within the filter matrix,

in an attempt to mitigate failure of the PRD-66 filter element during continued process operation.

As a result, during conduct of the originally proposed contract with DOE/FETC, DLC supplied six, 1.5 m, PRD-66 candle filters to Westinghouse on February 28, *1995*. Production modifications which had been made by. DLC included:

- Strengthening of the flange and end cap(2 Standard or baseline filter elements identified as D-337 and D-338)
- Strengthening of the flange and end cap, and providing a higher permeability outer surface (o.d.) membrane (2 Improved membrane filter elements identified as D-325 and D-331)
- Strengthening of the flange and end, providing a higher permeability o.d. membrane, as well as an inner surface (i.d.) membrane (2 Improved dual membrane filter elements identified as D-328 and D330).[2]

Westinghouse initially performed room temperature permeability measurements on the six modified PRD-66 filter elements to confirm DLC's measurements (Task 3.1). One filter type

[2] Fabrication of the dual membrane candle was recommended by Westinghouse as a result of ash penetration along the i.d. surface of intact fitter elements (i.e., AEP Test Segments 1-3) after failure. of alternate candles had occurred within the filter array during process operation. Westinghouse patent pending.

of each element was then returned to DLC and sectioned. Sections were returned to Westinghouse for characterization of fines penetration into the matrix, as well as permeability measurements (Task 3.1). Following this effort, one element of each filter type was subjected to high temperature, high pressure (HTHP), simulated pressurized fluidized-bed combustion (PFBC) testing at the Westinghouse test facilities in Pittsburgh, PA (Task 3.3). After two hours of simulated PFBC exposure, and cooldown of the test facility, debonding of the outer membrane was evident. As a result continued HTHP testing was terminated, and DLC undertook an extensive effort to reformulate the manufacture and application of the membrane along the o.d. surface of the PRD-66 filter elements.

In 1997, DLC provided Westinghouse with newly formulated filter elements for qualification testing under simulated PFBC test conditions in Task 3.1. The viability and performance of the filter elements during qualification testing in Pittsburgh, PA, served as the basis for acceptance or rejection of elements for possible inclusion within Westinghouse's APF array which was installed at the Foster Wheeler pressurized circulating fluidized-bed combustion (PCFBC) test facility in Karhula, Finland. Twelve candles were subsequently manufactured and shipped directly to Karhula, Finland. After initial inspection, seven elements were identified for installation and operation in the PCFBC environment.

Development, Qualification, and Testing of Hot Gas Filters

Material Qualification

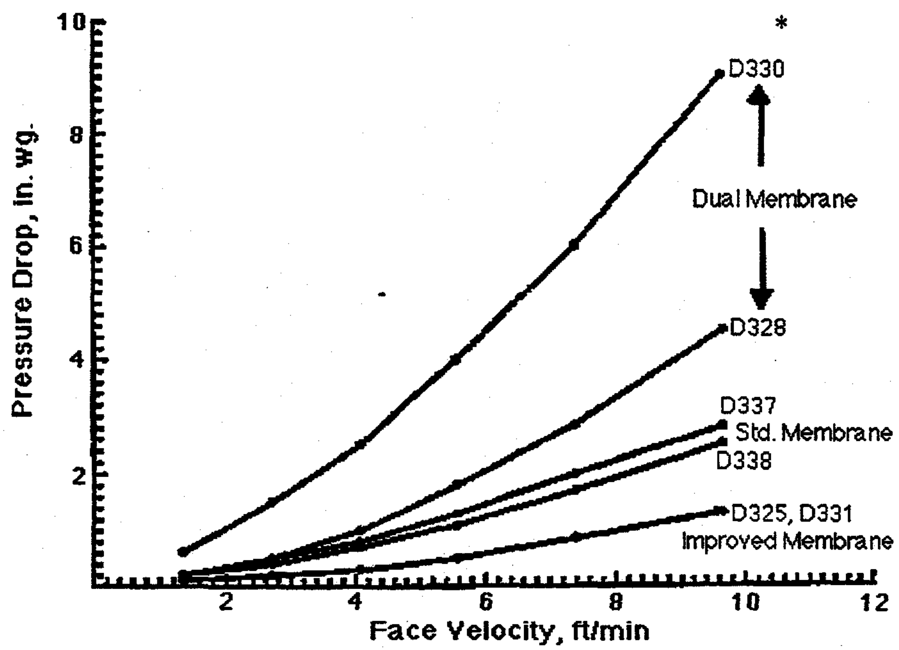
Candle Filter Permeability Measurements Task 3.1)

Westinghouse specifications for an initial pressure drop across an as-manufactured 1.5-m candle filter is 6+/-2 mbar at 52 m^3/hr/candle at 70^{0}F air (2.41+/-0.8 in-wg at 30.6 scfm at 70^{0}F air). With an outer filtration surface area of 2.76 ft^2/candle filter, and a flow of 30.6 scfm, a face velocity of 11.1 fpm results.

Initial room temperature gas flow resistance measurements were conducted on the following filter elements:

- Standard or baseline candles identified as D-337 and D-338 (Strengthened flange and end cap candles)
- Improved membrane candles identified as D-325 and D-33 1 (Strengthened flange and end cap candles with a higher permeability o.d. membrane)
- Improved dual membrane candles identified as D-328 and D-330 (Strengthened flange and end candles with a higher permeability outer surface membrane, and an inner membrane).

As shown in Figure 1, relative homogeneity resulted for the standard PRD-66 candle filters which had undergone flange and end cap strengthening or densification (i.e., D-337 and D-338). Extrapolating from the gas flow resistance measurements presented in Figure 1, the pressure drop across the standard filter elements at a face velocity of 11.1 fpm ranged between 3 and 3.4 in-wg (i.e., 7.5-8.5 mbar). Based on the room temperature gas flow resistance measurements, the standard PRD-66 candles were considered to be within the Westinghouse pressure drop specifications for as-manufactured candle filter elements.



* Westinghouse As-Manufactured Specifications

Figure 1 – Room temperature gas flow resistance measurements

With respect 10 candles that had been manufactured with an improved membrane, as well as a strengthened or densified flange and end cap (i.e., D-325 and D-331), a lower gas flow resistance resulted. As shown in Figure 1, the gas flow resistance through these elements was quite reproducible. For the improved membrane filters, the pressure drop across the candle at a face velocity of 11.1 fpm was 1.6 in-wg (i.e., 4 mbar). This was considered to be acceptable in view of the Westinghouse as-manufactured filter element pressure drop specifications.

When the improved membrane was applied to the outside surface of the PRD-66 filament wound filter element, and an internal membrane was also applied to the i.d. surface of the filter wall, the gas flow resistance across the filer matrix increased. As shown in Figure 1, a relatively wide range in gas flow resistance resulted between the two as-manufactured, dual membrane candle filters (i.e., D-328 and D-330). Based on the extrapolated gas flow resistance shown in Figure 1, the pressure drop across the dual membrane candles ranged between 5.6 and 11.0 in-wg (i.e., 14-27.4 mbar) for a gas face velocity of 11.1 fpm, which exceeded the Westinghouse pressure drop specifications for as-manufactured candle filters.

Based on these results, Westinghouse recommended:

- Establishing reproducibility in the manufacturing process for production of the dual membrane filter elements
- Further reduction of the gas flow resistance through the as-manufactured dual membrane candle filters while maintaining bulk material strength.

Coupon Gas Flow Resistance and Particle Collection (Task 3.1)

Table 1 provides a summary of the room temperature gas flow resistance measurements for twelve cylindrical PRD-66 filter samples that were supplied to Westinghouse by DLC on April 25, 1995 (i.e., D-35813, D-358C, D-358G, D-358H, D-358L, D-358M, D-359B, D-359C, D-359G, D-359H, D-359L, and D-359M). The higher gas flow resistance of samples that were designated as D-358 was supported by the visibly tighter filament winding pattern along the inner surface of the cylinders. The visibly tighter i.d. winding indicated that this series of cylinders had been manufactured with a dual membrane. In contrast, the lower gas flow resistance observed for the D-359 test sample series, as well as the open diamond weave, indicated that these samples were manufactured with only a single outer surface membrane.

The room temperature gas flow resistance of the D-359 single membrane PRD-66 cylinders was determined to be 0.51 +/- O.08 in-wg/fpm which indicated the relative uniformity of the six samples that were removed from various locations along the length of a single candle filter body. The room temperature gas flow resistance of the dual membrane D-358 PRD-66 cylinders was determined to be l.Ol +/- O.20 in-wg/fpm. The greater scatter in the gas flow resistance measurements for the dual membrane samples tended to indicate a reduction in production homogeneity along the length of the 1.5 m candle filter.

As shown in Table 1, four sections out of six of the D-358 cylinder series were within the Westinghouse gas flow resistance specifications (i.e., <1 in-wg/fpm), while two exceeded the as-manufactured gas flow resistance specifications. The wide range in gas flow resistance may be expected to possibly cause uneven dust cake removal. Perhaps the manner in which the membrane was applied (i.e., wetter yarn applied in one area versus another; variation in yarn

TABLE 1

GAS FLOW RESISTANCE MEASUREMENTS FOR THE IMPROVED o.d. AND i.d./o.d. MEMBRANE-COATED CYLINDERS

Filter Identification Number	System Pressure, psig	Velocity, fpm	Pressure Drop, in-wg	Gas Flow Resistance, in-wg/fpm
D-358B	8.5	12.29	16.0	1.30
D-358C	8.3	12.24	12.0	0.98
D-3580	5.7	11.51	10.0	0.87
D-358H	7.8	12.10	12.0	0.99
D-358L	5.7	11.51	8.5	0.74
D-358M	5.8	11.54	13.5	1.17
			Average +/- 1⇔	1.01 +/- 0.20
D-359B	6.0	11.58	6.0	0.52
D-359C	7.5	12.02	7.0	0.58
D-359G	5.7	11.51	5.0	0.43
D-359H	6.5	11.74	5.0	0.43
D-359L	5.6	11.48	5.5	0.48
D-359M	7.5	12.02	7.5	0.62
			Average +/- 1⇔	0.51 +/- 0.08

Cylinders: 58 mm o.d.; 50 mm length; Assumed uniform effective surface area during bonding/sealing along edge.

thickness; closer wrap positioning etc.), or possibly the extent of "sealing" which was added along the edges of each cylinder to provide an adequate test sealing surface were responsible for The gas flow resistance variations which led to what appeared to be a non-homogeneous filter body.

In an attempt to demonstrate particle collection efficiency, dust was delivered to each of the twelve cylindrical samples at room temperature for a period of 3 minutes. Both the clean inner surface appearance, as well as the absence of detectable fines in the off-gas stream indicated excellent particle collection efficiency of the PRD-66 matrix (Figure 2). When a particle challenged cylinder from the D-358 and D-359 series was fast fractured, fines were evident below the outer membrane-coated surface. As shown in Figure 3, the depth of fines penetration into the 6 mm filter wall varied from 1 to 3 mm indicating that the PRD-66 matrix had bulk rather than barrier filtration characteristics. Examination of the fast fractured surface indicated that the fines did not permeate across the entire 6 mm filter wall during the 3 minute dust exposure. Continued dust exposure testing would be needed to demonstrate the extent of fines penetration and/or plugging which may result during extended process operation.

High Temperature, High Pressure Simulated PFBC Testing (Task 3.3)

Three full length filters were subjected to high temperature, high pressure (HTHP) testing in Westinghouse's pressurized fluidized-bed combustion (PFBC) simulator in Pittsburgh, PA. These included candle filters D328 (improved, lower flow resistance dual membrane candles with a strengthened flange), D338 (standard membrane candles with a strengthened flange), and D325 (improved, lower flow resistance outer surface membrane candles with a strengthened flange). All three filter elements were mounted in the HTHP test facility, and the system was brought to temperature (1550^{0}F), and maintained at steady state conditions for two hours of operation with dust feed. After cool-down of the unit, areas along the outer surface of candle filter D328 and D325 were seen to have spalled off (Figure 4), while the standard outer surface membrane along candle filter D338 remained intact. The standard D338 membrane had typically been used at Tidd during the 1705 hour, Test Segment 4, and 1110 hour, Test Segment 5 campaigns. The failed membrane areas along D328 and D325 typically extended 1-2 inches, running parallel with the outer membrane winding pattern, and for 3-4 filament winding turns. Removal of the subsurface diamond pattern support structure was not evident (i.e., absence of initiation/propagation of "divoting"). Further development was recommended by Westinghouse to manufacture low gas flow resistance filter elements which maintained the integrity of the outer surface membrane.

Modified Filter Membrane Evaluation (Task 3.1)

Manufacturing modifications were undertaken to improve the bonding and integrity of the outer surface membrane of the PRD-66 candle, while maintaining the Westinghouse gas flow resistance criteria for as-manufactured filter elements. On October 16, 1996, two, 2 inch, PRD66 filter sections were received at Westinghouse. These were identified as:

- PRD-66 Combination membrane filter sample (492-5D)
- PRD-66 Particulate membrane filter sample (490-C).

Figure 5 illustrates the general appearance of both production configurations. The combination membrane consisted of:

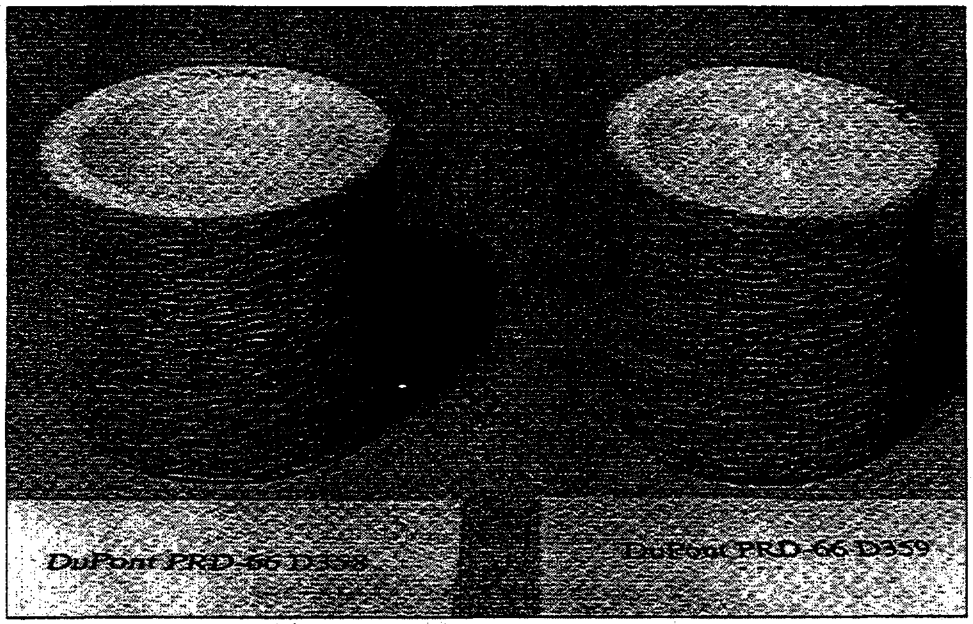


Figure 2 – DuPont PRD-66 filter matrices after room temperature particle collection and gas flow resistance testing.

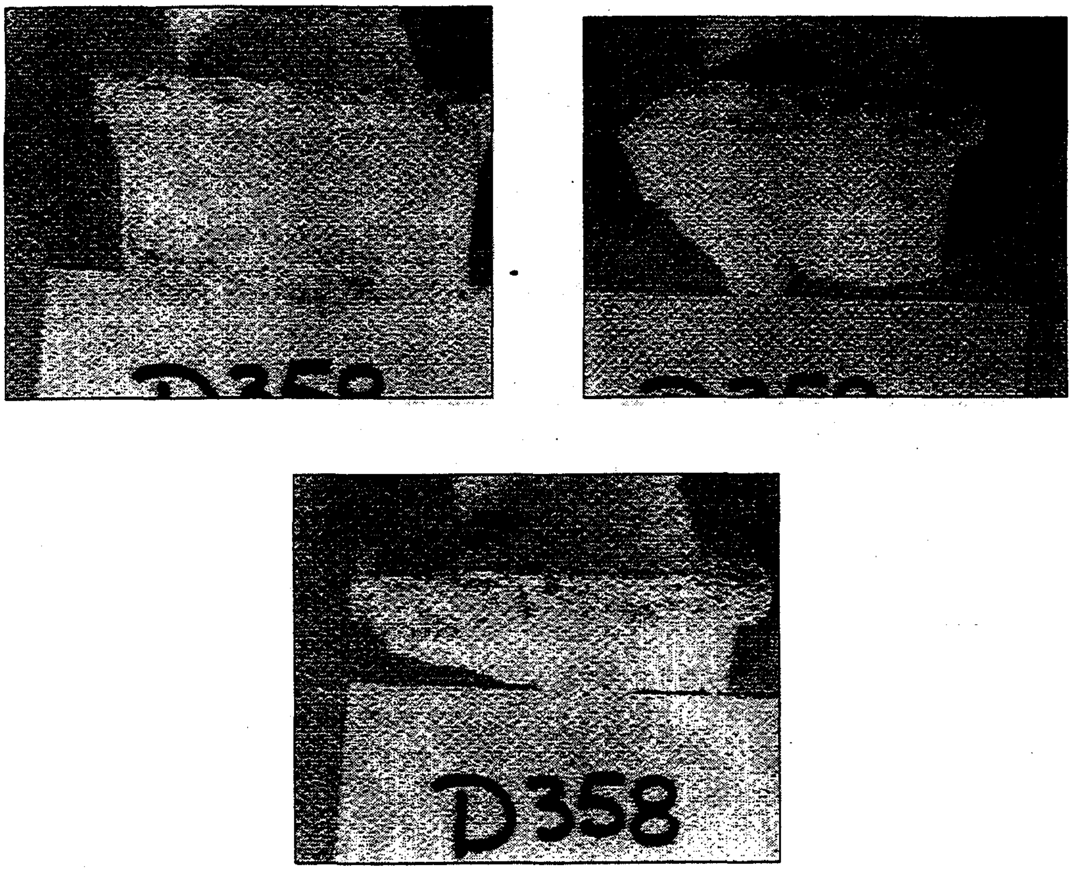


Figure 3a – Fresh fractured surface of the particle challenged D-358 filter matrix.

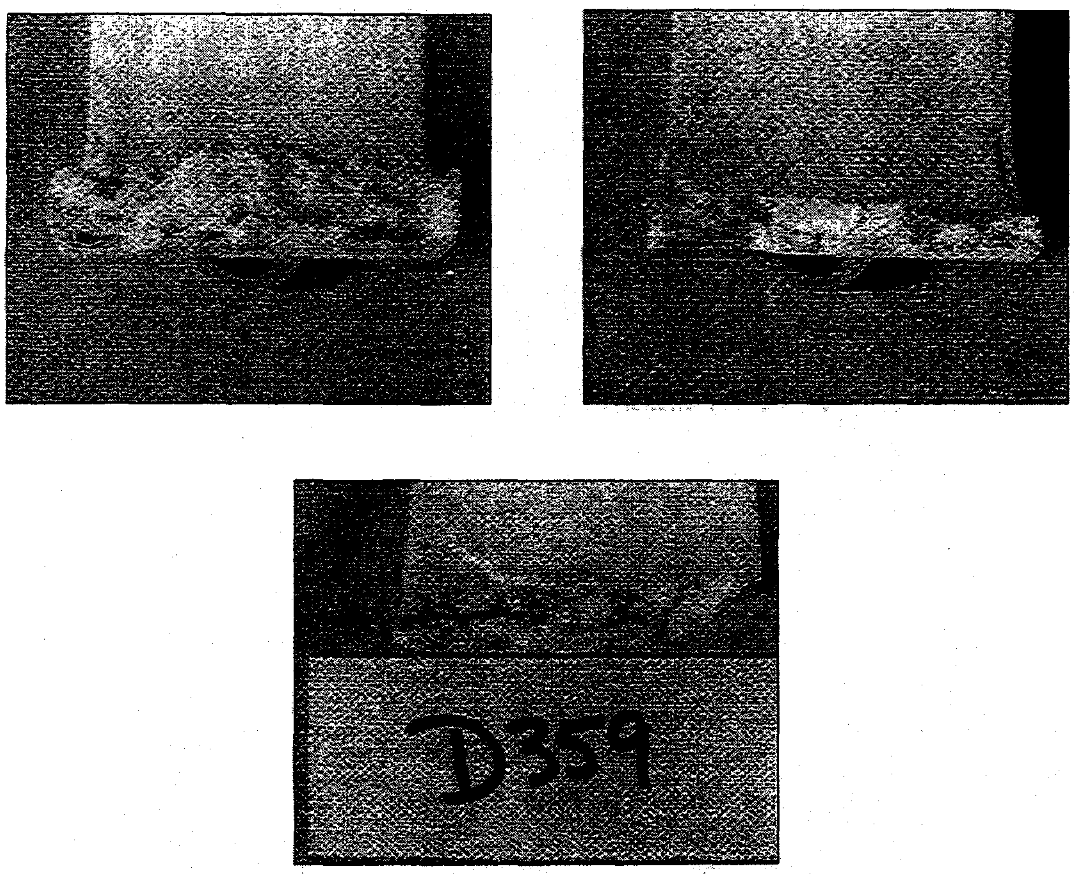


Figure 3b -- Fresh fractured surface of the particle challenged D-359 filter matrix.

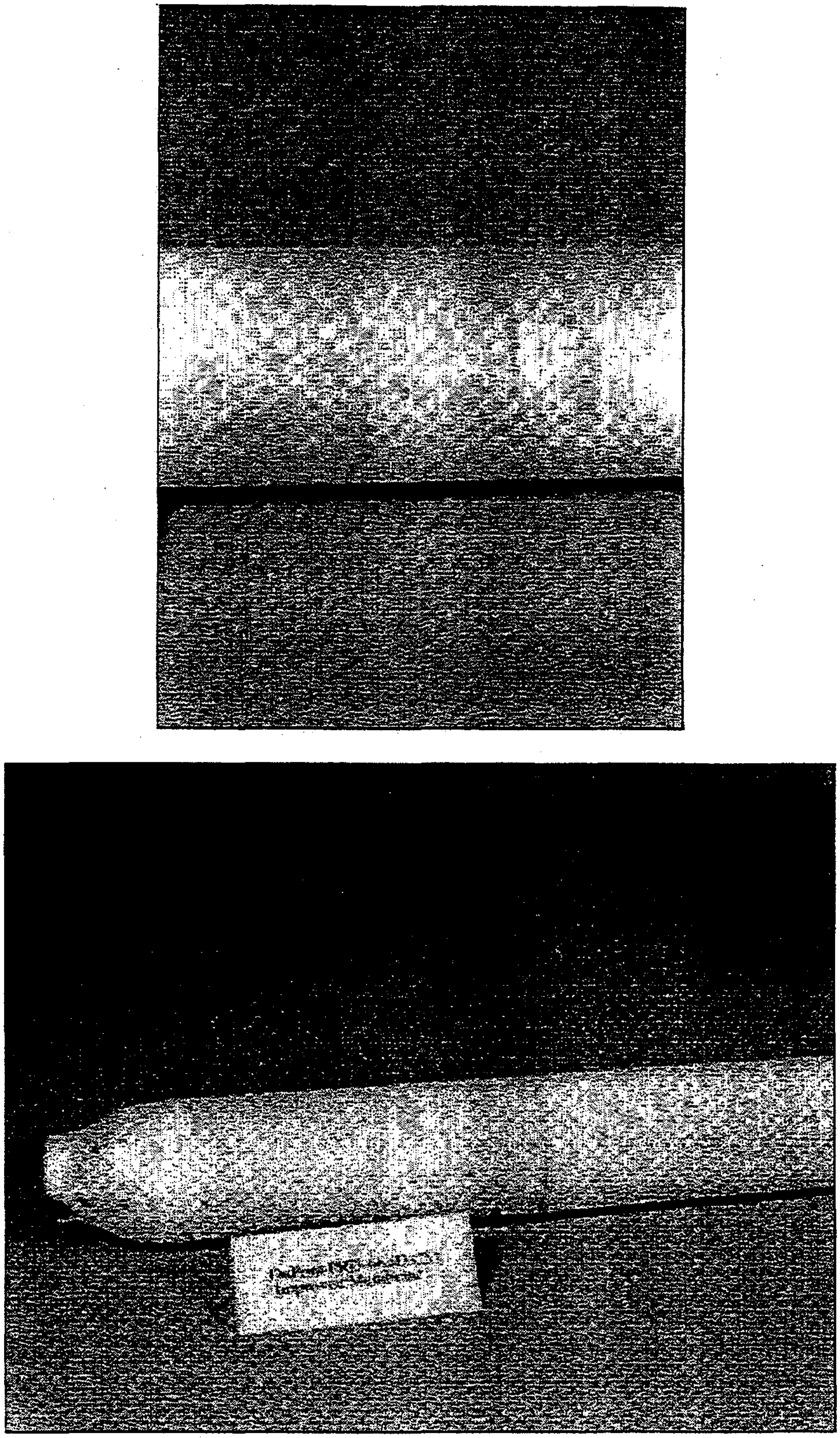


Figure 4a – HTHP-tested DuPont PRD-66 candle filter (Improved o.d. membrane; Strengthened flange).

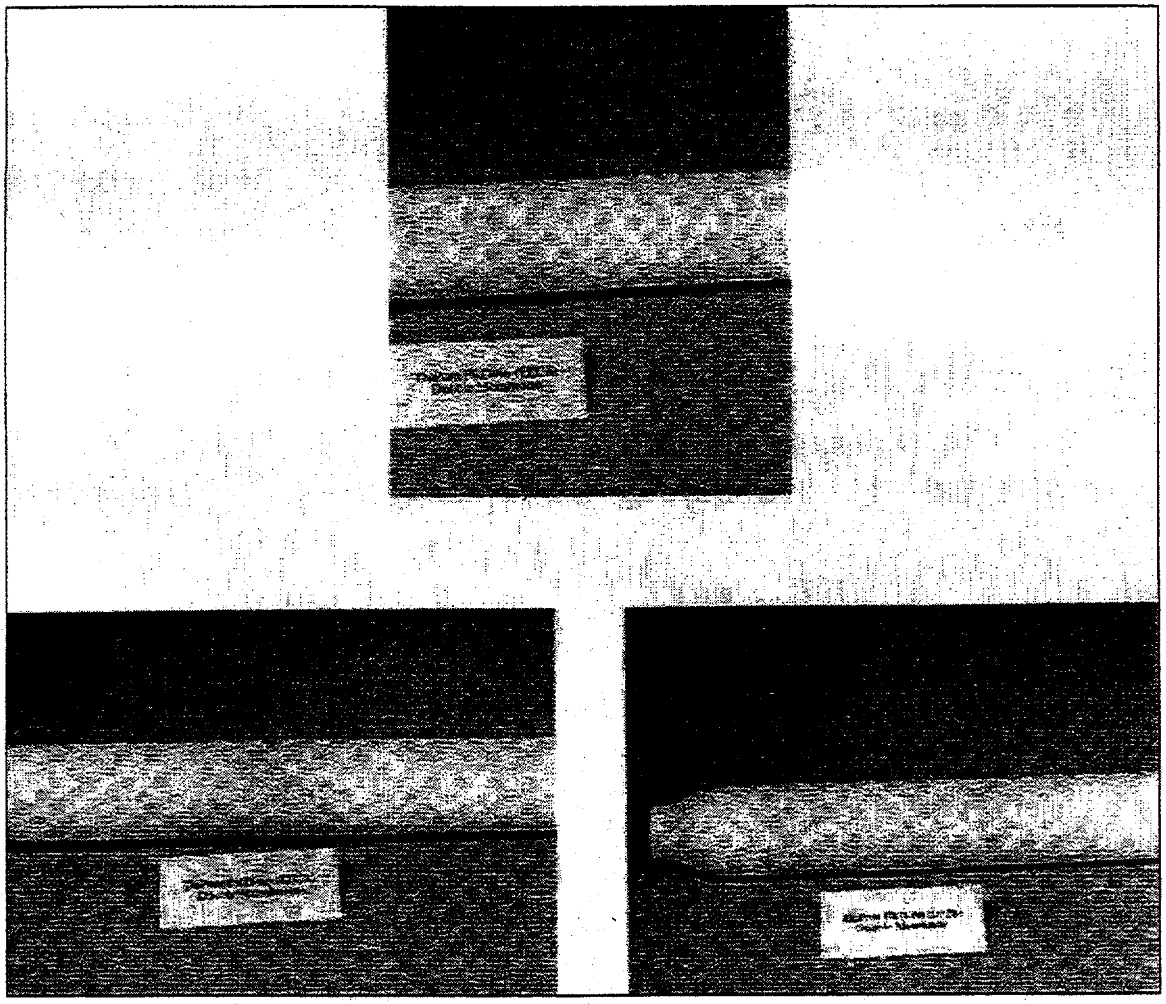


Figure 4b – HTHP-tested DuPont PRD-66 candle filter (Improved dual membrane; Strengthened flange).

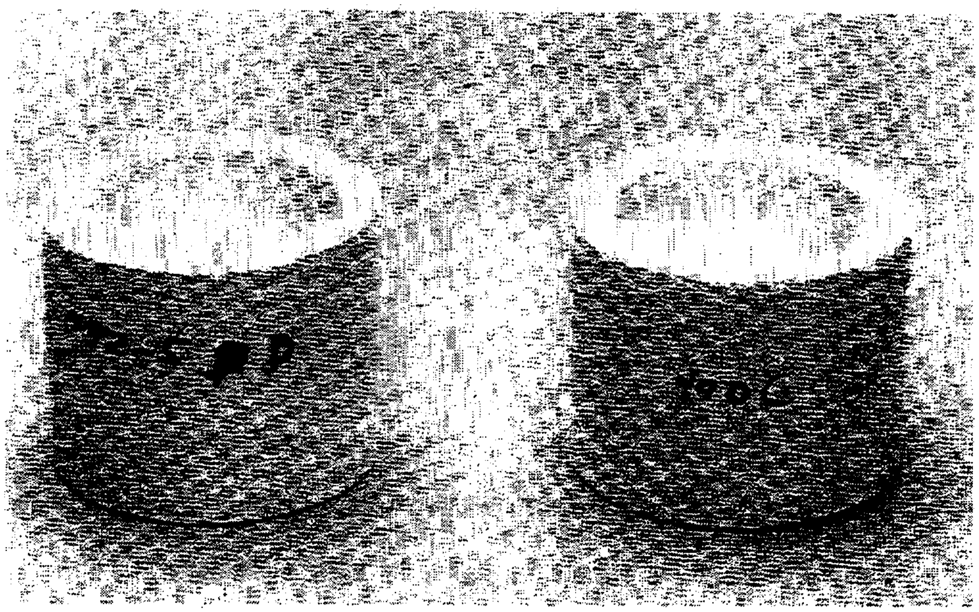


Figure 5 – PRD-66 combination membrane and particulate membrane filter concepts.

- The prior diamond winding pattern which served as the bulk or support matrix
- An additional external hoop winding which formed a smooth surface outer membrane
- The application of an additional particulate slurry infiltration which was expected to reduce the gaps between the outer hoop winding, resulting in the formation of the combined hoop wrap and particulate membrane.

In contrast the particulate membrane filter concept consisted of:

- The diamond support matrix
- The infiltration of particulates to form the membrane.

The hoop winding was not applied along the outer surface of the diamond winding. Both matrices were developed in an attempt to circumvent "divoting" and subsequent filter element failure which had previously been experienced in the Westinghouse APF system at Tidd during Test Segment 5.

Initially 8-inch sections of each material were shipped to Westinghouse for consideration and/or evaluation. The uneven edges along the 2-inch pieces which resulted from cutting of the filter sections at DLC were ground at Westinghouse in order to provide a smooth sealing surface prior to conduct of the room temperature gas flow resistance measurements. After testing and inspection, both samples were returned to DLC on October 21, 1996.

Table 2 provides comments regarding the PRD-66 combination membrane and particulate membrane filter concepts. Based on not only general appearance, but also the gas flow resistance measurements, Westinghouse recommended continued future development and manufacture of the combination membrane filter element with enhanced strengthening of the PRD-66 matrix along the flange of the candles.

Issues which remained to be addressed, however, included:

- Demonstrating the relative strength of both membrane filter concepts to identify if differences existed
- Demonstrating the relative load-to-failure for both membrane filter concepts to identify if differences existed
- Manufacturing of the filter sections and/or body with comparable o.d. dimensions. For the samples provided, the o.d. dimensions were not identical.

Based on the above information, Westinghouse supported production of the PRD-66 filter element with the combination membrane for use in future process simulation and/or field testing. Should the hoop wrap prove to be ineffective (i.e., bulk filtration vs. complete barrier filtration performance), additional modifications to the PRD-66 particulate membrane filter would be needed.

[3] Both the diamond winding pattern and external hoop were conceptually similar to what had previously been utilized to manufacture the filter elements installed at AEP.

TABLE 2 COMPARISON OF PRD-66 FILTER MEMBRANE CONCEPTS	
Combination Membrane Hoop Wrap with Particle Infiltrate	**Particulate Membrane**
W-STC Gas Flow Resistance: 0.5 in-wg/fpm	W-STC Gas Flow Resistance: 1.07 in-wg/fpm
DLC Gas Flow Resistance: 0.9 in-wg/fpm	DLC Gas Flow Resistance: 1.2 in-wg/fpm
Gaps Between Hoop Wrap Winding Were Evident. Potential Issues Include: -- Penetration of Submicron Fines -- Divot Formation Due to Thermal Expansion of Penetrated Submicron Fines -- Divoting Leading To Failure of The Element	Particulate Infiltrate May Be More Evenly Distributed Along The External Diamond Wrap Pattern. If So, Then -- Areas For Fines Penetration Into The Matrix Which May Mitigate Or Reduce Divoting/Failure Of The Filter Elements May Be Eliminated
Relatively Smooth Outer Surface -- A Conditioned Ash Cake Layer May Not Form Which May Lead To Penetration Of Submicron Fines Into The Interior Of The Filter Wall, Potentially Causing Divoting and/or I Failure Of The Element	Stepped Surface Due To Diamond Patterns May -- Be Potential Areas To Accumulate and/or Retain Ash Fines -- Lead To The Formation Of A Conditioned Ash Layer Which Could Possess Bulk Filtration Characteristics -- Pending Accumulation Of Fines Along The Diamond Weave Edges, Localized Removal Of Fines May Not Occur Leading To A High Pressure Drop Across The Filter Element.
-- Minimal "Crumbling" Of Cut Surfaces In Contrast To Original Matrices	
-- Along Cut Surfaces, Potential Delanimation Areas Still Exist Most Likely As A Result Of Bulk Substrate Winding Patterns.	

* Differences between the Westinghouse and DuPont gas flow resistance measurements may be due to variations in the uniformity of the 2-inch vs. 8-inch sections, or alternately the measurement technique.

Qualification Testing for PCFBC Applications (Task 3.3)

Eight, 1.5 m, PRD-66 candle filters were received from DuPont on March27, 1997. In the manufacturing process, either a coarse or medium grade hoop wrapped membrane was applied to the outer surface of the filter elements. The results of the room temperature gas flow resistance measurements of the eight, as-manufactured, 1.5 m, candle filters are shown in Figures 6 and 7. Both sets of filter elements met the Westinghouse gas flow resistance tolerance of<1 in-wg/fpm for as-manufactured candles.

During April 1997, one candle of each filter element type was subjected to high temperature, high pressure (HTHP), simulated pressurized fluidized-bed combustion (PFBC) testing in Westinghouse's test facility in Pittsburgh, PA. Testing included exposure of the PRD-66 candle filters with alternate monolithic and advanced fiber reinforced candle filter elements in order to support pressurized circulating fluidized-bed combustion (PCPBC) test initiatives in Karhula, Finland. The filter array was subjected to 120 hours of steady state operating conditions at temperatures of 1550^{0}F, and subsequently 2200 accelerated pulse cycling, and 12 mild thermal transients events.

Post-test inspection of the filter army indicated that both PFBC-exposed PRD-66 filter elements remained intact. As a result, both elements, and an unexposed filter of each element type were subsequently subjected to mechanical strength characterization, and x-ray diffraction and microstructural analyses. The results of these efforts are summarized in the following sections.[4]

Figure 8 provides photographs of the residual dust cake layer that remained along the outer surface of the qualification-tested filter elements. Due the manner in which the qualification test was performed, the thin dust cake layer was considered to reflect the conditioned layer that generally remains attached to the outer surface of the candle during field exposure. Post-test gas flow resistance measurements of the qualification-tested candles are provided in Figure 9. The coarse membrane-coated filter element initially had a lower pressure drop in comparison the medium membrane-coated filter element. After qualification testing, this relationship was retained

Bulk Strength Analysis

As shown in Table 3, the strength of the coarse and medium membrane qualification tested DLC PRD-66 candle filters tended to be greater than the strength of comparable as-manufactured filter elements. As previously demonstrated by Westinghouse, the bulk strength of the DLC PRD-66 matrix tended to increase during simulated or field exposure [2] This was considered to result from the bulk vs. barrier filtration characteristics of the material, whereby submicron and micron fines penetrated through the membrane of the PRD-66 filter element and become entrapped within the filter wall. Although divot formations along the outer membrane did not occur during the qualification test program, the potential may still exist during extended

[4] Sections of both the coarse and medium membrane-coated, qualification-tested, PRD-66 filter elements were also returned to DLC on June 20, 1997, for additional inspection and characterization.

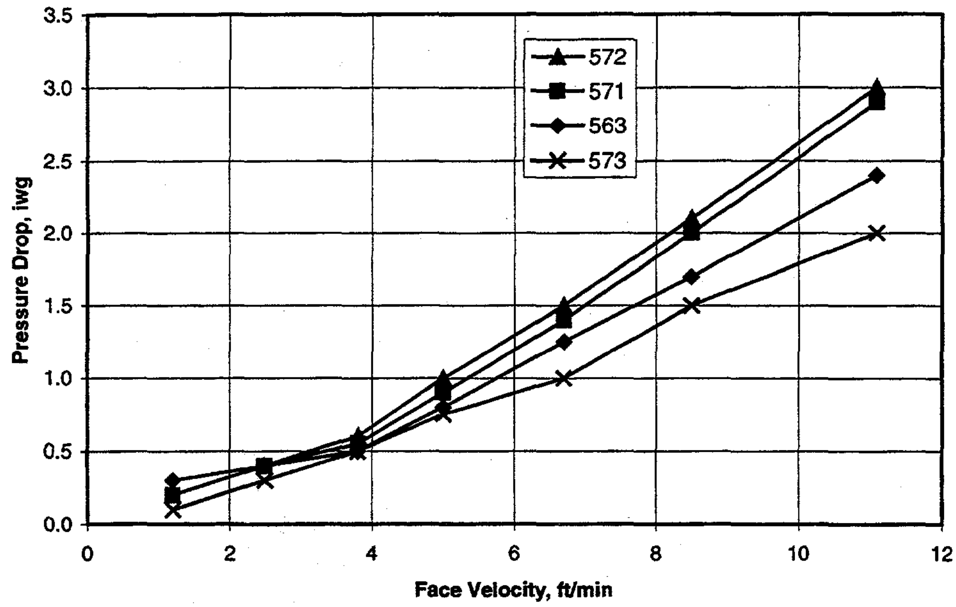


Figure 6 – Room temperature gas flow resistance measurements of the course membrane PRD-66 candle filters.

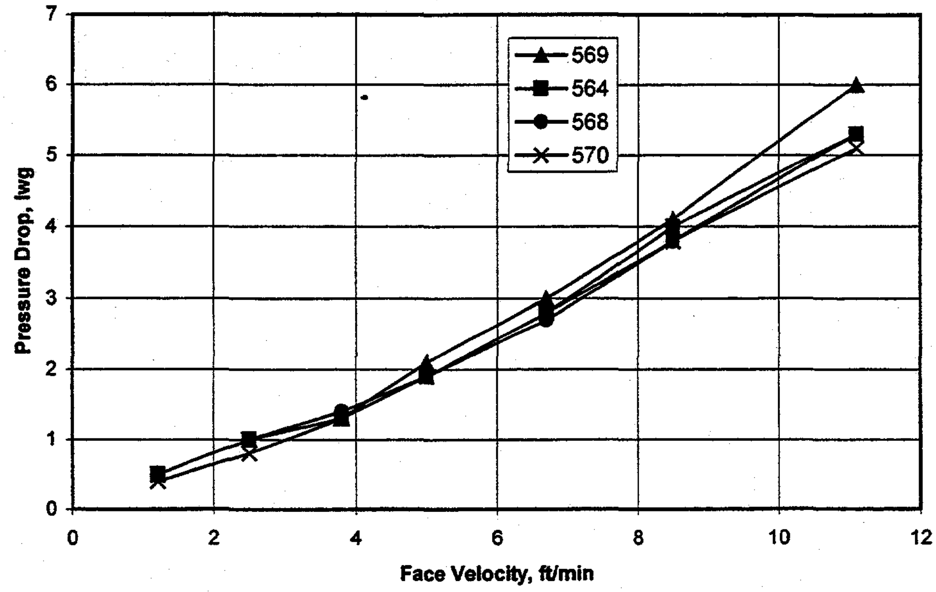


Figure 7 – Room temperature gas flow resistance measurements of the medium membrane PRD-66 candle filters.

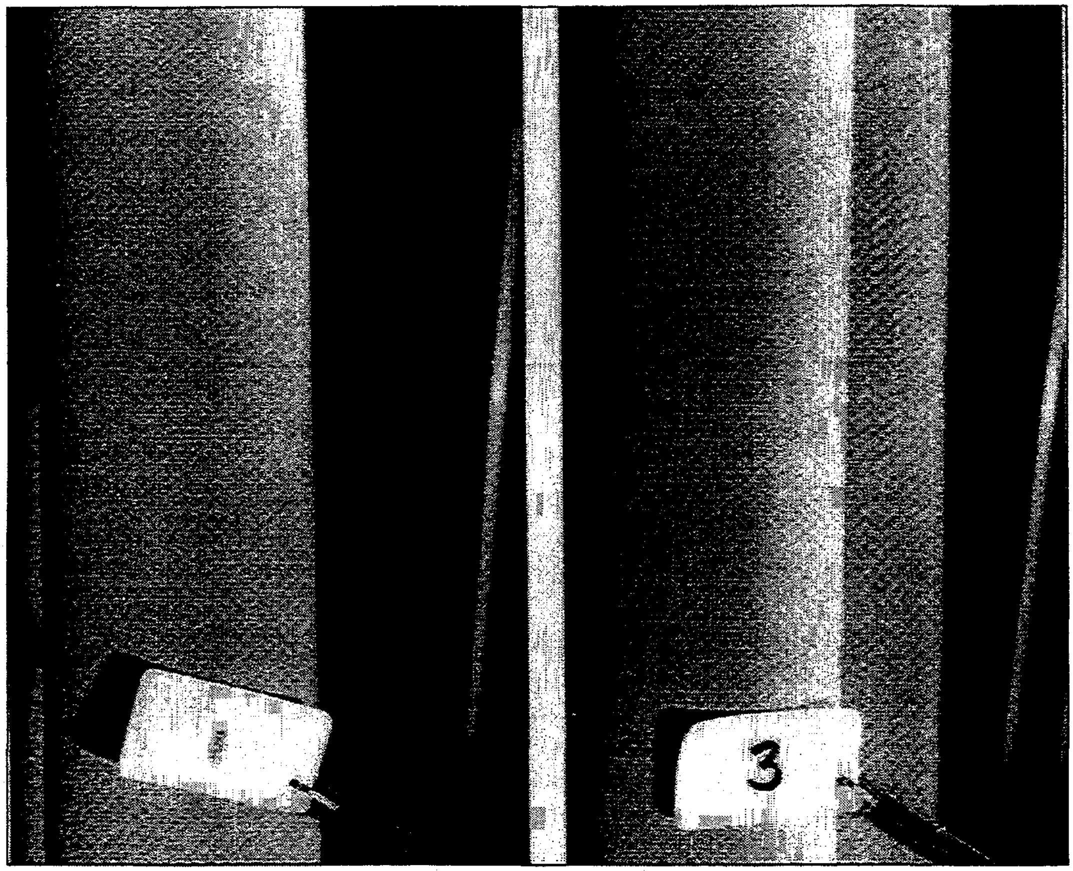
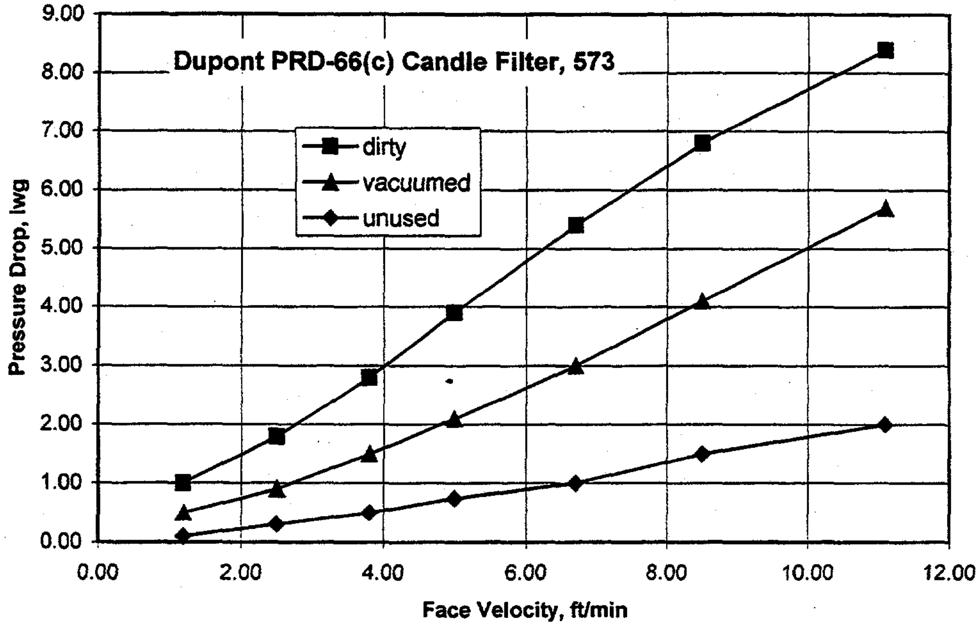


Figure 8 – Photograph illustrating the residual ash cake layer that remained along the outer surface of the PRD-66 candle filters after qualification testing that was conducted under simulated PFBC conditions.



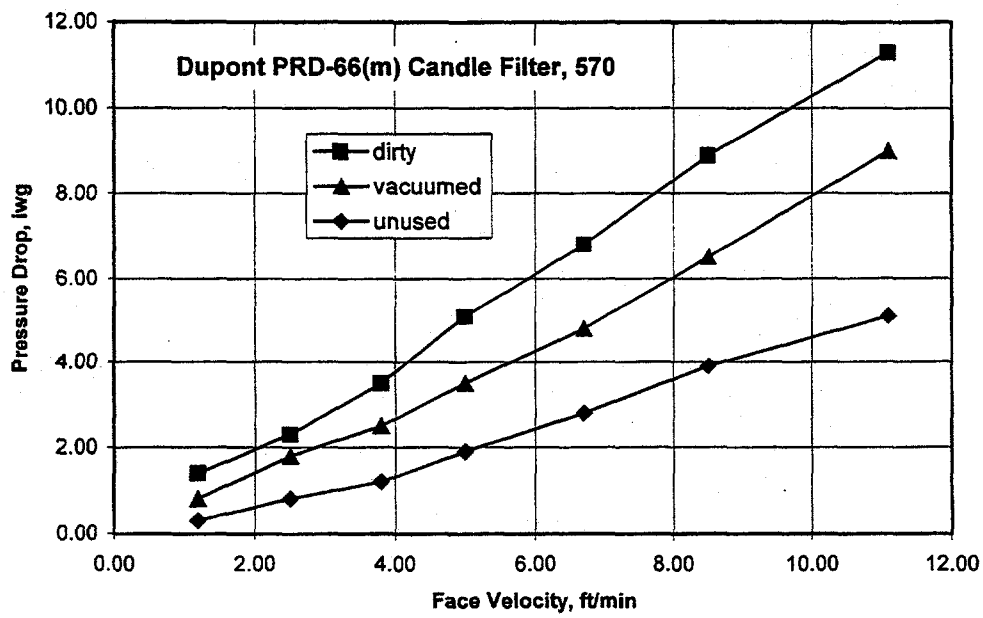


Figure 9 – Gas flow resistance measurements of the as-manufactured and qualification-tested PRD-66 candle filters.

TABLE 3

ROOM TEMPERATURE AND PROCESS STRENGTH OF THE AS-MANUFACTURED AND QUALIFICATION-TESTED DUPONT PRD-66 CANDLE FILTERS					
Candle Identification Number	Status	C-Ring Compressive Strength, psi		C-Ring Tensile Strength, psi	
		25-degC	843-degC	25-degC	843-degC
DuPont PRD-66 (Coarse Membrane)					
D-563c	As-Manufactured	955+/-62 (9)	962+/-92 (8)	809+/-154 (9)	1009+/-103 (7)
D-573c	Qualification Tested	1214+/-67 (9)	1210+/-86 (9)	990+/-82 (9)	1195+/-166 (9)
DuPont PRD-66 (Medium Membrane)					
D-564m	As-Manufactured	990+/-130 (9)	883+/-79 (9)	846+/-105 (9)	918+/-104 (9)
D-570m	Qualification Tested	1021+/-127 (9)	1019+/-88 (9)	973+/-165 (9)	1193+/-149 (8)

TABLE 4

ULTIMATE LOAD APPLIED DURING STRENGTH CHARACTERIZATION OF THE AS-MANUFACTURED AND QUALIFICATION-TESTED DUPONT PRD-66 CANDLE FILTERS					
Candle Identification Number	Status	C-Ring Compressive Load-to-Failure, psi		C-Ring Tensile Load-to-Failure, psi	
		25-degC	843-degC	25-degC	843-degC
DuPont PRD-66 (Coarse Membrane)					
D-563c	As-Manufactured	8.2+/-0.5 (9)	8.2+/-0.9 (8)	5.2+/-1.1 (9)	6.7+/-0.7 (7)
D-573c	Qualification Tested	10.3+/-0.6 (9)	10.3+/-0.6 (9)	6.4+/-1.2 (9)	7.6+/-1.0 (9)
DuPont PRD-66 (Medium Membrane)					
D-564m	As-Manufactured	8.0+/-0.9 (9)	7.3+/-0.6 (9)	5.2+/-0.6 (9)	5.7+/-0.6 (9)
D-570m	Qualification Tested	8.3+/-1.0 (9)	8.3+/-0.8 (9)	6.1+/-0.9 (9)	7.4+/-0.8 (8)

field operation, particularly if thermal expansion of the ash fines occurs within the filter wall during plant startup cycles [3], or hydration of the ash resulted during shutdown cycles.

In relation to alternate filter elements [4], the PRD-66 candle filter body was considered to be a moderately low load bearing matrix (Table 4). Additional material properties as burst strength, modulus, and Poisson's ratio, which were developed at Westinghouse are provided in Table 5.

X-ray Diffraction Analysis

An alternate explanation for increased strength conceivably is through crystallization of the matrix as a response of the material to the process gas chemistry and operating temperature. X-ray diffraction (XRD) analyses of the PRD-66 filter matrix identified the presence of 30% cordierite and ~50% α-alumina, with mullite as a minor phase. The XRD patterns for the as-manufactured coarse and medium membrane matrices, and qualification-tested coarse and medium matrices appeared to be virtually identical. Since neither the qualification test exposure nor coarseness of the membrane affected phase assemblage, the concept of increased bulk strength as a result of fines infiltration was supported.

Microstructural Characterization

Sections of the PRD-66 filter matrices were removed from the qualification-tested filter elements, and were subjected to microstructural analyses via scanning electron microscopy energy disperse x-ray analyses (SEM/EDAX). Figures 10 and 11 illustrate the surface morphology of the coarse membrane-coated, qualification-tested, PRD-66 filter element. Random areas of ash were identified along the outer surface of the "cleaned" filter element (i.e., Area 1, Figure 10: relatively ash-free surface; Area 2, Figure 10: presence of fines). Although what appeared to be limited adherence of ash along the outer surface of the element, when viewed at higher magnification (Area 1, Figure 11), fines were readily seen to entrapped between adjacent, slurry deposited alumina-rich grains which formed the outer membrane surface. When viewed in cross-section, the fine graine membrane was seen to be adherently bonded to the underlying filament wound support fiber bundle structure (Figure 12). At higher magnification, ash fines were seen to be attached to individual grains contained within the membrane layer(Figure 13). Based on the microstructural analyses of the "cleaned", coarse membrane-coated, PRD-66 filter, the open porosity of the element was nearly completely retained after being subjected to simulated PFBC, qualification testing.

Similar microstructural analyses were conducted on the medium membrane-coated, qualification-tested, PRD-66 filter element. As shown in Figure 14 (i.e., Area 1), areas of ash were retained along the outer surface of the candle. When viewed at higher magnification, ash fines (Area 1, Photo 3, Figure 15; Photo 4, Figure 15) were seen to be contained between adjacent alumina-rich grains that were present in the outer membrane (Area 2, Photo 3, Figure 15). When fresh fractured, the cross-sectioned PRD-66 filter wall appeared to retain its relatively open porosity through both the membrane, as well as underlying filament wound structural support (Figure 16). At higher magnification (Figure 17), isolated ash fines were identified to adhere to either the outer surface of the alumina-rich membrane grains, or to the outer surface of the filament wound fiber bundles.

TABLE 5

MATERIAL PROPERTIES OF THE AS-MANUFACTURED AND QUALIFICATION-TESTED DUPONT PRD-66 CANDLE FILTERS					
Candle Identification Number	**Status**	**Burst Pressure, psi**	**Ultimate Hoop Stress, psi**	**Modulus, psi x 10^6**	**Poisson's Ratio**
DuPont PRD-66 (Coarse Membrane)					
D-563c	As-Manufactured	148	555	7.96	0.86
D-573c	Qualification Tested	158	597	6.11	0.82
DuPont PRD-66 (Medium Membrane)					
D-564m	As-Manufactured	180	691	7.09	0.84
D-570m	Qualification Tested	170	653	5.42	0.84

TABLE 6

Pressurized Circulating Fluidized-bed Combustion Testing at the Foster Wheeler Test Facility in Karhula, Finland - TS2-97	
Date	September 4, 1997 – November 7, 1997
Number of Filter Elements Tested	8
Filter Operating Temperature, deg.C	700 - 750
Filter Operating Pressure, bar	9.5 - 11
Coal Feed	Eastern Kentucky
Sorbent	Florida Limestone
Time, hrs	581 (6)*, 342 (1), 239 (1)
Face Velocity, cm/sec	2.8 - 4.0
Particle Load, ppmw	6000 - 9000
Particle Size, microns	< 1 - 150
Thermal Excursions	None
Number of Startup/Shutdown Cycles	7

* All elements remained intact. The number in parentheses indicates the number of elements exposed for the respective PCFBC operating hours.

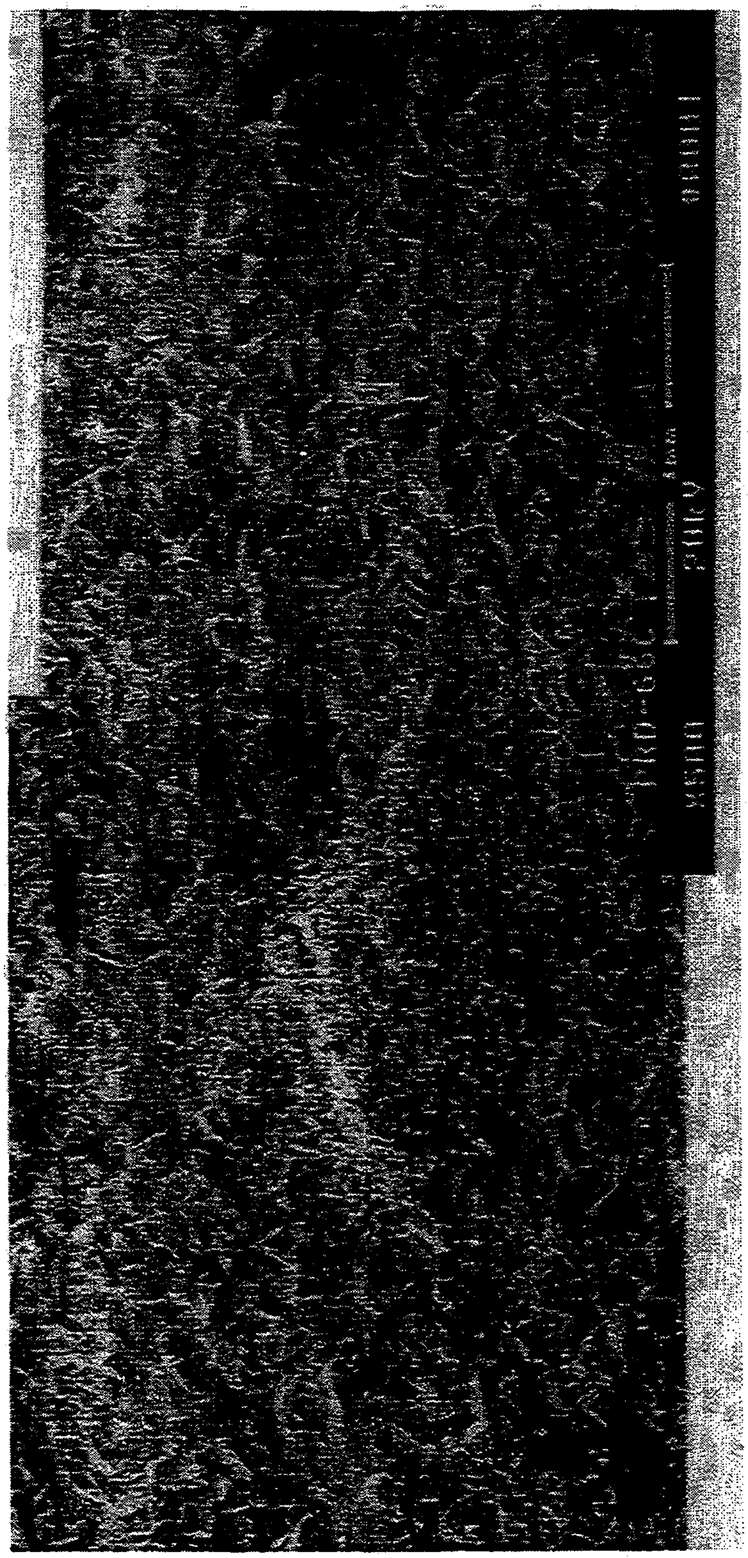


Figure 10 – Micrograph montage illustrating localized adherence of ash fines along the outer surface of the qualification-tested, coarse membrane-coated, PRD-66 filter element.

Figure 11 – Higher magnification micrograph montage illustrating the adherence of ash fines between adjacent alumina-rich grains present along the outer surface of the qualification-test, coarse membrane-coated, PRD-66 filter element.

Figure 12 – Micrograph montage illustrating the morphology of the cross-sectioned filter wall of the qualification-test, coarse membrane-coated, PRD-66 filter element.

Figure 13 – Adherence of ash fines along the surface of the alumina-rich grains that were present within the outer surface membrane of the qualification-tested PRD-66 filter element.

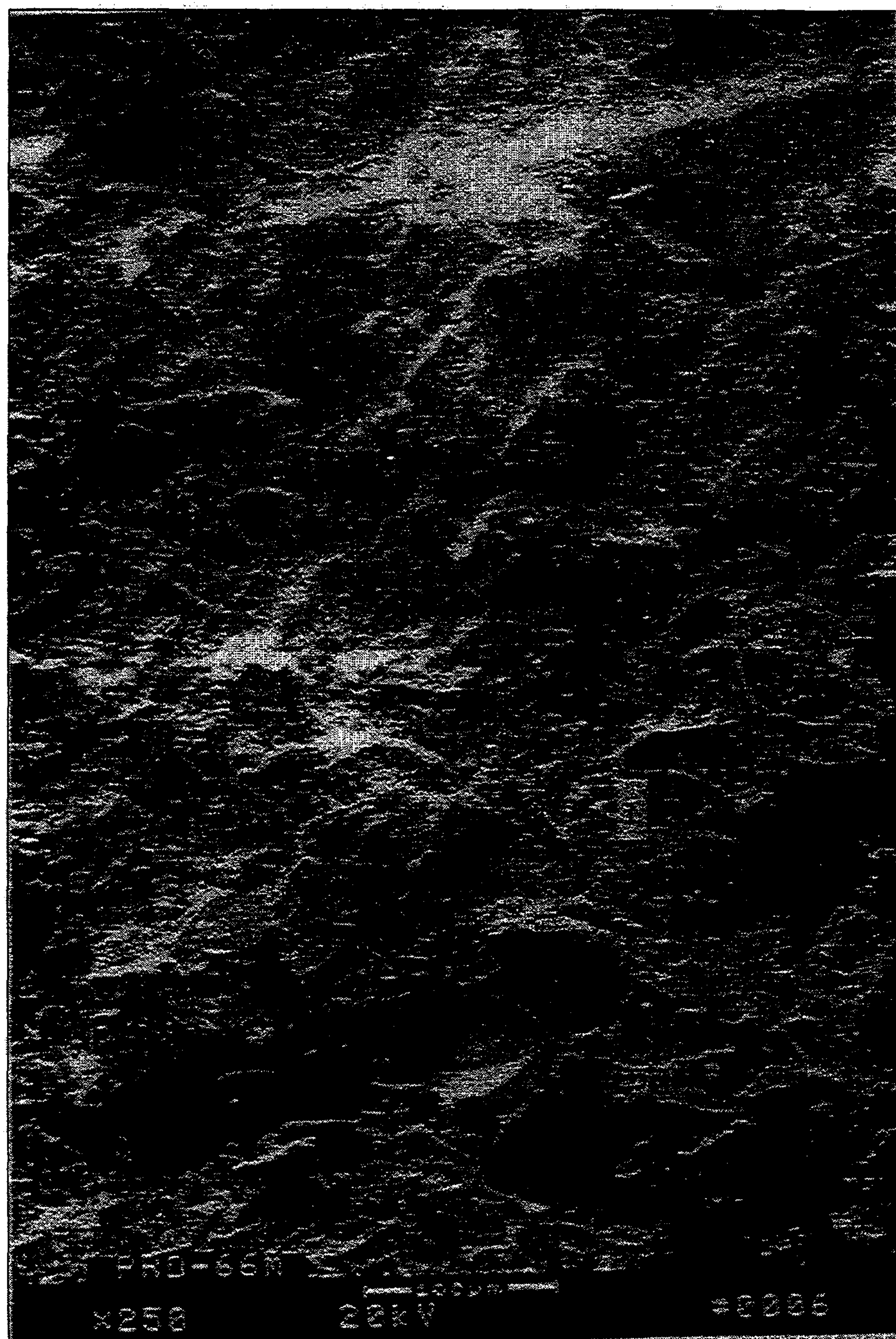


Figure 14 – Micrograph montage illustrating localized adherence of ash fines along the outer surface of the qualification-test, medium membrane-coated, PRD-66 filter element.

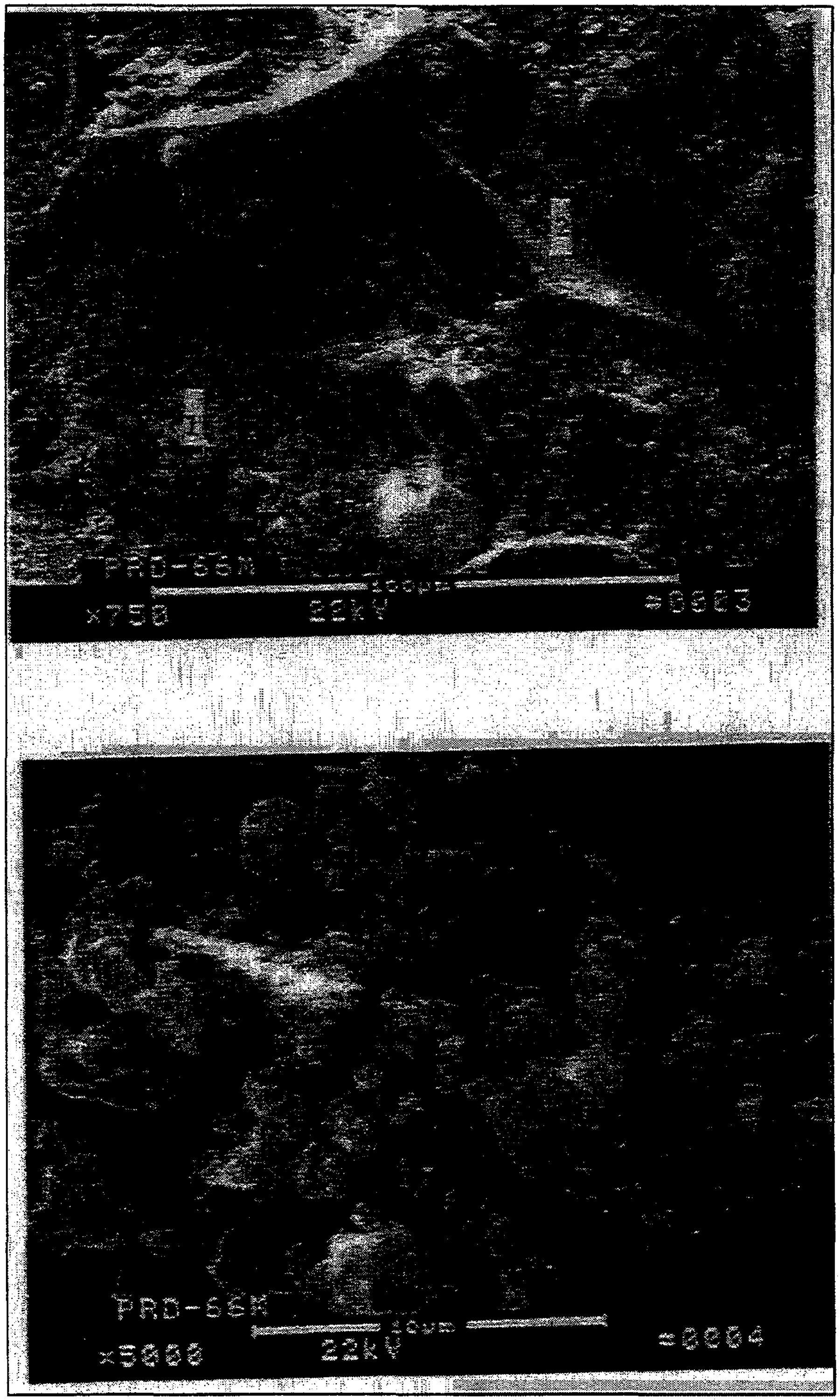


Figure 15 – Higher magnification micrographs illustrating the adherence of ash fines between adjacent alumina-rich grains present along the outer surface of the qualification-test, medium membrane-coated, PRD-66 filter element. The highly porous network of ash fines is shown in the lower micrograph.

Figure 16 – Micrograph montage illustrating the morphology of the cross-sectioned filter wall of the qualification-test, medium membrane-coated, PRD-66 filter element.

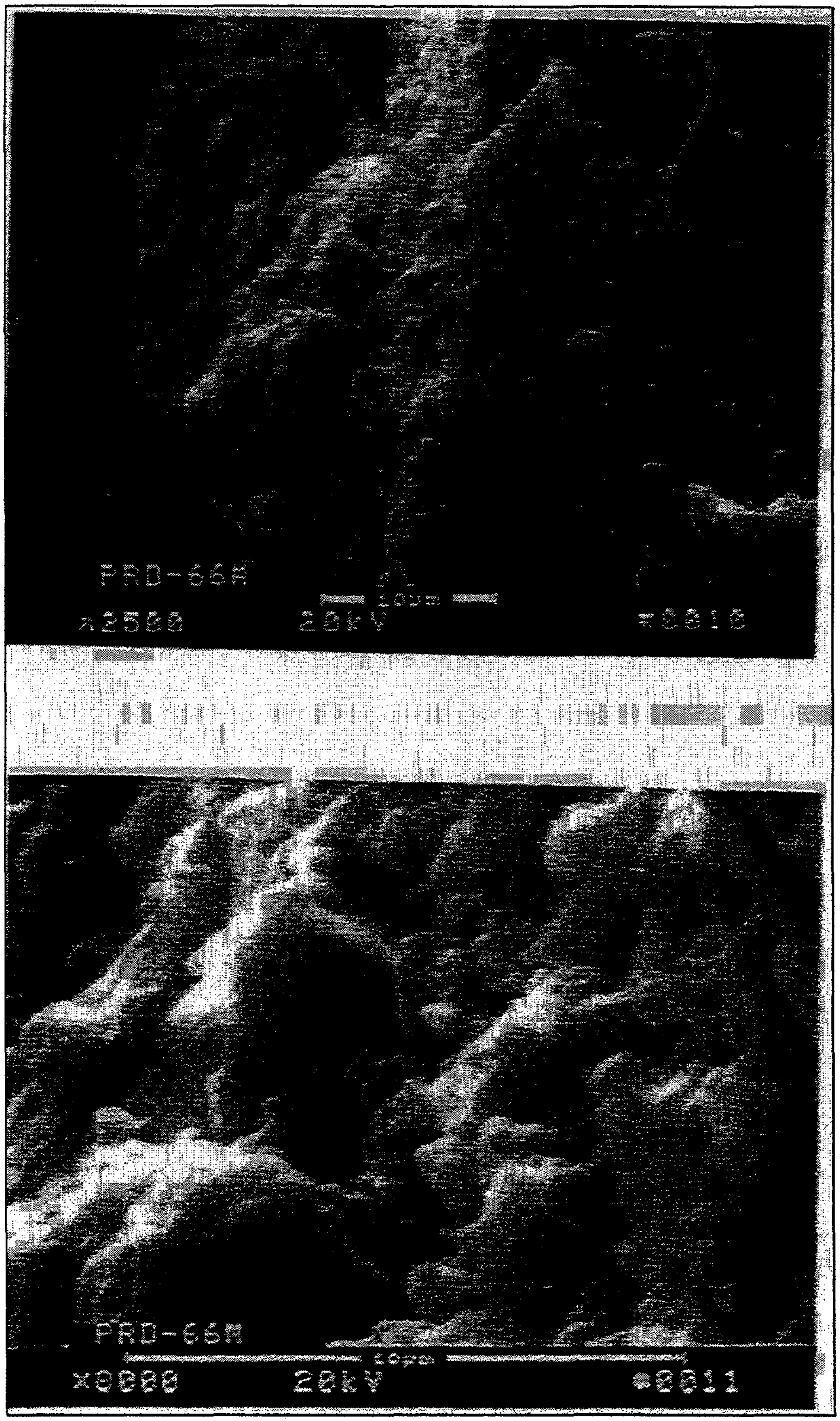


Figure 17 – High magnification micrographs illustrating the adherence of ash fines along the outer surface of the alumina-rich grains that were present within the membrane of the qualification-tested, medium membrane-coated, PRD-66 filter element.

Comment

Limited penetration of ash fines into the membrane-coated filament wound filter matrix was identified for sections of the PRD-66 filter elements examined in this effort. Characterization of additional sections removed from the qualification-tested filter elements, and extended field operation (i.e., >500-1000 hours) are needed to confirm whether the DLC PRD-66 element performs as a barrier vs. bulk filter.

Based on the results of the qualification testing, both coarse and medium membrane-coated filter elements were considered to be acceptable for use in Westinghouse's APF system at the Foster Wheeler PCFBC test facility in Karhula, Finland. In view of the gas flow resistance measurements for the as-manufactured candles, production of the coarse membrane-coated elements was selected as the filter type of choice for use at Karhula.

PCFBC Candle Filter Testing

Twelve, 1.5 m, DuPont PRD-66 candle filters were manufactured with the coarse membrane coating, and shipped to Karhula at the end of July 1997. All twelve filter elements arrived intact, and were initially inspected, prior to consideration for inclusion within the Westinghouse APF. During inspection of the elements, the following comments were made:

- Generally all elements had a smooth outer surface finish
- Questions arose as to whether there would be an acceptable fit of the candle within the metal filter holder due to the extended length of the DLC hemispherical flange
- High intensity light source inserted along the i.d. of each filter element indicated general uniformity along the length of each candle
    - On one or two of the elements, bands of denser areas of matrix were evident near the end caps
    - On several elements, the intensity of the light appeared to be greater than along the body, possibly indicating a thinner area of the matrix
    - If discontinuities existed, they were located at the bottom of the elements, near the end cap
- All end caps were generally uniform
- A section of the matrix (~1-2 mm wide) was removed from the bottom end cap of one element during ultrasonic evaluation. This technique was modified to eliminate material removal during continued testing of the PRD-66 filter elements.
- Only one element had a slightly rougher outer membrane surface.

Seven DLC PRD-66 candles were installed in the bottom array of the Westinghouse APF, and were operated for a period of 342 to 581 hours (i.e., Test Segment 2: September 4, 1997 through November 7, 1997). Table 6 identifies the PCFBC operation conditions during conduct of this test campaign. At the conclusion of the test program, the filter vessel was slow cooled and inspected. All PRD-66 filter elements had remained intact during operation in the PCFBC environment. During removal from the filter array, one element failed at the base of the flange due to binding of the candle with ash in the filter holder mount, and the force required for disassembly. Divoting was not evident along the outer surface of the filter elements, implying that the integrity of the combination membrane had been retained during the first 581 hours of service life. Due to the relatively "soft" and fragile nature of the PRD-66 filter matrix, removal of the membrane (i.e., "nicks") occurred along several areas of the candles during disassembly of the elements from the filter array, as well as during cleaning and subsequent handling.

Summary and Conclusions

- The as-manufactured, outer membrane-coated DLC PRD-66 filter elements achieved the gas flow resistance specifications identified by Westinghouse.
- Continued production modifications have lead to the development and application of a coarse membrane coating along the hoop wrapped, outer surface of the filter elements. After 581 hours of exposure in the PCFBC environment, the integrity of the coarse membrane was retained.
- Further efforts are needed to address the barrier vs bulk filtration characteristics, of the PRD-66 filter element during long-term operation in PFBC, PCFBC, or gasification applications. This includes extensive microstnictural analyses of the elements which have experienced greater than 500-1000 hours of field test exposure.
- Additional efforts remain to be focused on the development and production of the dual membrane, barrier candle filter; further strengthening of the flange; and the incorporation of a chip resistant outer surface.

References

I. M. A. Alvin, "Performance and Stability of Porous Ceramic Candle Filters during PFBC Operation," *Materials at High Temperatures,* 1997, 14 (3), *355-364.*

2. M. A. Alvin, "Advanced Ceramic Materials for Use in High Temperature Particulate Removal Systems,"*Ind. Eng. Chem. Res.* 1996, 35, 3384-3398.

3. M. A. Alvin, T. E. Lippert, B. S. Diaz, E. B. Smeltzer, and R. E. Tressler, "Durability of Ceramic Filters," *Proceedings of the Coal-Fired Power Systems '94-Advances in IGCC and PFBC Review Meeting, 545-571,* DOE/METC-94/10()8.

4. M. A. Alvin, T. B. Lippert, E. S. Diaz, B. B. Smeltzer, and G. J. Bruck, "Filter Component Assessment," *Proceedings of the Advanced Coal Based Power and Environmental Systems 397 Contractor's Review Meeting,* Pittsburgh, PA, July 22-24, 1997.