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Advanced Hot Gas Filter Development

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PATENT STATUS

In July 1997, a patent was filed which covered the development of an improved surface filtration membrane for hot gas filters. This patent was filed as a "continuation-in-part" to the original Hot Gas Filter Patent (5,460,637) owned by DuPont Lanxide Composites Inc. In September 1997, the United States Department of Energy granted DuPont Lanxide Composites Inc. a waiver request for the subject invention, DOE Docket No. S-88,782. In November 1998, this patent was approved by the United States Patent Office. At the time of this publication, the patent number had not yet been assigned.

TECHNICAL STATUS

This technical report is being transmitted in advance of DOE review and no further dissemination or publication shall be made of the report without prior approval of the DOE Project/Program Manager.

CONTRACTOR'S NOTE

Contract #DE-AC21-94MC31214 was awarded to DuPont Lanxide Composites Inc. in September 1994. In August 1998, DuPont Lanxide Composites Inc. was acquired by AlliedSignal Inc., and renamed AlliedSignal Composites Inc. Novation of this contract was performed by DCMC in January 1999.

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1. EXECUTIVE SUMMARY

DuPont Lanxide Composites, Inc. undertook a forty-month program, under DOE Contract DE-AC21-94MC31214, in order to develop hot gas candle filters from a patented material technology know as PRD-66. The goal of this program was to extend the development of this material as a filter element and fully assess the capability of this technology to meet the needs of Pressurized Fluidized Bed Combustion (PFBC) and Integrated Gasification Combined Cycle (IGCC) power generation systems at commercial scale.

The principal objective of Task 3 was to build on the initial PRD-66 filter development, optimize its structure, and evaluate basic material properties relevant to the hot gas filter application. Initially, this consisted of an evaluation of an advanced filament-wound core structure that had been designed to produce an effective bulk filter underneath the barrier filter formed by the outer membrane. The basic material properties to be evaluated (as established by the DOE/METC materials working group) would include mechanical, thermal, and fracture toughness parameters for both new and used material, for the purpose of building a material database consistent with what is being done for the alternative candle filter systems. Task 3 was later expanded to include analysis of PRD-66 candle filters, which had been exposed to actual PFBC conditions, development of an improved membrane, and installation of equipment necessary for the processing of a modified composition.

Task 4 would address essential technical issues involving the scale-up of PRD-66 candle filter manufacturing from prototype production to commercial scale manufacturing. The focus would be on capacity (as it affects the ability to deliver commercial order quantities), process specification (as it affects yields, quality, and costs), and manufacturing systems (e.g. QA/QC, materials handling, parts flow, and cost data acquisition).

2. INTRODUCTION

Advanced, coal-based power plants will require durable and reliable hot gas filtration systems to remove particulate contaminants from the gas streams to protect downstream components such as turbine blades from erosion damage. It is expected that the filter elements in these systems will have to be made of ceramic materials to withstand goal service temperatures of 1600°F or higher. Recent demonstration projects and pilot plant tests have indicated that the current generation of ceramic hot gas filters (cross-flow and candle configurations) are failing prematurely. Two of the most promising materials that have been extensively evaluated are clay-bonded silicon carbide[1, 2] and alumina-mullite porous monoliths. These candidates, however, have been found to suffer progressive thermal shock/fatigue damage, as a result of rapid cooling/heating cycles. Such temperature changes occur when the hot filters are back-pulsed with cooler gas to clean them, or in process upset conditions, where even larger gas temperature changes may occur quickly and unpredictably.[9] In addition, the clay-bonded silicon carbide materials are susceptible to chemical attack of the glassy binder phase that holds the SiC particles together, resulting in softening, strength loss, creep, and eventual failure.[1]

To address these issues, Du Pont Lanxide Composites (DLC) developed a unique and innovative new candle filter made from a ceramic material called PRD-66. This material, an extensively microcracked structure comprising a mixture of crystalline oxide phases (primarily mullite, cordierite, and corundum). It combines the high chemical stability inherent in the oxide ceramics with a thermal shock resistance typically found only in state-of-the art, fiber-reinforced, ceramic matrix composites. The highly microcracked structure provides an effective mechanism for stopping crack propagation through the material, resulting in a toughened structure that responds to high impacts, that would cause catastrophic brittle fracture in monolithic structures, by forming dents.[7]

An additional attribute of PRD-66 ceramic structures is that unlike many whisker-reinforced ceramic composites, they contain no respirable ceramic fibers. This makes handling, installation, and removal of the filters a simpler task, requiring no special protective equipment or record keeping, necessary to comply with the increasing health concerns and likely regulations governing personnel exposure to non-asbestos respirable fibers (NARFS).[7]

Based on its low-cost ingredients and relatively simple manufacturing process, commercial quantity costs of PRD-66 hot gas filters are expected to be fully competitive with the clay-bonded SiC and alumina-mullite monolithic filters that have been involved in recent demonstration programs.

Prototype PRD-66 candle filters are comprised of a cleanable porous membrane structure over a core that is inherently a bulk filter. Should the membrane become locally damaged by an impact e.g., during installation. The exposed core structure would continue to filter out particulates, until it eventually "blinds", effectively healing the damaged section while the rest of the filter continues to perform as designed.

Early development activity included a preliminary material characterization and the demonstration of acceptable permeability and dust retention properties. One-meter working prototypes were manufactured and tested in cooperation with Westinghouse Science and Technology Center.[2] Testing included short-term, high temperature, high pressure exposure to simulated Pressurized Fluidized Bed Combustion conditions under steady state and thermal transients (accelerated pulse cleaning and turbine trip simulations). Although limited, this testing was sufficiently encouraging to stimulate production of 1.5-meter prototypes with a flange configuration that was designed to allow retrofit in existing demonstration units.

Based on the initial development successes of PRD-66 hot gas candle filter prototypes,[2] the goal of this program was to extend the development of PRD-66 candle filters and fully assess the capability of this technology to meet the needs of PFBC and IGCC power generation systems at commercial scale. The work will emphasize optimizing the filter body and flange configurations, demonstrating goal mechanical durability in qualification testing under normal and "upset" operating conditions, and defining and addressing the key issues involved in manufacturing PRD-66 hot gas filters at commercial scale.

The scientific and engineering rationale for developing PRD-66 as a hot gas filtration media is supported by the following evidence:

- The chemical stability of these oxides in coal combustion environments is well known.[3]

- PRD-66 has an extended use temperature of over 1200 degrees Celsius (2200° F). This service temperature significantly exceeds the goals of current coal combustion programs, and keeps the way open to higher temperature higher thermodynamic efficiency combustion processes in the future.[7,8]

- Microcracked structures such as this, in addition to being inherently porous filtering structures, are very effective at preventing crack propagation. Because of this microstructure, the thermal shock resistance of PRD-66 is outstanding. In catalyst support applications PRD-66 was subjected to multiple thermal downshocks (theoretically exceeding 10,000°C/second) in turbine trip simulations without damage.[7,8]

- By using highly developed textile and composite forming technologies, the precise location of each yarn can be controlled and structures fabricated with independent control of gas paths, porosities, and backpressure. This allows for the creation of filters having a thin, low pressure drop surface barrier, backed up by a bulk-filter core that acts as a secondary, backup filter to protect the turbine, should the filter surface be mechanically damaged during installation or operation.[7,8]

- The manufacturing process is environmentally clean and neither uses nor generates hazardous chemicals or respirable fibers.

- The manufacturing process is simple, well controlled, and readily scaleable.

- The ingredients (fiberglass yarn and alumina) are inexpensive and readily available. This offers a route to advanced filters that will be price competitive with the current generation of hot gas filters.[7,8]

- DLC has installed capacity that is sufficient to meet the industry's development needs for the next several years. Capacity can be readily expanded with minimal new investment. This offers a clear path to scale-up without requiring the industry to support large capital investments or wait a long time to evaluate or adopt the technology on a commercial scale.

3. TECHNICAL RESULTS AND DISCUSSION

3.1 Background Technology

PRD-66 all-oxide ceramic materials were invented and patented by DuPont and assigned to Du Pont Lanxide Composites (DLC), a joint venture company owned by E. I du Pont de Nemours, Inc. and Lanxide, Inc. A fiberglass yarn is coated with a suspension of alumina in water, and placed by high precision fiber handling techniques, in this case, filament winding, into the net shape of the filter. This preform is allowed to dry, then fired through a proprietary firing cycle. In this firing process, the silica and magnesia in the fiberglass react with the alumina in the slurry to form mullite and cordierite. The surface of the material is unreacted alumina. It should be noted that the fiberglass is consumed in this chemical reaction, and the resulting product is not fiber reinforced.[7]

For several years prior to the initiation of this project, DuPont, DLC, and Westinghouse Electric Corporation cooperated in the fabrication and early testing of hot gas candle filters based on the PRD-66 technology. The result of that collaboration will, hereafter in this report, be referred to as the "baseline" PRD-66 Candle Filter.

The raw materials required to produce a "baseline" PRD-66 Candle Filter are fiberglass yarn (S-2 type, produced by Owens Corning), calcined alumina power (A-17, produced by Alcoa), fumed alumina powder (produced by Degussa), and deionized water.

The flange, body and membrane portions of the PRD-66 Candle Filter are all produced by coating the fiberglass yarn with a precise amount of alumina slurry and winding the coated filament onto a spinning mandrel.[7]

The first step in producing a PRD-66 Candle Filter is the fabrication of the flange segment. This operation is performed, as shown in Figure 1, on a small winder (max. unit length = 6 inches). The slurry-coated yarn is wound onto a 46mm diameter mandrel with a removable plastic sleeve. When the cylindrical structure is 60mm in diameter, the winding is stopped. The "integral flange" and the plastic sleeve are then removed from the flange mandrel, and slid onto the filter mandrel, which had been previously covered with a plastic sleeve along its entire length. The integral flange is positioned at the appropriate position from the tip end of the mandrel.

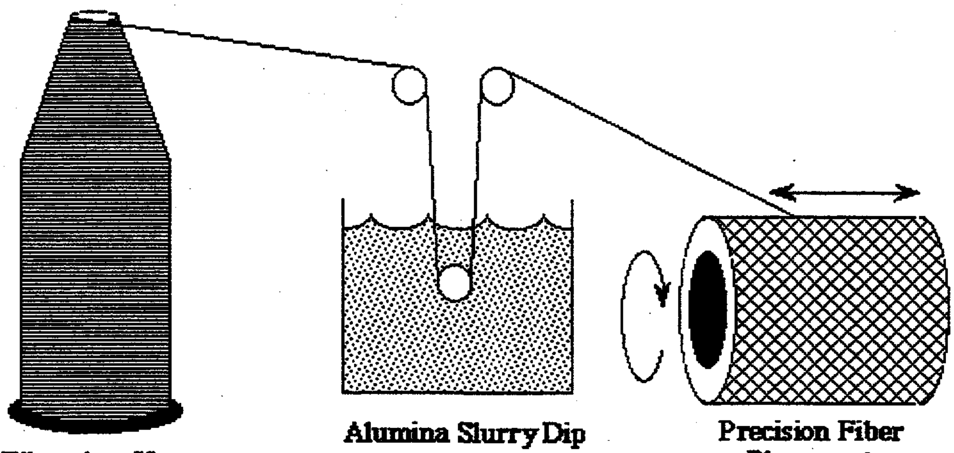


Figure 1 - Schematic of the basic process for winding PRD-66 cylindrical structures.

The winding of the filter support is then performed, as shown in Figure 1, on a winder capable of producing 65-inch long cylindrical structures. As the slurry-coated yarn is applied to the mandrel, it encases the integral flange. Winding proceeds until the outside diameter of the tube is 60mm, yielding a flange diameter of 74mm.

The winding of the membrane yarn is then performed, as shown Figure 2, on a winder which has been specially designed for laying down the yarn at approximately 90° to the axis of the mandrel. The winding begins at the tip end of the candle support structure; each successive "hoop" is laid down immediately adjacent to the previous one. Winding proceeds along the straight portion of the filter, then over the flange portion of the filter, creating a single layer of membrane yarn.

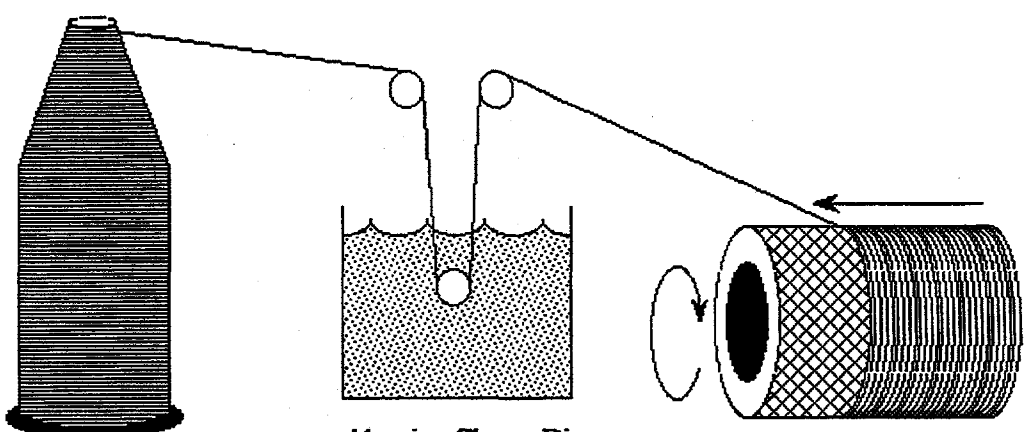


Figure 2 - Schematic of the PRD-66 membrane winding process.

The filter is then dried overnight on the mandrel, cut to length, and removed from the mandrel. A paste-like substance (comprised of the same raw materials as the filter itself) is then used to fill the hole left in the tip of the candle by the mandrel. The filter is then heated to approximately 1400°C in air. During this firing, the alumina coating reacts with the silica, magnesia, and alumina in the glass yarn to form a layered, microcracked structure comprising primarily cordierite, mullite, and corundum.

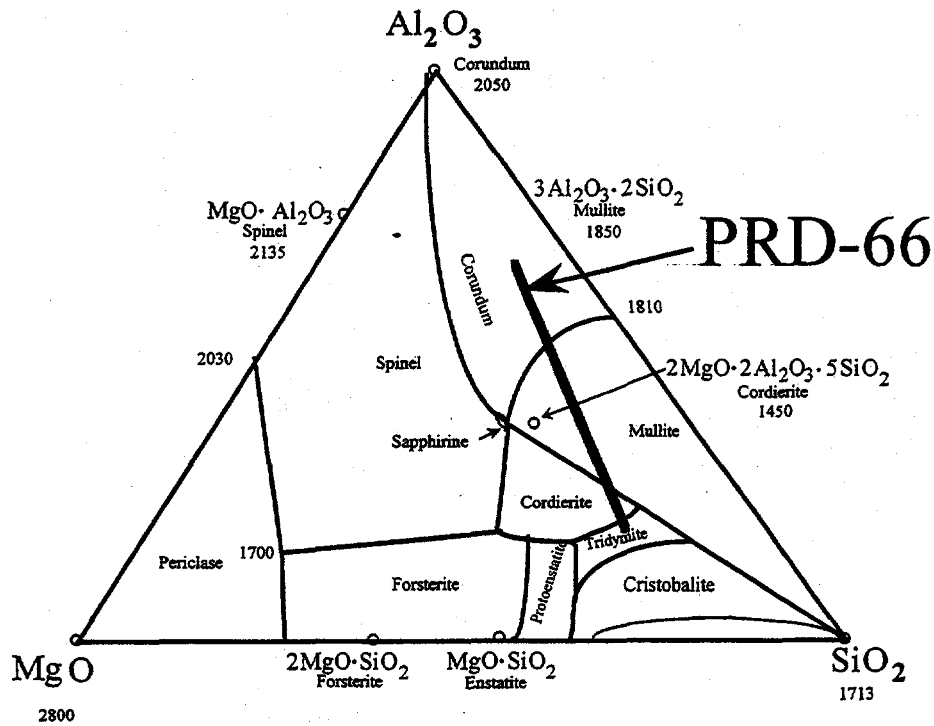


Figure 3 - Phase diagram including PRD-66 composition.

This "baseline" PRD-66 filter was successfully tested in Ohio Power's TIDD PFBC facility in the late summer through early fall of 1994.[2]

3.2 Material Qualification (Subtask 3.1)

In Subtask 3.1, attempts were made to improve the design of the baseline candle filter. The design improvements sought included:

1. improved surface filtration membrane for reduced pressure drop

2. a "dual membrane" filter (with membranes on the inside and outside surfaces) having acceptable backpressure

3. increased strength of the flange region

Full size candle filters, which incorporated these attempts at design changes, were fabricated. These filters were then tested by our subcontractor, Westinghouse Science and Technology Center, to assure that the improved filters still met the fundamental requirements of acceptable permeability and filtration efficiency. After this testing, a decision on which improvements were successful were made, and full sized candle filters incorporating the selected improvements were produced for testing in subsequent tasks.

Also in Subtask 3.1, mechanical property tests suitable for monitoring progress toward stronger filters, and ultimately for process control, were surveyed. After choosing the best test, the mechanical properties of the baseline filter were determined and an evaluation of strength improvements was performed.

3.2.1 *Improving the Surface Membrane*

In attempting to improve the surface filtration membrane on the PRD-66 candle filter, while retaining good filtration characteristics, two properties had to be considered. Firstly, a lower backpressure membrane is desirable. Secondly, a membrane that will release the ashcake more easily is desirable. In the grossest qualitative sense, a smooth appearance on the surface of the filter is thought to be important to good cake release, and can be assessed visually. In the absence of an effective quantitative test, DLC attempted to maintain the same degree of smoothness in the membrane based on visual appearance. DLC had equipment in house to determine if a reduction in backpressure has been achieved and efforts concentrated on reducing the backpressure of the surface membrane.

There were essentially three "knobs" to turn in an attempt to reduce the backpressure of the membrane. They were the type of yarn used in the construction, the ratio of alumina slurry-to-yarn (the matrix ratio), and the spacing of the yarns on the surface of the filter body. Experiments were carried out to turn all three of these knobs in a systematic manner. The results of those experiments are presented in Figure 4.

To vary the yarn type, we chose to hold yarn denier constant at the level in the baseline filter, and vary the yarn twist. The two variations chosen are a twisted yarn and an untwisted yarn. It was expected that the untwisted yarn would flatten on the filter surface yielding a smoother membrane. The matrix ratio is determined by the size of the orifice in a stripper die, which controls the amount of alumina slurry applied to the yarn. To retain proprietary information regarding our process, we'll describe the matrix ratio values as "low" and "high." Finally, we can control the spacing of the surface yarns by adjusting the speed at which the yarn is wrapped around the support. To control proprietary information, we will refer to these yarn spacing as "A" and "B," where "B" has fewer wraps per inch and a larger space between yarns. In these terms, the baseline filter membrane would be described as having been made with twisted yarn, high matrix ratio, and yarn spacing "A".

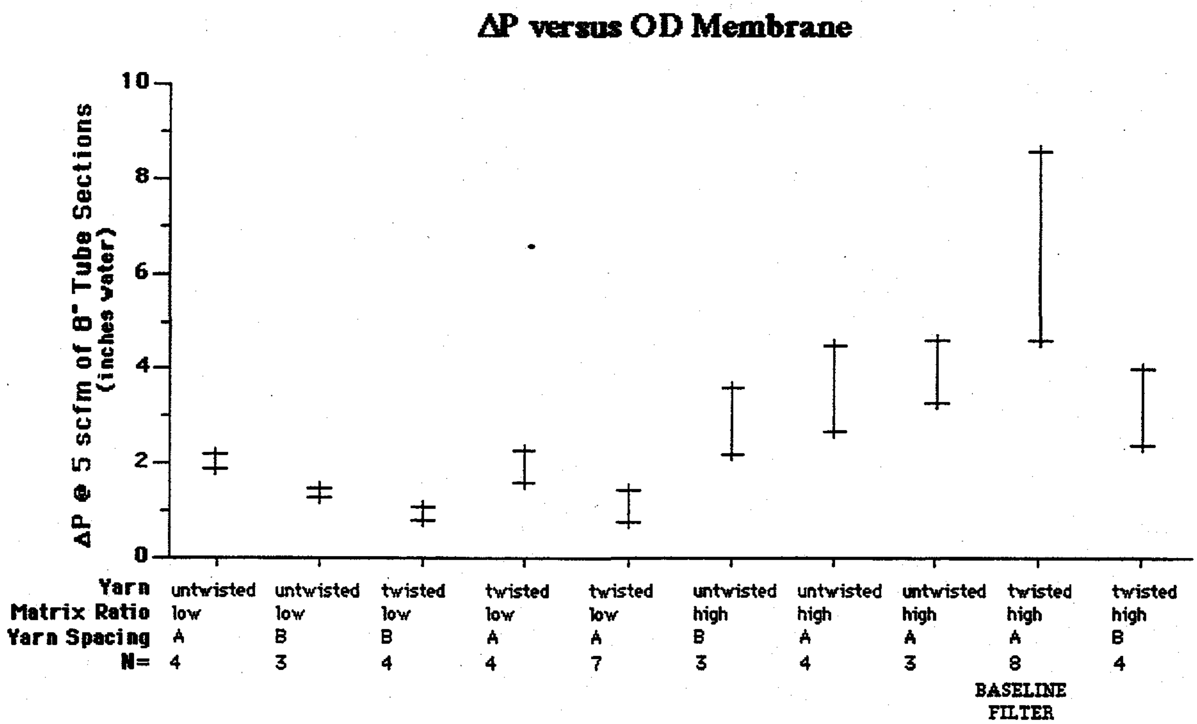


Figure 4 - Impact of three PRD-66 variables on backpressure (ΔP), where "N" is the number of samples.

As seen in Figure 4, every combination of yarn twist, matrix ratio, and yarn spacing examined in these experiments resulted in a reduction in backpressure when compared to the baseline filter. In these experiments, the new combinations were also less variable than the baseline filter. It should be noted that the backpressure measurements were made are on 8" long samples taken from full size filters; the backpressure values presented in Figure 4 ARE NOT EQUAL TO values found on full filters, but they are proportional to them, so comparisons are meaningful.

There is no apparent correlation with yarn type seen in the data, a mild correlation with yarn spacing, and a strong correlation with matrix ratio. Lower matrix ratios have less alumina on the yarns, which probably results in less matrix bridging between adjacent yarns, and a more permeable membrane. It could not be determined if this "lower matrix ratio membrane" would provide an acceptable surface filtration. If it did, the results of these experiments indicate a reduction of surface membrane backpressure by a factor of four is possible.

The choice of yarn spacing is less clear. Yarn spacing 'B' generally gave slightly lower backpressure, but resulted in a visual appearance with randomly spaced gaps in the membrane. These gaps are likely to provide dust leak paths, and therefore poor filtration performance and may adversely effect cake release. DLC, therefore, elected to forego the small drop in backpressure and remain with the baseline yarn spacing.

The untwisted yarn did flatten on the surface of the filter body as anticipated, but was more difficult to manufacture, leading to lower yields and higher costs. It also did not lead to an additional improvement in backpressure when used with the low matrix ratio.

Based on these results, DLC recommended the combination of a twisted yarn, low matrix ratio, and yarn spacing A, because of the low backpressure, retention of a smooth membrane, and ease of manufacture of such filters. DLC manufactured two filters having these parameters for examination by Westinghouse Science and Technology Center; results are discussed in "3.2.5 Filtration and Permeability Testing".

Serious concerns were raised by Westinghouse over poor adhesion of the reduced backpressure membranes. In response, test were conducted with an intermediate matrix ratio, which was more adherent, but still had significantly lower backpressure then the baseline filter; data is shown below in Table 1. New samples were produced for testing; two-inch segments were cut from three locations (flange end, middle, and closed end). Each was sealed around the cut edge and shipped to W-STC for bench-scale permeability and particle filtration efficiency testing.

Position within Candle	**ΔP of 8" Segment** (iwg, inches-water gauge)	**ΔP of 8" Baseline Segment** (iwg, inches-water gauge)
Flange End	2.4	4.7
Mid-Candle	2.0	5.8
Tip End	2.2	5.3

Table 1 - Impact of "intermediate" matrix ratio on backpressure (ΔP) at 5 scfm

3.2.2 Development of a Dual Membrane Candle Filter

During earlier development efforts, Westinghouse expressed a desire to have a membrane along the inside, as well as outside, surfaces of the filter element; this configuration was referred to as a "dual membrane" filter. As a starting point for experiments leading to a "low backpressure, dual membrane" hot gas candle filter, we wound a bulk filter body identical to the baseline filter body, but with no inner or outer membrane. As shown below in Figure 5, this filter segment has an extremely low backpressure. This demonstrates that overall filter backpressure is dominated by the pressure drop at the surface membranes. Efforts were focused, therefore, on developing a “low backpressure, dual membrane” filter with “low pressure drop” outside diameter membranes.

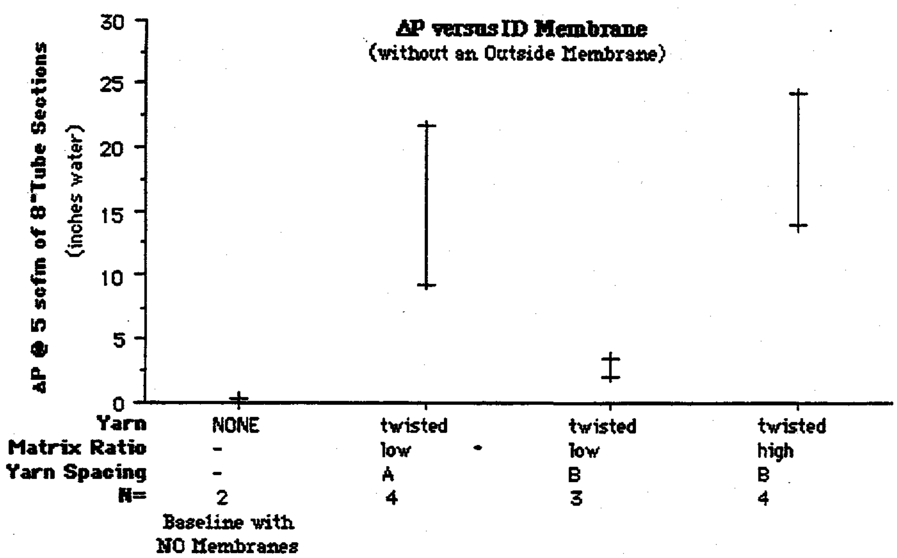


Figure 5 - Impact of inside diameter membrane on backpressure (ΔP), where “N” is the number of samples.

In a manner similar to the experiments described above, experiments were completed to fashion an internal membrane by the same filament winding techniques used for the outer membrane. Instead of winding on the outer body of the candle filter, the internal membrane is wound on the mandrel, and the body of the candle is wound on top of the membrane. Since the wet yarns conform to the surface of the smooth mandrel, a very smooth membrane surface is obtained. We therefore expect excellent cake release from this inner membrane.

Figure 5 shows a backpressure dependence of inner membranes on both “matrix ratio” and “yarn spacing”. No winding conditions could be found which would allow us to make a satisfactory inner membrane with an untwisted yarn. No gaps were formed in the membrane with yarn spacing "A" or "B". The combination of twisted yarns, low matrix ratio, and yarn spacing "B" for the inner membrane, was chosen based on the low backpressure.

Samples of a dual membrane filter using these conditions for the inner membrane and the "medium matrix membrane" conditions described earlier are shown in Table 2.

Position within Candle	ΔP of 8" Segment (iwg, inches-water gauge)	ΔP of 8" Baseline Segment (iwg, inches-water gauge)
Flange End	6.3	4.7
Mid-Candle	9.9	5.8
Tip End	10.3	5.3

Table 2 - Impact of dual membranes on backpressure (ΔP) at 5 scfm.

3.2.3 Mechanical Testing

In order to judge the effectiveness of our experiments to strengthen the flange region of our filters, a reliable mechanical property test was necessary. It was desirable for such a test to minimize the effect of machining damage incurred in fashioning the test specimen, and to be amenable for quality control in future production. Because PRD-66 hot gas filters are made by a process that produces only tubular shapes, it was impossible to manufacture a flat coupon that closely mimicked the internal structure of a PRD-66 filter. Only tests that use cylindrical samples, therefore, were considered. This limited the range to o-ring or c-ring tests. C-ring tests were subjectively evaluated, but cutting the 1-inch slot from the coupon incurred machining damage and an additional fixturing cost would have been necessary to achieve reproducible slot geometries. O-ring tests were ideal in that they required only two, easily controllable cuts to sample a tubular product. Since o-ring tension tests require more complicated and costly fixtures, a simple o-ring compression test was most favored. Figure 6 shows a load deflection curve typical of the o-ring compression tests carried out in this project.

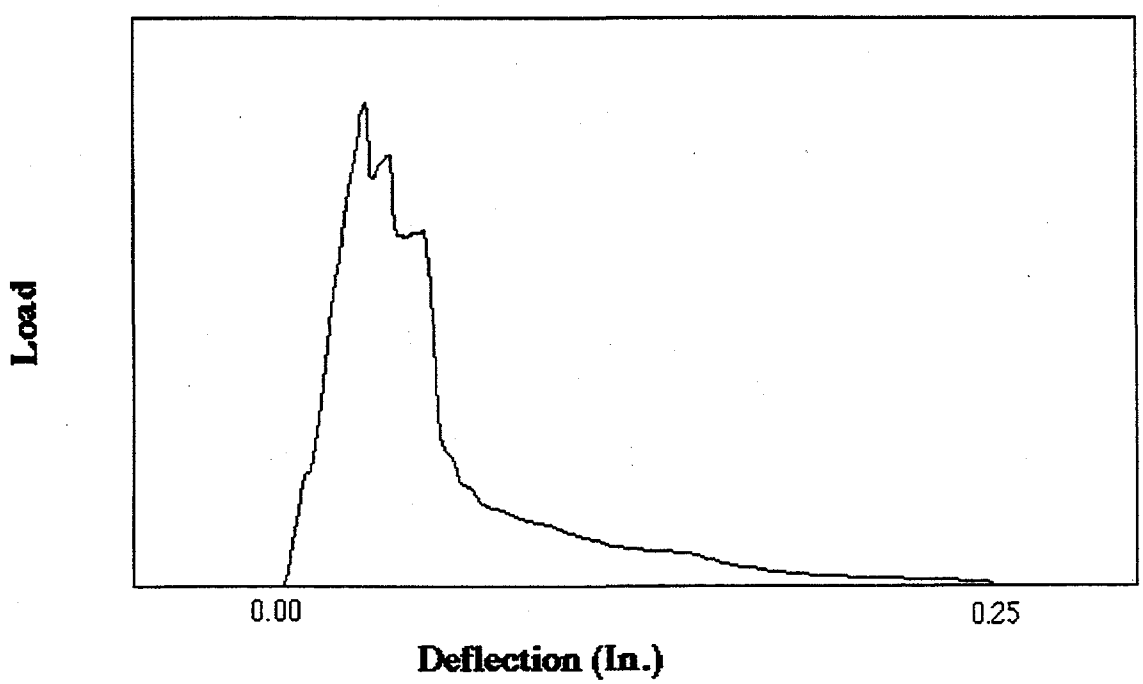


Figure 6 - Typical Load Displacement Curve for PRD-66 filter segment.

This displacement curve reveals a great deal of reloading and strain tolerance after peak load is achieved. Tests that were carried out until essentially no load resistance was encountered often had deflections as high as 0.25 inches, or roughly the same as the wall thickness of the sample. As shown in Figure 7, the samples were intact, though macroscopic cracks were readily visible. In the 100 or so mechanical tests conducted in developing this o-ring diametrical compression test, no sample fractured instantly into two or more pieces.

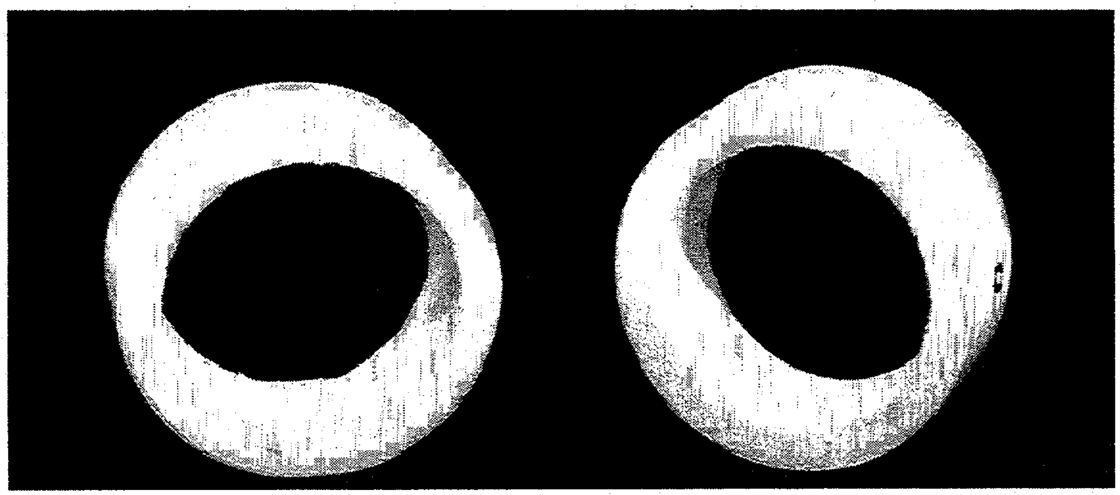


Figure 7 - O-rings AFTER (left) and BEFORE (right) diametrical compression testing.

The diametrical compressive strength was determined by the maximum peak at which the first crack occurred. To characterize PRD-66, forty-one 1-inch wide samples, from three different production filters were tested. The average crushing strength on the samples was 410 psi (std.dev. = 38 psi). This is significantly lower than the results of Westinghouse Science and Technology Center's tests, which reported strengths of 1050 psi on ¾" wide o-rings. Unfortunately, the DLC records, which detailed the exact calculations used, were not available, however, a more accurate equation was adopted approximately a year after the original data was generated. In the later equation, developed by O.M. Jadaan, et al.[10], stress is defined as follows:

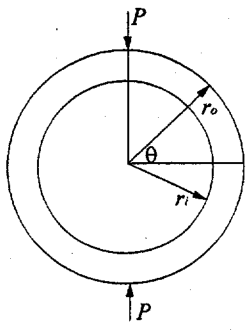
$$\sigma_\theta = \frac{P}{2}\left[0.637\,\frac{r_a y}{I} - \cos(\theta)\left(\frac{1}{A} + \frac{r_a y}{I}\right)\right]$$

where $I = \frac{1}{12}b(r_o - r_i)^3 = \frac{1}{12}bt^3$

$y = r_a - r$

$A = b(r_o - r_i) = bt$

$P = \text{load}$



Where I is the moment of inertia, t is the thickness, and A is the cross-sectional area.

Using the new equation, a sample of the old data was recalculated as shown in Table 3. These strength values agree much better with data reported at W-STC. All data analysis that was conducted during Task 3, however, was performed with the old equations, which yielded lower values.

	Load (lbs)	Old Strength Value (psi)	New Strength Value (psi)
Filter #316 (n=13)	33	417	1057
Filter #317 (n=16)	35	369	934
Filter #318 (n=16)	41	423	1071

Table 3 - Average o-ring diametrical compressive strength ("n" is the number of samples).

A Weibull analysis, shown in Figure 8, was conducted on the original data after calculating the failure strengths of each of the o-rings at the point of maximum stress on the load/deflection curve (Figure 6). The resulting failure stresses were then used to obtain parameter estimates associated with the underlying population distribution.[11] PRD-66 behaved as expected for a porous ceramic material, with a Weibull modulus around "4". Significantly more data would be necessary to correct for statistical bias errors and calculate confidence bounds.

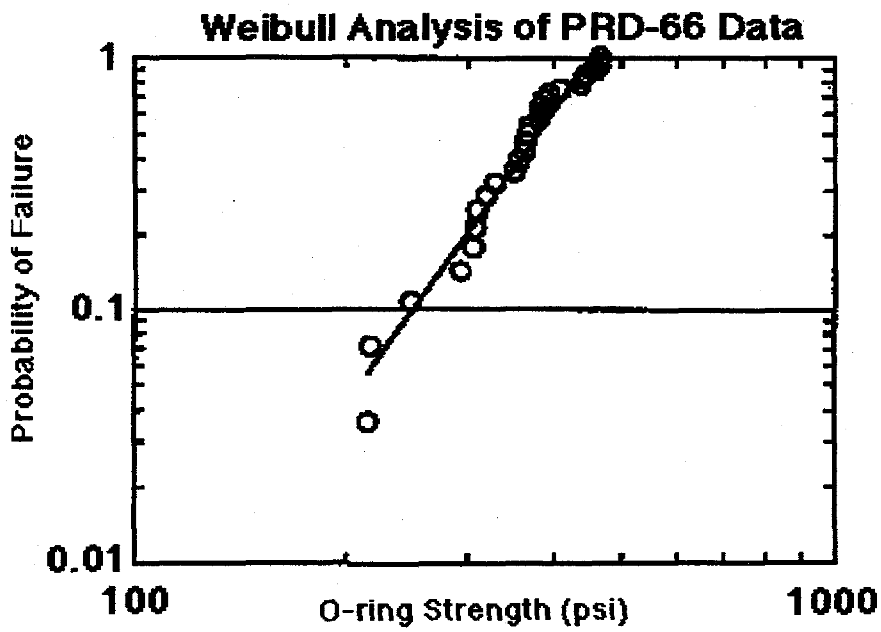


Figure 8 - Weibull Analysis of PRD-66 candle filter segment.

Additional o-rings were tested at various rates of applied stress, as determined by the crosshead speed of the apparatus. When the average strengths were plotted in Figure 9, their was no obvious strain rate dependence for PRD-66, additional data would be required, however, to verify statistical significance.

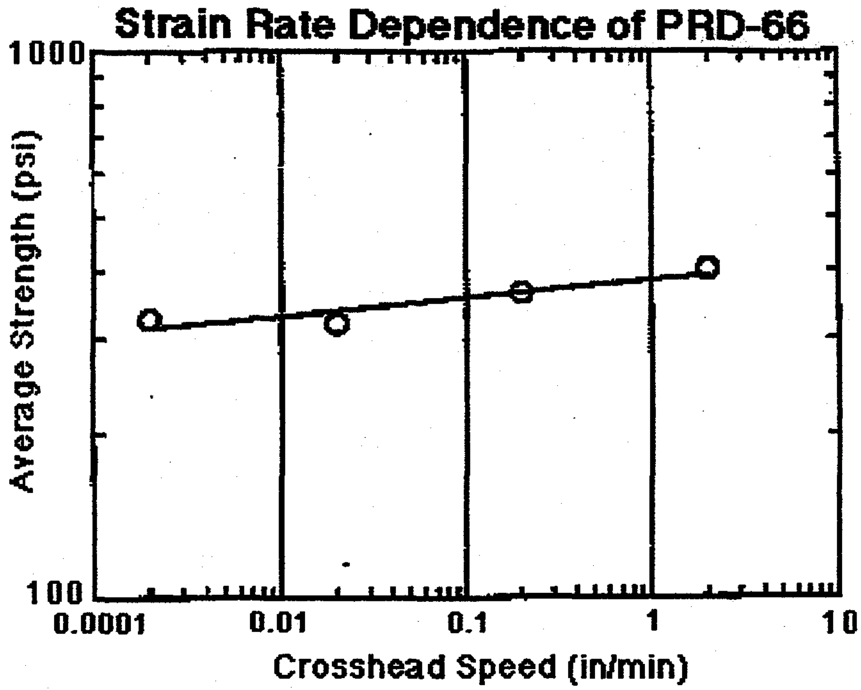


Figure 9 - Strain rate dependence of o-ring crushing test.

3.2.4 Strengthened Flanges

In previous experiments, it had been shown (although somewhat qualitatively) that selective reinforcement of PRD-66 filters can be obtained by adding slurry to portions of the filter in need of reinforcement after winding the filter body. This was of particular importance in view of early tests conducted at Westinghouse (and reported verbally to DLC) in which failure of the filter element occurred just below the flange. Since that time, the holder assembly was redesigned by W-STC and a method was developed by DLC to add controlled amounts of slurry to the areas requiring reinforcement.

Filter samples were fabricated with a range of slurry additions (10, 15, or 20 cc) introduced to portions of the bulk filter body. Three different slurry viscosities were also tested to examine whether the infiltrated slurry stayed where it had been applied or migrated into adjacent regions. To control for filter-to-filter variations, replicate samples were taken from several different filter bodies, and at different points along the body.

As seen in Figure 10 and Figure 11, there is a strong positive correlation between both the "weight gain" and "volume of added slurry" with o-ring crushing strength of 1-inch segments of the filter. Overall, strength increased from about 400 psi for uninfiltrated sections to about 600 psi for fully infiltrated samples, about a 50% increase in strength.

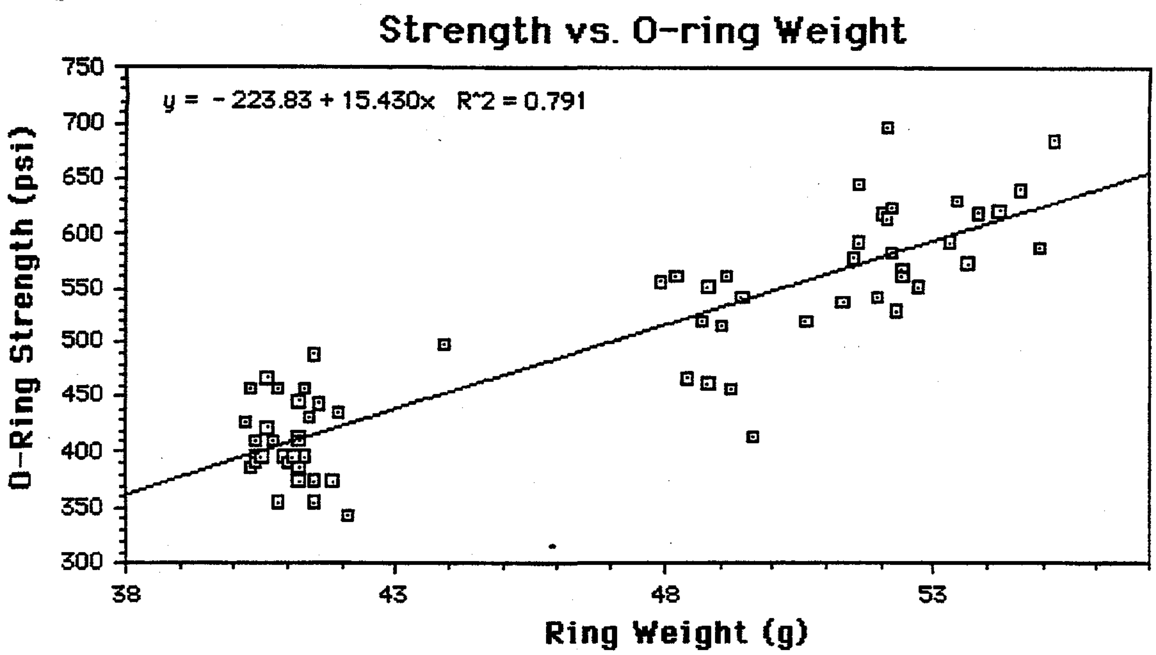


Figure 10 - O-ring diametrical compressive strength versus ring weight.

Higher viscosity slurries (Figure 11) achieved higher strengths with less slurry addition, and lower viscosities took more slurry to attain the same strength. This is probably due to migration of the slurry out of the test segment into the regions adjacent to it, which would result in less effective reinforcement.

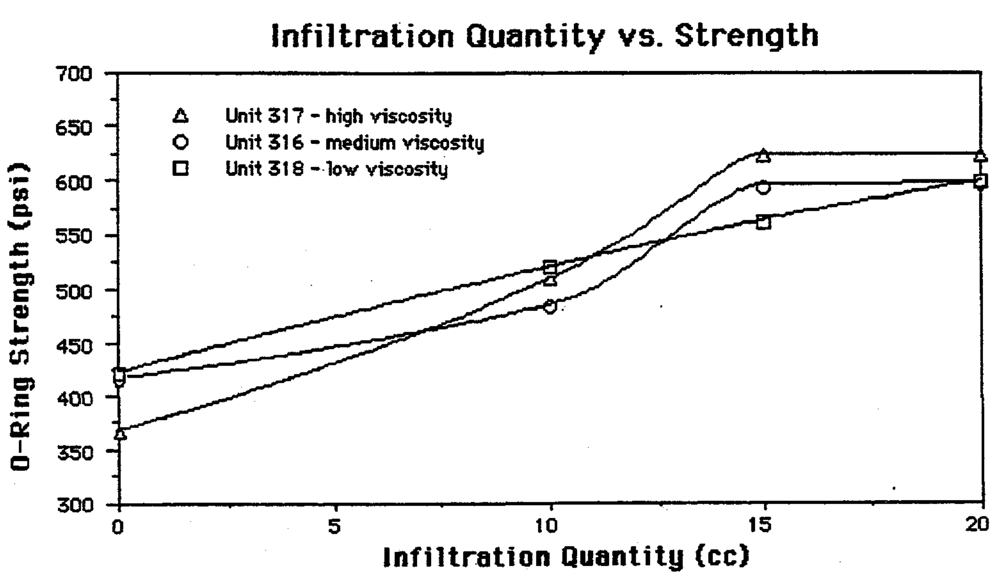


Figure 11 - O-ring diametrical compressive strength vs. amount & viscosity of infiltrate

There appears to be a greater tendency toward brittle failure with the infiltrated material, but, as shown in the load displacement curve of Figure 12, there is still quite graceful failure. We interpret the more triangular shape plot after maximum load (compared to Figure 6) as an indication of more brittle failure, but the fact that there is significant reloading after "peak load" still suggests graceful failure. As with the uninfiltrated material, these samples never broke into two pieces, even with deflections as large as 1/4 inch and as many as four independent cracks per specimen.

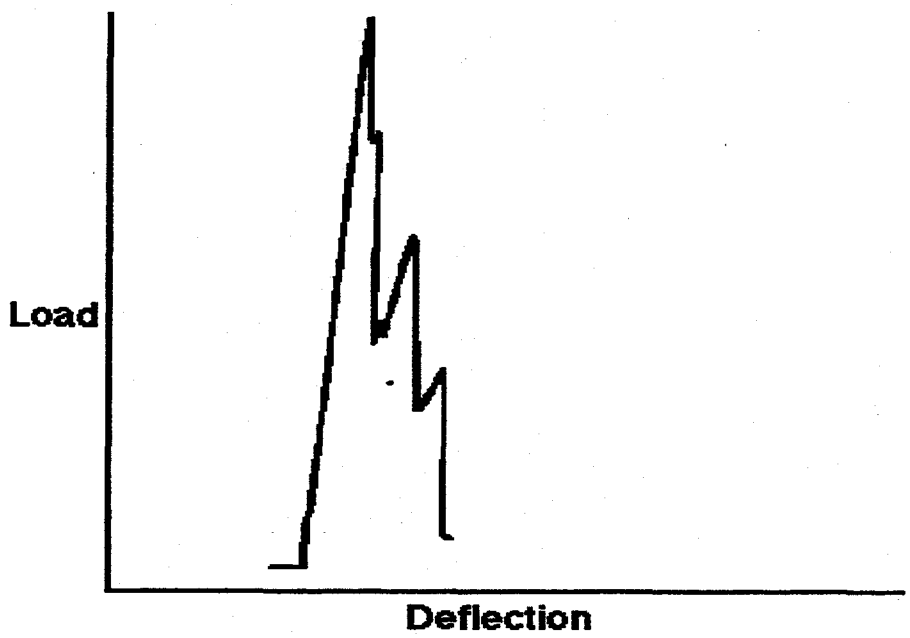


Figure 12 - Load Displacement Curve for infiltrated PRD-66 filter segment.

Seeing no real negative factors in using this new infiltration technique, and a significant benefit, strengthened flanges were incorporated into all three of the improved filter designs, mentioned earlier. Two "baseline" candles with the improved reinforcement technique at the flange were submitted to Westinghouse for testing.

3.2.5 Filtration and Permeability Testing

(Note: the following information, with regard to testing performed by Westinghouse Science and Technology Center (W-STC), was conducted under a subcontract between DLC and Westinghouse, a full copy of the Final Report is provided in Appendix 2.)

Preliminary tests were conducted by Westinghouse Science and Technology Center on 2-inch long filter segments that had low pressure and dual membranes. Dust was delivered to each sample's outside diameter at room temperature for ~3 minutes. Both the clean ID appearance, as well as the absence of detectable fines in the off-gas stream indicated excellent particle collection efficiency, by Westinghouse standards. When a tested specimen was fast-fractured, fines were evident below the outside diameter surface. Penetration within the 7-mm thick wall was apparent to a depth of 1 to 3 mm.

As mentioned earlier, DLC fabricated the following 1.5-meter candles for testing: two with improved (low pressure) outside membrane only, two dual membrane candles, and two "baseline" candles, ALL with strengthened flanges. Westinghouse performed room temperature gas flow resistance measurements on all six candles; results are show in Figure 13. These results parallel measurements conducted at DLC.

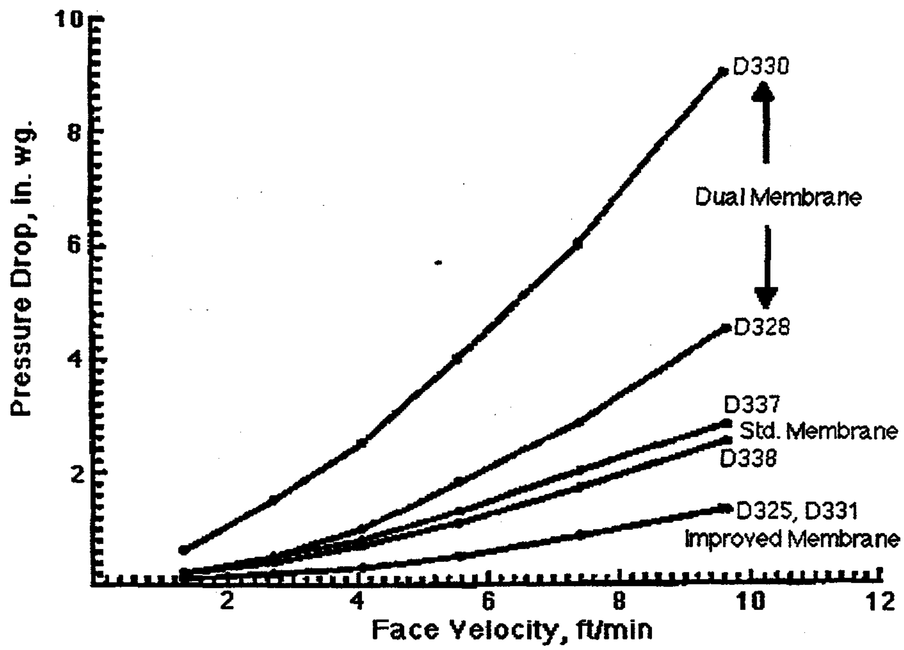


Figure 13 - W-STC Room Temperature Gas Flow Resistance measurements of 1.5-meter PRD-66 filter elements with various membranes.[12]

Westinghouse concluded that the "baseline" and the reduced backpressure membrane filters had flow restrictions within their specification range. The flow restriction of the two dual membrane filters did not agree with each other and one exceeded the pressure drop specification of <1 in-wg/fpm.

After two hours of high temperature exposure in Westinghouse's HTHP facility, outer membranes on the reduced backpressure and dual membrane filters delaminated. This was the most probable failure mode of these candles. The "strengthened flange" filter, which had the baseline surface membrane, did not delaminate.

3.3 Field Testing of "Baseline" PRD-66 Filter Elements

Prior to the beginning of this program, PRD-66 hot gas candle filters (baseline filters) were tested at Westinghouse Electric Corporation's Science and Technology Center.[2] Testing on two-inch filter segments confirmed that PRD-66 filters had acceptable particle filtration efficiency and permeability characteristics in lab scale testing. Westinghouse then exposed full-size, 1.5-meter candle filters to simulated coal combustion filtration conditions in their high temperature high pressure (HTHP) test chamber. That testing confirmed that full-scale candle filters also performed well in filtration efficiency and permeability. Accelerated pulsing and process interruption testing revealed the need for strengthening of the flange region of the filter. After DLC took steps to increase the strength of the filter's flange, further accelerated tests which simulated 6000 hours of filtration were successful.

To identify the thermal/chemical stability of the PRD-66 material, W-STC subjected 10" mini-candles to 400 hours at 870°C, in a 5-7% steam/air environment at 1 Atm. Additional samples were subjected to 400 hours at 870°C, in a 20ppm NaCl/5-7% steam/air environment at 1 Atm. X-ray diffraction was used to compare the crystalline compositions of the materials. Neither of the test conditions had any measurable effect on the PRD-66 material.[3]

3.3.1 Tidd Test Segment 4

After the testing at Westinghouse, three PRD-66 candle filters were placed into field testing at American Electric Power's Tidd Pressurized fluidized bed combustor (PFBC) filter vessel. The PRD-66 candles were placed in the middle array of the vessel. They were exposed to temperatures up to 760°C and operated for the entire duration of the test segment, 1700 hours with ash loading of 3200 ppmw. All three of the PRD-66 filters survived the test segment and suffered no damage. Upon inspection of the filters after exposure, only a loose, thin (approximately 1/8" thick) ashcake clung to the candles. Despite significant ash bridging problems in the test, no ash bridges were found on the PRD-66 candles. Mechanical property tests performed by Westinghouse on ring segments cut from the exposed filters showed no decrease in mechanical properties after the 1700-hour exposure. The only significant negative finding in the test was that the wall of the PRD-66 filters had become filled with trapped ash. At the time, this was attributed to ash penetration from the inside of the filter, due to ash reaching the "clean side" of the filter vessel from other broken candle filters tested in the same plenum.[2,8]

From the results of this test segment, DLC concluded that PRD-66 candle filters were resistant to attack by the corrosive atmosphere resulting from coal combustion. Further, it was concluded that PRD-66 filters had the necessary mechanical strength to survive filtration and backpulse cleaning for at least 1700 hours of operation. The complete retention of mechanical properties in post-exposure testing suggests that under the conditions in Tidd Test Segment 4, significantly longer useful lives would be possible.

3.3.2 Tidd Test Segment 5

Concurrent with the development of the low-pressure and dual-membrane filter elements under this program, twenty-two "baseline" PRD-66 candle filters (identical to those used in Test Segment 4) were placed in service in Tidd Test Segment 5. After the test, it was discovered that all of the PRD-66 candle filters had experienced significant damage. Two types of failure were observed. The first was a classic flange failure, with filters broken in the holder area where the flange transitions to the filter body. The second failure mode was observed mid-body, with approximately half the filter body remaining intact. In this failure mode, "divots" were taken out of the filter body, appearing as lenticular avulsions greater than a millimeter deep, as shown in Figure 14. In filters with mid-body failures, fracture occurred at these thinned spots in the body wall, often where a "divot-in-a-divot" had removed most of the wall thickness.[2,8]

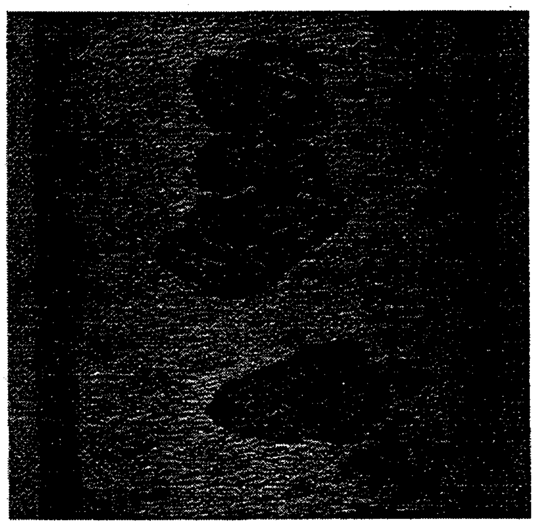


Figure 14 - "Divots" in PRD-66 filter tested in Tidd Test Segment 5.

3.3.3 Analysis of Field Exposed Elements (Subtask 3.4)

To understand the cause of the discrepancy between the results of Tidd Test Segments 4 and 5, DLC undertook Task 3.4 of this program, entitled "Analysis of Field Exposed Filters". This task was carried out in five phases: Consultation, Elimination of Known Faults, Hypothesis Formulation, Hypothesis Verification, and Correction.[8]

3.3.3.1 Phase 1 - Consultation

In the Consultation phase, DLC held discussions with numerous experts in the field of hot gas filtration, including Ted McMahon, Rich Dennis and Dwayne Smith of FETC, Mary Anne Alvin and Rich Newby of Westinghouse Science and Technology Center, Tina Watne and John Holmes of the University of North Dakota's Energy and Environmental Research Center, and Dick Tressler of Penn State. Valuable evidence and insight was gained from these discussions, which is incorporated into following summary.

3.3.3.2 Phase 2 - Elimination of Known Faults

In Phase 2, DLC undertook detailed evaluations of all the manufacturing records for filters supplied to Tidd Test Segment 5 to seek any anomalies in manufacturing which might explain the differences in performance. While some minor changes in the process were found, no process variations correlated with performance. X-ray diffraction tests on the filters fired in the same run with Test Segment 5 filters showed no difference with those in Test Segment 4.

3.3.3.3 Phase 3 - Hypothesis Formulation

Unable to find any significant differences in the filters, Phase 3 focused on physical evidence found in filters which survived Test Segment 5 in whole or in part, and documented differences in run conditions between Test Segments 4 and 5. As shown in Table 4, there were significant differences between Test Segments 4 and 5.

Test Segment	**Tidd 4**	**Tidd 5**
Test Duration	1700 hrs.	1100 hrs.
Survival Rate	100%	10%
Ash Cake	Thin, uniform	Thin, patchy
Damage	None	Divots, mid-body Broken, flange
Bridging	None	None
Operating Temperature	660 - 760°C	760 - 845°C
Ash Loading	3,200 ppmw	18,000 ppmw
Primary Cyclone	De-tuned	Inactive

Table 4 - Comparison of test conditions in Tidd Test Segments 4 and 5.

Ash loading increased from 3,200 ppmw to 18,000 ppmw because of the inactivation of the primary cyclone upstream of the filter vessel. The mean particle size of the ash increased significantly. The highest run temperature increased from 760 to 845°C. Different adsorbents and coals were used. In Test Segment 4, the PRD-66 candle filters were placed in the middle array, while in Test Segment 5, they were in the top array. Two failure modes were observed. One was a classic flange failure, with the fracture locus high up in the holder. These filters, in order to remain identical to the ones tested in Test Segment 4, did not use the selective reinforcement of the flange area described in Section 3.2.4. This reinforcement technique would have increased the strength of the PRD-66 material by about 50%. A second, more puzzling failure, was that found in along the body of the filters. The physical evidence seen on the filters included "divots", as shown in Figure 14.

"Divots" are pieces of the candle filter membrane and body, avulsed from the filter. Such "divots" were found aligned along the filter body on roughly opposite sides. A "divot" was also found under the sock and holder, which eliminates mechanical impact as a cause of the damage. There was no visible evidence of corrosion. The filter body walls were filled with ash, as they had been in Test Segment 4. The body of the filter was covered with a thin layer of loose ash, roughly 2mm thick in most regions. There were also denser ash deposits, aligned with the "divots" described above. All "divots" were packed with dense ash, though some ash-packed "divots" were covered with loose ash. Finally, in Test Segment 5, all filters of all types in the top array were somehow "glued" in place (strongly adhered to their holders). This was not observed in the middle or bottom arrays. Filter segments tested by Westinghouse showed no decrease in mechanical properties after exposure. Finally, micrographs taken at EERC by Tina Watne showed inclusions of a white material, identified by EDX as containing magnesium, calcium, sulfur, and oxygen, well inside the filter body, see Figure 15. This white deposit was of a physical size far too large to have penetrated the undamaged filter above it intact. Undamaged filter areas showed no such deposits.

Based on this evidence, a hypothesis of the failure mechanism of PRD-66 candle filters in Test Segment 5 was formulated. Despite earlier results of room temperature and high temperature tests to the contrary, ash that contained adsorbent penetrated the surface membrane of the PRD-66 filters. This ash then became trapped in the bulk filtering body of the candle. Once trapped there, it was subjected to long term exposure of hot SO2 gas, causing in situ sulfation of the ash to calcium and/or magnesium sulfates in the pores and microcracks of the filters.

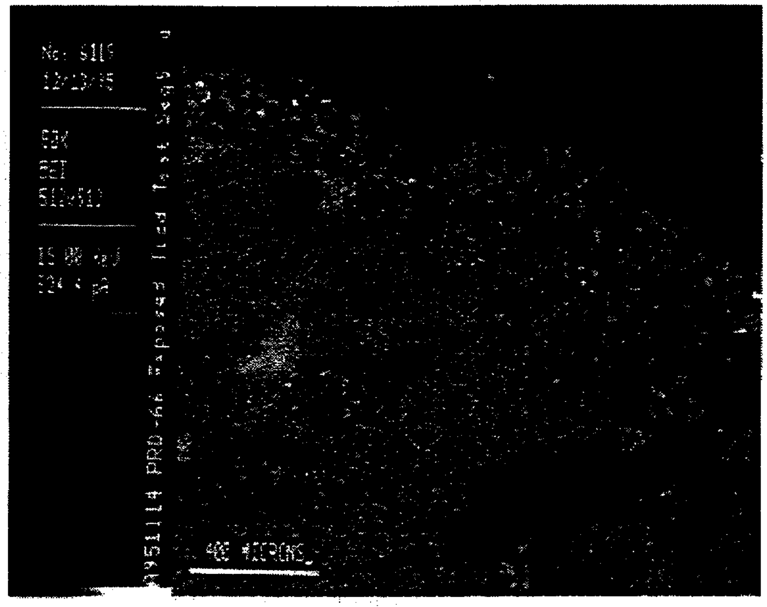


Figure 15 - White deposit (middle left) in vicinity of "divot" (upper right).

Once lodged in a microcrack at high temperature, these deposits could change in size by several mechanisms. One possible damage mechanism is by thermal expansion and contraction of the sulfate deposit during process interruptions, of which there were several in Test Segment 5. A second possible mechanism is by crystal growth from the hydration of sulfates during cooling in a moisture-containing atmosphere, which also would occur on process interruptions. Figure 16 shows how the unit-cell volume of anhydrous magnesium sulfate increases as it picks up waters of hydration.

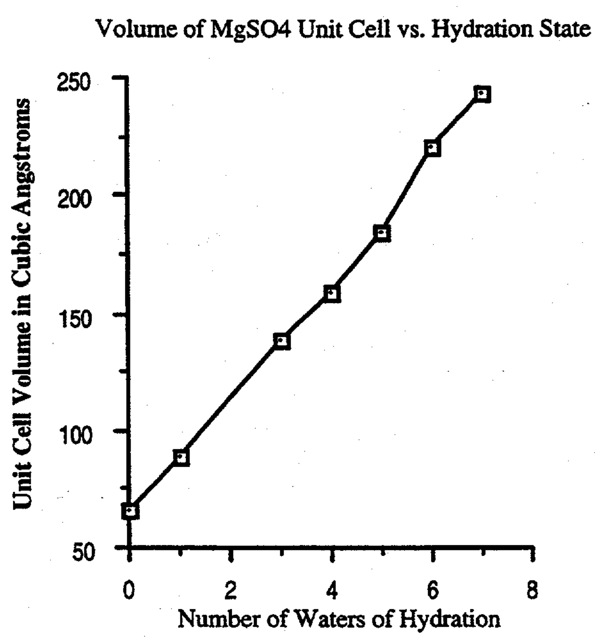


Figure 16 - Unit-cell size of magnesium sulfate versus state of hydration.

The roughly four-fold volume increase associated with formation of the hexahydrate salt would induce a linear strain in a microcrack of over 150%, far larger than the strain tolerance of most ceramics. By either of these mechanisms, severe internal stresses could be placed on the filter body, causing localized failure near a sulfate deposit. In areas where multiple deposits formed, a "divot-in-a-divot" could occur, either fracturing the wall or weakening the wall enough to cause mechanical failure during a backpulse.

3.3.3.4 Phase 4 - Hypothesis Verification

In Phase 4, DLC set out to verify that 1) this hypothesis is in keeping with the known conditions of Test Segment 5, and 2) the possibility of penetration of ash through the surface membrane, contrary to previous test results. DLC found that all conditions necessary for the hypothesis to be true existed in the Tidd test conditions. All that was required was the presence of trapped ash in the filter, the presence of gas phase SO2, and moisture, plus rapid temperature excursions. All these circumstances can be verified from knowledge of the system, the run history, and physical examination of the field exposed filters. To verify that it was possible that ash leaked through what was thought to be 'leak proof' surface membrane, DLC devised a room-temperature test of surface filtration characteristics more rigorous than the ones it had previously passed. In the previous tests, filter segments were exposed to gas flows containing ash. Once a smooth filter cake built up, it was supposed that the ashcake would strongly adhere and then take over filtration. A sample passes the test if no ash penetrates to the inner diameter. Since physical evidence from Tidd Test Segments 4 and 5 showed that the ashcake was thin and only loosely adhered, DLC worked under the assumption that, the surface of the PRD-66 filters released the ash essentially completely on each backpulse. To mimic this ash removal in DLC's laboratory, after exposing filter segments to ash by applying a vacuum to the inner diameter, the resulting ashcake was physically removed with light brushing. This ash exposure/cleaning cycle was repeated 25 times. The intent was to simulate the effect of complete ashcake release after a series of cleaning backpulses. Figure 17 illustrates the apparatus used to conduct this test.

In this test, ash consistently penetrated the membrane of the "baseline" filter and accumulated in the filter wall. Figure 18 shows an example of a 2"-segment, exposed to 25 PIT cycles, viewed with transmitted light; the light source had been inserted into the sample and the examination was performed in a darkened room.

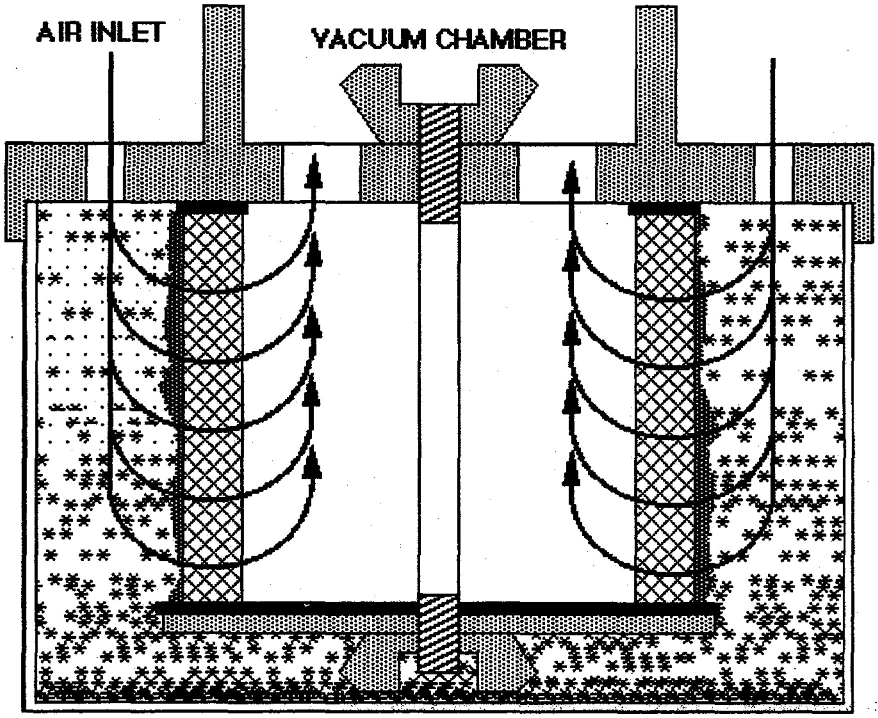


Figure 17 - Particle Infiltration Test (PIT) Device

Figure 18 - PIT-exposed sample viewed in transmitted light.

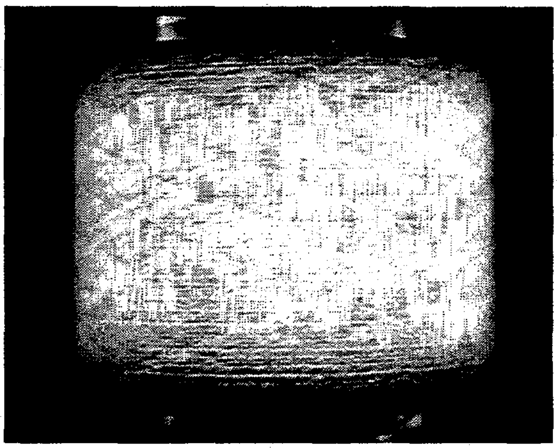


Figure 19 - Untested sample viewed in transmitted light.

When compared to an untested filter segment (Figure 19), areas of ash infiltration appear as dark streaks and spots; in the case of the "baseline" membrane, these areas are many and widespread. Even after the extensive penetration shown in the figure, however, ash still did not penetrate to the inner diameter after 25 cycles. This indicates that the bulk filtering body does trap ash in the wall. Because of the expense associated with recreating the in-situ sulfation of the penetrated ash, no such experiments were conducted.

Further verification of this hypothesis was found by Westinghouse's independent investigation of the failure mechanism. Westinghouse discovered differences in the ash adhered to the filters and uncleaned surfaces in the top array, versus the ash in the two lower arrays. They verified that the filters of the top array were 'glued' in place. Westinghouse also reported the presence of magnesium sulfate hexahydrate in the ash, as found by X-ray diffraction, on uncleaned, stagnant surfaces of the top array, such as the holders and tubesheet. As described above, DLC hypothesized the formation of magnesium sulfate hexahydrate in the filter body as a potential cause of damage, without formally verifying the existence of the compound by XRD. Westinghouse's proof of the formation of the hexahydrate salt verifies that actual system conditions present in Test Segment 5 could cause its formation, and therefore supports the likelihood of DLC's hypothesis. The fact that no such compound was found in the middle array could explain why ash-filled PRD-66 candles in the middle array of Test Segment 4 showed no damage.

The presence of factors that may have contributed to the formation of "divots" was confirmed, but this theory alone could not explain the presence of "divots" in localized areas. The PIT evaluation indicated that ash penetration would occur in over half of the filter surface and examination of the exposed filters showed that the ash was thoroughly imbedded throughout the wall of the entire unit. As a percentage of the outside diameter, the "divots" would account for less than 5% of the surface. A significant contributing factor may have been the presence of regions, within the wall, of poor interlaminar strength. When a PRD-66 candle filter is cut into rings, it is common to observe regions where adjacent layers of yarn have separated from each other, as shown in Figure 20. Occasionally, these defects might extend approximately a quarter of the way around the circumference, and continue for 1-2" inches along the length of the filter element. They have been observed at random depths and positions within the support body and could never be correlated with any process variables.

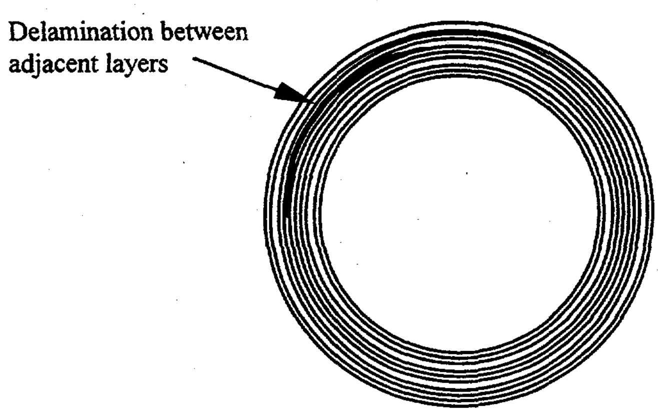


Figure 20 - Exaggerated illustration of a PRD-66 delamination

It is possible that the "divots" were caused by the combined presence of three things: an environment conducive to sulfate formation and hydration, ash entrainment, and localized interlaminar weaknesses. Since DLC has no control over the PFBC environment, corrective action was focused on improving the surface filtration quality of the membrane and reducing the presence of delaminating within the support wall.

3.3.3.5 Phase 5 - Correction

The composition of the "baseline" slurry was fumed alumina, calcined A-17-grade alumina, and deionized water. Observations made during Subtask 3.1 suggested the resulting alumina matrix might not have had adequate bonding strength. It was also noted that in the green state (dried, but not fired), bonds between coated filaments could be damaged when removing the filter from the mandrel. An alternate composition was evaluated in which the fumed alumina in the slurry was replace by aluminum chlorohydrate, as an alumina precursor. This ingredient imparts significant "green strength", unfortunately environmental controls were necessary to deal with the evolution of HCl that results during heating. To remove this hazardous byproduct from the effluent stream, an HCl scrubber was installed and tied-in to a furnace capable of heating to 800°C (the "low-fire" step), under Subtask 3.5. With the use of this new slurry, virtually no delaminations were apparent within the wall of the filter elements, fewer candles were damaged during mandrel removal, and better adhesion between adjacent yarns was been observed.

With regard to the membrane quality issue, Westinghouse's filtration efficiency test exposed the filter to only one ash penetration challenge, and showed no penetration to the inner body. The test protocol assumed that once a smooth ash layer was built up, it would adhere to the filter surface, and thereby take over future surface filtration. The thin, loose ash cakes on PRD-66 filters after Tidd exposures, however, brought that assumption into question. The Westinghouse test protocol also assumed that if ash penetrated the surface membrane, it would immediately show up on the inner diameter. Based on the hypothesis described above, the standards by which a membrane is deemed "acceptable" needed to be changed, at least where PRD-66 was concerned. The PRD-66 membrane would need to function as a much better ash barrier to minimize the risk of "divots" and to reduce the pressure buildup caused by accumulated entrained ash.

For the "baseline" filter, the leakage through the outer membrane appeared to occur through tiny gaps between the adjacent yarns of the "wound-on" membrane. Apparently, the alumina slurry coating on the fiberglass yarns did not consistently bridge the gaps between the yarns and an incomplete membrane formed. Furthermore, gaps appear to occur more frequently, where the membrane yarn covers a primary crossover point in the pattern of the support winding underneath.

Several options were evaluated for improving the quality of the membrane layer

1. a different filament winding pattern for the body
2. a double outer membrane
3. a different type of membrane yarn
4. additions to the membrane layer

To test the efficiency of such alternate membrane technologies, 2-inch test segments were exposed to the Particle Infiltration Test (PIT), described in Figure 17. All samples were examined in transmitted light for areas of ash penetration; a subjective scale of appearance, ranking from "1" (many large wide-spread infiltration areas) to "10" (no detectable areas of ash infiltration), was established. Several specimens of each candidate were generally prepared to evaluate reproducibility.

Another critical aspect of the evaluation was to quantify the backpressure of the experimental membranes. 8-inch specimens of the promising candidates were prepared. Many of the membranes, which were studied, had excellent PIT ratings, but resulted in backpressure above Westinghouse's acceptable limits. For 8-inch long units, tested at 5 scfm, the target was 10 inches water gauge. In some cases, new membranes were evaluated for permeability first; only acceptable candidates were leak tested in the PIT.

Almost one hundred different combinations of the variables mentioned above were tested. A statistical evaluation was not feasible, however, certain conclusions, concerning the effectiveness of the varying approaches, could be drawn.

<u>Filament Winding Patterns.</u> It had been observed that many gaps occurred where the membrane yarn covered a primary crossover point in the pattern of the support winding underneath. Attempts were made to alter the winding pattern of the body to create a smoother surface on which to wind the membrane yarn. Although initial changes looked promising, each new pattern was very time-consuming to model and implement, and produced only marginal improvements. Consequently, no changes were made to the "baseline" winding pattern.

<u>Double Outer Wound Membrane.</u> The addition of a second layer of membrane yarn, on top of the first, was evaluated using a variety of slurry types, yarn spacings, and yarn types. Although several combinations produced units with good PIT ratings, the backpressure exceeded the 10-iwg

target. Consequently, the winding of two outer layers of membrane yarn was not incorporated into the "baseline" product.

Different Membrane Yarns. PRD-66 filters use fiberglass yarn, which is available with varying amounts of twist. It was hoped that by using a less twisted grade, the yarn would lie flatter on the surface of the filter body, the edges of adjacent yarns could overlap, and the gaps could be eliminated. Although this concept was demonstrated, the untwisted yarn was very difficult to work with and broke frequently during winding. Consequently, no yarn changes were incorporated into the "baseline" product.

Additions to the Wound Membrane Layer. Initially, the focus was on filling the gaps between adjacent membrane yarns with ceramic fibers, ceramic particles or ceramic precursors. Although many combinations were effective filters, they had poor permeability (high backpressure). By using these filler materials INSTEAD OF a hoop-wound membrane yarn, permeabilities that are more reasonable were achieved. The contours on the surface of the filter body, however, made reproducibility difficult. The most effective solution was to apply a hoop-wound membrane with intentional gaps between adjacent yarns and then fill those gaps with a material that gave appropriate filtration and backpressure. This membrane modification was incorporated into the "baseline" product and was commonly referred to as a "combination" membrane.

In summary, to correct the problem of the leaky membrane, identified in Subtask 3.4, the most promising approach chosen for further study was a membrane comprised of a "hoop-wound" yarn with a ceramic filler material in-between adjacent windings. To improve the interlaminar strength of the support body underneath, the filter would be fabricated using the modified slurry composition.

3.4 Development of High Efficiency Membrane

To facilitate this addition of a ceramic filler material, a new pattern was chosen for the 'hoop-wound' yarns allowing broader spacing between adjacent yarns. Instead of relying on the microcracks in the alumina slurry to provide adequate filtration, a more controlled material would be used to fill in the gaps and provide a uniform porosity. The approximate relationship of this new spacing to the original membrane spacing is depicted in Figure 21 and Figure 22, showing the additional filler material between the 'wound-on' yarns, and the additional membrane area created in this process.

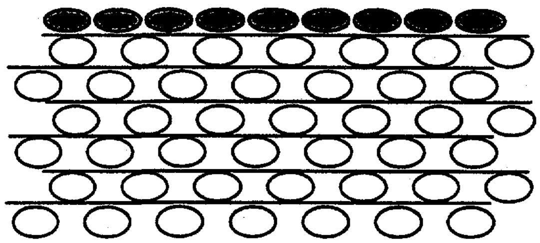


Figure 21 - Original wound membrane (wall cross-section).

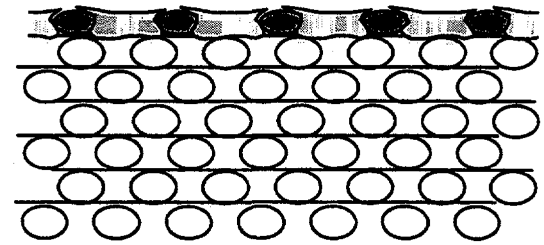


Figure 22 - Membrane with added filler (wall cross-section)

The composition of the filler material was varied over as wide a range of options and a variety of application techniques were attempted. Some of the variables evaluated included:

1. Particulate Alumina: 220-grit, 320-grit, 400-grit, 100-grit tabular alumina, fumed alumina
2. Ceramic Precursors: aluminum chlorohydrate, colloidal alumina, colloidal silica
3. Application Technique: brushing, hand-rubbing, spraying, immersion, squeegeeing
4. State of Filter Body: unfired, partially-fired, fully-fired

The criteria used for comparison consisted of "ease of application", "uniformity", "reproducibility", "adherence", "permeability", and "filtration efficiency". Candidate membranes were selected for further evaluation only if they scored a PIT rating >"9" after 25 exposure cycles. Figure 23 illustrates a unit with a rating of "10". The specimen pictured in Figure 18 would be representative of a rating of "3".

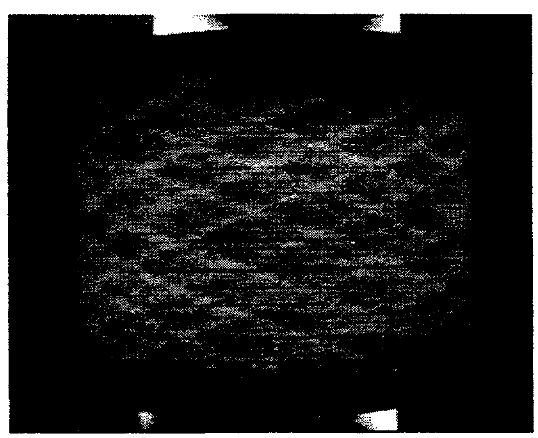


Figure 23 - Modified membrane with PIT rating of "10".

After assessment of a large number of filter segments, another advantage of transmitted light inspection became readily apparent. Any defects, which appeared as ash-infiltrated darkened areas in the PIT tested samples, had also been apparent in the untested samples when examined by transmitted light. Although small membrane defects on the order of 100-200μ diameter were not readily apparent on routine visual inspection (Figure 24), they became visible as intensely bright points of light in transmitted light inspection (Figure 25). Further, these defects were detectable in the filters prior to firing, allowing for the application of additional membrane filler before the final ceramic conversion firing.

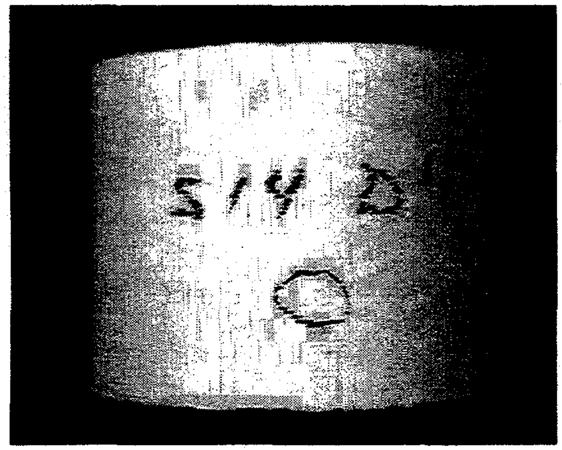


Figure 24 - Hole in membrane, undetectable under direct light.

Figure 25 - Hole in membrane, detectable under transmitted light.

Controlled testing of specimens with membrane defects was conducted. Each sample was examined in transmitted light prior to firing, some pinholes were filled with additional material, and some were left open. Specimens were subjected to 25 PIT cycles. All sites where ash penetration occurred, during PIT exposure, had been easily located prior to firing. None of the filled pinholes showed signs of leakage. No additional defects developed during the final ceramic conversion firing. Figure 26 shows the result of testing a defective segment where a pinhole, detected prior to firing, was allowed to remain.

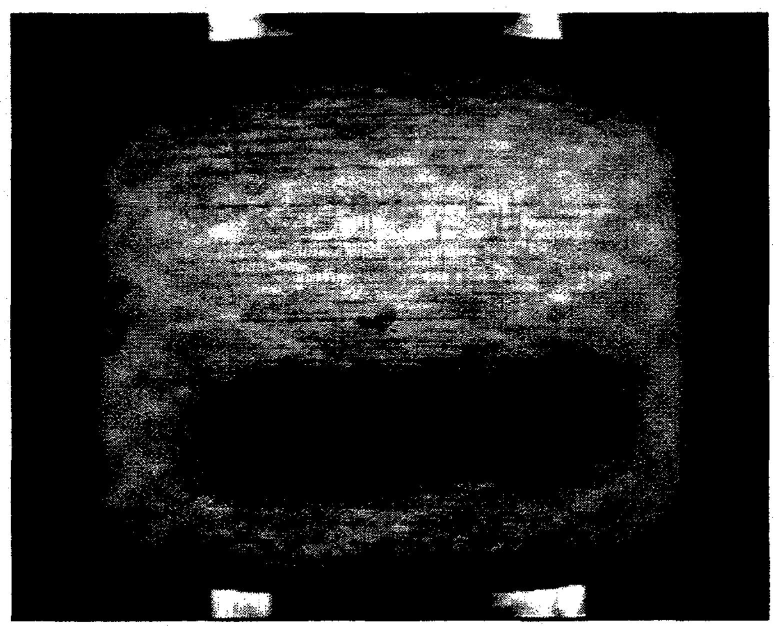


Figure 26 - Hole in membrane after 25 PIT cycles, viewed in transmitted light.

This defect was virtually undetectable when examined in direct light, but immediately obvious in transmitted light. This test and defect elimination procedure was added to DLC's standard manufacturing protocol for 100% of PRD-66 production filters.

From the many candidate membranes tested, two variants were selected for further evaluation. PRD-66M and PRD-66C were selected for their excellent, but different, combinations of filtration performance and flow resistance characteristics. Both of these membrane candidates were processed into full size filter elements for testing at the Westinghouse HTHP facility. PRD-66M has a mean pore size for filtration of about 10.5μ (Figure 27) with flow resistance comparable to the close wound membrane filters. Flow resistance of 1.5-meter filters was tested both before and after HTHP testing, as shown in Figure 28.

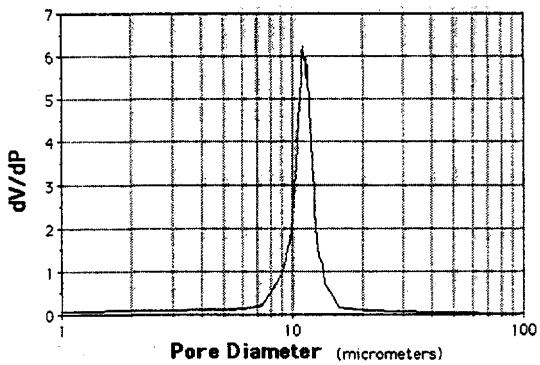


Figure 27 - PRD-66M membrane, measured pore distribution.

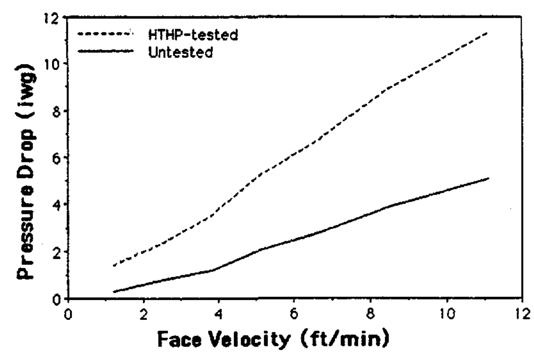


Figure 28 - PRD-66M flow resistance for 1.5-meter candles.

The second membrane candidate, PRD-66C, was chosen because of its unusually low flow resistance in combination with excellent filtration performance. With a mean pore size of about 25μ (Figure 29) its flow resistance is less than half that of filters with PRD-66M membranes (Figure 30).

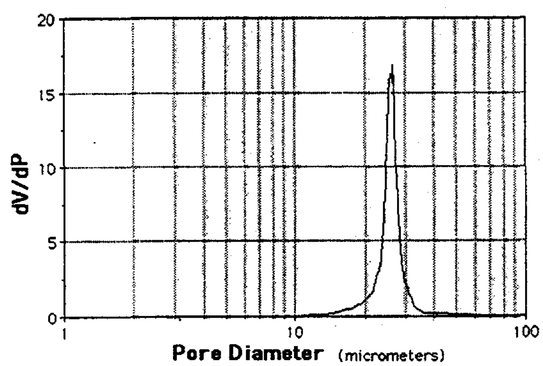


Figure 29 - PRD-66C membrane, measured pore distribution.

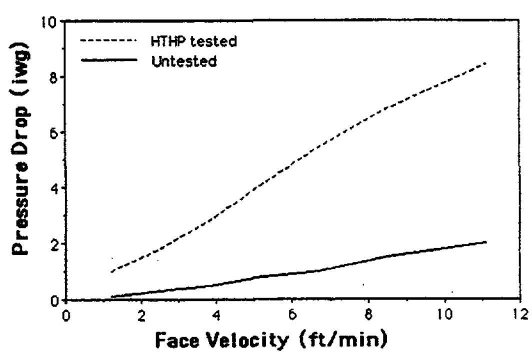


Figure 30 - PRD-66C flow resistance for 1.5-meter candles.

Both membrane types are considered viable candidates for future commercialization. The choice of which to use would depend on system requirements. Further refinements of the membrane composition are detailed in Section 3.5.3.2 "Variables".

3.5 Manufacturing Hot Gas Filters (Task 4)

The focus of Task 4 was to lay the foundation for the repeat manufacturing of PRD-66 Hot Gas Candle Filters. The effort was divided into six areas: raw materials plan, process instrumentation, process variable experiments, process capability demonstration, equipment analysis and improvement, and evaluation of long-term degradation.

3.5.1 Raw Materials Plan (Subtask 4.1)

Discussions were held with DLC's quality organization to align the PRD-66 product with the company's overall quality plan. Copies of DLC's documentation requirements for raw materials specifications are detailed in Appendix 1. DLC will develop specifications for all raw ingredients necessary to the production of PRD-66 Hot Gas Candle Filters and require Certificates of Conformance (COC) and/or Certificates of Analysis (COA) with each shipment which document conformance of the incoming raw ingredients with specifications. Raw material suppliers were contacted about our requirements and were very cooperative in meeting them.

3.5.2 Process Instrumentation (Subtask 4.2)

The goal of this effort was to identify any critical equipment used to perform in-process measurements and establish methods to assure the level of calibration necessary to maintain process control.

The most important instruments used in fabricating hot gas candle filters are the electronic balances. Several balances, with different accuracy ranges, are utilized at different stages of the process. When winding candle filters, the bobbins of feed yarn (see Figure 1) are positioned on balances, which have a maximum load of 2,000 grams (+/-0.1 gram). The amount of yarn that is used in the preform is determined by the net change in the indicated weight of the feed bobbin. This weight, when compared to the weight of the actual candle, is used to calculate the amount of alumina picked-up when the yarn is dipped into the alumina slurry (see Figure 1). Adequate pickup is necessary to insure the strength of the product.

A larger capacity balance, with a +/- 5-gram readout, is necessary for weighing the raw materials that comprise the alumina slurry. This balance is also used for the weighing of the candle filters; although, a more accurate balance with +/- 1-gram accuracy would be preferable for this purpose, if one was available.

All balances are calibrated annually in accordance with NIST HB44, ISO 10012-1 and ANSI/NCSL Z540 requirements. During the period of this contract, balances were calibrated several times and all were found to be within acceptable tolerances.

The only other critical instrument used in the PRD-66 process is a Brookfield viscometer. This devise measures the viscosity (resistance-to-flow) of liquids. Viscosity standards were purchased from Brookfield with known viscosities similar to that of the alumina slurry used in the PRD-66 process. No measurable deviations from calibration were observed throughout the period of this contract.

Although the viscosity of the slurry is critical in a broad sense, experiments performed during Task 4.3 (3.5.3 - Process Variables Experiments) indicate that variations as high as 50% from nominal have no impact on the process. For this reason, the viscometer does not require routine calibration checks. It is critical, however, to ensure that the settings on the instrument are always appropriate for the spindle being used. An incorrect setting, for example, could lead one to believe that the viscosity is 100 cps, when if fact it is 1,000 cps. For this reason, use of this equipment is restricted to the PRD-66 project staff, and is used only for alumina slurries having similar viscosities.

3.5.3 Process Variables Experiments (Subtask 4.3)

The focus of this subtask was to identify critical process parameters and vary them systematically to learn their effect on the product. In order to identify which variables the process was most sensitive to, ranges were chosen to encompass and exceed the existing specifications. If there was minimal sensitivity at the values tested, the existing specifications would be deemed acceptable. If sensitivity was detected, a more thorough evaluation would be conducted in order to define appropriate parameter limits.

The standard conditions for winding the fiberglass yarn included the use of the improved slurry composition, which had improved strength in the dry-state and better interlaminar adhesion, see Section 3.3.3.5.

3.5.3.1 Variables Impacting the Support Winding

The variables studied for their impact on the winding of the filter support were slurry viscosity, winding speed and atmospheric humidity. The ranges investigated were chosen based on current process capability to control them, see Table 5.

Variable	Lower Limit	Upper Limit
Winding Speed	- 22%	+ 22%
Alumina Slurry Viscosity	- 50%	+ 50%
Relative Humidity	20%	80%

Table 5 - Process variables investigated for winding filter support.

Candle filter support structures were wound, without flanges and without membranes. Winding was terminated when the weight of the fiber wound reached 1100 grams. Any unusual events that occurred were noted during the course of each run. After overnight air drying, tubes were each cut into eight, 8"long sections and the two end pieces retained as scrap. All portions were fired to 700° C ("low-fired"), held for one hour and allowed to cool to room temperature. All portions were weighed and measured, then high-fired to approximately 1400°C. Alumina pickup was calculated based on the low-fired weights and the known weight of the fiberglass yarn and high-fired materials were flow tested and inspected for delaminations. A summary of the results is depicted in Table 6.

Variable	Lower Limit	Upper Limit
Winding Speed	very slight increase in diameter	no detectable effect
Alumina Slurry Viscosity	statistically significant decrease in alumina pickup	no detectable effect
Relative Humidity	slight increase in diameter	slight decrease in diameter

Table 6 - Observed impact of process changes on support winding.

The lower and upper limits, which were tested, are all outside the normal process limits, yet, only the use of an "alumina slurry with half of the normal viscosity" resulted in a statistically significant change. No statistically significant variations in the product occurred within the nominal viscosity range. None of the other changes were statistically significant, suggesting that the normal process control limits are adequate for the reproducibility of PRD-66 candle filters.

Experiments were also conducted to determine the effect of "process interruptions during the winding operation" on process quality. The most critical type of interruption is an unattended yarn break during winding. To simulate this type of problem, the winding was intentionally stopped approximately half way into an otherwise routine winding run. The package was allowed to sit for approximately 15 minutes while still rotating, although a five-minute interruption would be more typical of current process norms. This experiment was conducted under a range of humidity conditions. Winding was restarted following standard procedures, and stopped at the target diameter. After the tube was dried overnight, it was cut, low-fired, weighed, and measured as described earlier; the specimens were then high-fired through a standard cycle to approximately 1400°C.

The only sample impacted by the winding interruption was the unit wound at the lowest humidity condition, which was outside of the normal operating range. When the completely fired material was cut, and the cross-section examined, a slight delamination could be discerned at approximately the mid-way point in wall, closely corresponding to the point at which the winding had been interrupted. Apparently, process interruptions of up to 15 minutes can be tolerated without adversely affecting the product, except in humidity conditions which are generally outside the normal range. Besides the resulting improvement in product yields, the insensitivity to interruptions will allow the use of "short bobbins" of fiberglass yarn. Standard bobbins of S-2 yarn typically have about 25% more yarn than is actually required for winding one candle filter. To stop the winding, and string-up a new bobbin of yarn usually takes approximately three minutes. The ability to do this without jeopardizing product quality will lead to less wasted yarn and lower costs.

An additional variable, which had to be added to this experiment, was the impact of fiberglass yarn "twisted" by a different company. Owens-Corning FiberGlas (supplier of S-2 glass yarn) decided that they would no longer directly supply yarn that is "twisted" in a wide assortment of configurations, including that required by this process. Two alternate sources of this twisted yarn were identified; only one, however, was reasonably priced. Three candle filters were fabricated from yarn twisted by the Varflex Corporation (Owens-Corning is still the sole manufacturer of the S-2 glass filaments). The run information was compared to the database that had been generated in earlier portions of this task. Evaluations were conducted on " alumina pickup", diameter growth rates, frequency of yarn breaks, and integrity of the overall structure. The twisted yarn from Varflex appeared to be either equivalent or superior to the original material in all tests. DLC's current inventory of yarn (purchased from Owens-Corning) is adequate to complete the fabrication of candles required for this program, but future purchases will be made from Varflex.

3.5.3.2 Variables Impacting the Membrane

As discussed in Section 3.4 "Development of High Efficiency Membrane" several variables were identified as being critical to the formation of a satisfactory membrane for the PRD-66 Hot Gas Candle Filter. Under Task 4.3, extensive tests were conducted to identify a membrane-filler formulation that would consistently yield low backpressure units with good filtration. The variables explored included:

1. 4 different solid-to-liquid suspension ratios
2. applying the particulate material to low-fired or high-fired candles
3. 2 different particle or grit sizes of alumina
4. 2 different levels of a fusible binder addition

Evaluations of items "1" and "2" were based on subjective comparisons of the ease of preparation and application of the filler material. The preferable solid-to-liquid ratio (2:1) was an aqueous suspension with a consistency similar to very, smooth peanut butter. More consistent results were achieved by applying this filler material to the surface of low-fired candles. Samples prepared in this manner with the medium-grit membrane, however, frequently developed extremely fine cracks in the membrane during the final firing, visible only with intense scrutiny using transmitted light. These cracks were so fine that no TIDD ash penetrated after 25 PIT cycles. Evaluations of items "3" and "4" were conducted in a more quantitative fashion, as shown in Table 7.

Both the "coarse" and "medium" grit alumina particulate are capable of producing membranes with a PIT rating of "10". The two grit sizes, however, had different ashcake release characteristics in the PIT evaluation, with the ash being more adherent to the coarse-grit membrane. In the Karhula field trial, this type of candle exhibited the formation of a traditional "conditioned ash cake layer". Tests of the original "baseline" candle in TIDD did not form such a layer; the repeated exposure of the imperfect membrane surface, after backpulsing, was thought to have contributed to the entrainment of ash in the filter wall.

PRD-66 Membrane Variations and Reproducibility

8" Unit ID	Full Length Candle ID#	Membrane Grit Size	wt% Binder	Weight of Added Membrane	Backpressure in-wg @5scfm
1	553	Medium	5%	9.03	5.6
2	553	Medium	5%	8.50	7.0
3	553	Medium	5%	8.55	5.6
4	553	Medium	5%	8.94	5.5
5	553	Coarse	5%	8.14	2.2
6	553	Coarse	5%	7.23	5.2
7	553	Coarse	5%	8.53	3.4
8	534	Coarse	5%	7.67	2.6
9	534	Coarse	5%	7.38	1.8
10	534	Medium	5%	7.85	4.0
11	555	Medium	5%	7.22	5.4
12	555	Medium	5%	7.85	5.6
13	534	Coarse	5%	5.73	3.0
14	555	Coarse	5%	5.83	1.9
15	555	Coarse	10%	-	2.1
16	555	Coarse	10%	-	3.2
17	555	Coarse	10%	-	3.1
18	555	Coarse	10%	-	1.9

Table 7 - Impact of grit-size and binder content on backpressure.

The data shown in Table 7 was also used to evaluate the impact of applying reproducible amounts of the particulate membrane. A correlation of the weight of the membrane filler and the backpressure was plotted in Figure 31. In general, the exact amount of the added membrane filler did not directly effect backpressure, at the quantities being used; in severe cases, however, excess material has been observed to crack during the high-fire step.

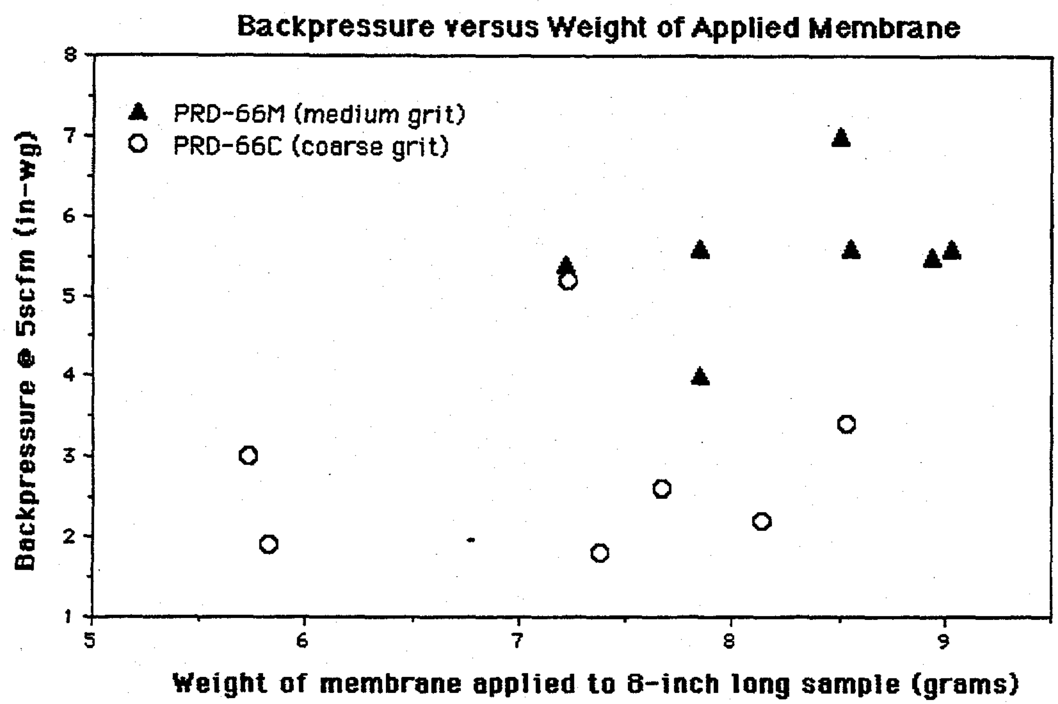


Figure 31 - Impact of membrane weight and type on backpressure.

Backpressure also appeared to be relatively unrelated to the binder content in the larger grit size composition, see Table 8. The higher level of fusible binder addition seemed to be preferable for the coarse-grit filler; the resultant material adhered better to the surface of the candle, as observed in the repeated brushing involved in the PIT evaluation. This level of fusible binder was not necessary with the medium-grit filler-material, probably because the higher surface area of the finer particles sintered more readily. Fortunately, a higher level of fusible binder did not seem to significantly impact backpressure.

Backpressure of Coarse Samples Only vs. Binder Content

Binder Content	No. of samples	Backpressure @ 5scfm (in-wg)	
		Average	Std. Dev.
5%	n=7	2.9	1.2
10%	n=4	2.6	0.7

Table 8 - Impact of binder content on backpressure of PRD-66C.

Based on the experiment described above the membrane formulations chosen for further evaluations were: "medium grit with 5% binder" and "coarse grit with 10% binder"

In addition to the membrane experiments described above, an evaluation was conducted to determine the effectiveness of filling "pin holes" in the unfired membrane. Eighteen low-fired, 8" filter segments were coated with either the "coarse" or "medium" grit membranes. After the membrane dried, each unit was checked with transmitted light for "pin holes". Additional membrane filler was then applied to those areas and marked with a high-temperature marking pencil, to make later identification possible. After high-firing, all specimens were examined again. All patched areas appeared completely sealed and no additional "pin holes" developed.

Earlier in this section, mention was made of the formation of extremely fine membrane cracks after high-firing the PRD-66M candle filters. The reason for their occurrence was not determined. In general, these flaws were only visible using transmitted light, and then, only if you knew exactly what to look for. If significant amounts of excess filler-material remained on the surface, the cracks were more severe and visible to the eye under normal lighting conditions. Preparation of multiple samples, from virtually identical tubes, has yielded significant information. Only the membrane made with the medium-grit, or finer, alumina particulate exhibits the problem, under normal conditions. The problem is minimized by using lower levels of the fusible binder addition, but not eliminated. When several 8" samples, from the same candle, were prepared in the same way with the medium-grit filler, and fired side-by-side, only one sample in the batch had cracks. As noted earlier, a specimen with a crack was PIT-tested with TIDD ash; the ash was trapped in the membrane and did not penetrate into the support wall. It is unknown whether or not this condition jeopardizes the successful operation of the candle. Aggressive investigation was discontinued due to the time constraint of providing filters to Westinghouse for testing. The best-known formulations and application methods would be used. General and specific information, with regard to handling of the candles, placement within the furnace, etc., would be monitored and correlations would be sought with any incidence of cracking.

3.5.4 Process Capability Demonstration (Subtask 4.4)

The focus of this subtask was to produce three batches of candle filters, according to the specifications required by the Westinghouse Advanced Particulate Filtration (APF) System, as shown in Figure 32. Each batch consisted of ten candles, manufactured under identical conditions. Before beginning each batch, critical components of the process equipment was inspected. Where feasible, new parts were put into service and process changes were incorporated to improve the product quality and process yields. An evaluation was conducted on all measurable features of the filters to assess controllability and product uniformity. Significant aspects of the process, which effected final yields, were identified. Eight of the first-quality candles were used for high-temperature, high-pressure (HTHP) testing at Westinghouse Science and Technology Center (see Section 3.6.1). Twelve of the first-quality candles were field tested at the Foster Wheeler 10 MWt PCFBC facility in Karhula, Finland (see Section 3.6.2).

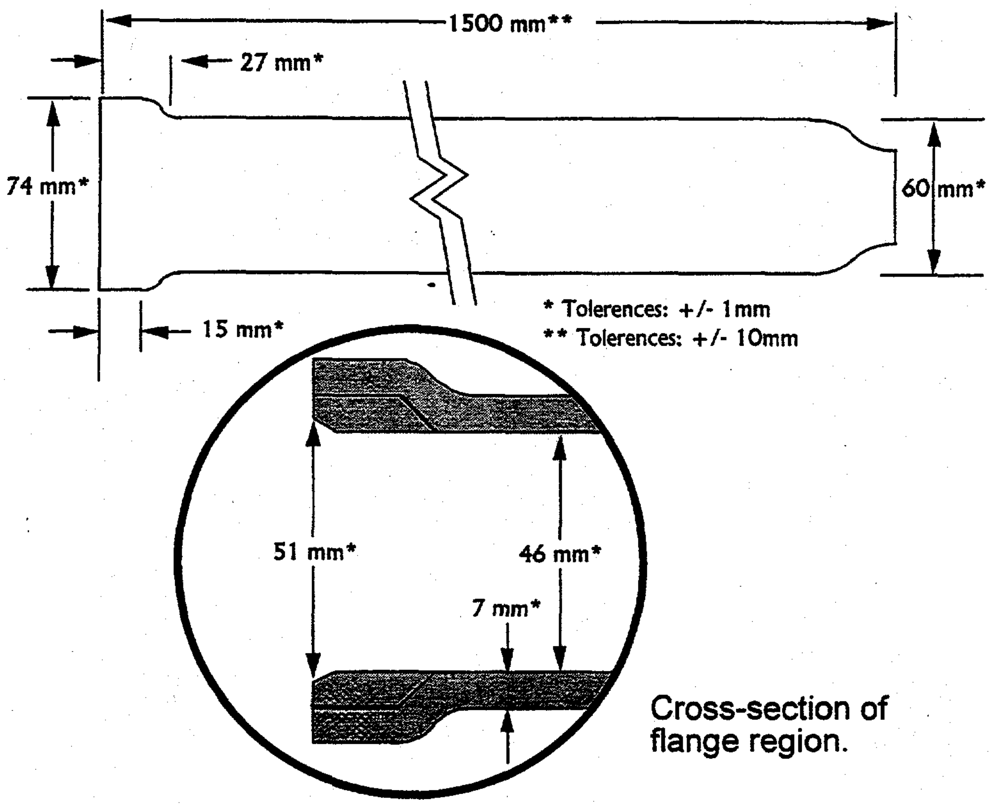


Figure 32 - PRD-66 Candle Filter dimensions

During this capability study, twenty-one good candles were produced, out of a possible 30, or 70% yield. Table 9 gives a detailed evaluation of all elements fabricated. Table 10 summarizes the data into the three, ten-unit runs, which were conducted.

Seven elements were rejected as a result of physical damage incurred during some stage of the processing. One element was rejected because the flange was out-of-spec (too long). One element was rejected for a poor quality membrane. Although, the "inside edge diameters" of nine flanges were out-of-spec, Westinghouse felt confident that their holder assembly could accommodate them, so they were not rejected.

PRD-66 Hot Gas Candle Filters

Process Capability Demonstration - 30 Candles

Candle	Weight (g)	Flange (mm) OD	ID	Length (5)	Length (4)	Bend (mm)	Mem. Type	Backpressure (iwg@50scfm)	$\%Al_2O_3$ Pickup	Visual Exam	Pass/ Fail
564	2610	73.3	50.7	27.0	15.5	2.0	M	7.4	55.9		
565	2575	73.1					M	6.8	56.5	bumped in mid-candle after winding	Fail
566	2600	73.4					C	2.8	55.7	bumped in mid-candle after winding	Fail
567	2505	73.6	51.5	27.1	15.3	1.0	M	6.0	55.0		P
568	2565	73.1	50.2	27.9	15.9	2.0	M	7.2	56.3		P
569	2520	73.2	50.3	26.8	15.7	0.5	M	7.9	56.0		P
570	2590	73.8	50.5	27.4	15.8	1.0	M	7.5	57.3		P
571	2540	73.2	50.3	27.3	15.8	1.0	C	3.6	56.0		P
572	2515	74.0	51.4	26.2	15.0	0.5	C	3.7	55.4		P
573	2485	74.0	50.6	26.6	14.7	1.5	C	2.1	54.9		P
574	2555	74.0	50.2	32.3	27.6	1.5	C	2.4	54.9	Flange cut too long	Fail
575	2555	73.4	**53.5**	26.1	14.1	2.0	C	5.1	56.6		P
576	2445	73.8	**52.5**	27.3	15.3	1.5	C	4.4	55.4	chip in memb. (7.5"from tip)	Fail
577	2495	73.2	**52.9**	27.4	15.4	0.0	C	4.3	56.0	finger prints	P
578	2365	74.2	52.0	26.7	14.7	0.5	C	3.2	53.5	chip in memb.(6.5"from flange)	Fail
579	2520	73.4	51.3	26.5	14.5	2.0	C	4.3	55.6		P
580	2570	73.0	50.5	27.5	15.5	0.0	C	4.2	56.4	poor bulk support wind pattern	P
581	2390	74.0	**53.8**	26.6	14.6	2.0	C	3.4	55.0	finger prints	P
582	2515	73.3	**53.1**	27.7	15.7	2.0	C	4.1	55.9	few scars, finger prints	P
583	2515	73.6	**53.0**	27.0	15.0	1.5	C	3.0	55.6	tip crack, fing. pr.	Fail
584	2550	74.0	**53.3**	27.6	15.6	2.0	C	4.1	56.6		P
585	2490	74.2	51.8	28.0	16.0	0.5	C	3.5	55.7	1/2" wide chip @ tip, finger prints	Fail
586	2555	73.1	51.8	27.7	15.7	1.0	C	5.2	56.6	large patches of excess memb. finger prints	P
587	2615	73.9	**53.0**	26.5	14.5	1.5	C	4.2	56.9	finger prints	P
588	2560	73.5	50.1	27.9	15.9	1.0	C	4.3	56.3		P
589	2595	73.9	51.1	27.6	15.6	2.0	C	3.3	56.6	chip in memb.(5.5"from flange)	Fail
590	2545	74.0	51.5	27.1	15.1	2.0	C	3.0	55.0	SEVERE support winding irregularities	Fail
591	2455	73.8	**53.0**	27.1	15.1	2.0	C	3.9	54.9	poor bulk support wind pattern, finger prints	P
594	2585	73.5	45.9*	26.8	14.8	2.5	C	3.0	56.6		P
595	2565	74.0	45.9*	27.9	15.9	2.5	C	4.0	56.7		P
Average	2522	73.7	52.1	27.5	15.8	1.5		3.7	56.6		
StDev.	60	0.4	1.2	1.1	2.4	0.7		0.8	0.8		
* at Westinghouse's request, the open end was not bevelled								TYPE-C only			

<u>Dimensions not detailed above</u>

	Ave.	StDev.		Ave.	StDev.
Overall Length	1502	2	Tube OD	59.7	0.3
Length of open filter	1417	7	Tube ID	45.8	0.2

Table 9 - Process Capability Demonstration

PRD-66 Hot Gas Candle Filters *Process Capability Demonstration - Summary*												
	Weight	Length (mm)		Tube (mm)		Flange (mm)				Bend	Backpressure	%Al2O3
	(g)	Overall	Open*	OD	ID	OD	ID	L (5)	L (4)	(mm)	(iwg@50scfm)	Pickup
TOTAL (30 candles)											**TYPE-C only**	
Average	2522	1502	1417	59.7	45.8	73.7	52.1	27.5	15.8	1.5	3.7	56.6
StDev.	60	2	7	0.3	0.2	0.4	1.2	1.1	2.4	0.7	0.8	0.8
Target		1500		60.0	46.0	74.0	51.0	27.0	15.0	0.0		
Spec.		+/- 10		+/- 1.0	+/- 1.0	+/- 1.0	+/- 1.0	+/- 1.0	+/- 1.0	< 3.0		
RUN 1 (10 candles)												
Average	2551	1502	1405	59.6	46.0	73.5	50.7	27.0	15.5	1.2	3.1	55.9
StDev.	43	3	4	0.3	0.0	0.4	0.5	0.5	0.4	0.6	0.8	0.7
Max	2610	1505	1411	60.1	46.0	74.0	51.5	27.9	15.9	2.0	3.7	57.3
Min	2485	1499	1397	59.2	46.0	73.1	50.2	26.2	14.7	0.5	2.1	54.9
RUN 2 (10 candles)												
Average	2493	1501	1417	59.7	45.7	73.6	52.3	27.5	16.2	1.3	3.8	55.5
StDev.	70	3	6	0.3	0.2	0.4	1.2	1.8	4.0	0.8	0.8	0.9
Max	2570	1508	1426	60.0	46.0	74.2	53.8	32.3	27.6	2.0	5.1	56.6
Min	2365	1499	1405	59.3	45.5	73.0	50.2	26.1	14.1	0.0	2.4	53.5
RUN 3 (10 candles)												
Average	2552	1502	1418	59.8	45.9	73.8	52.0	27.4	15.4	1.7	3.9	56.2
StDev.	65	3	9	0.3	0.2	0.4	1.2	1.8	4.0	0.7	1.5	1.0
Max	2615	1505	1426	60.5	46.2	74.2	53.3	28.0	16.0	2.5	5.2	56.9
Min	2455	1500	1410	59.2	45.7	73.1	50.1	26.5	14.5	0.5	3.0	54.9
* Open Length is defined as that portion of the filter which provides active filtration.												

Table 10 - Process Capability Summary

The physical damage to the filter elements appeared to have two distinct sources. The first occasion for significant damage to occur was during the transfer of the developing candle from the bulk support winder to the membrane hoop winder (while it was still soft, damp and easily dented). Any obstructions on the equipment or between the winders increased the risk of damage. When two candles were dented, it was immediate and obvious.

Of more serious concern were several filter elements which each had a single chip (approximately 1/8"-1/4" long and 1/16"- 1/8" wide) in the membrane, discovered during final inspection. After final firing, the damaged areas "puckered" and the membrane easily flaked off when rubbed. Based on historical observations, the damage probably occurred while the candle was in the unfired or low-fired state. Possible causes include excessively tight gripping during a difficult mandrel removal or contact of the membrane with an inadequately padded area of the storage cart. In either case, damage would not have been apparent prior to the final high temperature firing.

Examining the standard deviation of the data, most of the features were within 3% of the average and within the acceptable range established by the Westinghouse protocols. The inside diameter of the flange and the length of the flange, however, were much more difficult to keep in-spec:

Inside diameter of the flange. The inside diameter of the flange was out-of-spec on 30% of the candles fabricated. The open end of each candle was finished-off by grinding a bevel on the inside edge, such that, the finished edge of the ID was 51mm +/- 1mm. The hand grinding technique, which was employed to create this bevel, was not adequately reproducible; machining was not a viable economic option. The original reason for the grinding was that the inside edge of the flange was occasional too friable, resulting in an irregular surface. Throughout the course of this program, however, with the adoption of the Chlorhydrol®-containing alumina slurry (see Section 3.3.3.5), the inside edge became much denser than with the original composition. The added step of grinding this area no longer appeared to be necessary; both DLC and Westinghouse agreed to eliminate this feature in future production runs.

Length of the flange. The data does not wholly reflect the difficulty encountered in meeting the required tolerances. Because the outside contour of the PRD-66 flange has no distinct edges, defining the precise location for cutting is not simple. It was also difficult to establish whether the flange was "in-spec" or "out-of-spec". All measurements were taken based on how the flange aligned with a plastic tool having a similar contour. Several candles which seemed to be slightly too long were hand-ground into spec. No problems were encountered, by Westinghouse or Foster Wheeler, mounting any of these candles for field trials. Eventually, better measurement techniques and better-defined specifications will be needed.

The data collected during the process capability run (see Table 9) indicated that the alumina matrix pickup varied from 53.5% to 57%. A possible link between diametrical compressive strength and alumina matrix pickup was investigated. 1" wide o-rings were cut from the candle with the 53.5% pickup and o-ring diametrical compressive tests were performed. The strength values were within the range of all measurements previously taken. During the course of Task 5 additional tests will be conducted on the candle with the lowest matrix, pickup to see if any impact on strength can be observed. Furthermore, any finished candle having a damaged portion, making it unsuitable for field use, will be cut up into 1" o-rings and tested in order to define the nominal strength range of PRD-66 filter elements. This information will be essential in determining if field-exposed elements are any stronger or weaker than the as-manufactured material.

An important objective of this task was to gain a better understanding of the process economics of manufacturing PRD-66 Hot Gas Filters. The most dramatic finding was that the utilization of the winding equipment was well below expectations due to the high level of equipment maintenance required. While some problems were anticipated as a result of wear, the biggest difficulties encountered were inherent in the basic winder design. Many of the features that make this devise very versatile compromise its reliability under routine operating conditions. A simpler winder, designed specifically for PRD-66 candle filters, would require significantly less time, labor, and materials to maintain.

3.5.5 Equipment Analysis and Improvement (Subtask 4.5)

During "Task 4.4 - Process Capability Demonstration", described in the previous section, an analysis of the rate of wear of critical components was conducted. Attention was initially focused on surfaces that were in contact with abrasive slurry-coated yarn and the moving components of the winder itself. As part of "Task 4.5 - Equipment Analysis and Improvement", the feasibility and cost of making improvements was be evaluated and changes made where appropriate.

The first issue addressed was an increase in the frequency with which the slurry-coated yarn would break during the winding process. Breaks usually occurred when the traverse changed direction and the yarn needed to slide from one side of the guide to the other. The most obvious reason for this problem was that the alumina guide would develop grooves on either side, because of abrasion from the particulate alumina in the slurry. The deeper the grooves became the more likely the yarn was to break when the traverse changed direction. Two potentially more abrasion-resistant materials were evaluated: metal-matrix composite and polycrystalline diamond. The metal-matrix composite material turned out to be even more susceptible to abrasion. The polycrystalline diamond guide was never actually tried; it was prohibitively expensive to achieve a sufficiently rounded surface that would not cut the yarn. Since neither material offered any advantages over the high purity alumina, the alumina guide was changed out more frequently to keep yarn breaks to a minimum.

During this investigation, however, another reason for yarn breaks was observed. The yarn would most frequently break during the first 20 minutes of winding, when the guide changed direction at the tip-end of the mandrel. The mandrel on which the PRD-66 filter element was wound had a hemispherical shape at the tip end, going from 45 mm down to 6 mm in diameter in approximately 1" of length, as shown in Figure 33.

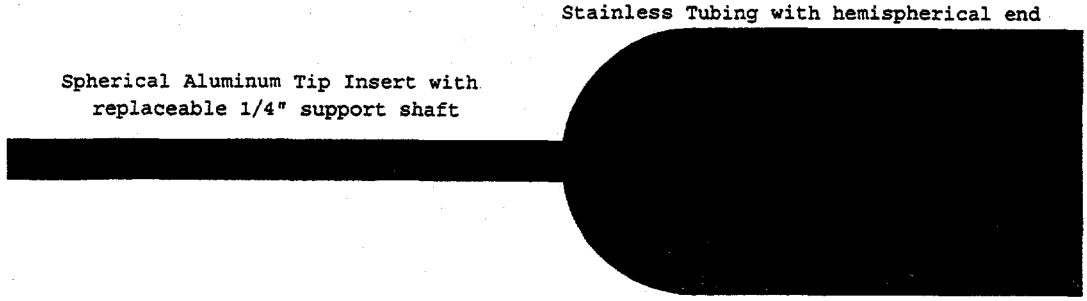


Figure 33 - Tip of original steel mandrel

When the yarn wound down to the narrow support shaft, the speed at which it was being removed from the yarn bobbin (see Figure 1) would slow dramatically; as the guide carried it back up to the 45 mm tubing, the yarn would be "tugged" suddenly, often breaking the yarn. As a layer of yarn accumulated on the shaft, thus increasing its diameter, this became less of a problem. During the first twenty minutes of winding, however, constant supervision and slower winding speeds were required. To address this problem a design change was made, to use a conical-shaped tip instead of a hemispherical one; this change was instituted along with other changes intended to create more easily removable mandrels. After the changes were implemented, the frequency of breaks dropped dramatically.

Another problem this task sought to address was the difficulty with which the wound filter was removed from the mandrel. In several cases, damage to the candle could result, which was not always easily detected until much later in processing (see Section 3.5.4). After unsuccessfully trying to find an outside vendor who could supply a mandrel that would meet DLC's needs, an in-house program was initiated. Several combinations of steel tubing, plastic tubing and rubber were evaluated. The mandrel chosen for future manufacturing use was made from readily available sizes of tubing, with a rubber conical tip, and could easily be removed from the filter after spending about 30 minutes in a freezer. Because of the use of standard tubing sizes, the filters were approximately 1 mm smaller in the inside diameter. Sample candles made on the prototype mandrel were send to Westinghouse to determine if they anticipated any problems with the design. Westinghouse did have to modify the design of their "fail-safe devise" to accommodate the inside diameter change.

Another issue addressed in this task was the inadequacy of the procedure and tools used to cut the scrap ends from the dried candle filters. The standard procedure required the use of a razor knife, while rotating the candle (while still on the mandrel). After the finished candles were checked for perpendicularity, however, many flanges required hand grinding in order to meet the specification. A new concept was evaluated involving the use of a rotating, circular blade, while rotating the candle/mandrel. A silicon carbide blade and a diamond wafering blade were both tested. The diamond blade was the most effective and was used with later candles made in the "Process Capability Demonstration". The need for hand finishing of the final filters was reduced.

A major equipment issue involved the repair of DLC's 15-ft long X 4-ft wide high-fire furnace. The deterioration of the roof insulation over the previous six years led to detectable temperature non-uniformities along the length. To compound this problem the furnace had to be relocated to a more suitable manufacturing area, this move caused additional damage to the roof insulation. Since there are no other furnaces readily accessible to DLC for firing 1.5-meter candle filters and the PRD-66 Hot Gas Filter Program was only user of this equipment, repairs were conducted under this program.

While some of these modifications were implemented during the "Process Capability Demonstration", all had been put in place by the start "Task 5 - Manufacturing 50 Candles".

3.6 Field Testing of "Improved" PRD-66 Filter Elements

3.6.1 High Temperature High Pressure (HTHP) Testing at W-STC

Eight filter elements (four of each membrane type), manufactured during the first 10-candle run of the "process capability demonstration", were submitted to Westinghouse Science and Technology Center. Upon arrival, all candles were measured for room temperature gas flow resistance, as shown in Figure 34 and Figure 35. Both sets of filter elements met the W-STC tolerance of <1 in-wg/fpm for as-manufactured candles.

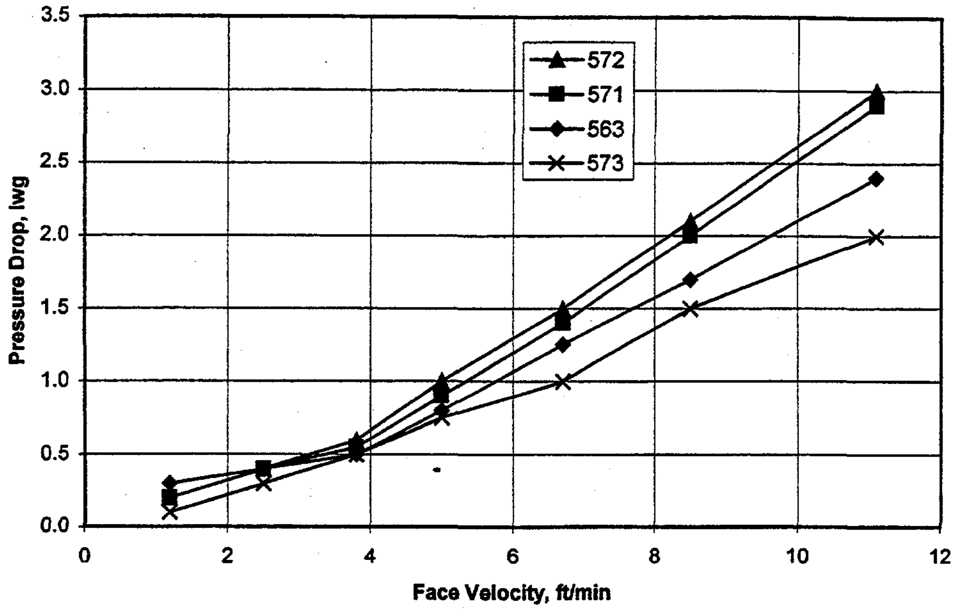


Figure 34 - PRD-66C - Room temperature gas flow resistance[12]

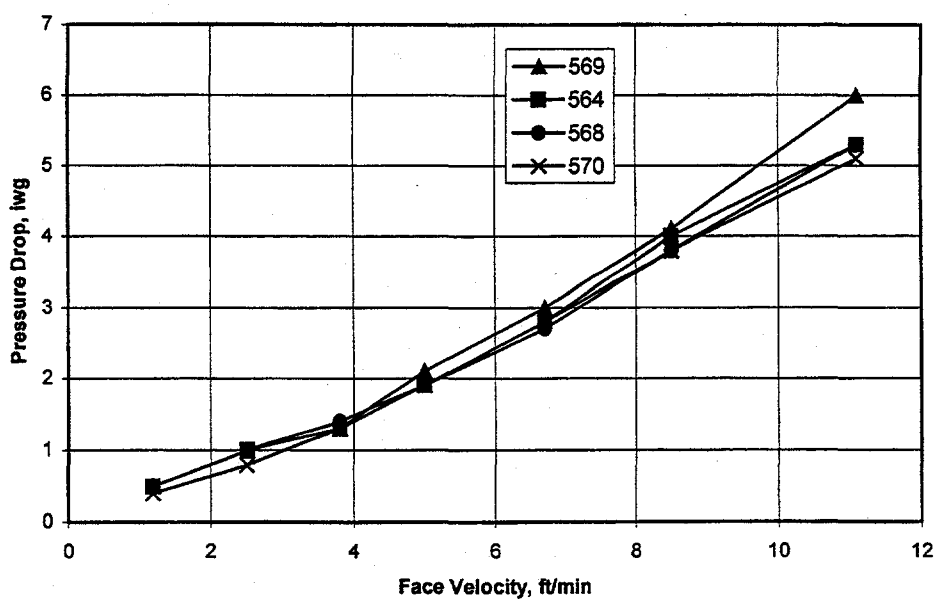


Figure 35 - PRD-66M - Room temperature gas flow resistance[12]

During April 1997, one candle of each membrane type was subjected to a high temperature, high pressure (HTHP), simulated pressurized fluidized-bed combustion (PFBC) environment. Testing included exposure of the PRD-66 candles with alternate monolithic and advanced fiber reinforced candle filter elements in order to support pressurized circulating fluidized-bed combustion (PCFBC) test initiatives in Karhula, Finland. The filter array was subjected to 120 hours of steady state operating conditions at 843°C (1550°F), and subsequently 2,200 accelerated pulse cycles, and 12 mild thermal transient events.

Post-test inspection of the filter array indicated that both exposed PRD-66 filter elements remained intact. The following comments were noted:

- thin dust cake layer on both considered to be a "normal conditioned layer"
- no debonding or "divoting" of the outer membrane occurred
- no cracks were identified along the flange or body
- apparent heavier retention of fines in diamond pattern of PRD-66C versus PRD-66M

Post-test gas flow resistance measurements of the qualification-tested candles are provided in Figure 36. The coarse membrane (PRD-66C) element initially had a lower pressure drop in comparison to the medium membrane (PRD-66M) element; after qualification testing, this relationship was retained. These elements were subsequently subjected to mechanical strength characterization, x-ray diffraction, and microstructural analysis.

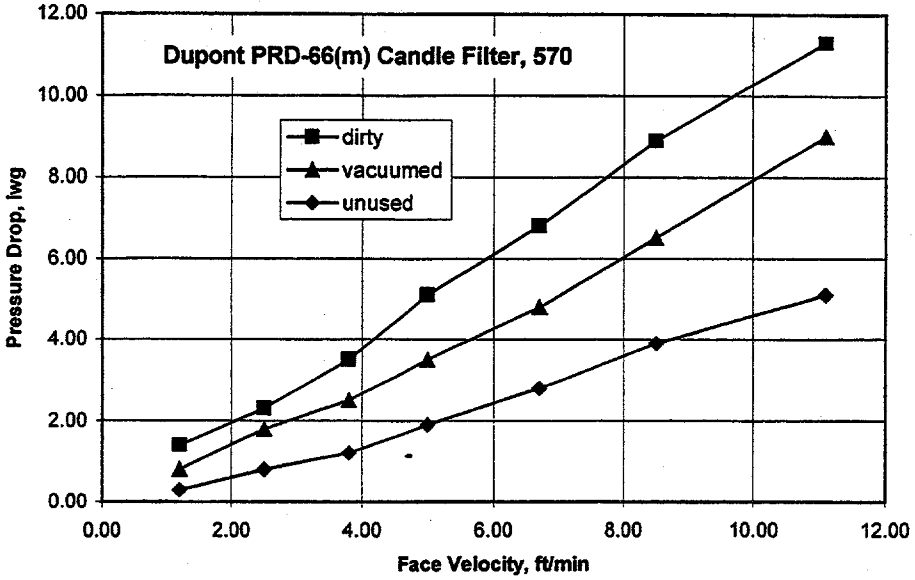


Figure 36 - Gas flow resistance of as-manufactured and HTHP-exposed PRD-66M elements[12]

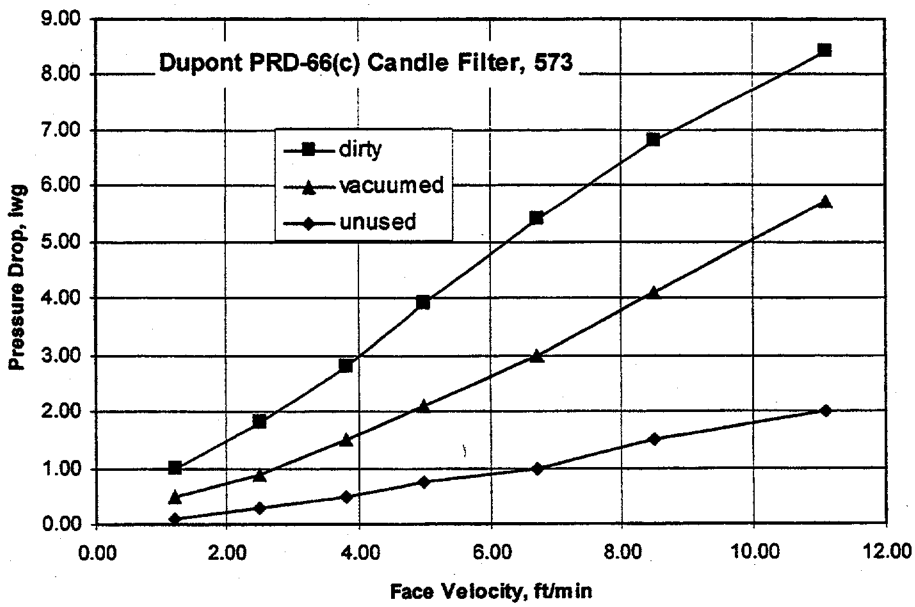


Figure 37 - Gas flow resistance of as-manufactured and HTHP-exposed PRD-66C elements12

W-STC characterized the mechanical properties the two tested elements, along with one as-manufactured candle of each membrane type (see Appendix 2). Table 11 summarizes the compressive and tensile c-ring tests that were conducted; the data suggests that the strength of the coarse and medium membrane "exposed" elements tended to be greater than the strength of comparable as-manufactured elements. M. A. Alvin of W-STC feels that this conclusion is supported by similar results obtained during other simulated and field exposures.[4] It had been postulated that an increase in strength could result from the bulk versus barrier filtration characteristics of the material, whereby submicron and micron fines penetrate through the membrane of the PRD-66 filter element and become trapped within the filter wall. Under these conditions, trapped ash could cause significant problems during field operation, particularly if thermal expansion occurs within the filter wall during plant startup cycles,[5] or hydration of the ash resulted during thermal shutdown cycles (Section 3.3.3). In relation to alternate filter elements,[6] the PRD-66 candle filters were considered to be "moderately low" load-bearing (Table 12). Additional material properties as burst strength, modulus, and Poisson's ratio, which were developed at Westinghouse, are provided in Table 13.

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 11 - W-STC Room temperature and process strength of PRD-66 elements [12]

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)

Table 12 - W-STC Ultimate load applied during strength characterization [12]

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 13 - W-STC Material properties of PRD-66 elements [12]

Additional strength testing was conducted by DuPont Lanxide Composites on segments of the same "exposed" filter elements tested by W-STC and on two different as-manufactured candles. These results, shown in Table 14, DO NOT support the Westinghouse conclusions. The "exposed" PRD-66C had a higher strength, however the "exposed" PRD-66M had a lower strength. The data suggests that the candle-to-candle strength variability of the material outweighs any effect of exposure. It was interesting to note, however, that the W-STC c-ring strength values and the DLC o-ring strength values for candles #570 and #573 were very similar.

Candle ID#	Status	O-Ring Comp.Str	Load-to-Failure
PRD-66C			
566C	As-Manufactured	1087 ± 80 (11)	41.5 ± 3.1 (11)
573C	Qualification Tested	1252 ± 44 (5)	45.6 ± 3.6 (5)
PRD-66M			
567M	As-Manufactured	1229 ± 117 (11)	44.7 ± 3.9 (11)
570M	Qualification Tested	1095 ± 184 (5)	37.2 ± 6.7 (5)

Table 14 - DLC Diametrical compression testing of HTHP-exposed & unexposed candles

3.6.2 PCFBC Exposure at Karhula

A 581-hour exposure of PRD-66C filter elements was conducted in Foster Wheeler's pressurized circulating fluidized-bed combustion (PCFBC) test facility in Karhula, Finland. Analysis of an exposed filter was conducted under Task 3.2.

Seven candles began the test in early September. Table 15 (provided by Westinghouse) identifies the operating conditions experienced by the PRD-66C Hot Gas Candle Filters in Westinghouse's Advanced Particulate Filter cluster during the TS2-1997 test campaign

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

* The number in parentheses indicates the number of elements exposed for the respective operating hours.

Table 15 - Karhula PCFBC test conditions

After 239 hours, the system was turned off and all elements were examined. Significant quantities of ash were found on the "clean side" of the system. All candles were removed and cleaned by vacuuming and washing. One PRD-66C candle broke at the flange when it was removed;

some force had been necessary to dislodge the flange from the holder assembly. When the run was restarted, a new PRD-66C candle was put in its place. The test concluded 342 hours later.

At the conclusion of the run, the six PRD-66C elements that were exposed for the entire 581 hours, and the one candle that was exposed for a total of 342 hours, all looked good. All but one of the elements had been cleaned by brushing and vacuuming prior to inspection, see photograph in Figure 38. There was no sign of any material deterioration in the possible forms of "divots", abrasion, poor membrane adhesion, or cracking. A significant amount of ash, however, was observed in the wall of the inside diameter, though it was much less for the element that was only exposed for 342 hours.

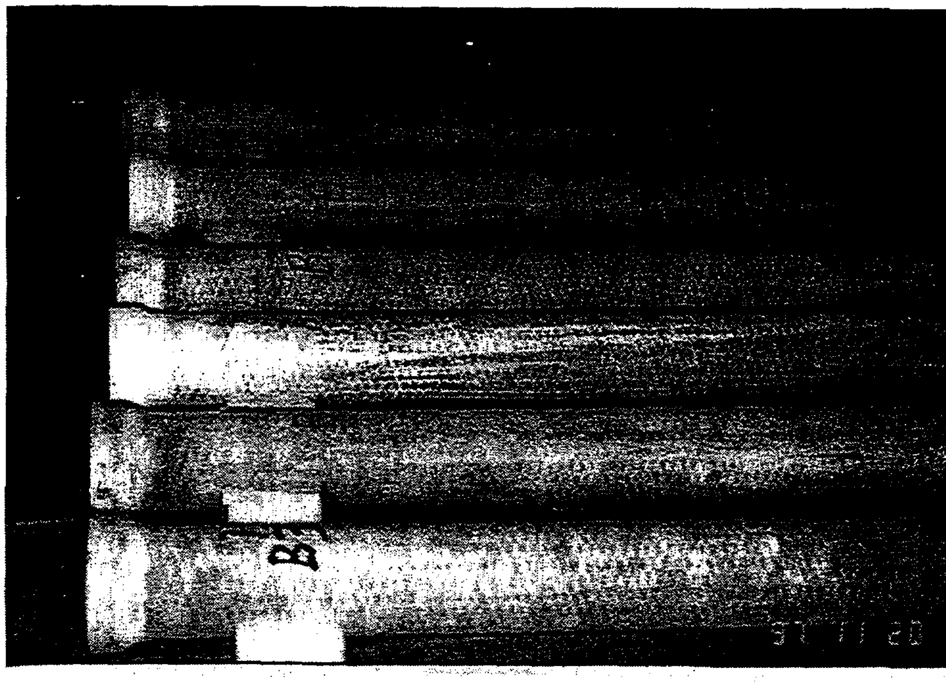


Figure 38 - Karhula-exposed PRD-66C filters

A single candle was examined before any ash had been cleaned from the material. A conditioned ash cake layer, approximately 2mm thick, had formed along the outside diameter, see photograph in Figure 39. The ash was soft and easily removable by handling or by brushing. The inside diameter was also caked with ash, approximately 2mm thick, with at least six inches of loose ash present in the tip of the candle.

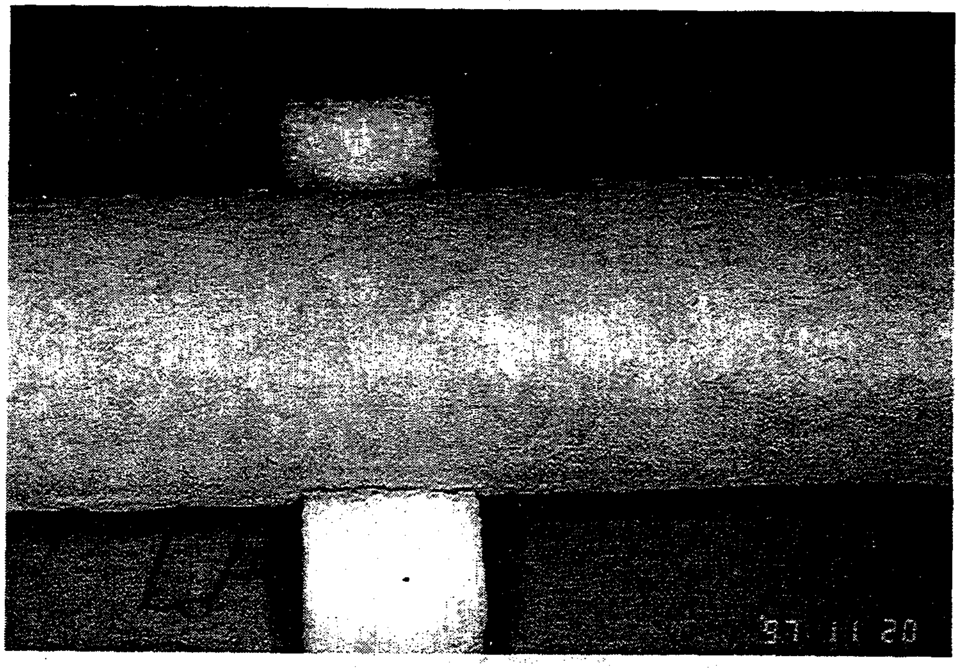


Figure 39 - Outside diameter of Karhula-exposed element before ash removal

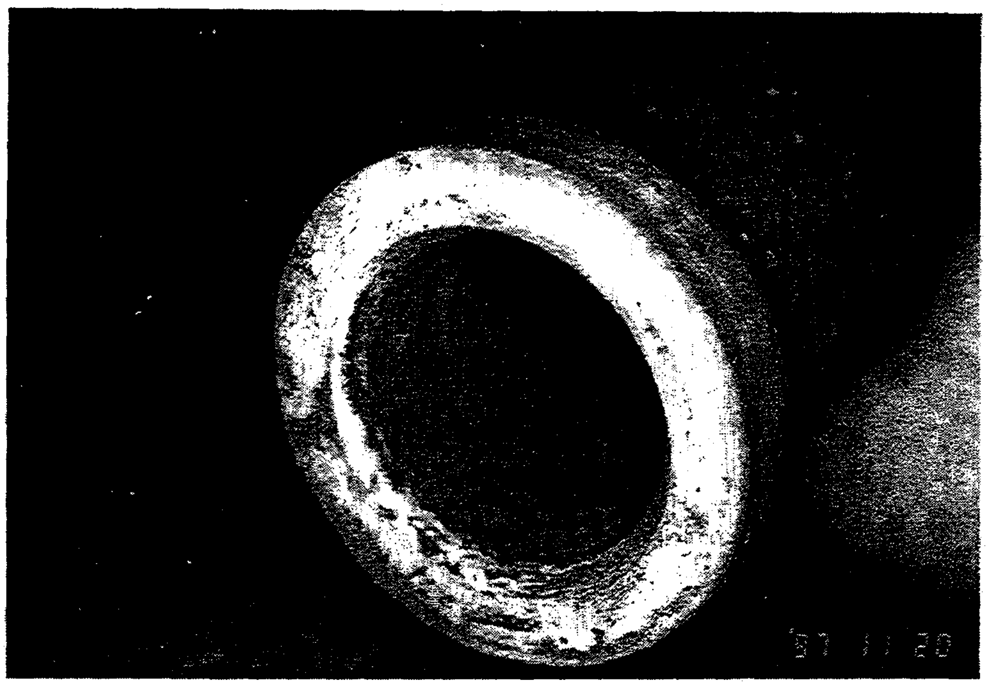


Figure 40 - Inside diameter of Karhula-exposed element before ash removal

All candles were vacuum-cleaned, inside and out, prior to inspection, after which, differential pressure measurements were conducted by Foster Wheeler personnel, see Figure 41. In summary, all elements showed significantly higher backpressure, with the exception of the single candle that was installed after the "239-hour shutdown", which had a slight increase in backpressure.

FW has attributed the plugging of the other filters to the presence of significant quantities of ash on the "clean side", rather than the length of exposure.

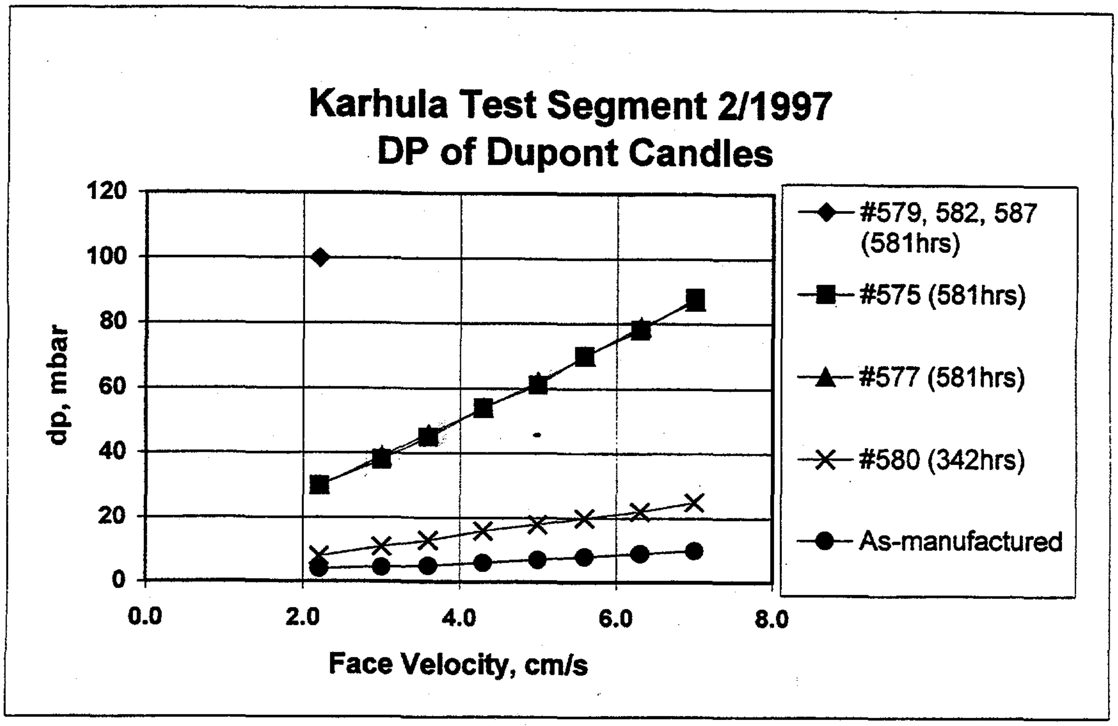


Figure 41 - Differential pressure of Karhula filters measured by Foster Wheeler

One of the candles with the full exposure time (#577), and the candle, which broke during removal after 239 hours (#591), were shipped to DLC for analysis. Unfortunately, both broke into at least three pieces during transport.

3.6.2.1 Visual Inspection for Ash Penetration in Karhula-Exposed Element

Samples of candle #577 (with 581 exposure hours) were prepared by "fast-fracture", to expose a cross-section of the wall. The contrast between the dark (orange-brown) ash and the white PRD-66 support material made it easy to determine where obvious ash penetration had occurred. Figure 42 is a photograph of a particular sample in which the support yarn was exposed at two distinct levels: just below the membrane and approximately 4mm below the membrane (mid-way through the wall). The presence of ash mid-way through the wall was no surprise, since a process upset had occurred during the Karhula exposure, which introduced large quantities of ash into the ID of the filter elements. The most significant observation was that there was no ash within 1-2mm of the membrane. Figure 43 is an enlargement of that area shown in Figure 42. The ash is clearly seen trapped in the membrane, while the yarns of the support structure immediately below are clean and white. This indicates that the new PRD-66C membrane (with nominal 25-micron pores) is an effective surface filter for PCFBC applications. It is significant that, no "divots" occurred despite the large volumes of ash that penetrated from the "clean side".

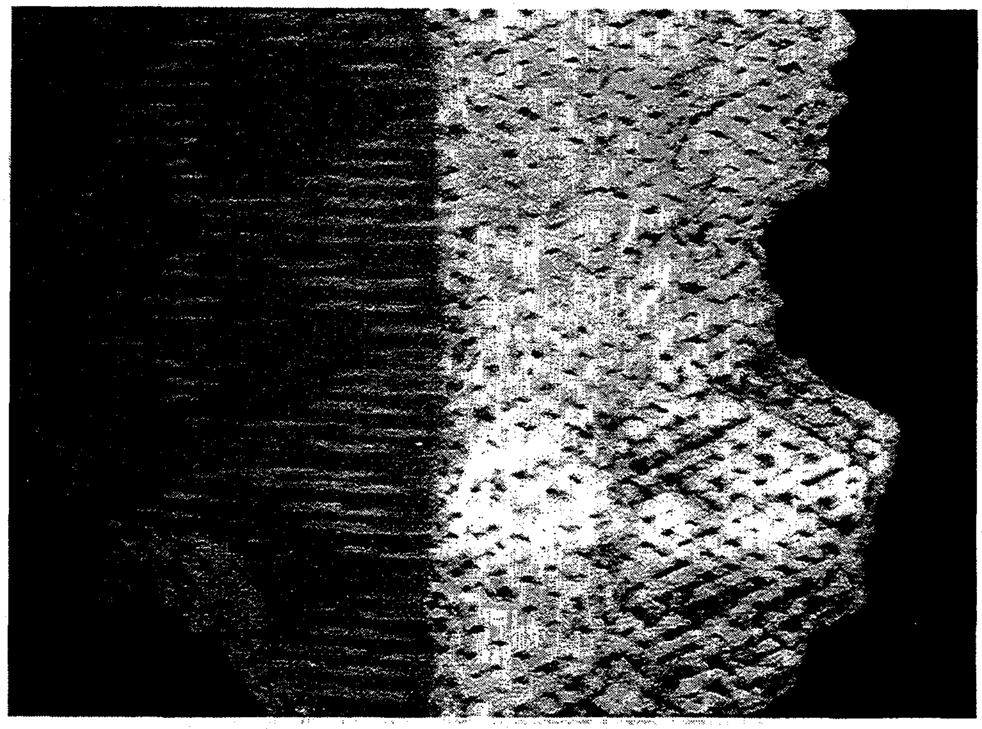


Figure 42 - Wall interior of Karhula-exposed candle #577

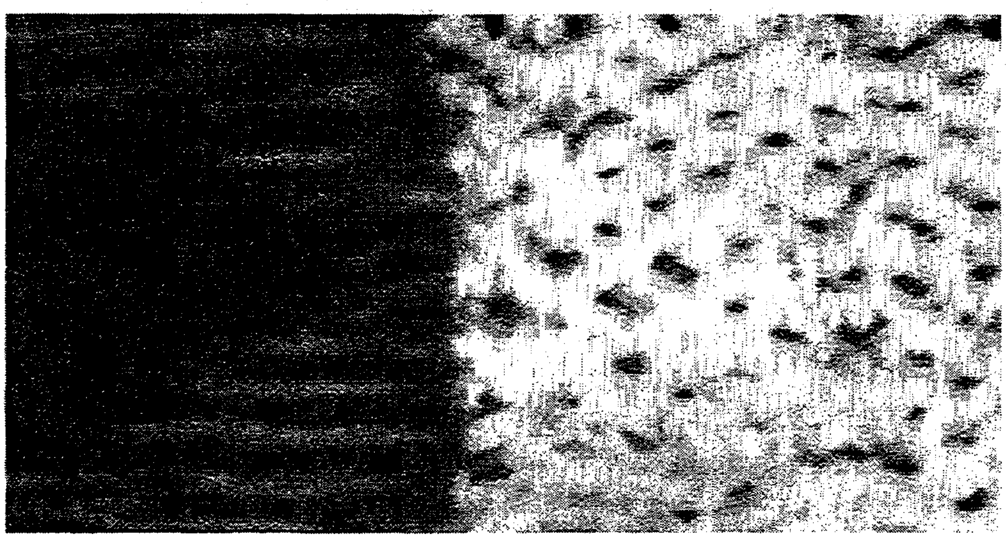


Figure 43 - Close-up of #577 - OD surface and 1-2mm below

FW also shipped approximately one liter of PCFBC ash that could be used to conduct a particle infiltration test (PIT) on a "sister" candle filter. The test was performed on a two-inch segment of unused candle #576. The results confirmed the observations made on the Karhula-exposed candle; no penetration of ash through the membrane was detected.

3.6.2.2 Microstructural Analysis of Karhula-Exposed and Unexposed Elements

A series of scanning electron microscopy (SEM) photographs were taken of different features of the exposed candle #577 and the unexposed candle #576.

In the following photos, comparisons were made of the exposed outside diameter surfaces. In Figure 44, the structure of the unexposed membrane has coarse alumina grains speckled with fine grains of the fusible binder, when viewed at 300X. By comparison, the exposed candle in Figure 45 and Figure 46 show similar irregularities which have been "smoothed-over" by the presence of ash.

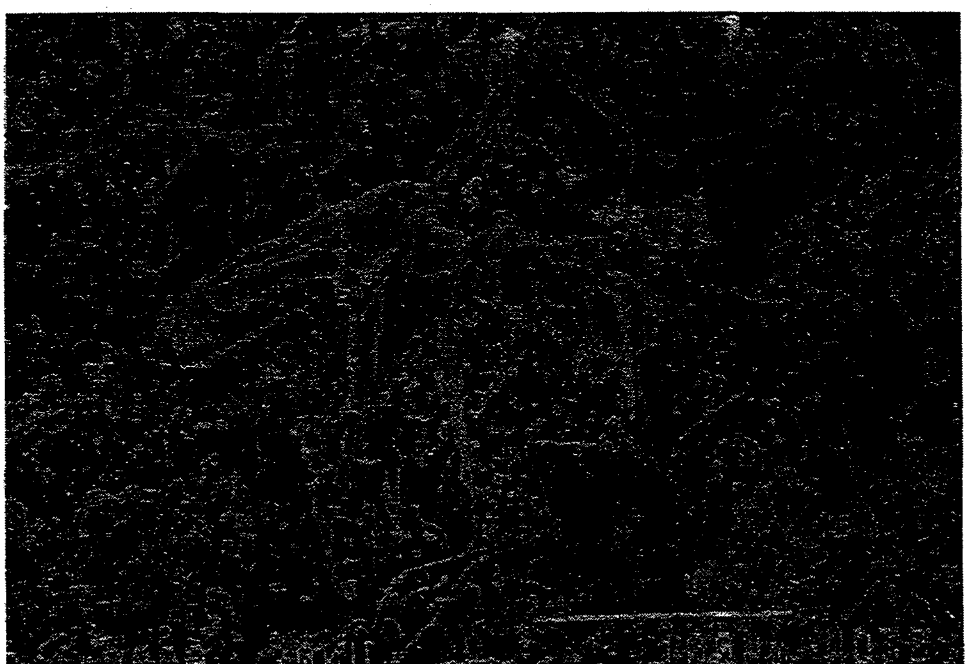


Figure 44 - 300X - UNEXPOSED candle surface

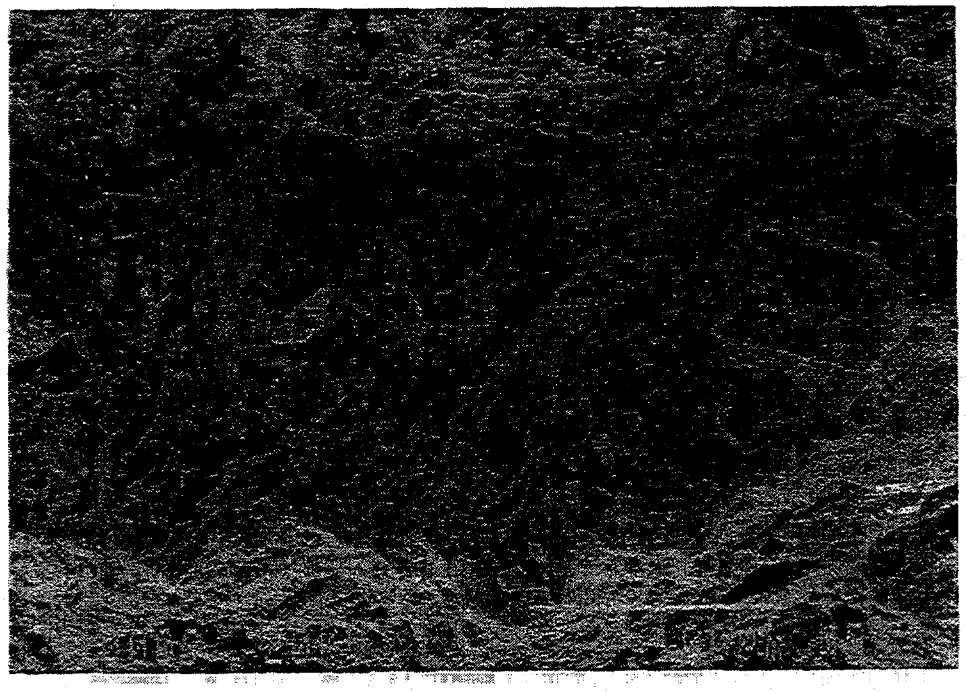


Figure 45 - 300X - EXPOSED candle surface

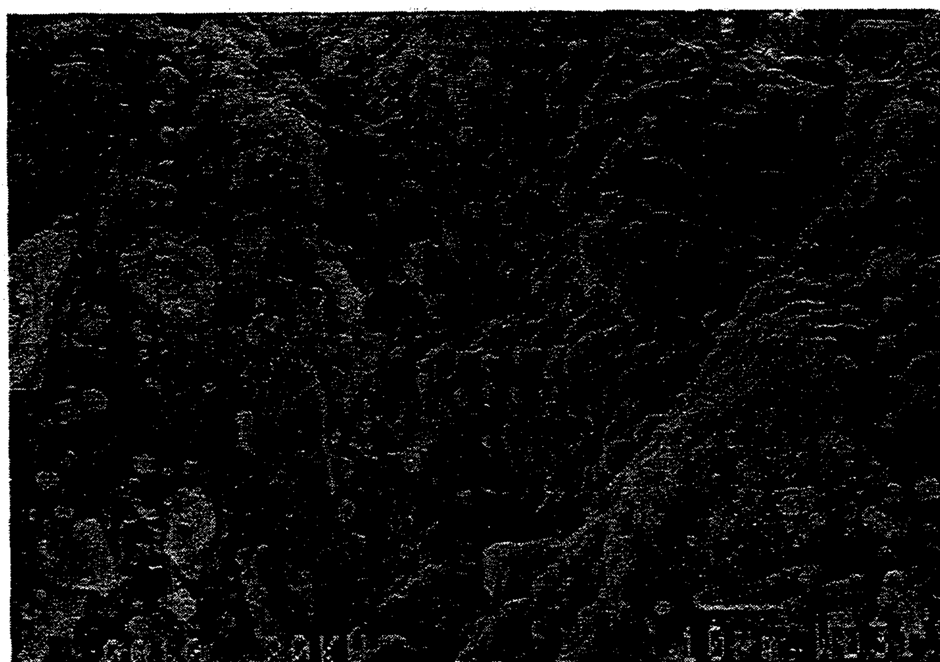


Figure 46 - 1,000X - EXPOSED candle surface

In the following photos, cross-sections of the particulate membrane filler were exposed by fast-fracture and evidence of any ash deposits were sought. By making comparisons with an unexposed filter (Figure 47), no obvious trace of ash could be discerned in Figure 48; no significant difference in the sharp edges of the alumina particles of the membrane was observed. Mary Anne Alvin, of Westinghouse, has suggested that an elemental scan for calcium would be more conclusive but the high amount of gold coating necessary on the sample for SEM obscures the calcium peaks. The assistance of an outside lab would be required and, unfortunately, was not budgeted for.

Figure 47 - UNEXPOSED CANDLE, cross-section of membrane filler (300X)

Figure 48 - EXPOSED CANDLE, cross-section of membrane filler (300X)

In the following photos, the SEM was focused on the region of the support wall within 3mm of the OD surface. The exposed candle in Figure 50 showed no obvious evidence of ash entrainment when compared to the unexposed candle in Figure 49.

Figure 49 - 25X, fast-fracture - UNEXPOSED CANDLE, interior of support wall

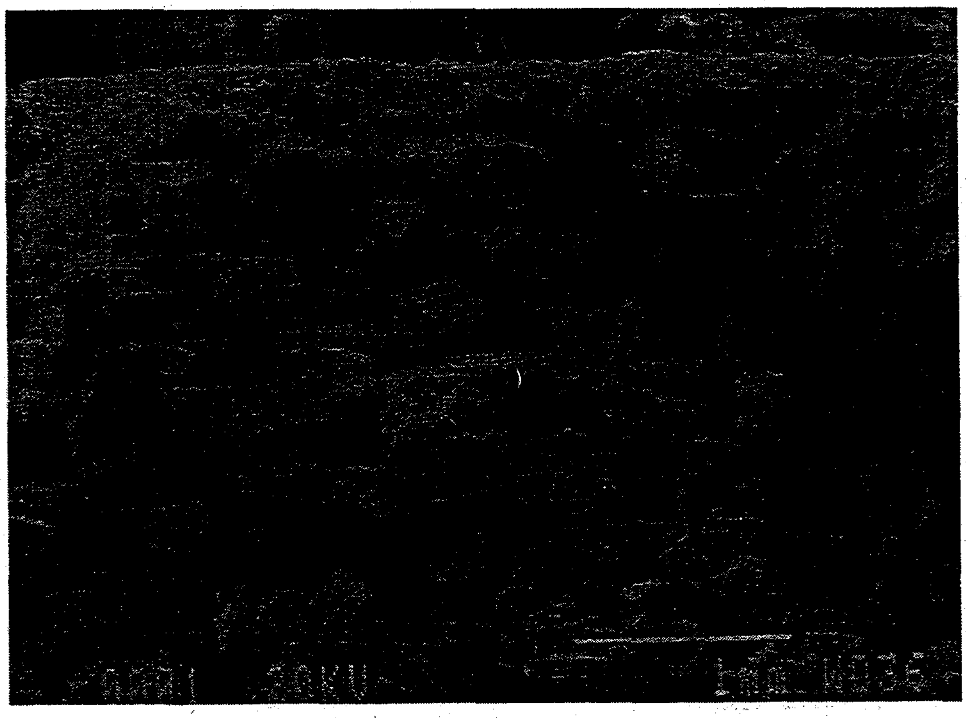


Figure 50 - 25X, fresh-fracture - EXPOSED CANDLE, interior of support wall

Upon closer examination of the 1mm area directly below the membrane, the natural microcracks in the unexposed material are visible along the surface of the filament structures (Figure 51). These microcracks were also visible in the Figure 52 photo of the exposed candle; if ash penetration had occurred, a smoothening or filling of those features may have resulted. These micrographs support the observation that no detectable penetration of ash through the membrane layer occurred.

Figure 51 - 50X, fast-fracture - UNEXPOSED CANDLE, interior of support wall

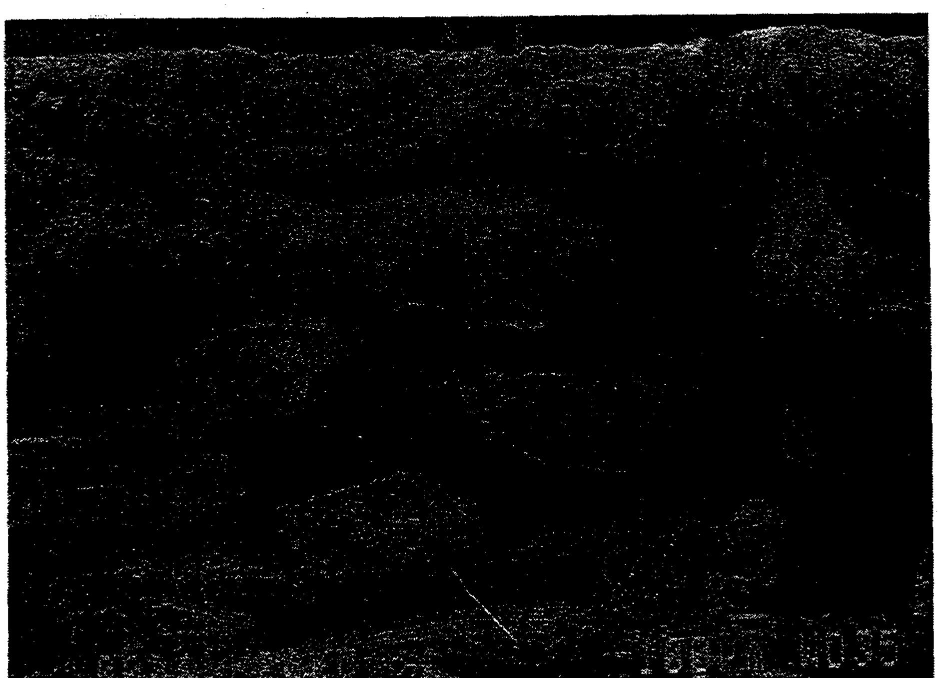


Figure 52 - 50X, fast-fracture - EXPOSED CANDLE, interior of wall support

In Figure 53 through Figure 56, the conditions of the filament structures were examined for evidence of any change resulting from the exposure environment. Figure 53 and Figure 54 each show the cross-section of a single "yarn bundle" at 300X magnification. Each yarn bundle originally consisted of hundreds of filaments. During the firing process, the individual amorphous filaments, coated with alumina, are converted to crystalline phases, primarily cordierite and alumina, with some mullite. The mullite is evident as "needle-shaped" crystals, as seen in the higher magnification photos (Figure 55 and Figure 56). Under conditions which challenge the stability of the PRD-66 microstructure, these needle-like formations are the first to degrade and holes begin to form in the centers of the individual yarn filaments. Neither sign of reaction was observed in either photo of the exposed candle. As a result of this analysis, it was concluded that the microstructure of the PRD-66 material was stable in the Karhula PCFBC environment.

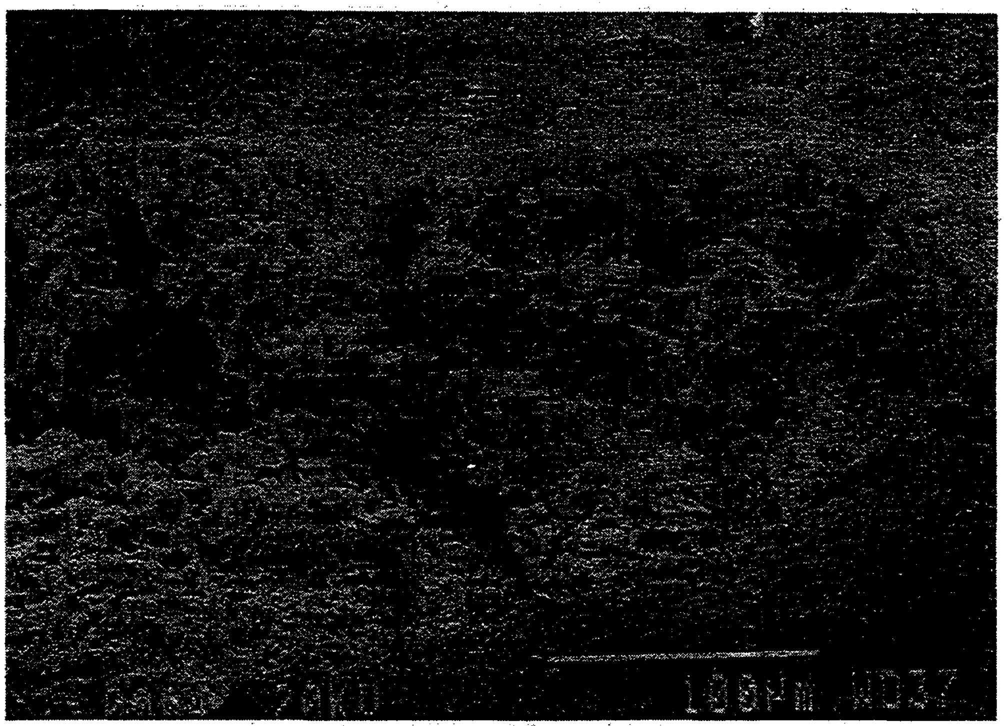


Figure 53 - UNEXPOSED CANDLE, individual "yarn bundle" (300X)

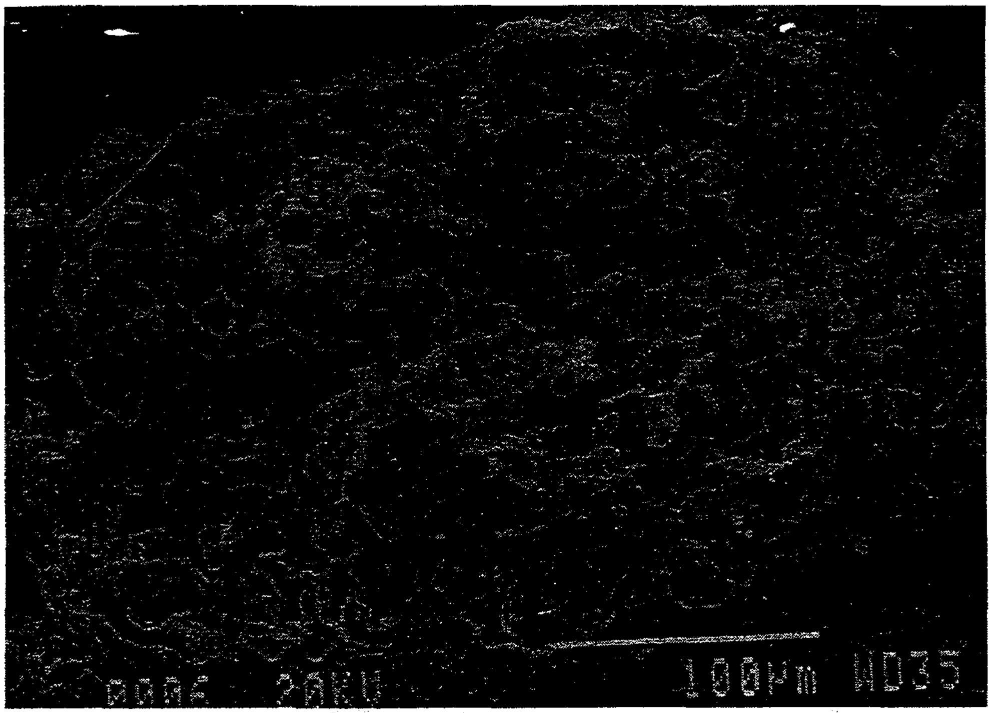


Figure 54 - EXPOSED CANDLE, individual "yarn bundle" (300X)

Figure 55 - UNEXPOSED CANDLE, individual "yarn bundle" (1,000X)

Figure 56 - EXPOSED CANDLE, individual "yarn bundle" (1,000X)

3.6.2.3 Diffraction Analysis of Karhula-Exposed and Unexposed Elements

The stability of the PRD-66 material was further evaluated by qualitative x-ray diffraction (XRD). Specimens of candle #576 (unexposed) and candle #576 (581-hr exposure) were ground into powder and scanned from 5-90 degrees two theta. Both samples contained alumina, cordierite, mullite, and small amounts of cristobalite, in virtually identical amounts. The "exposed" material showed no evidence of any other crystalline phases that may have formed from a reaction of the PRD-66 with the PCFBC environment. The presence of coal ash in the "exposed" sample was not apparent since the material is not crystalline in nature. This analysis supports the visual SEM observation that the material was stable under the Karhula PFBC conditions.

3.6.2.4 Strength Testing of Karhula-Exposed and Unexposed Elements

As previously mentioned, two tested filter elements had been returned by Foster Wheeler to DLC. Candle #577 had been exposed to 581 hours on coal. Candle #591 had been exposed to 239 hours on coal and was broken at the flange when all candles were removed from the vessel for cleaning. 1-inch wide o-rings were sectioned from each candle and tested by o-ring diametrical compression. Average strengths and "load-to-failure" values are compared to unused candles as shown in Table 16. No apparent change in strength was observed.

Unit No.	Condition	Average (psi)	Std. Dev. (psi)	Load-to-Failure (lbs.)	Samples
C566	Unexposed	1087.6	80.8	41.5	11
C576	Unexposed	1256.2	64.7	45.6	6
C578	Unexposed	1352.9	65.2	48.1	5
C590	Unexposed	1076.1	47.8	47.4	6
C577	Exposed-581hrs	1246.6	49.9	50.0	6
C591	Exposed-239hrs	1315.0	103.9	57.0	6

Table 16 - O-ring diametrical compressive testing of Karhula-exposed & unexposed candles

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.

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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:

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

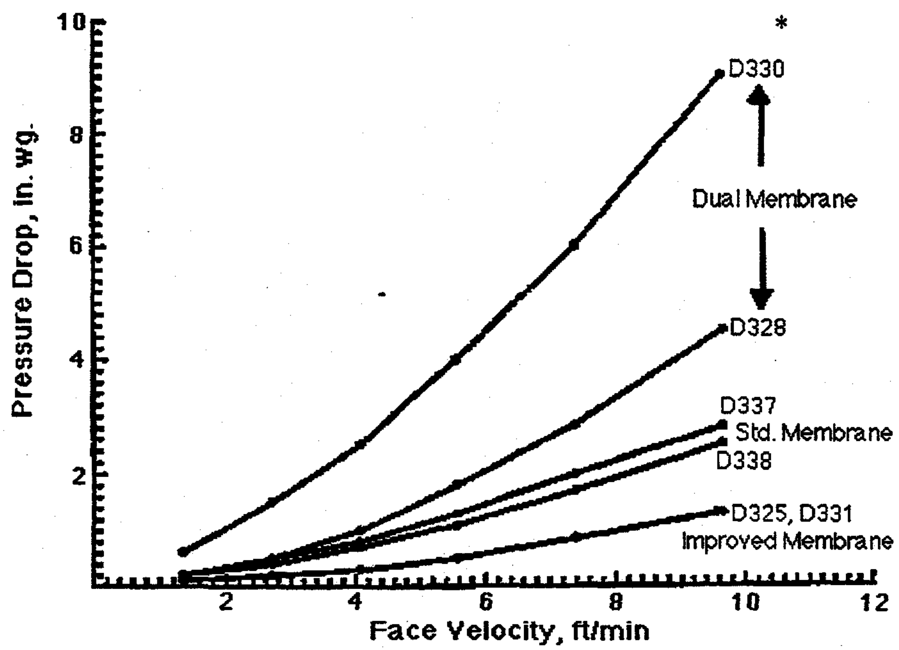
Candle Filter Permeability Measurements Task 3.1)

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 70^0F air (2.41+/-0.8 in-wg at 30.6 scfm at 70^0F 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:

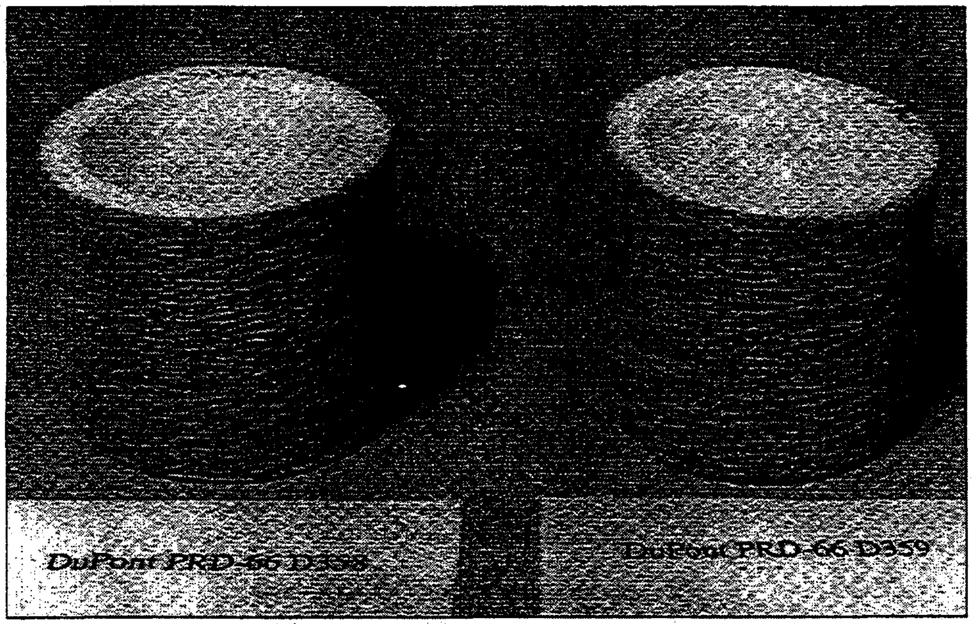


Figure 2 – DuPont PRD-66 filter matrices after room temperature particle collection and gas flow resistance testing.

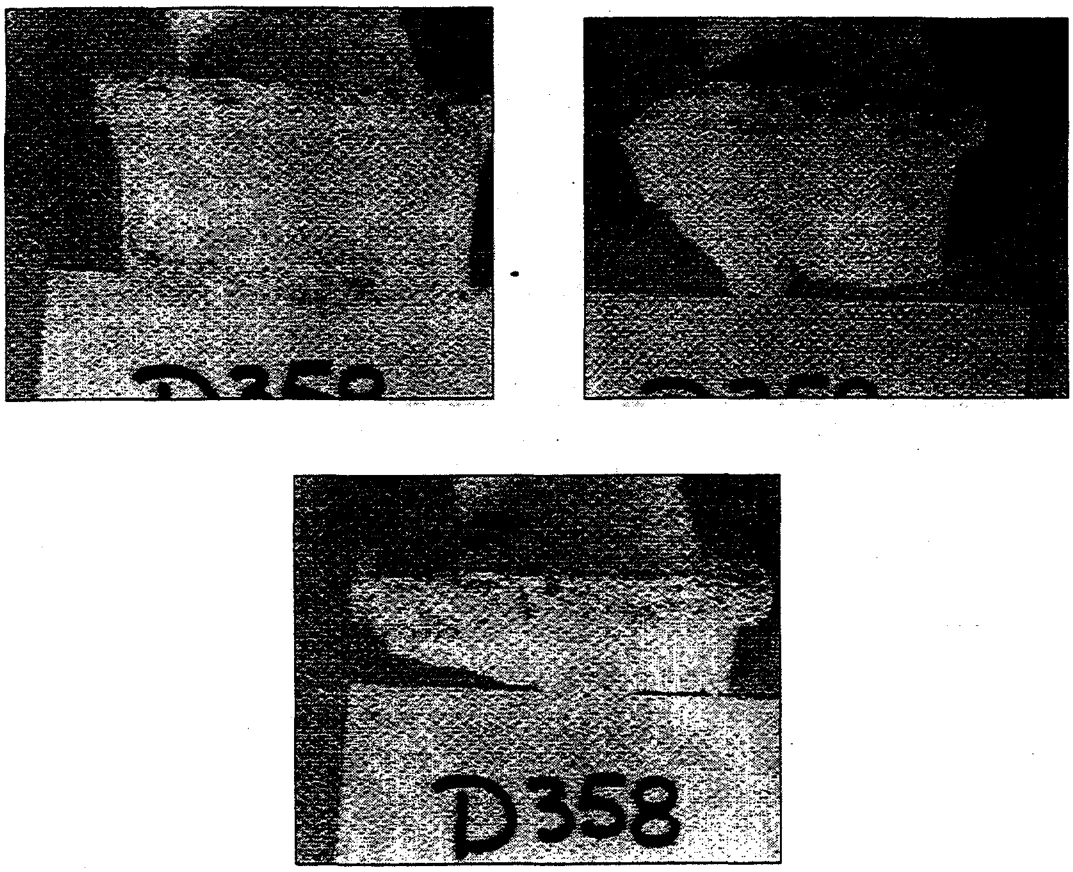


Figure 3a – Fresh fractured surface of the particle challenged D-358 filter matrix.

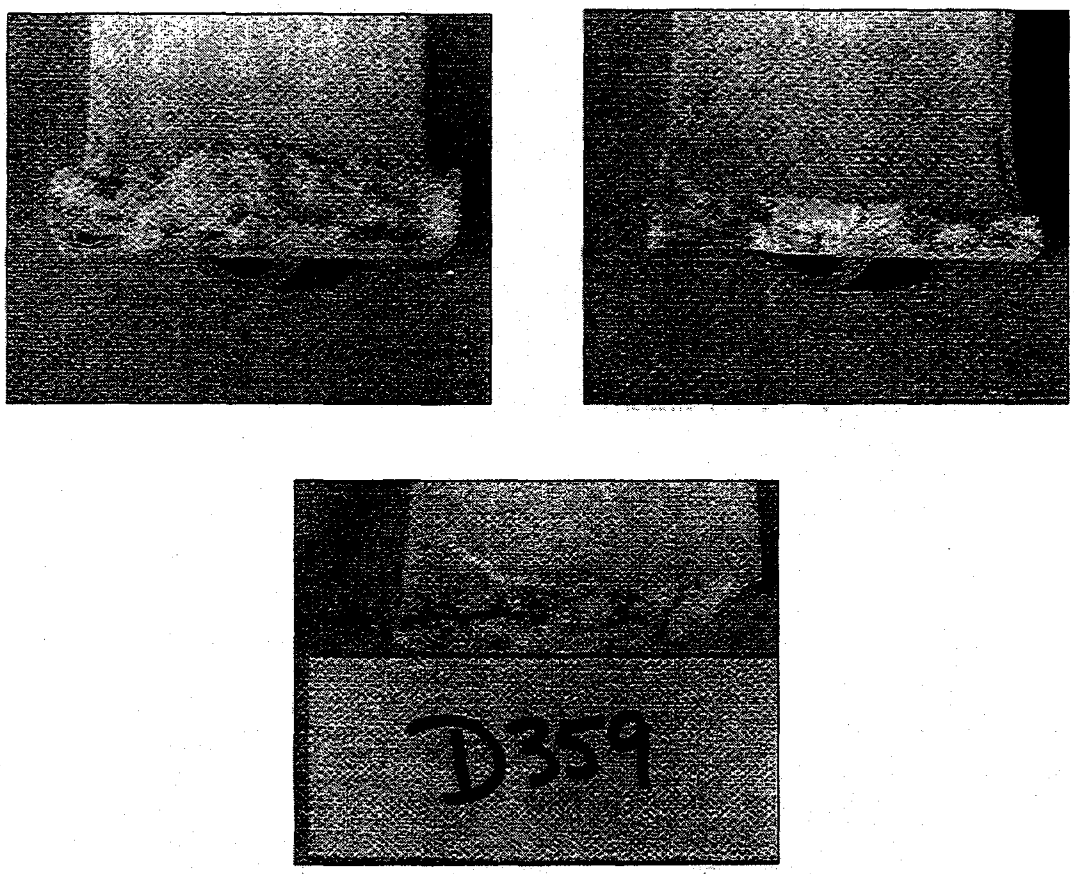


Figure 3b -- Fresh fractured surface of the particle challenged D-359 filter matrix.

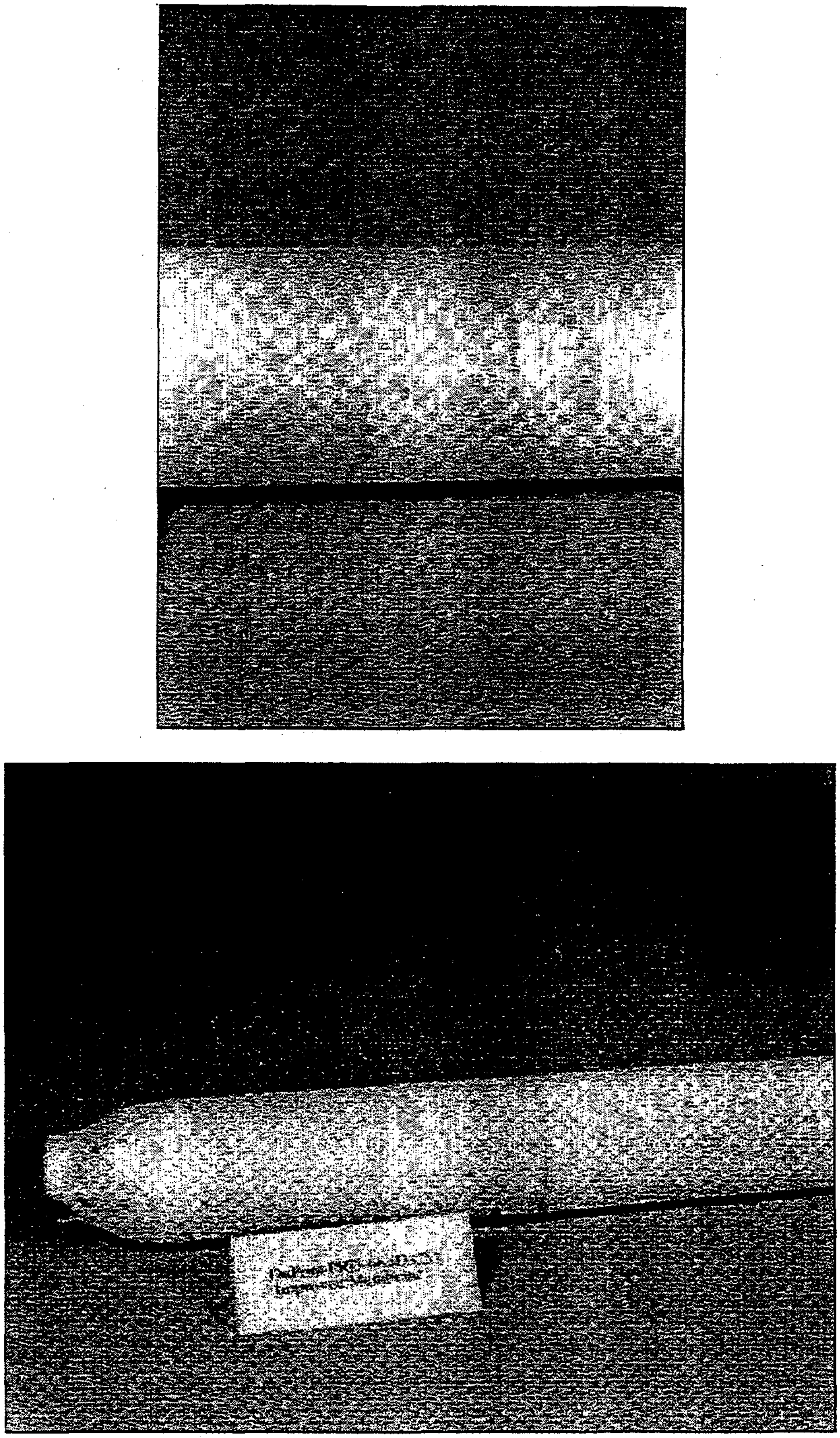


Figure 4a – HTHP-tested DuPont PRD-66 candle filter (Improved o.d. membrane; Strengthened flange).

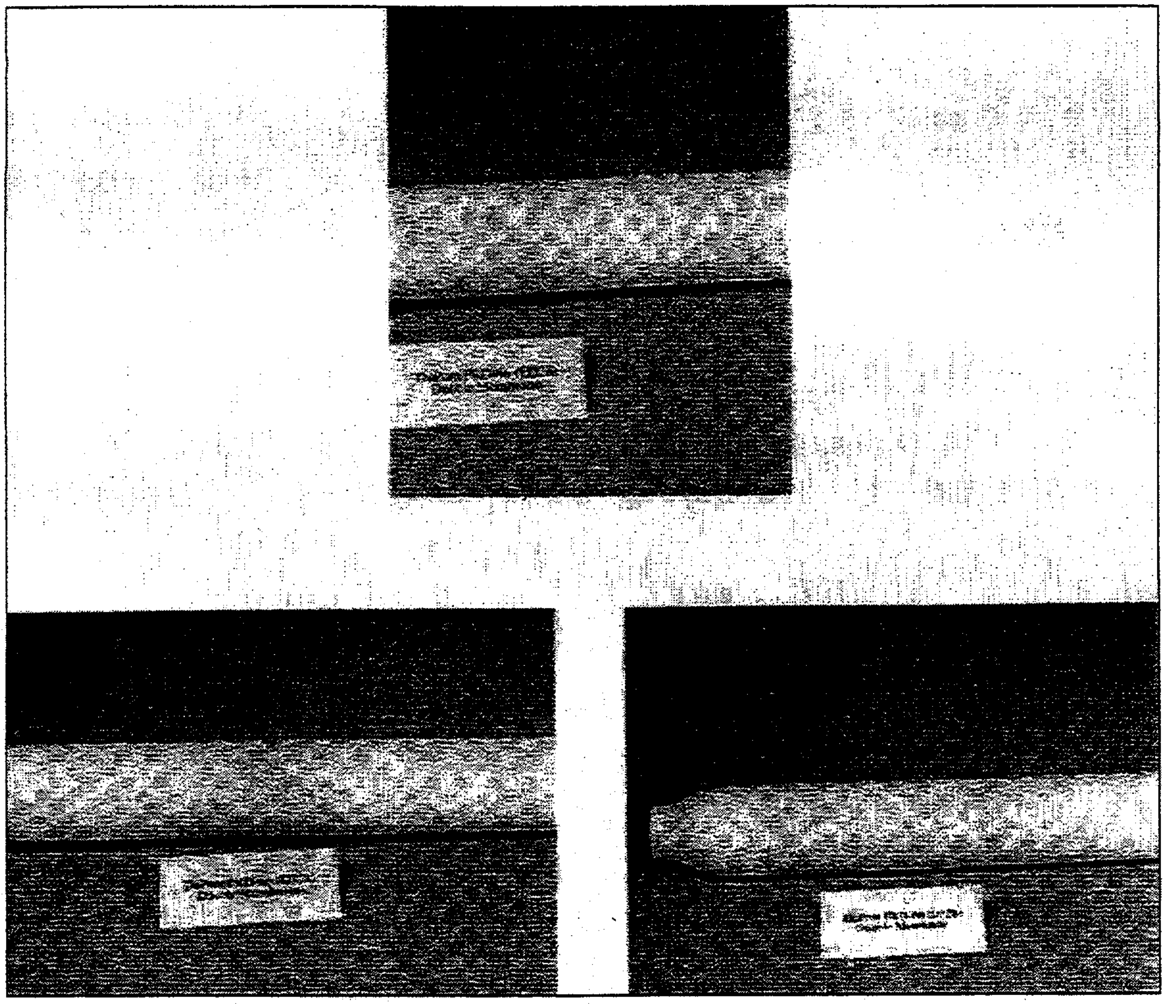


Figure 4b – HTHP-tested DuPont PRD-66 candle filter (Improved dual membrane; Strengthened flange).

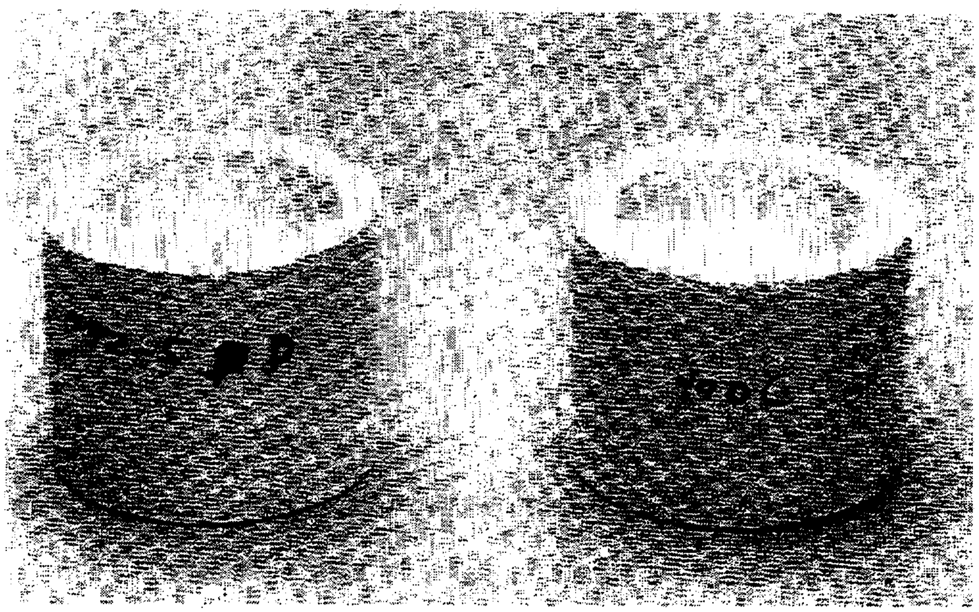


Figure 5 – PRD-66 combination membrane and particulate membrane filter concepts.

- 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.

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[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.

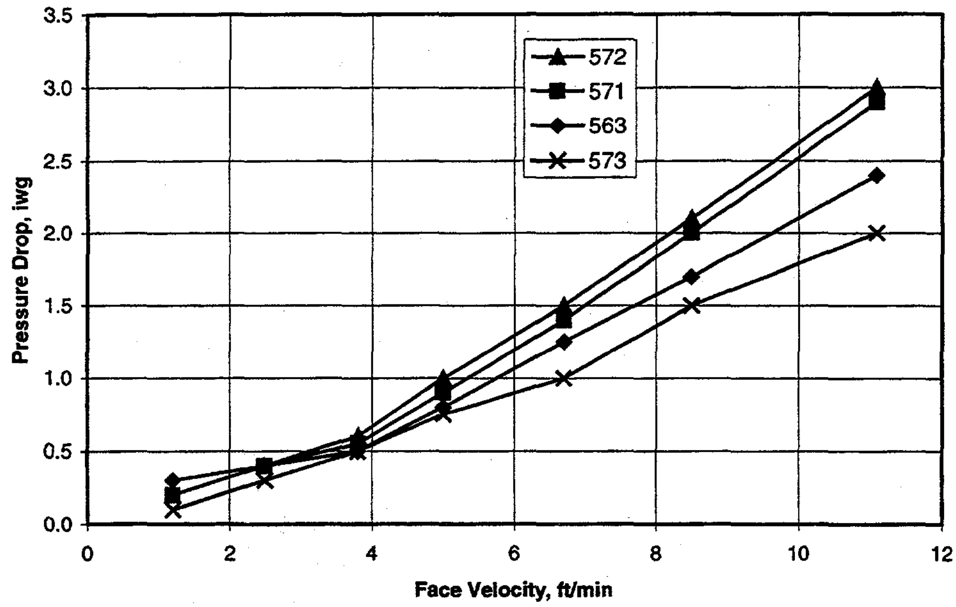


Figure 6 – Room temperature gas flow resistance measurements of the course membrane PRD-66 candle filters.

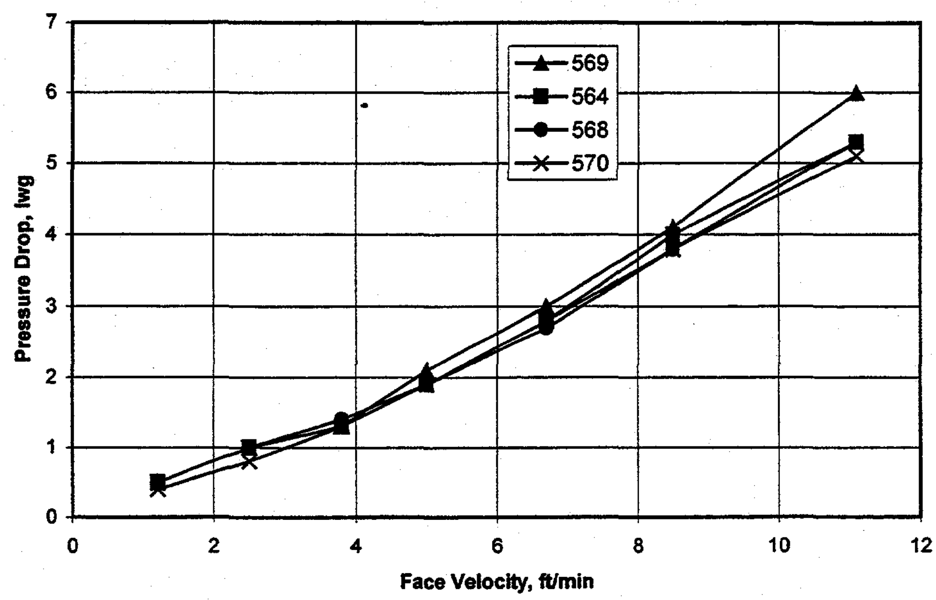


Figure 7 – Room temperature gas flow resistance measurements of the medium membrane PRD-66 candle filters.

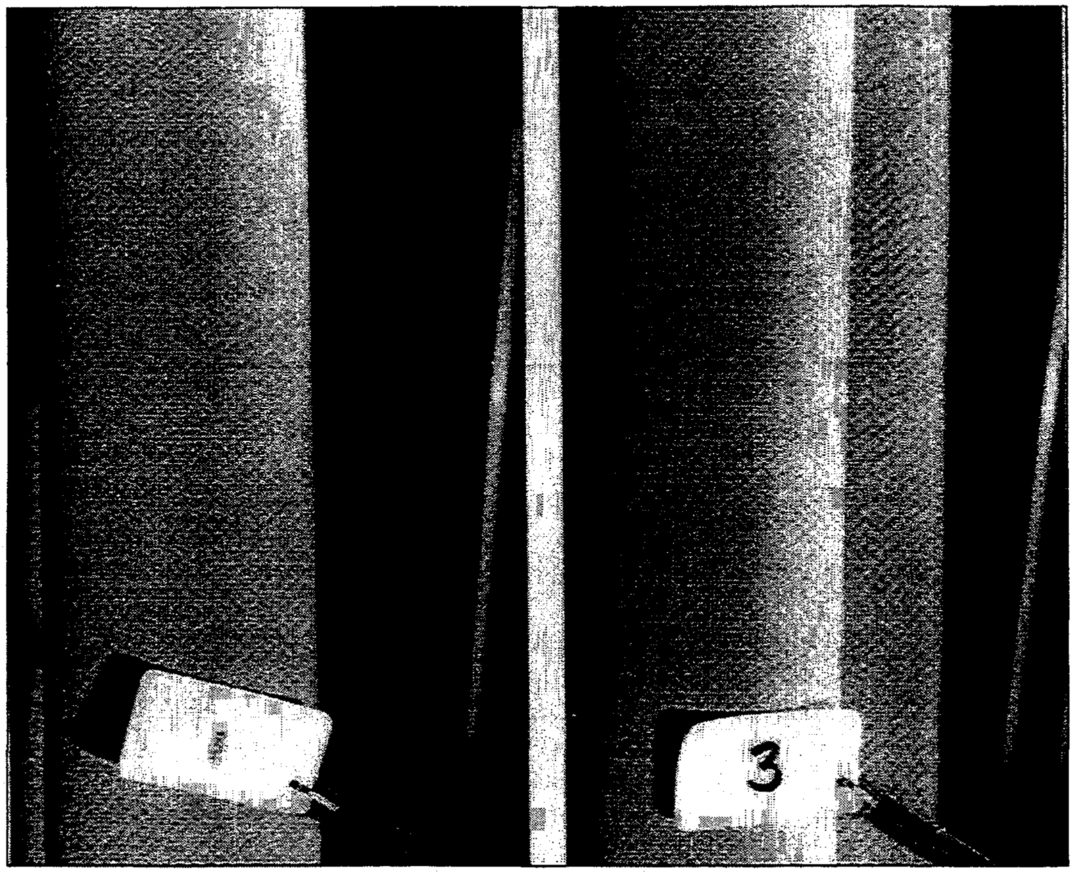
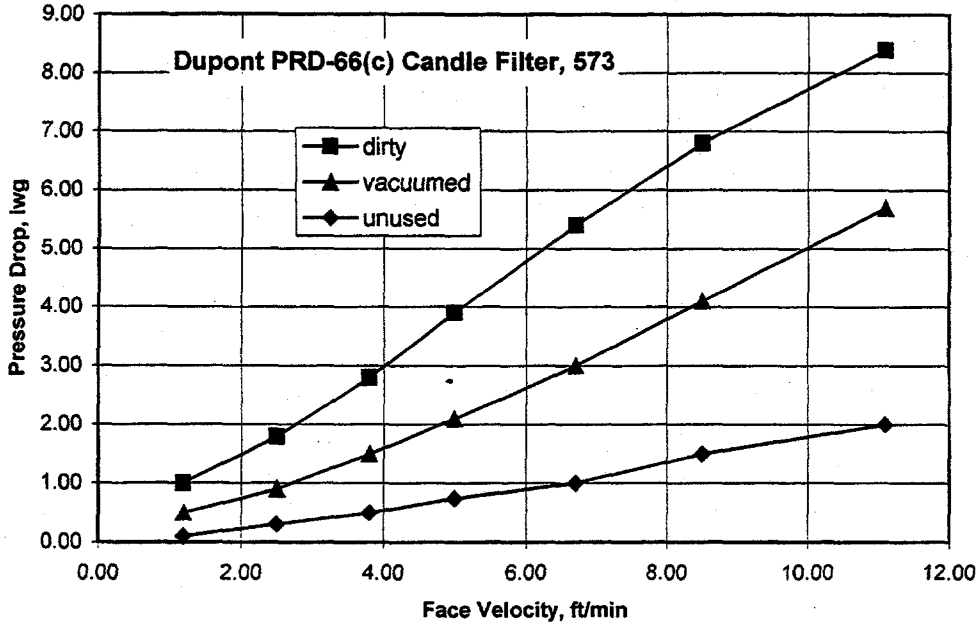


Figure 8 – Photograph illustrating the residual ash cake layer that remained along the outer surface of the PRD-66 candle filters after qualification testing that was conducted under simulated PFBC conditions.



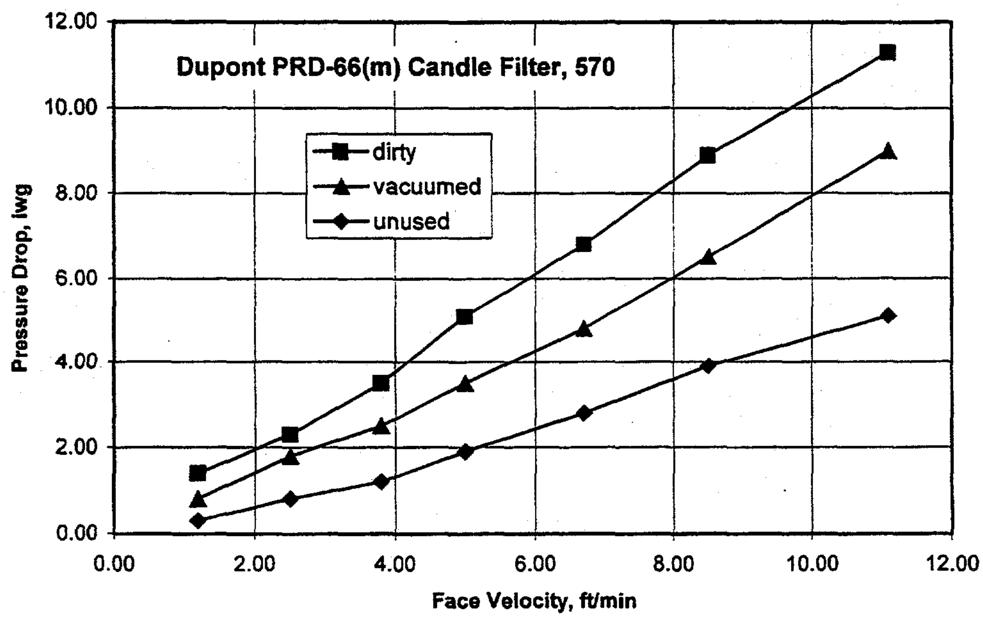


Figure 9 – Gas flow resistance measurements of the as-manufactured and qualification-tested PRD-66 candle filters.

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.

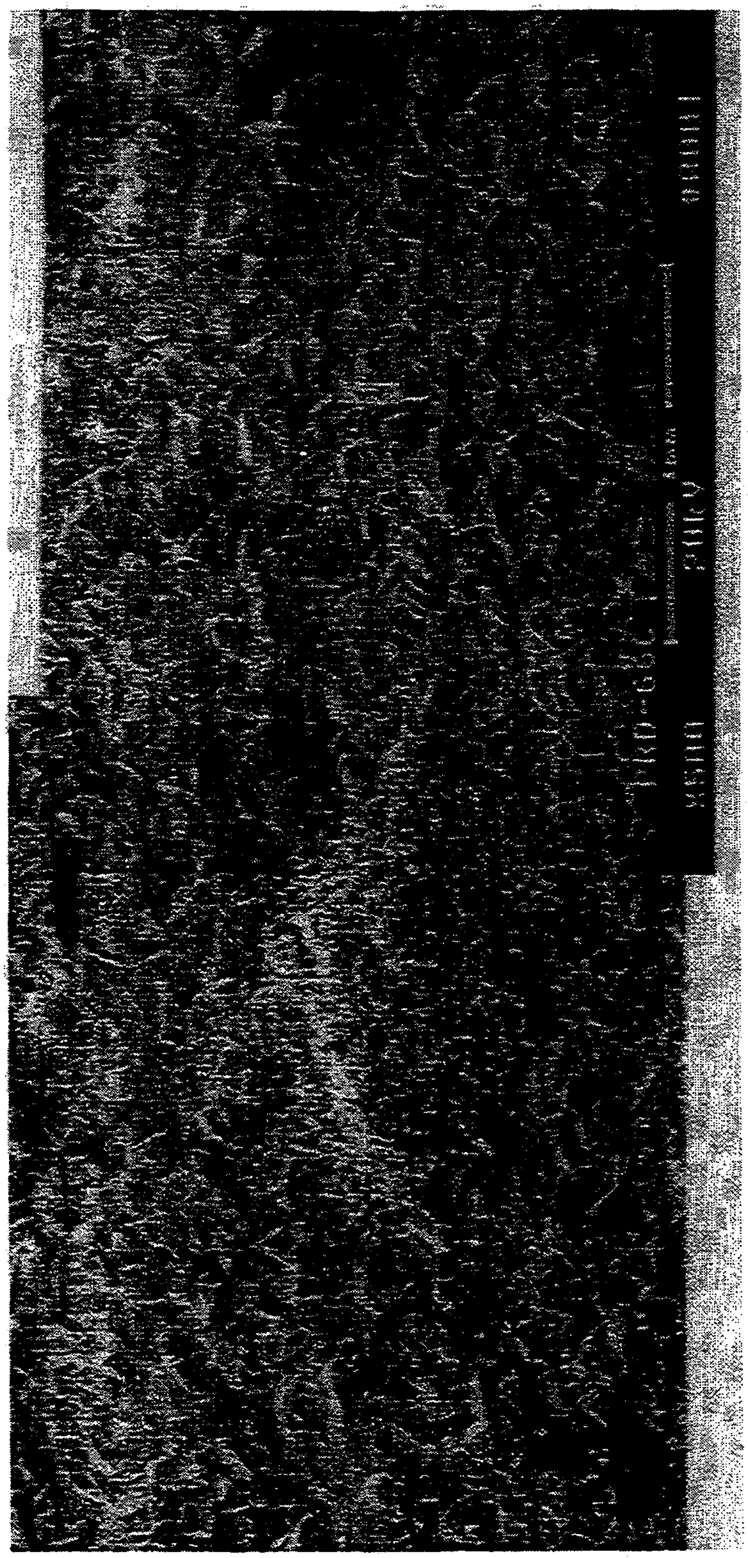


Figure 10 – Micrograph montage illustrating localized adherence of ash fines along the outer surface of the qualification-tested, coarse membrane-coated, PRD-66 filter element.

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.

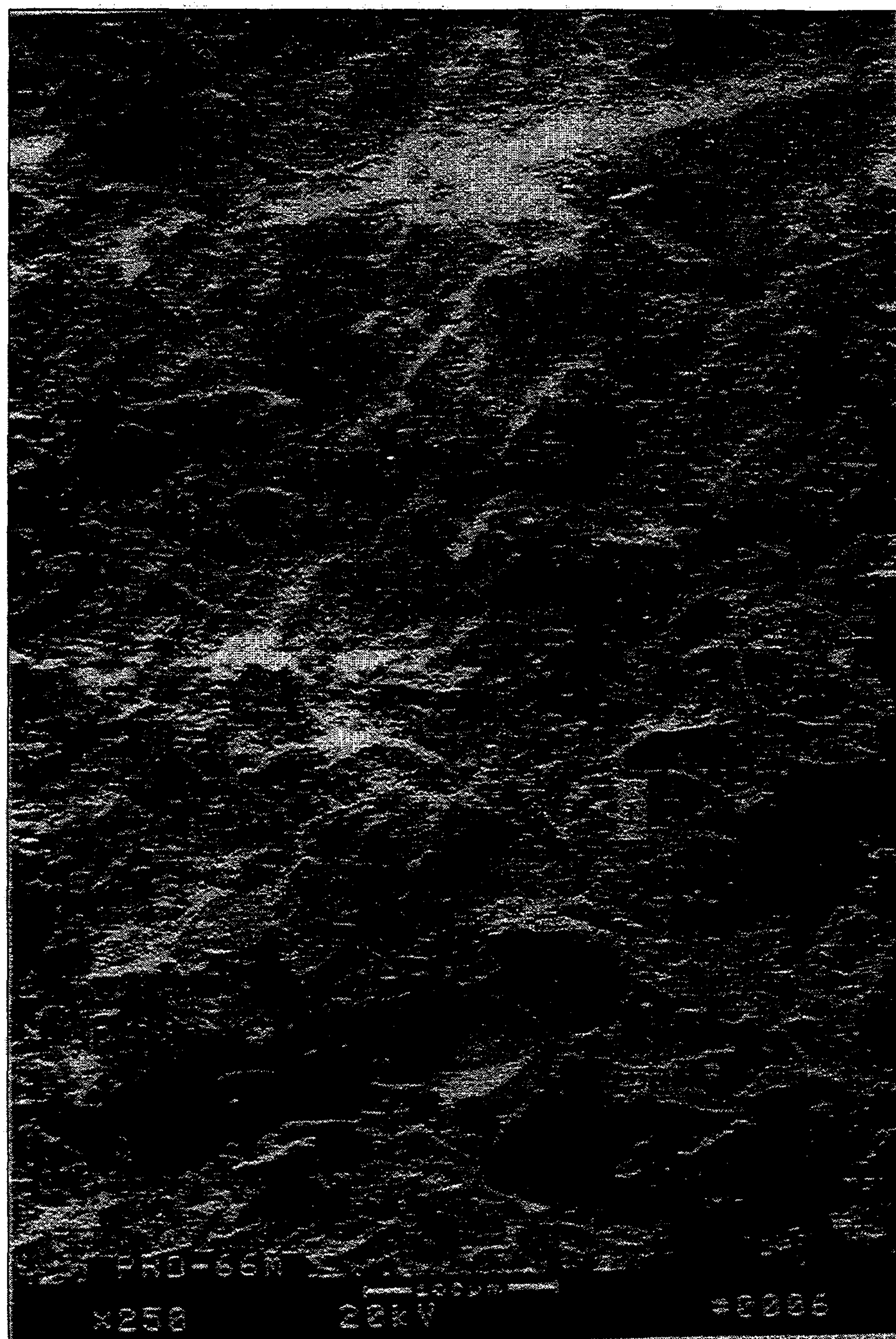


Figure 14 – Micrograph montage illustrating localized adherence of ash fines along the outer surface of the qualification-test, medium membrane-coated, PRD-66 filter element.

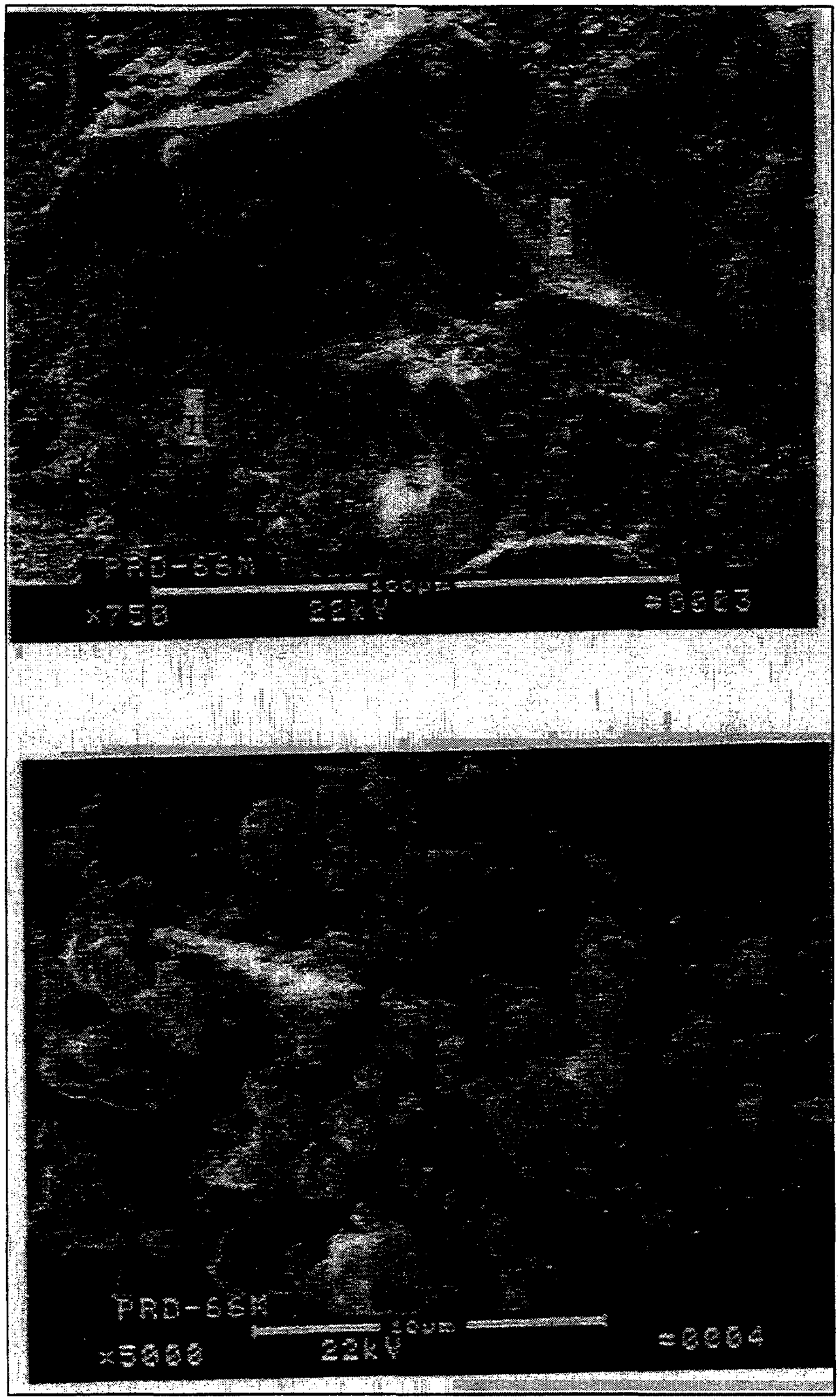


Figure 15 – Higher magnification micrographs illustrating the adherence of ash fines between adjacent alumina-rich grains present along the outer surface of the qualification-test, medium membrane-coated, PRD-66 filter element. The highly porous network of ash fines is shown in the lower micrograph.

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

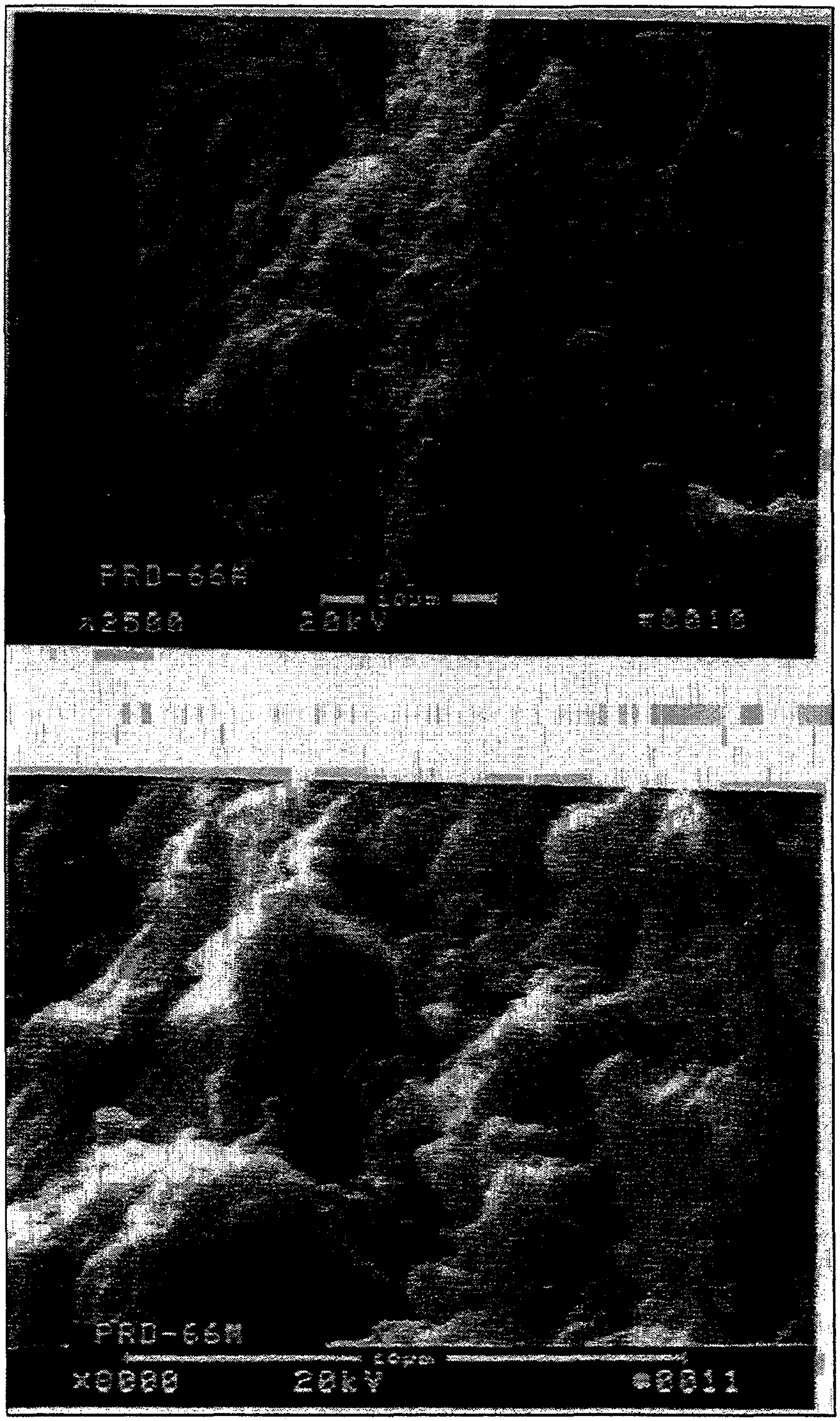


Figure 17 – High magnification micrographs illustrating the adherence of ash fines along the outer surface of the alumina-rich grains that were present within the membrane of the qualification-tested, medium membrane-coated, PRD-66 filter element.

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.

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