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Development and Testing of PRD-66 Hot Gas Filters

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Contract Information

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Abstract

The overall objective of this program is to develop and commercialize PRD-66 hot gas filters for application in pressurized fluidized bed combustors (PFBC) and Integrated Gas Combined Cycle (IGCC) power generation systems. The work is being carried out in phases with the following specific objectives:

1. Demonstrate acceptable mechanical, chemical, and filtration properties in exposure tests.
2. Produce and qualify selected prototype design filter elements in high temperature high pressure (HTHP) simulated PFBC exposure tests.
3. (Option) Generate a manufacturing plan to support commercial scale-up.
4. (Option) Recommend process equipment upgrades and produce 50 candle filters.

Since the beginning of this program, a parallel evaluation of DuPont Lanxide Composites Inc. (DLC) PRD-66 hot gas candle filters took place using AEP's TIDD PFBC facility. Several PRD-66 filters experienced damage during the final testing phase at TIDD, after highly successful testing in earlier runs. During the past year, DLC has undertaken a study under this contract to understand the mechanism of damage sustained in TIDD Test Segment 5. DLC has formulated a hypothesis for the damage mechanism based on the available evidence, and verified that the damage mechanism is possible given the conditions known to exist in TIDD. Improvements to the filter design to eliminate the root cause of the failure have been undertaken. This report details DLC's conclusions regarding the failure mechanism, the evidence supporting the conclusions, and steps being taken to eliminate the root cause.

FY 1996 Program Schedule

	O	N	D	J	F	M	A	M	J	J	A	S
Analysis of Field Exposed Filters	—	—	—	—	—	—	—	—	—	—	—	
Corrosion Testing												—

During the performance of this contract, DuPont Lanxide Composites Inc. (DLC) has been involved in a series of tests at American Electric Power's TIDD pressurized fluidized bed combustor system, exposing PRD-66 hot gas candle filters to pilot scale testing in Westinghouse's filter vessel. After a completely successful test of three candles in Test Segment 4, numerous failures occured in Test Segment 5. In TIDD Test Segment 4, PRD-66 candle filters had a 100% survival rate, with no noticeable damage or decrease in mechanical properties after a run which lasted 1700 hours and operated at temperatures up to 760_C. However, in Test Segment 5, filters essentially identical to those which survived Test Segment 4 had a <10% survival rate, with all filters showing some damage after only 1100 hours and peak temperatures of 845_C. There were many other significant differences between these two runs. For instance, ash loadings were more than five times higher in Test Segment 5 than in previous runs, due to inactivation of the primary cyclone. While Test Segment 4 suffered significant bridging events (though none was observed involving PRD-66 filters) Test Segment 5 was essentially free of bridging. Many other differences will be discussed below.

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, titled Analysis of Field Exposed Filters. This task was carried out in five phases. They were Consultation, Elimination of Known Faults, Hypothesis Formulation, Hypothesis Verification, and Correction.

Phase 1 - Consultation

In the Consultation phase, DLC held discussions with numerous experts in the field of hot gas filtration. They included Ted McMahon, Rich Dennis and Dwayne Smith of METC, Mary Anne Alvin 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. Valuable evidence and insight was gained from these discussions.

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.

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 1, there were significant differences between Test Segments 4 and 5. Ash loading increased

Test Segment	TIDD 4	TIDD 5
Test Duration	1700 hr.	1100 hr.
Survival Rate	100%	10%
Ash Cake	Thin, uniform	Thin patchy
Damage	None	Divots Mid-body Flange
Bridging	None	None
Operating Temperature, °C	660-760	760-845
Ash Loading, ppmw	3,200	18,000
Primary Cyclone	Detuned	Inactive

Table 1. Comparison of test conditions in TIDD Test Segments 4 and 5.

from 3,200 ppmw to 18,000 ppmw because of the inactivation of the primary cyclone before 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 technique developed by DLC under this contract, and described ion our paper last year. This reinforcement technique increased the strength of PRD-66 materials by about 50%. DLC is confident the the reinforcement will ameliorate this problem. A second, more puzzling failure mechanism is found in mid-body failures, as well as filters which survived the full duration of the test. The physical evidence seen on the filters included 'divots,' as shown in Figure 1.

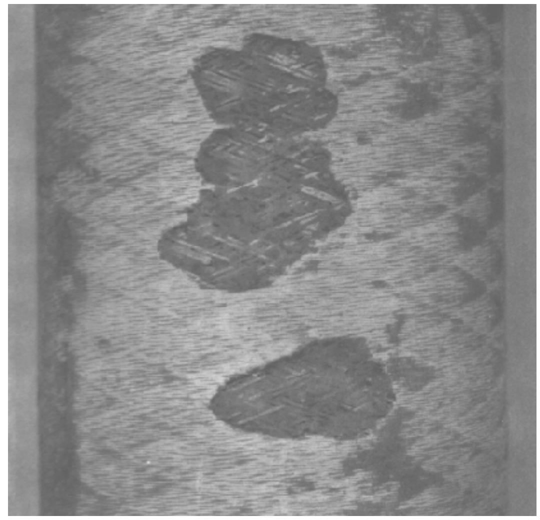

Figure 1. Divots in a PRD-66 filter segment.

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, i.e., they were 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 2) 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.

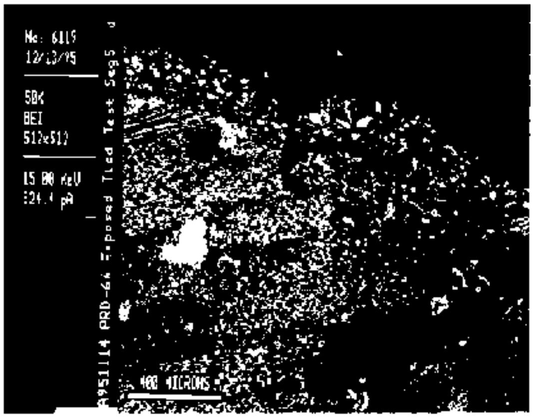

Figure 2. White deposit (middle left) in vicinity of divot (upper right.)

Based on this evidence, DLC formulated a hypothesis of the failure mechanism of PRD-66 candle filters in Test Segment 5.
DLC hypothesized that despite earlier results of room temperature and high temperature tests to the contrary, ash containing adsorbent penetrated the surface filtration membrane of 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 to hot SO_2 gas, causing *in situ* sulfation of the ash to calcium and/or magnesium sulfates in the pores and microcracks of the filters. 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 by successive hydration of the sulfates during cooling in moisture containing atmosphere, which also would occur on process interruptions. Figure 3 shows how the unit cell volume of anhydrous magnesium sulfate increases as it picks up waters of hydration.

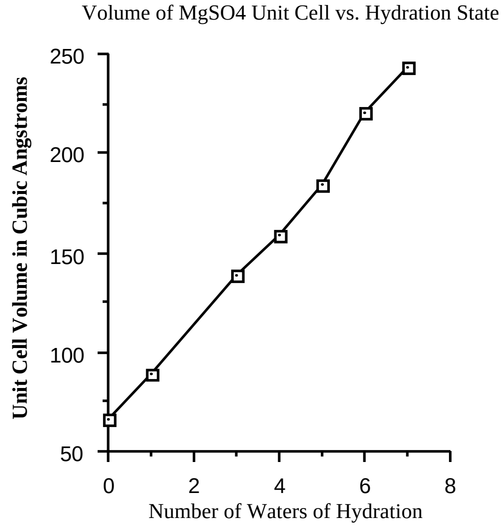

Figure 3. Unit cell size of magnesium sulfate vs. state of hydration.

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 in the vicinity of 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 in a backpulse. (See Figure 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

SO_2, and possibly moisture, plus

Figure 4. Wall thickness reduced by divot at fracture surface, lower left.

excursions of temperature. 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 ash cake would strongly adhere and then take over filtration. A sample passes the test if after one exposure, no ash penetrates to the inner diameter. Since physical evidence from TIDD test segments 4 and 5 showed that the ash cake was thin and only loosely adhered, DLC worked under the assumption that the crystalline alumina surface of our filter released the ash essentially completely on each backpulse. Therefore, after exposing filter segments to ash by applying a vacuum to the inner diameter, the resulting ash cake was physically removed with light brushing. This ash exposure/cleaning cycle was repeated 25 times. The intent was to simulate the effect of complete ash cake release after a series of cleaning backpulses. This more stringent test showed that ash consistently penetrated into the filter body and was trapped in the wall using the filter design tested in TIDD. Figure 5 shows the results of a non-destructive test which images where ash penetrates the membrane surface.

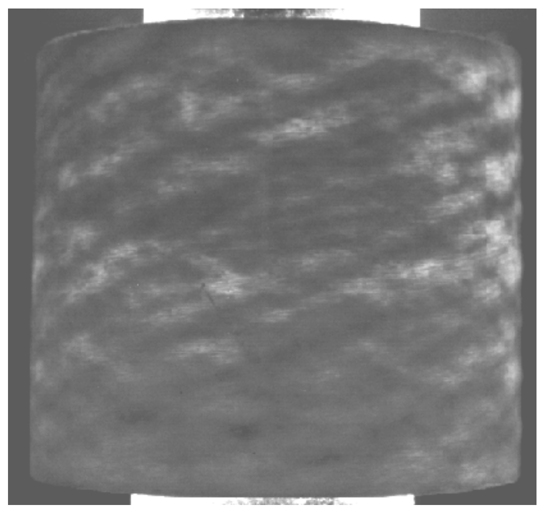

Figure 5. Non-destructive evaluation of ash penetration. Dark areas show ash penetration, light areas show no penetration.

Even after the extensive penetration shown in the figure, ash still does not penetrate to the inner diameter after 25 cycles. This shows 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.

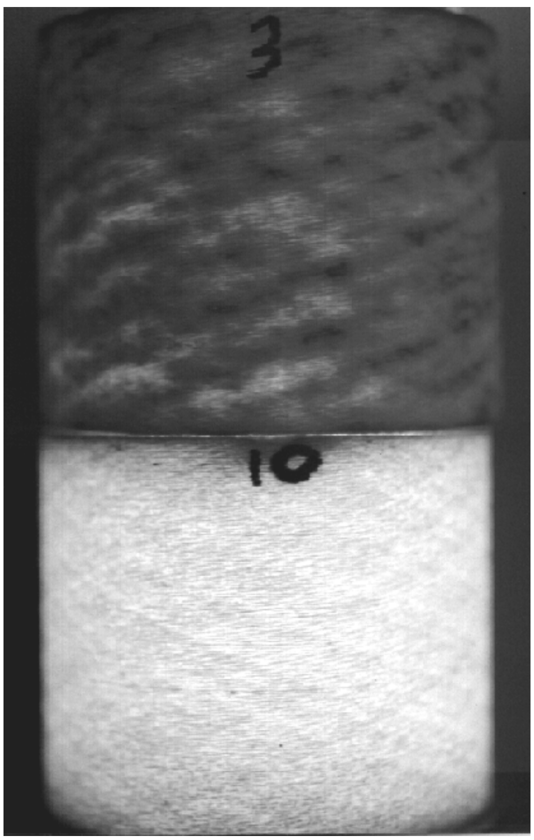

Figure 6. NDE test showing: ash penetration in TIDD filter design (top) no penetration in improved design (bottom).

Phase 5 - Correction

The task for Phase 5 is therefore obvious; to stop the penetration of ash through the membrane into the body of the filter. Without ash in the body of the filter, the damage caused by the hypothesized failure mechanism could not occur. To this end, DLC is designing improved surface filtration membranes. We have already designed several membranes which pass our ash infiltration test, and offer acceptable pressure drops. Figure 6 shows a filter segment of the type tested in TIDD, which shows significant penetration, and one of the new membranes which show no ash penetration. DLC will fabricate full scale filters incorporating the new membrane for corrosion and high temperature, high pressure testing at Westinghouse's test facilities, which will fit us back into the original program design for this contract.

Conclusion

After an investigation of filter failures in TIDD Test Segment 5, DLC formulated a hypothesis that *in situ* sulfation of ash trapped in the filter wall caused deposits which damaged the filter material on thermal excursions. DLC has verfied that necessary and sufficient conditions to bring about that failure condition existed in TIDD. To correct the problem, DLC has invented new surface filtration membranes to prevent the problem in the future.

Acknowledgements

The authors and DuPont Lanxide Composites Inc. gratefully acknowledge conversations with Ted McMahon, Rich Dennis and Dwayne Smith of METC, Mary Anne Alvin 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. DLC gratefully acknowledges financial support from METC under contract DE-AC21-94MC31214.