CONF-9506162--55

DOE/MC/31214-96/C0521

Development and Testing of PRD-66 Hot Gas Filters

Authors:

Jeffrey A. Chambers, Ph.D.

Contractor:

DuPont Lanxide Composites, Inc.
1300 Marrows Road
P.O. Box 6077
Newark, DE 19714-6077

Contract Number:

DE-AC21-94MC31214

Conference Title:

Advanced Coal-Fired Power Systems '95 Review Meeting

Conference Location:

Morgantown, West Virginia

Conference Dates:

June 27-29, 1995

Conference Sponsor:

U.S. Department of Energy, Morgantown Energy Technology Center (METC)

6A.1 Development and Testing of PRD-66 Hot Gas Filters

Contract Information

Contract Number DE-AC21-94MC31214

Contractor DuPont Lanxide Composites Inc.
1300 Marrows Road PO Box 6077
Newark, DE 19714-6077
(302) 456-6235 (telephone)
(302) 456-6480 (FAX)

Other Funding Sources

Contract Project Manager Jeffrey A. Chambers, Ph.D.

Principal Investigator Jeffrey A. Chambers, Ph.D.

METC Project Manager Theodore J. McMahon

Period of Performance September 29, 1994 to November 30, 1995

Schedule and Milestones

FY 95 Program Schedule

Objectives

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.

Background Information

For several years prior to the initiation of this project, DuPont, DuPont Lanxide Composites Inc. (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 was what will be referred to hereafter as the 'baseline' PRD-66 candle filter.

PRD-66 ceramic oxides are materials with a unique combination of high continuous use temperature and high thermal shock resistance in a non-fiber reinforced material (Figure 1). To be sure, there are monolithic ceramics with higher continuous use temperature, but they tend to have poor thermal shock resistance. Certainly there are metals with better thermal shock resistance, but these metals tend to melt and/or oxidize at high

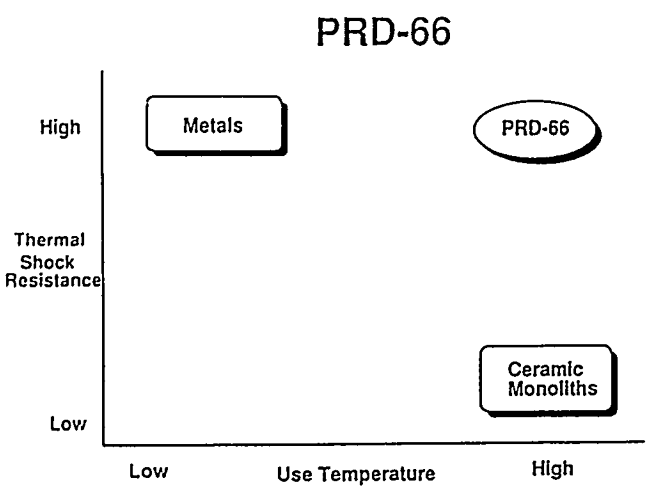

Figure 1. Thermal Properties of PRD-66

temperatures. PRD-66 is comprised of a layered, microcracked structure of an all-oxide chemical composition of silica, magnesia, and alumina (Figure 2). It consists of alternating layers in the crystal forms of corundum, mullite and cordierite, each layer being 50-100 microns thick. These layers repeat throughout the body of the filter. The all-oxide chemical composition is thought by

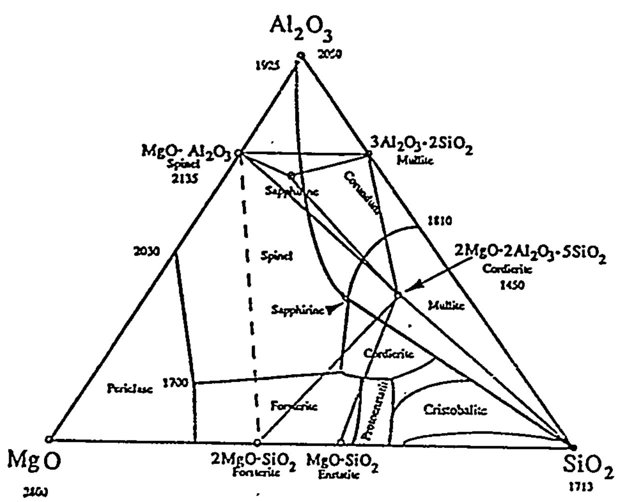

Figure 2. PRD-66 Composition

experts in the field to be favorable for survival in the coal combustion gas environment. The extensive network of microcracks and interfaces between the oxide layers is thought to provide the outstanding thermal shock resistance and damage tolerance of PRD-66, as any cracks that may form in the material do not progress very far before they encounter a crack or interface which helps reduce crack propagation energy.

PRD-66 is manufactured through a simple, patented process (Figure 3). A fiberglass yarn is coated with a slurry 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

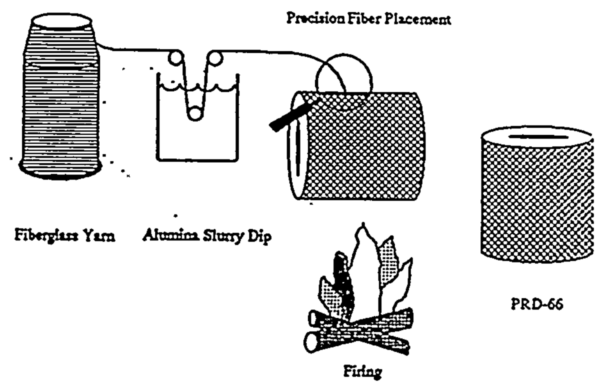

Figure 3. PRD-66 Manufacturing Process

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.

The resulting ceramic material has a continuous use temperature of approximately 1200-1300°C in oxidizing atmospheres. In other applications, PRD-66 has survived numerous thermal shock cycles in excess of 1000°C/sec. It is believed that these thermal properties are in great excess of those experienced in this application. Through careful control of the filament winding process, DLC has been able to produce a filter body which is an absolute bulk filter, as well as having a surface membrane which is a completely efficient particle filter. In the event that the surface filter should be damaged, the bulk filter would take over the filtration.

Project Description

The project is being carried out in five tasks, two of which have been completed and the third is underway. Task 1 provided the management plan and the necessary NEPA information for the project. In Task 2, the test plan was written. Laboratory work began in Task 3, which consists of three subtasks. In Subtask 3.1, attempts at design improvements of the baseline candle filter were made. Among the design improvements sought in the project were increased strength of the flange area of the filter, and a reduced pressure drop filter. Full size candle filters incorporating these attempts at design improvements were fabricated. These filters would then be tested by our subcontractor, Westinghouse Corporation, 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 will be made, and full sized candle filters incorporating the selected improvements will be made 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, mechanical properties of the baseline filter would be obtained.

In Subtask 3.2, corrosion testing will be carried out on the improved candle filters at Westinghouse. In Subtask 3.3, HTHP testing simulating PFBC conditions will also be carried out. After exposure testing, mechanical properties will be measured to determine residual mechanical properties.

In Task 4, which is an option to be exercised at METC's discretion, basic manufacturing issues necessary for scale-up to commercial production will be investigated. These issues include raw ingredient quality, process economics, and manufacturing equipment reliability, among others. In Task 5, also an option, recommendations for process improvements will be made, and 50 filters will be manufactured.

Results

As of this writing, the project is in the latter stages of Subtask 3.1. Tasks 1 and 2 were completed per the projected schedule. DLC has completed development of design improvements on both filter subelements and full sized filters, as described below. Sections of those filters have been tested for filtration and permeability by Westinghouse, and those results are presented below. Mechanical tests have been surveyed, and a preferred test has been selected and applied to understand the mechanical properties of the baseline PRD-66 hot gas filter.

Reduced Pressure Drop Filter

The objective of this effort was to reduce the pressure drop of PRD-66 filters compared to the baseline filter, while maintaining good filtration, cake formation and release characteristics. As shown in Figure 4, those efforts have been successful. Through a series of process modifications, it was demonstrated that virtually all the pressure drop of the candle is developed by the surface membrane. Thus the task of reducing the overall backpressure was reduced to lowering the pressure drop of the membrane. Figure 4 shows that pressure drops could be lowered as much as 85%. In the very lowest pressure drop candles, problems developed with adhesion of the

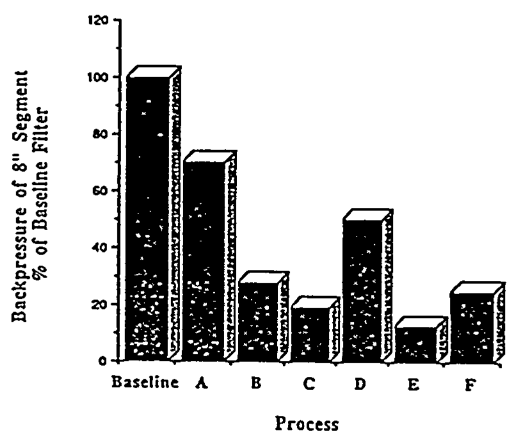

Figure 4. Pressure Drop vs. Process Iteration

membrane to the body of the candle filter, and it has been shown that a 'medium' level of pressure drop reduction can be obtained with a membrane that is strongly adhered. Full scale filters incorporating the lower pressure drop membrane have been manufactured.

Strengthened Flanges

The objective of this effort was to increase the strength of the flange region of the filter while maintaining a graceful failure mode. It was deemed desirable to be able to control the location and degree of reinforcement. As shown in Figure 5, DLC was able to increase the strength of the filter body material by up to 50% relative to the baseline filter. We were also to able to vary the degree of reinforcement over a relatively smooth curve. Within a reasonable degree of control, we were also able to provide a gradient of reinforcement from the strongest section to the filter body. Load deflection curves of o-ring sections showed graceful similar to the baseline material.

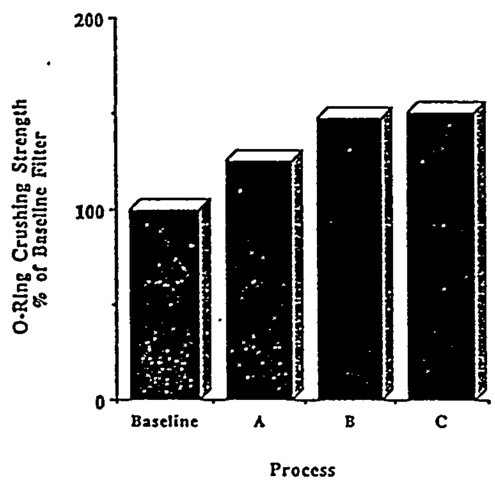

Figure 5. O-ring Crushing Strength vs. Process Iteration

Mechanical Property Testing

The objective of this effort was to identify a mechanical property test which is precise and accurate for the PRD-66 material. In addition, it is 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.

PRD-66 hot gas filters are made by a process which, at a fundamental level, produces only tubular shapes. It is impossible to manufacture a flat coupon which closely mimics the internal structure of a PRD-66 filter, and therefore only tests which are based on a cylindrical sample were considered. This limits the range to o-ring or c-ring tests. C-ring tests were evaluated, but cutting the 1 inch notch from the coupon incurred machining damage and additional cost. O-ring tests are ideal from that standpoint, with only two cuts required 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. It shows a great deal of reloading and strain tolerance after peak load. Tests 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. In the 100 or so mechanical tests conducted in developing this o-ring section test, no sample displayed catastrophic failure, i.e., failure into two or more pieces. Figure 7 shows the test

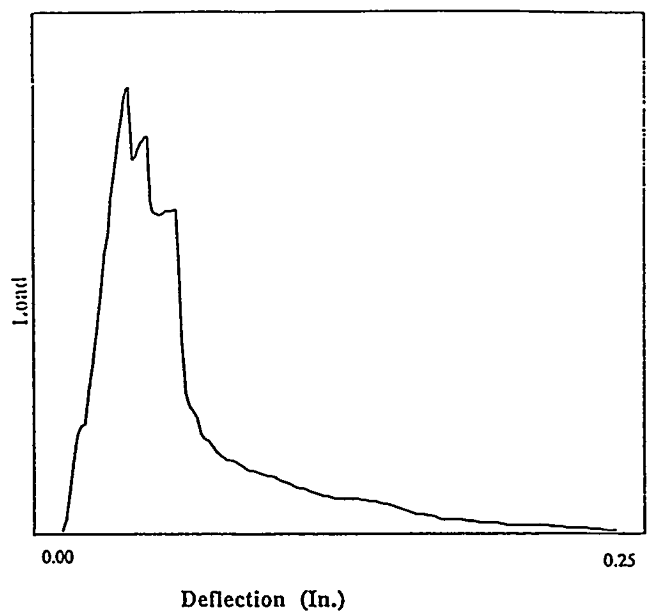

Figure 6. Load Displacement Curve of Typical PRD-66 Filter Section

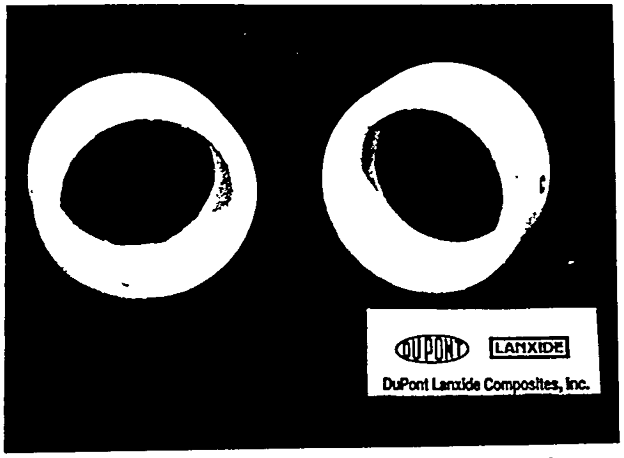

Figure 7. PRD-66 Filter Section Before (right) and After (left) O-ring Compression Test.

specimen from Figure 6 after failure, displaying a permanent deflection of 0.25 inches, but still intact. Method plus product variability of the o-ring compression test was on the order of ±10%, fairly remarkable for mechanical tests on porous ceramics. Other laboratories have also conducted o-ring and c-ring tests on PRD-66 filter samples and calculated different strengths. DLC's estimate is the most conservative. DLC hopes to reconcile the different testing results in the near future.

Filtration and Permeability Testing

The objective of this effort is to verify that the design improvements developed in the work described above do not adversely impact the fundamental properties needed in a hot gas filter, permeability and filtration efficiency. To demonstrate this, 2 inch segments of full sized filters were tested on bench scale equipment at Westinghouse. While permeability results are not finalized, they appear to be acceptable. Filtration tests showed the filters to be of acceptable efficiency, with very smooth and uniform cake formation.

Future Work

In the near future, DLC, Westinghouse and METC will reach a decision on which of the design improvements to incorporate into a final filter design. A number of those filters will then be manufactured for corrosion and HTHP filtration testing at Westinghouse. Assuming those tests are successful, and METC exercises the optional portions of the contract, DLC will address manufacturing issues in Task 4, and begin commercial manufacturing of 50 filters in Task 5.