Contract No. DE-AC21-94MC31160--30

PARTICULATE HOT GAS STREAM CLEANUP TECHNICAL ISSUES

QUARTERLY REPORT

October 1998 - December 1998

Prepared for

UNITED STATES DEPARTMENT OF ENERGY
Federal Energy Technology Center - Morgantown
Post Office Box 880, 3610 Collins Ferry Road
Morgantown, West Virginia 26505

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Contract No. DE-AC21-94MC31160
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Abstract

This quarterly report describes technical activities performed under Contract No. DE-AC21-94MC31160. The analyses of hot gas stream cleanup (HGCU) ashes and descriptions of filter performance studied under Task 1 of this contract are designed to address problems with filter operation that are apparently linked to characteristics of the collected ash. This report includes a description of a device developed to harden a filter cake on a filter element so that the element and cake can subsequently be encapsulated in epoxy and studied in detail. This report also reviews the status of the HGCU data base of ash and char characteristics. Task 1 plans for the remainder of the project include characterization of additional samples collected during site visits to the Department of Energy / Southern Company Services Power Systems Development Facility (PSDF), encapsulation of an intact filter cake from the PSDF, and completion and delivery of the HGCU data bank.

Task 2 of this project concerns the testing and failure analyses of new and used filter elements and filter materials. Task 2 work during the past quarter consisted of hoop tensile and axial compressive stress-strain responses of McDermott ceramic composite and hoop tensile testing of Techniweave candle filters as-manufactured and after exposure to the gasification environment.

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EXECUTIVE SUMMARY

This quarterly report describes technical activities performed under Contract No. DE-AC21-94MC31160. The analyses of Hot Gas Stream Cleanup (HGCU) ashes and descriptions of filter performance studied under this contract are designed to address problems with filter operation that are apparently linked to characteristics of the collected ash. Task 1 is designed to generate a data bank of the key characteristics of ashes collected from operating advanced particle filters (APFs) and to relate these ash properties to the operation and performance of these filters and their components. APF operations have also been limited by the strength and durability of the ceramic materials that have served as barrier filters for the capture of entrained HGCU ashes. Task 2 concerns testing and failure analyses of ceramic filter elements currently used in operating APFs and the characterization and evaluation of new ceramic materials.

Task 1 research activities during the past quarter included development and testing of a device for the hardening of filter cake on the surface of a filter element prior to the impregnation of the filter cake with low-viscosity epoxy. (Experience has shown that found that without prior hardening, most filter cakes in barrier filters are too fragile to withstand the force of the surface tension of the epoxy. Instead of infiltrating the filter cake and preserving its structure, the application of the epoxy causes the cake to lose its shape and strength.) Data, photographs and discussions were added to the HGCU data bank, and refinements were made to its presentation on the computer screen and its capability for hardcopy outputs.

Task 2 work during the past quarter consisted of hoop tensile and axial compressive stress-strain responses of McDermott ceramic composite and hoop tensile testing of Techniweave candle filters as-manufactured and after exposure to the gasification environment.

INTRODUCTION

This quarterly report describes technical activities performed under Contract No. DE-AC21-94MC31160. Task 1 of this contract concerns analyses of HGCU ashes and descriptions of filter performance that are designed to address problems with filter operation linked to characteristics of the collected ash. Much of the work planned for Task 1 builds directly on work performed under a prior contract (No. DE-AC21-89MC26239) with the Department of Energy's Federal Energy Technology Center in Morgantown, WV (DOE/FETC-MGN). Task 2 of this contract includes characterization of new and used filter elements. Some of the problems observed at PFBC facilities include excessive filtering pressure drop, the formation of large, tenacious ash deposits within the filter vessel, and bent or broken candle filter elements. These problems have been attributed to ash characteristics, durability of the ceramic filter elements, and specific limitations of the filter design. In addition to the problems related to the characteristics of PFBC ashes, laboratory characterizations of gasifier and carbonizer particulates have shown that these ashes and chars also have characteristics that might negatively affect filtration. Specifically, gasifier particulates may form filter cakes that accumulate in thickness quite rapidly and also may reentrain following cleaning pulses.

To identify which particulate characteristics can lead to problems with filtration, over 350 particulate samples from fourteen facilities involved in FETC's HGCU program have been assembled. Three samples from gasification studies being carried out by Herman Research Pty. Ltd. (HRL) of Melbourne, Australia have also been included in the data bank. The most recent facility included in the data bank is the Westinghouse filter at Sierra Pacific Power Company's Piñon Pine Power Project. Many of the samples in the data bank have been analyzed with a variety of laboratory tests. Physical attributes of the particles that have been examined include size distribution, specific surface area, particle morphology, and bulk ash cohesivity and permeability. A range of chemical analyses of these samples, as well as characterizations of agglomerates of particles removed from filter vessels at Tidd, Karhula and Foster Wheeler's pilot-scale combustion facility located in Livingston, New Jersey have also been performed. The data obtained in these studies are being assembled into an interactive data bank which will help the manufacturers and operators of high-temperature barrier filters tailor their designs and operations to the specific characteristics of the particulate materials they are collecting.

Under Task 2 a wide variety of filter elements and filter materials have been evaluated. Recently, hoop tensile testing of one-inch long McDermott specimens was conducted and resulted in improper failure modes. Some two-inch long specimens were tested and the results are given in this report. For the composite materials, compressive stress-strain responses are also important. Axial compressive stress-strain responses were measured for the McDermott material and the results are included in this report. Hoop tensile strength measurements on Techniweave, as-manufactured and after gasification exposure, are given also.

OBJECTIVES

Task 1 of this project is explicitly designed to address aspects of filter operation that are linked to the characteristics of the collected particles. This task has two primary objectives. The first is the generation of an interactive computerized data bank of the key characteristics of HGCU ashes collected from operating high-temperature, high-pressure, particle filters. The data bank is structured to identify, when possible, relationships between HGCU particulate properties and the operation and performance of these filters. Construction of the data bank is intended to help manufacturers and operators of high-temperature barrier filters tailor process design and operation to the specific characteristics of the particulate materials they are collecting. The second objective is to relate these measured properties and the contents of the data bank to the operation and performance of the advanced particle filters and filter components. The first objective includes formatting the data bank and collecting, analyzing, and maintaining particulate samples from operating HGCU facilities. The second objective of this task involves the collection of operating histories from advanced particle filters, correlating these histories with sample characteristics, interpreting these correlations, and communicating results in the various venues prescribed by DOE/FETC-MGN.

The objectives of the Task 2 test program at Southern Research are as follows:

- Provide material characterization to develop an understanding of the physical, mechanical, and thermal behavior of hot gas filter materials.

- Develop a material property data base from which the behavior of materials in the hot gas cleanup environment may be predicted.

- Perform testing and analysis of filter elements after exposure to actual operating conditions to determine the effects of the thermal and chemical environments in hot gas filtration on material properties.

- Explore the glass-like nature of the matrix material.

TASK 1 ASSESSMENT OF ASH CHARACTERISTICS

Task 1 research activities during the past quarter included development and testing of a device for the hardening of filter cake on the surface of a filter element prior to the impregnation of the filter cake with low-viscosity epoxy. (Experience has shown that found that without prior hardening, most filter cakes in barrier filters are too fragile to withstand the force of the surface tension of the epoxy. Instead of infiltrating the filter cake and preserving its structure, the application of the epoxy causes the cake to lose its shape and strength.) Data, photographs and discussions were added to the HGCU data bank, and refinements were made to its presentation on the computer screen and its capability for hardcopy outputs.

DEVELOPMENT OF A TECHNIQUE FOR PRESERVATION OF FILTER CAKES

Filter cakes that have been observed on-site at the PSDF (and most of the filter cakes from other facilities) are quite fragile. Although relatively strong nodular deposits obtained at the Tidd PFBC were successfully encapsulated and preserved for analysis by the infiltration of low-viscosity epoxy, efforts to apply this epoxy to more fragile cakes has resulted in their destruction. Therefore, laboratory experiments were undertaken to develop a method to strengthen these fragile cakes prior to the introduction of the low-viscosity epoxy. A method for hardening filter cakes with cynoacrylate “super” glue vapor was developed and successfully demonstrated for a small simulated PFBC filter cake. The application of the glue vapor allowed the simulated filter cake to maintain its original structure, but with significantly increased strength. When the hardened sample was exposed to the low-viscosity epoxy, the epoxy easily impregnated the simulated cake. Based on the success of this bench-scale trial, a device was designed that could allow hardening of the bottom 12 to 16 inches of a 1.5 meter filter element and its attached filter cake. This device, which is shown schematically in Figure 1, has been constructed and tested on a filter cake present on a filter element removed from the Westinghouse FL0301 filter vessel at the PSDF on August 1, 1997. (This test was successful, but because it was conducted in late January, 1999, this test will be discussed further in the next quarterly technical report.)

The design of the device shown in Figure 1 allows the element to be suspended in an acrylic tube with its cake intact. A foam collar is positioned around the element to separate the upper portion of the element, which will not be hardened, from the bottom portion of the element, which will be hardened with cynoacrylate vapor. After purging the lower portion of the tube with dry gas, glue vapor is generated by passing dry air (or nitrogen) over an open container of liquid glue maintained at about 250 °F. (A dry carrier gas is required because the presence of water is what cures the glue.) The vapor-laden gas is passed through the filter cake on the lower portion of the candle, where a portion of the vapor is adsorbed on the surfaces of the particles in the filter cake. Vapor which is not adsorbed by the cake is transported by the carrier gas through two water baths where it is cured into fine particles and collected. This collection is necessary to protect the pump from damage from the glue. The initial trials performed with this technique have demonstrated that the glue cures almost immediately when it contacts the filter cake. This rapid curing is believed to result from the contact of the glue with hydrated water in the filter cake. The system shown in Figure 1 also contains various flow controls and an overflow line for venting in case of failure of the pump or the source of dry carrier gas.

After strengthening the cake on the lower portion of a filter element, the entire lower portion of the element (candle and cake) will gradually be impregnated with epoxy. This must be done in relatively thin increments along the candle's length (about 2 inches thick), to allow for proper curing of the epoxy. Following encapsulation, sectioning, and polishing, fully encapsulated filter cake/filter element specimens should be available for distribution and analysis. If this technique is successful for PFBC filter cake preservation, there is every expectation that it will also be effective for preserving gasification filter cakes.

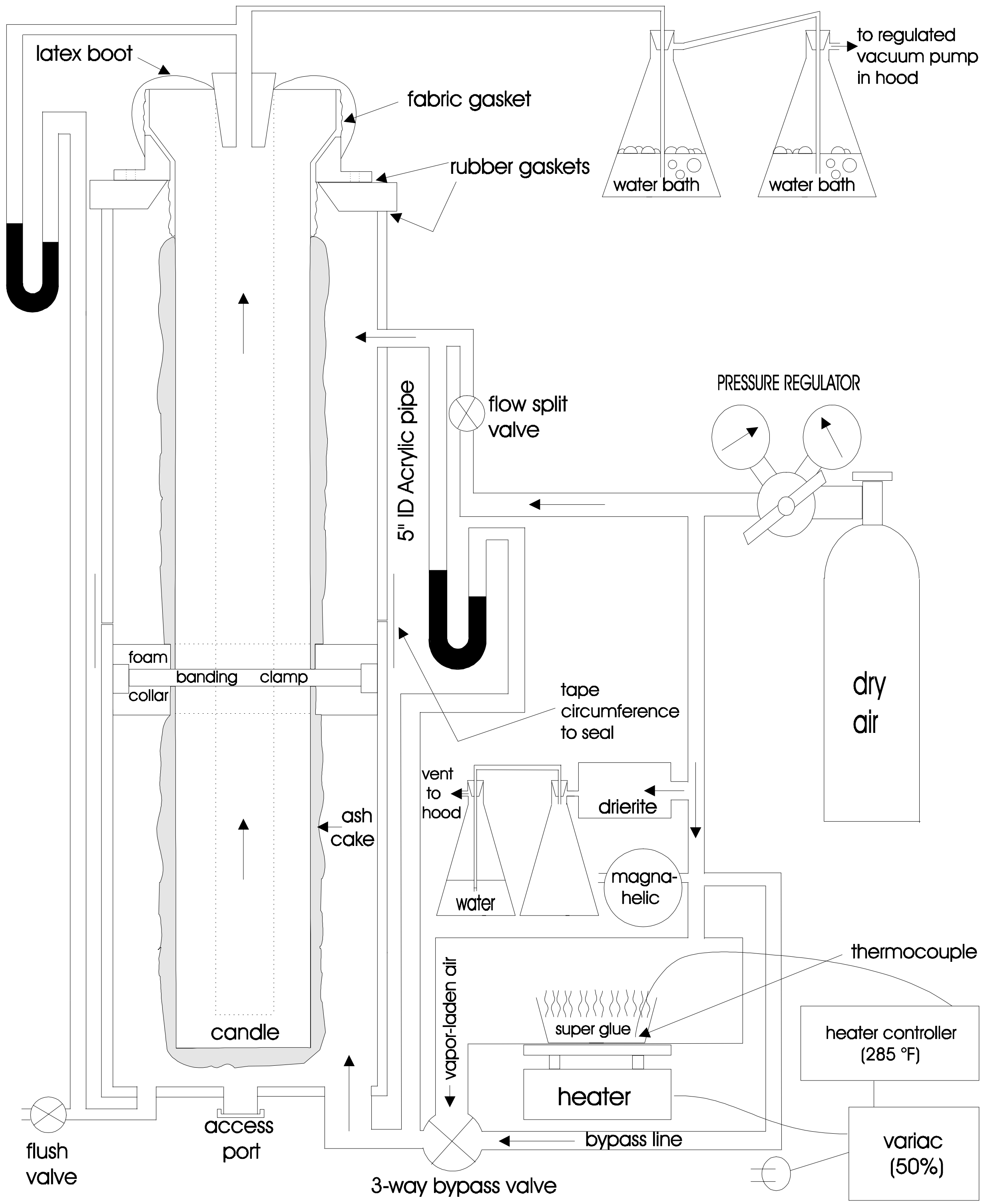

Figure 1. Schematic diagram of a device for using cynoacrylate glue to strengthen filter cakes formed on a candle filter element in preparation for subsequent impregnation and encapsulation with low-viscosity epoxy.

STATUS OF ASH DATA BANK DEVELOPMENT

Much of the work during this past quarter has focused on further development of the HGCU ash data bank. These efforts have included setting up an updatable file structure that will hold the input data for the MS-Access program to retrieve and display. The files in this structure will include text documents (in MS Word), numerical tables (in MS Excel), compressed files with combined text and graphics (in Adobe portable doument format), and photographs and schematics (in jpg, gif, or tif formats). The Access program is being configured to retrieve these files so that the data in the data bank can be easily updated, without modifying the main Access program, as more samples and information become available for inclusion.

The Access program is also being designed to allow hard-copy output of the variety of photographs, data summaries, and in-depth discussions that are being included in the data bank. Other recent work includes the compression of the files containing the SEM photomicrographs of the samples. In addition, a number of changes are being made to improve navigation between displays in the data bank. Figure 2 presents a flow chart relating the major components of the data bank. Upcoming work will include delivery of a draft version of the data bank to the DOE/FETC Program Manager for review and comments. As the data bank becomes finalized over the next several months, an instruction manual will be written describing the installation and use of the data bank. Prior to the conclusion of this project in September, 1999, a number of copies of the data bank will be produced on CD-ROM and provided to DOE/FETC for use and distribution.

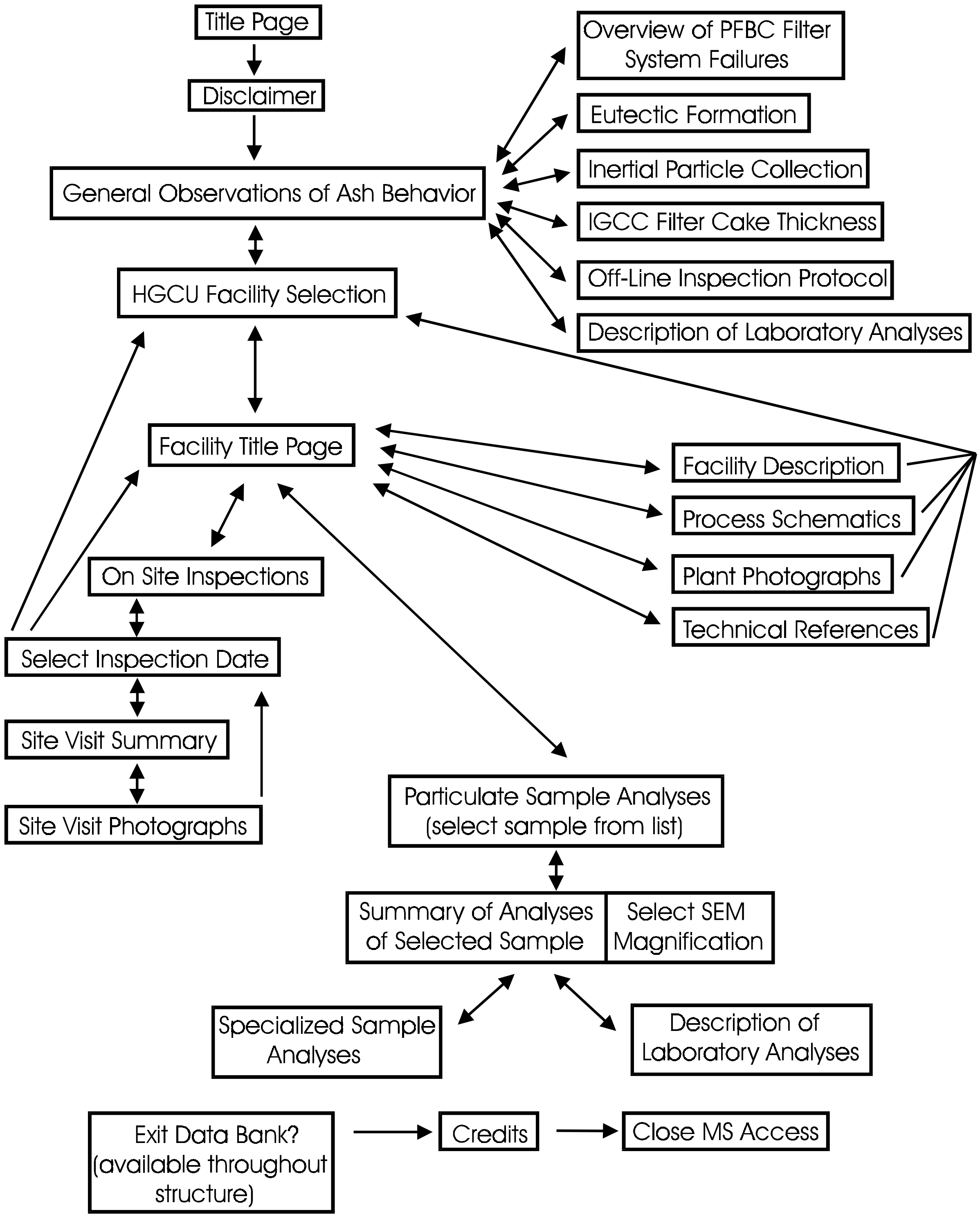

Figure 2. Structural diagram describing the navigation paths and principle components of the HGCU ash data bank.

TASK 2 FILTER MATERIAL CHARACTERIZATION

Hoop tensile stress-strain responses of McDermott ceramic composite are shown in Figure 3 and the results are summarized in Table 1. Endpoints shown in Figure 3 do not represent ultimate strengths. Specimen strain continued with little or no load increase considerably beyond the endpoints shown; however, it is unlikely that the material could function as an effective filter after these endpoints. The strength based on the initial failure points ranged from 490 psi to 760 psi and Young's modulus ranged from 1.1 msi to 1.4 msi. Axial compressive stress-strain responses are shown in Figure 4 and the results summarized in Table 2. The axial compressive strength ranged from 500 psi to 560 psi, axial Young's modulus ranged from 0.33 msi to 0.38 msi, and the axial compressive strain-to-failure ranged from 1.4 mil/inch to 1.9 mil/inch.

Hoop tensile strength measurements on two twenty-inch Techniweave candles, one virgin and one after exposure to gasification conditions, are summarized in Table 3. The strength of virgin material, denoted as Candle TW2 in Table 3, ranged from 1990 psi to 3020 psi. The strength of the material exposed to gasification conditions, denoted Gas in Table 3, ranged from 2140 psi to 3020 psi. No difference was seen after exposure to gasification conditions. Techniweave has changed material processing since these candles were manufactured. One 1.5 m virgin candle manufactured with the current processing is in testing now. These results will indicate whether the results from the twenty-inch candles is representative of current material.

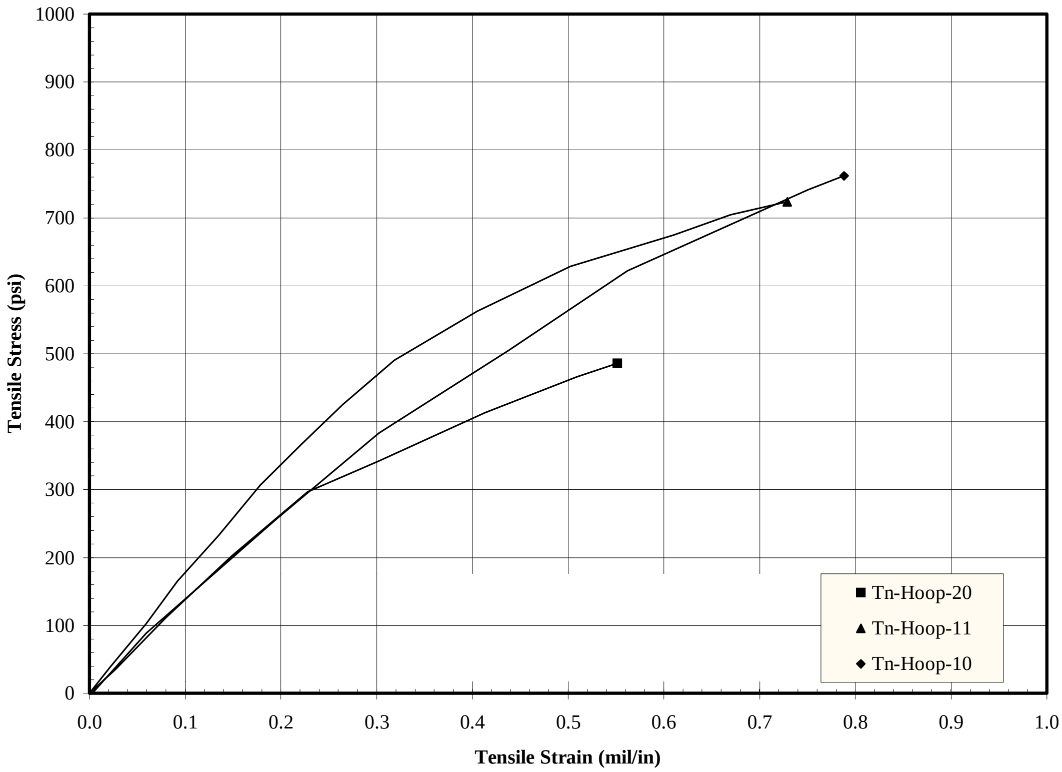

Figure 3. Hoop Tensile Stress-Strain Responses of Mc Demott Ceramic Composite

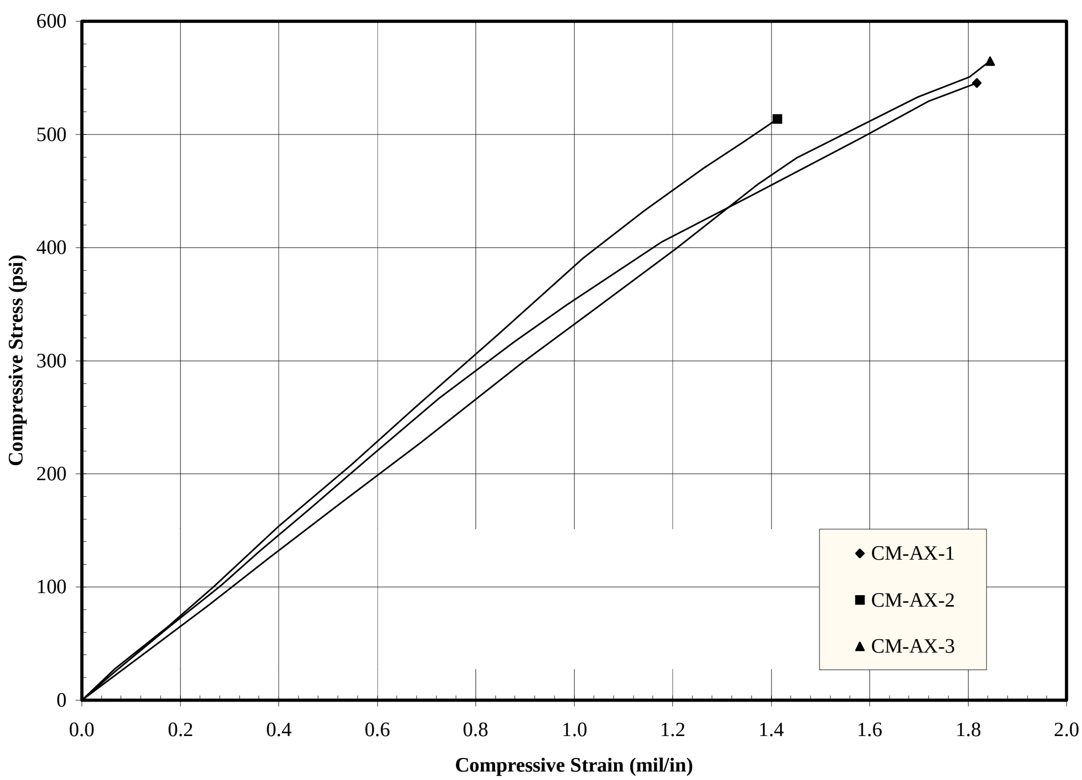

Figure 4. Axial Compressive Stress-Strain Responses of Mc Dermott Ceramic Composite

Table 1
Hoop Tensile Results for McDermott Ceramic Composite

Specimen Number	Candle	Temp. (°F)	I.D. (inch)	O.D. (inch)	Maximum Hydrostatic Pressure (psig)	Initial Failure Stress (psi)	Initial Young's Modulus (msi)
Tn-Hoop-10	8-1-23	RT	1.95	2.36	140	760	1.1
Tn-Hoop-11	8-1-30-1	RT	1.92	2.35	140	720	1.4
Tn-Hoop-12	8-1-30-1	RT	1.93	2.39	140	650	strain signal erratic
Tn-Hoop-20	8-1-30-1	RT	1.93	2.39	100	490	1.1

Table 2
Axial Compressive Results for McDermott Ceramic Composite

Specimen Number	Candle	Temp. (°F)	I.D. (inch)	O.D. (inch)	Initial Failure Stress (psi)	Initial Young's Modulus (msi)	Strain at Initial Failure (mil/inch)
Cm-Ax-1	8-1-30-1	RT	1.94	2.35	550	0.37	1.8
Cm-Ax-2	8-1-30-1	RT	1.94	2.35	500	0.38	1.4
Cm-Ax-3	8-1-30-1	RT	1.93	2.37	560	0.33	1.9

Table 3
Hoop Tensile Results for Techniweave Filters

Filter Identification	Specimen Number	Test Temperature (°F)	Specimen I.D. (in.)	Specimen O.D. (in.)	Isotropic Ultimate Tensile Strength (psi)
TW2	tn-h-1	RT	2.10	2.38	1988
TW2	tn-h-2	RT	2.12	2.41	2172
TW2	tn-h-3	RT	2.12	2.41	1792
TW2	tn-h-4	RT	2.11	2.40	2424
TW2	tn-h-5	RT	2.11	2.41	2589
TW2	tn-h-6	RT	2.11	2.41	3020
Gas	tn-h-7	RT	2.12	2.40	3022
Gas	tn-h-8	RT	2.12	2.43	2799
Gas	tn-h-9	RT	2.09	2.42	2235
Gas	tn-h-10	RT	2.13	2.40	2229
Gas	tn-h-11	RT	2.12	2.40	2139
Gas	tn-h-12	RT	2.13	2.40	2807

Table 4
Test Matrix for As-Manufactured
ABB Material

		Replications at Temp. (°F)	
Test	Orientation	RT	1400
Tn	Hoop	6	
Cm	Axial	5	5
TE	Axial	2---'> to approx. 1800 °F	
K	Radial	2---'> to approx. 1500 °F	
Cp		2---'> to approx. 1000 °F	
Light Mic.		Yes	

Legend:
Tn - Tensile
Cm - Compression
TE - Thermal Expansion
K - Thermal Conductivity
Light Mic. - Light Microscopy

Table 5
Test Matrix for PFBC Exposed
ABB Material

		Replications at Temp. (°F)	
Test	Orientation	RT	1400
Tn	Hoop	6	
Cm	Axial	5	5
Light Mic.		Yes	

Legend:
Tn - Tensile
Cm - Compression
Light Mic. - Light Microscopy

FUTURE WORK

Efforts under Task 1 during the next quarter will include analysis of particulate samples from Piñon Pine. A site visit to the PSDF for filter inspection and sampling occurred on January 26, 1999. Analyses of data and samples obtained on that visit will be included in the next technical report. Also during that site visit, a complete filter element was removed intact with its attached filter cake for hardening and encapsulation. The preservation of this filter element and its filter cake will be discussed in the next quarterly report. Improvements and modifications to the HGCU data bank will continue during the next quarter following review of a draft version of the data bank by the DOE/FETC Program Manager. In the upcoming quarter under Task 2, two “soft” candles supplied by ABB will be tested according to the test matrices shown in Tables 4 and 5. One of the candles is virgin and the other was exposed to simulated PBFC conditions. Hoop tensile testing of Schumacher TF20 with 1-inch long specimens, 3-inch long specimens, 6-inch long specimens, and 10-inch long specimens is in progress to evaluate machining effects, if any. One virgin Techniweave candle is also to be tested and the test matrix is currently being developed.

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