DE-AC21-94MC31160--2

Particulate Hot Gas Stream Cleanup Technical Issues

Quarterly Report
January 1 - March 31, 1997

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EXECUTIVE SUMMARY

This is the tenth in a series of quarterly reports describing the activities performed under Contract No. DE-AC21-94MC31160. Analyses of Hot Gas Stream Cleanup (HGCU) ashes and descriptions of filter performance address aspects of filter operation that are apparently linked to the characteristics of the collected ash or the performance of the ceramic barrier filter elements. Task 1 is designed to generate a data base 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. Task 2 concerns testing and failure analysis of ceramic filter elements.

Under Task 1 during the past quarter, analyses were performed on a particulate sample from the Transport Reactor Demonstration Unit (TRDU) located at the University of North Dakota Energy and Environmental Research Center. Analyses are in progress on ash samples from the Advanced Particulate Filter (APF) at the Pressurized Fluidized-Bed Combustor (PFBC) that was in operation at Tidd and ash samples from the Pressurized Circulating Fluid Bed (PCFB) system located at Karhula, Finland. A site visit was made to the Power Systems Development Facility (PSDF) to collect ash samples from the filter vessel and to document the condition of the filter vessel with still photographs and videotape. Particulate samples obtained during this visit are currently being analyzed for entry into the Hot Gas Cleanup (HGCU) data base. Preparations are being made for a review meeting on ash bridging to be held at Department of Energy Federal Energy Technology Center - Morgantown (DOE/FETC-MGN) in the near future.

Most work on Task 2 was on hold pending receipt of additional funds; however, creep testing of Schumacher FT20 continued. The creep tests on Schumacher FT20 specimens just recently ended and data analysis and comparisons to other data are ongoing. A summary and analysis of these creep results will be sent out shortly. Creep testing of two Refractron 326 specimens is now in progress. Among the tasks expected to be completed this quarter are analysis of the creep data obtained thus far, microstructural analysis of Refractron 326 and Schumacher FT20, definition of bending loads on candle filters, and characterization of additional candle filters from Karhula.

INTRODUCTION

This is the tenth quarterly report describing the 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 the problems with filter operation linked to the characteristics of the collected ash. 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 ashes have shown that these ashes also have characteristics that might negatively affect filtration. Problems with the durability of the filter elements are being addressed by the development and evaluation of elements constructed from alternative ceramic materials.

To identify which ash characteristics can lead to problems with filtration, over 250 particulate samples from thirteen facilities involved in FETC's HGCU program have been assembled. Samples from gasification studies being carried out by Herman Research Pty Ltd. (HRL) of Melbourne, Australia have also been included in the data base. Many of these samples 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 on 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 base 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.

In order to understand the thermal and mechanical behavior of the various types of ceramic materials used in hot gas filtration, hoop and axial tensile tests, thermal expansion, compression, and creep evaluations of these materials at temperatures up to 1800 °F have been performed. Nondestructive testing methods performed on filter specimens include density and ultrasonic velocity. To date various characteristics of Dupont/Lanxide PRD-66, Dupont composite, 3M composite, IF&P Fibrosics, Refractron, Schumacher, and Blasch ceramic materials have been evaluated.

Task 1 has two primary objectives. The first is to generate a readily accessible data base of the key characteristics of ashes collected from operating advanced particle filters. The second objective is to relate these ash properties and the contents of the data base to the operation and performance of the advanced particle filters and filter components. The first objective includes formatting the data base and collecting, analyzing, and maintaining ashes from operating HGCU facilities. The second objective of this task involves the collection of operating histories from advanced particle filters, correlating these histories with ash characteristics, interpreting these correlations, and communicating results in the various venues prescribed by DOE/FETC-MGN.

The objective of Task 2 is to develop an overall understanding of the thermal and mechanical behavior of hot gas filter materials. This objective includes the creation of a materials property data base which will allow the prediction of the behavior of these materials in hot gas cleanup environments. Pertinent tests will be carried out on specimens of unused filter material and also on filter elements that have been exposed in actual operating environments. Nondestructive test techniques will be applied to filter elements to characterize the strength and durability of these elements without rendering them unusable. This task will also evaluate the adequacy and completeness of manufacturers' quality assurance/quality control plans for manufactured filter elements.

TASK 1 ASSESSMENT OF ASH CHARACTERISTICS

Analyses were performed on a particulate sample from the TRDU located at the University of North Dakota Energy and Environmental Research Center. Analyses are in progress on ash samples from the Tidd PFBC and the Karhula PCFB. A site visit was made to the PSDF to collect ash samples from the filter vessel and to document the condition of the filter vessel with still photographs and videotape. Particulate samples obtained during this visit are currently being analyzed for entry into the HGCU data base. The samples characterized during the past quarter are briefly described in Table 1.

Table 1
Particulate Samples Characterized during the Past Quarter

ID #	Source	Brief description
4199	UNDEERC TRDU	PO50 filter vessel hopper ash (1/14/97)
4231	PSDF	filter cake ash (4/9/97)
4087	Tidd PFBC	ash from deposit under tubesheet (5/5/94)
4088	Tidd PFBC	ash from deposit under tubesheet (5/5/94)
4143	Tidd PFBC	filter cake ash (5/11/95)
4144	Tidd PFBC	filter cake ash (5/11/95)
4067	Karhula PCFB	filter cake ash (2/94)
4182	Karhula PCFB	filter cake ash (received 6/96)

The results of the various physical and chemical analyses performed on the samples listed in Table 1 are presented in Tables 2 and 3. (Some of these results have been presented previously and are included to support the discussions presented in this report.) Complete size distributions measured for sample # 4199 and sample # 4231 are given in Figures 1 and 2 and representative scanning electron micrographs of sample # 4199 are presented in Figure 3.

Table 2
Physical Characteristics of Tidd, Karhula, TRDU and PSDF Ashes

quantity	4144	4087	4182	4199	4231
specific surface area, m^2/g	--	--	1.2	105.3	3.80
Stokes' MMD, mm	11	4.9	9.9	2.4	4.7
uncompacted bulk porosity, %	84	91	81	89.3	--
filter cake porosity, %	--	--	--	--	87
drag-equivalent diameter, mm	--	--	2.22	0.719	--
specific gas flow resistance, in H_2O·min·ft/lb*	--	--	1.5	8.5	--
true particle density, g/cm^3	--	2.72	2.83	2.26	2.61

* calculated for an assumed filter cake porosity equal to the uncompacted bulk porosity

Table 3
Chemical Composition of Tidd, Karhula, and TRDU Ashes,% wt.

Constituent ID #	4087	4144	4182	4199
LiO_2	0.01	0.01	0.01	0.01
Na_2O	0.34	0.29	0.87	0.90
K_2O	1.6	1.3	1.6	0.18
MgO	8.7	8.3	0.72	11.0
CaO	13.2	14.1	17.4	44.0
Fe_2O_3	5.3	7.1	11	9.0
Al_2O_3	12.2	11.7	12.6	15.1
SiO_2	24.3	26.1	34.4	12.9
TiO_2	0.5	1.2	0.6	1.7
P_2O_5	0.08	0.15	0.1	1.0
SO_3	32	30.1	19.8	1.9
LOI	5.1	13.5	0.22	54.2
soluble $SO_4^=$	39.1	29.7	23.8	0.98
Equilibrium pH*	5.8	--	7.4	10.87

* dimensionless

Analyses of Tidd and Karhula Ashes

Ash bridging in high temperature filters collecting PFBC ashes has been linked to the formation in the filter vessels of ash nodules with high inherent strength. Nodules with this characteristic have been collected from several PFBC filters, including Karhula and Tidd (where ash bridging has been well documented). To determine whether nodule formation could be simulated in the laboratory, an experiment was performed to see if baking uncompacted beds of Tidd and Karhula ashes at 1600 °F could induce the beds to consolidate and strengthen. For each of the four samples evaluated in this test, two uncompacted beds of sifted ash were placed in a laboratory muffle furnace. The first of these two sample beds was prepared by simply sifting (through a 60-mesh screen) the ash into an open ceramic cup and scraping off the excess ash so that any change in the volume of the ash in the cup could be easily determined. The second sample bed was prepared in the same manner, except that the ash was thoroughly ground with a mortar and pestle before being sifted into the open cup. This grinding was intended to break apart as many of the particle-to-particle bonds as possible before exposing the sample to the 1600 °F environment in the muffle furnace. The samples used for this experiment were ID #'s 4088, 4143, 4067, and 4182.

After each of the samples was sifted and loaded into an open cup, the cups were baked at 1600 °F for 72 hours. The Tidd ashes consolidated slightly (about 3 % loss in volume for the filter cake ash and about 10 % loss in volume for the ash from the tubesheet deposit). The Karhula ashes did not measurably decrease in volume. The ashes were then baked for an additional 168 hours, but no additional losses in volume were observed for any of the samples.

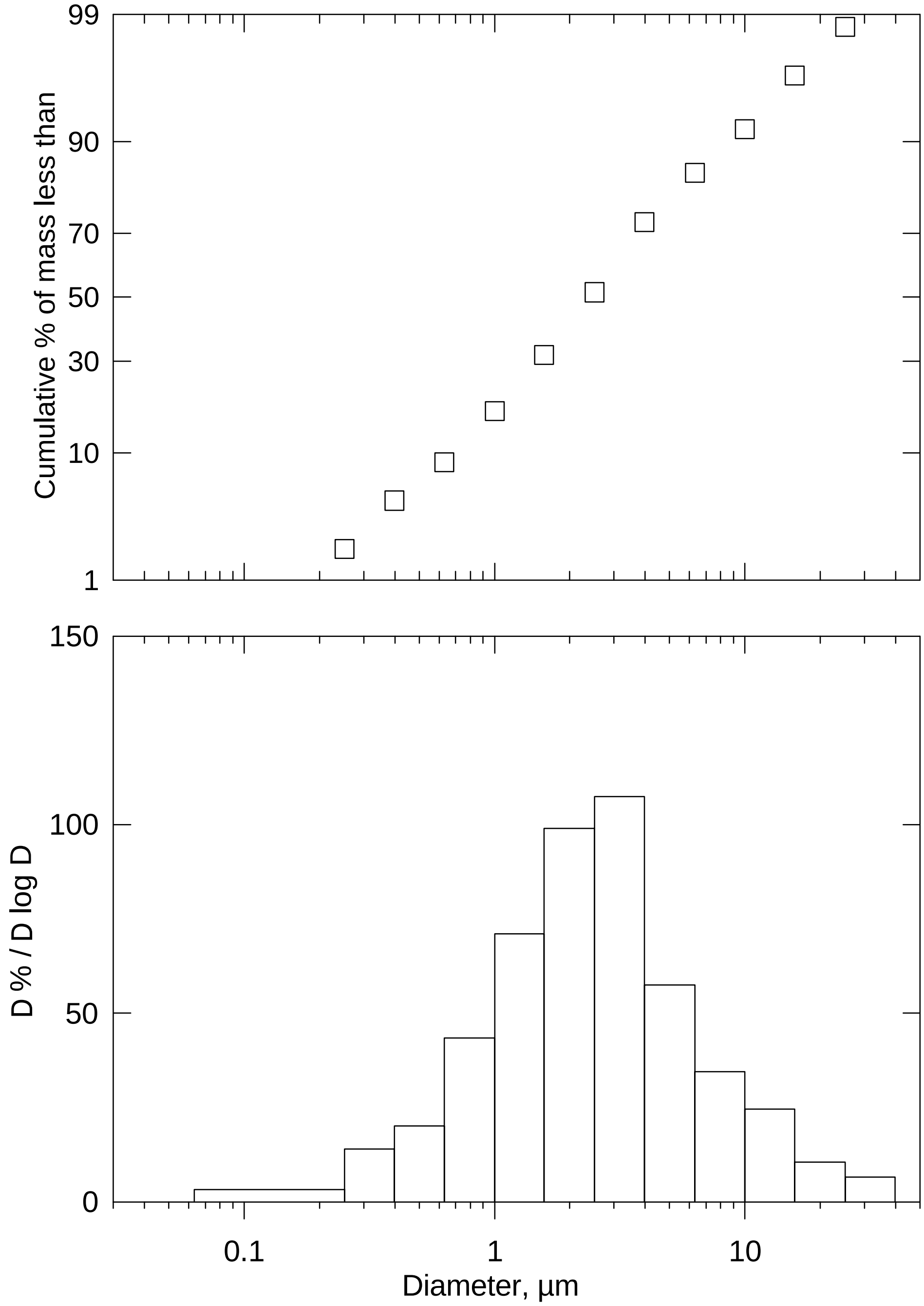


Figure 1. Cumulative and differential size distribution data measured for UNDEERC TRDU PO50 filter vessel ash (ID # 4199) measured with a Shimadzu SA-CP4 Centrifugal Particle Size Analyzer. The Stokes MMD of this distribution is 2.4 mm, and its geometric standard deviation is 2.6. The D_{16} of this distribution is 0.93 mm, and its D_{84} is 6.1 mm. (This size distribution data includes the assumption that the sample contains no particles smaller than 0.063 mm.)

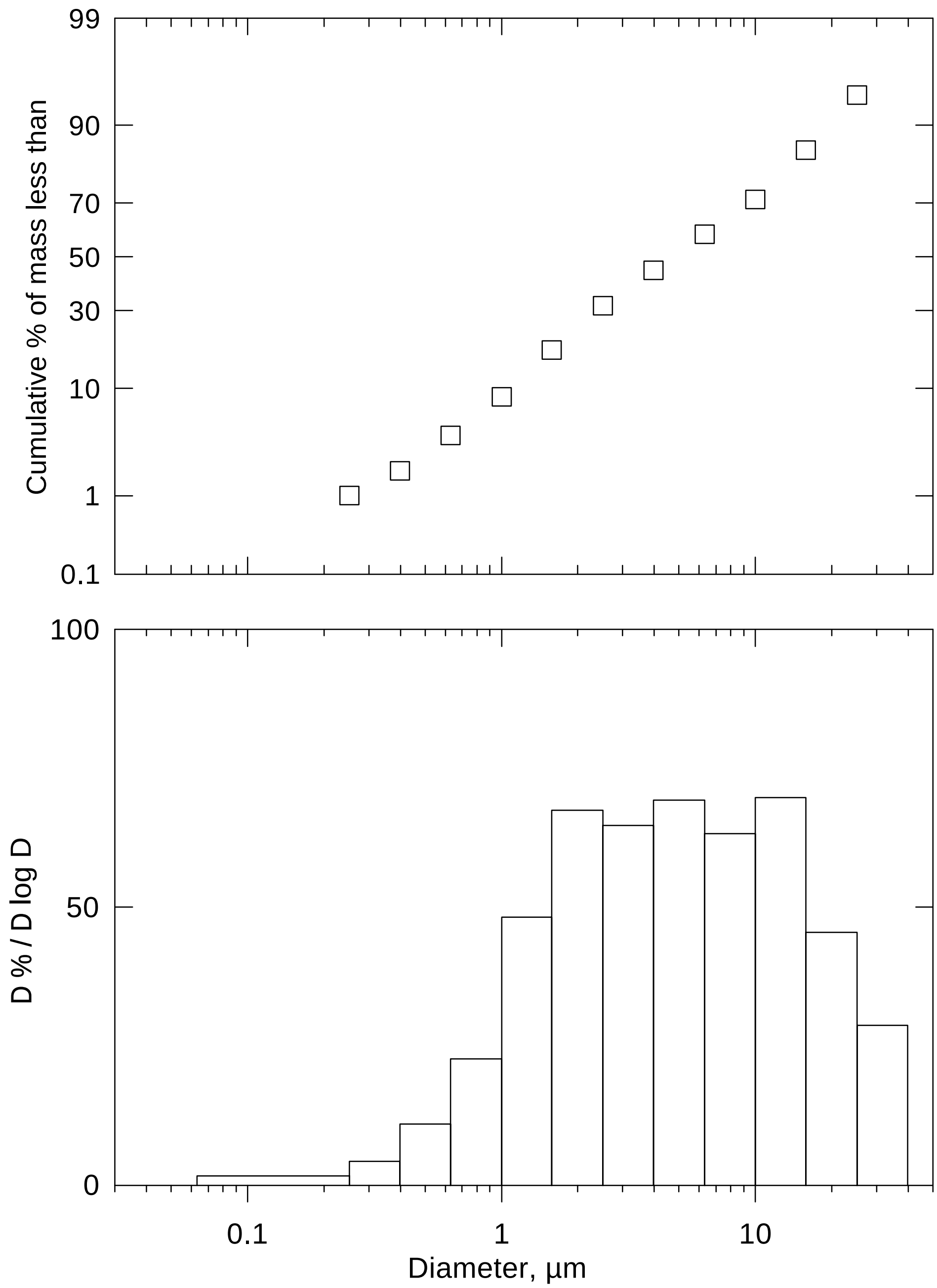
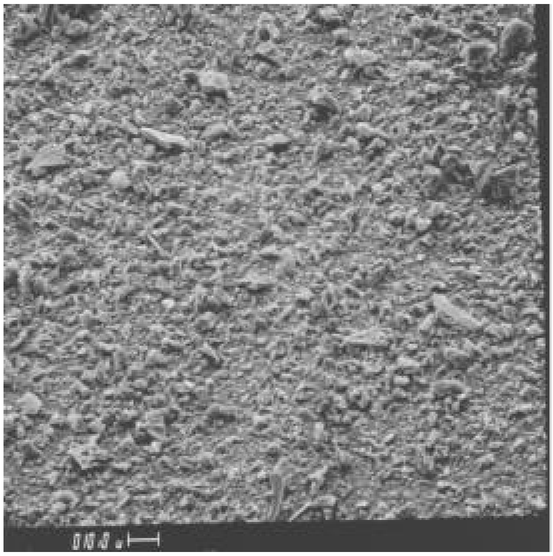


Figure 2. Cumulative and differential size distribution data measured for PSDF filter cake ash (ID # 4231) measured with a Shimadzu SA-CP4 Centrifugal Particle Size Analyzer. The Stokes MMD of this distribution is 4.7 μm, and its geometric standard deviation is 3.3. The D_{16} of this distribution is 1.4 μm, and its D_{84} is 15 μm. (This size distribution data includes the assumption that the sample contains no particles smaller than 0.063 μm.)



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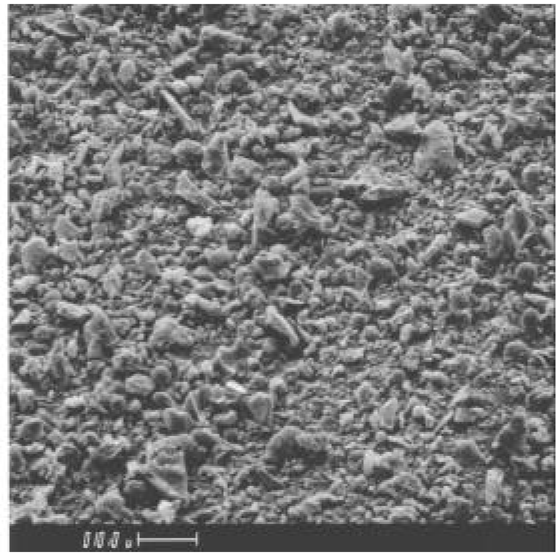

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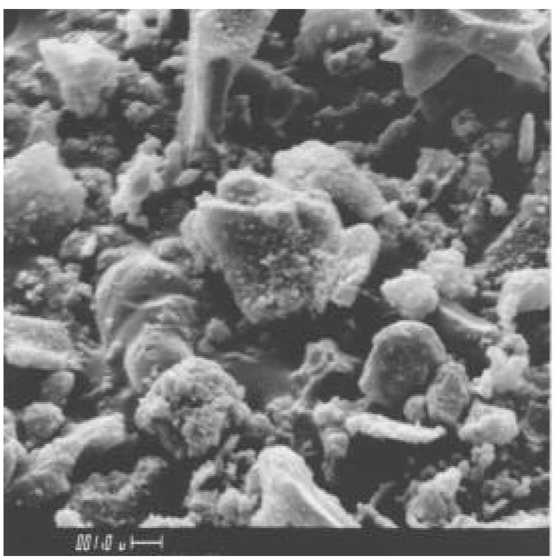

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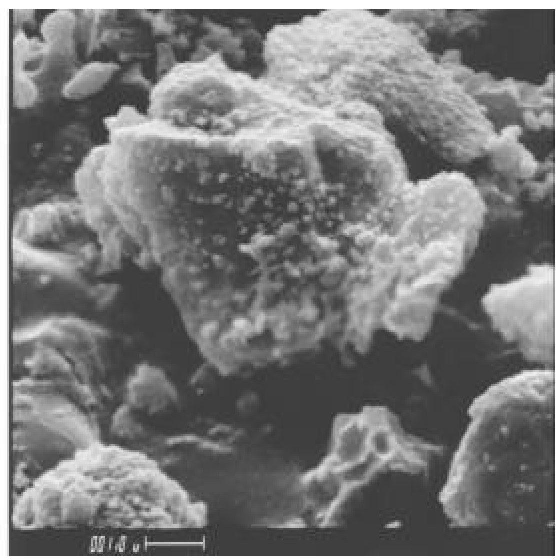

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Figure 3. Representative SEM photographs of UNDEERC TRDU PO50 filter vessel ash (ID # 4199) taken at a) 500X, b) 1000X, c) 5000X and d) 10000X.

In each case, the samples ground with the mortar and pestle behaved just like the unground ash samples. Additional baking tests may be performed on these ashes to determine if pressurizing the environment in the oven may induce greater consolidation than these atmospheric pressure tests. Pressurizing the samples during baking will increase the partial pressures of all gaseous species in contact with the particles. Literature describing boiler fouling has indicated that the availability of oxygen may influence the rate of liquefaction of particle surfaces. (Other factors believed to influence boiler tube fouling are the concentrations of iron, sodium, and calcium in the ash particles.)

Another test is in progress to compare the high-temperature behavior of these ashes. The ash deformation test commonly run on ashed coal samples during standard analyses of coal samples to determine their propensity to foul boiler tubes will soon be performed on these samples. Although this test subjects samples to temperatures up to 2800 °F, (much higher than high-temperature, high-pressure (HTHP) filters ever experience), the temperatures where these ash samples begin to soften may provide a ranking of their tendency to soften and potentially create viscous liquid or near liquid layers on their surfaces in HTHP filter vessels. The results of these tests will be presented in the next quarterly report.

Analyses of TRDU Ash

Sample # 4198 is similar to several other gasification samples that have been analyzed under this project. This TRDU sample is relatively fine (Figure 1), has a high uncompacted bulk porosity, and is dark black. Additionally, the sample has a relatively high specific surface area, a low drag-equivalent diameter, and a high uncompacted bulk porosity (Table 2). The specific gas flow resistance of this sample (calculated from drag-equivalent diameter and the assumption that filter cake porosity equals uncompacted bulk porosity) is not abnormally high. However, as with several other gasification samples that have been evaluated, the specific gas flow resistance of this sample could be much higher than the calculated value if the actual filter cake porosity is much lower than the measured uncompacted bulk porosity value of 89 %.

Site Visit to the PSDF and PSDF Ash Analyses

A site visit was made to the PSDF in April, 1997 to observe, document and characterize the filter cakes present on the candle filter elements and other filter vessel deposits when the filter vessel was opened for refitting. All of the candle filter elements were covered with patchy, thin cakes. The thickness of the filter cakes ranged from about 0.1 to 1 mm. (Because the filter was extensively pulse-cleaned prior to shutdown, the thickness of these cakes does not necessarily represent the condition of the cakes during operation.) Analyses of this filter cake ash (ID # 4231) is only partially complete, but preliminary indications are that filter cakes formed from this ash should not exhibit extraordinarily high filtering pressure drops. This statement is based on the relatively low specific surface area of this ash (3.80 m^2/g), even though the ash has a relatively fine size distribution (MMD = 4.7 mm, see Figure 2). Filter cake porosity was measured for nodules from the PSDF filter cake by impregnating weighed filter cake nodules with ethanol. This method yielded an average porosity of 87 %. Further interpretations of the filtration characteristics of this ash will be made once analyses are complete. These discussions will be included in the next quarterly report.

TASK 2 FILTER MATERIAL CHARACTERIZATION

Most work was on hold pending receipt of additional funds; however, creep testing of Schumacher FT20 continued. Specimen Creep-ax-4 broke while the heaters were being repaired after ~1100 hrs. at 250 psi and 1600 °F. Specimen Creep-ax-5 failed after ~730 hrs. at 250 psi. The creep tests on Schumacher FT20 specimens just recently ended and data analysis and comparisons to other data are ongoing. A summary and analysis of these creep results will be sent out shortly. Creep testing of two Refractron 326 specimens is now in progress. One is at 1600 °F, 500 psi, the other is at 1800 °F, 250 psi.

Additional funding has now been received and work will resume. Among the tasks expected to be completed this quarter are analysis of the creep data obtained thus far, microstructural analysis of Refractron 326 and Schumacher FT20, definition of bending loads on candle filters, and characterization of additional candle filters from Karhula.

FUTURE WORK

Plans for the next quarter include continued construction of the HGCU data base and entry of additional data, photographs, and text. Analyses of the samples from the PSDF mentioned in this report will be completed during the next quarter. Preparations are being made for a review meeting on ash bridging to be held at DOE/FETC-MGN in the near future. Other work during the next quarter will include preparation of a paper and poster for the Advanced Coal-Based Power and Environmental Systems ’97 Conference to be held in Pittsburgh in July.

PARTICULATE HOT GAS STREAM CLEANUP TECHNICAL ISSUES

QUARTERLY REPORT
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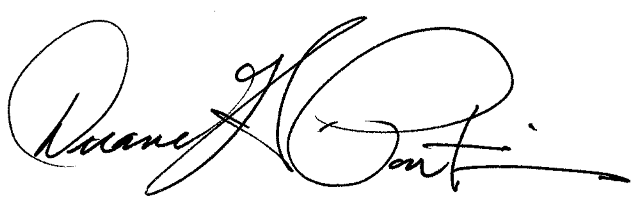

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