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Particulate Hot Gas Stream Cleanup Technical Issues

Quarterly Report

April 1 - June 30, 1996

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For
U.S. Department of Energy
Office of Fossil Energy
Morgantown Energy Technology Center
P.O. Box 880
Morgantown, West Virginia 26507-0880

By
Southern Research Institute
2000 Ninth Avenue South
Post Office Box 55305
Birmingham, Alabama 35255-5305

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EXECUTIVE SUMMARY

This is the seventh in a series of quarterly reports describing the activities performed under Contract No. DE-AC21-94MC31160. Our 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, we received and analyzed a hopper ash sample from the Transport Reactor Demonstration Unit (TRDU) located at the University of North Dakota's Energy and Environmental Research Center (UNDEERC). We also received six ash samples from the Ahlstrom 10 MWt Pressurized Fluidized Circulating Fluid Bed (PCFB) facility located at Karhula, Finland. We selected one of the filter cake ashes from this batch of samples for detailed analyses. We continued our work on the HGCU data base we are constructing in Microsoft Access®. We have been entering a variety of information into the data base, including numerical values, short or long text entries, and photographs. In addition to these activities, we prepared a paper and a poster summarizing our recent work for the 1996 DOE/METC Contractor's Conference. In addition, we have prepared a manuscript entitled *Particle Characteristics and High-Temperature Filtration* for publication and presentation at the Thirteenth Annual International Pittsburgh Coal Conference to be held in September of this year.

Task 2 efforts during the past quarter focused on hoop tensile testing of Schumacher FT20 and Refractron 326 candle filter elements removed from the Karhula APF after ~540 hours of service.

INTRODUCTION

This is the seventh 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, our 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, we have assembled 242 ash samples from twelve facilities involved in METC's HGCU program. We have analyzed many of these ashes with a variety of laboratory tests. Physical attributes of the particles that we have examined include size distribution, specific surface area, particle morphology, and bulk ash cohesivity and permeability. We have also performed a range of chemical analyses on these ashes, as well as characterizations of agglomerates of ash removed from filter vessels at Tidd, Karhula and Foster Wheeler's pilot-scale combustion facility located in Livingston, New Jersey. We are in the process of assembling the data obtained in these studies 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 ashes they are collecting.

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

OBJECTIVES

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 our conclusions in the various venues prescribed by the U.S. Department of Energy's Morgantown Energy Technology Center (DOE/METC).

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 RESEARCH ACTIVITIES

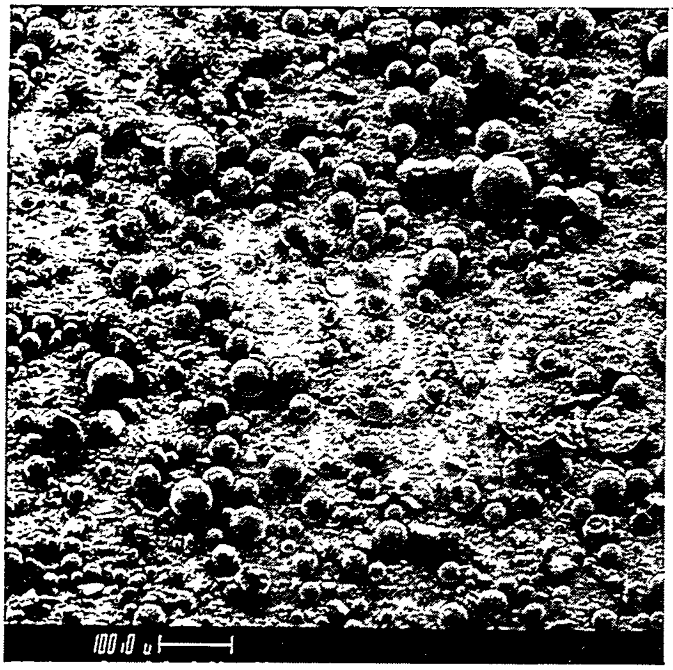
During the past quarter we received the samples described in Table 1 and performed detailed analyses of the UNDEERC TRDU filter hopper ash (ID # 4176) and the middle plenum filter cake ash from the Karhula PCFB (ID # 4182). We also continued development and data entry for our HGCU data base in Microsoft Access®.

Table 1
Ash Samples Received during the Past Quarter and Added to the HGCU Data Base

ID #	Source	Brief description
4176	UNDEERC TRDU	P047 filter hopper ash (4/18-20/1996)
4181	Karhula PCFB	filter cake ash with nodules from top plenum
4182	Karhula PCFB	filter cake ash with nodules from middle plenum
4183	Karhula PCFB	filter cake ash with nodules from bottom plenum
4184	Karhula PCFB	ash from filter vessel hopper
4185	Karhula PCFB	fly ash
4186	Karhula PCFB	bottom ash

ANALYSES OF UNDEERC TRDU ASH

As part of the preparation for evaluation of the M.W. Kellogg transport reactor at the Power Systems Development Facility, UNDEERC is in the process of evaluating the TRDU, an intermediate-scale transport reactor, at their in-house combustion laboratory. We received a hopper ash sample from tests conducted at the TRDU in mid-April. The analyses we performed on this sample are summarized in Tables 2 and 3. These data, in combination with observations we made in handling the sample and SEM photographs of the TRDU ash particles (Figure 1), indicate that this sample has a high bulk density, is medium gray, and has a very coarse size distribution. Although these characteristics were unexpected, they agree with quantitative evaluations we performed.



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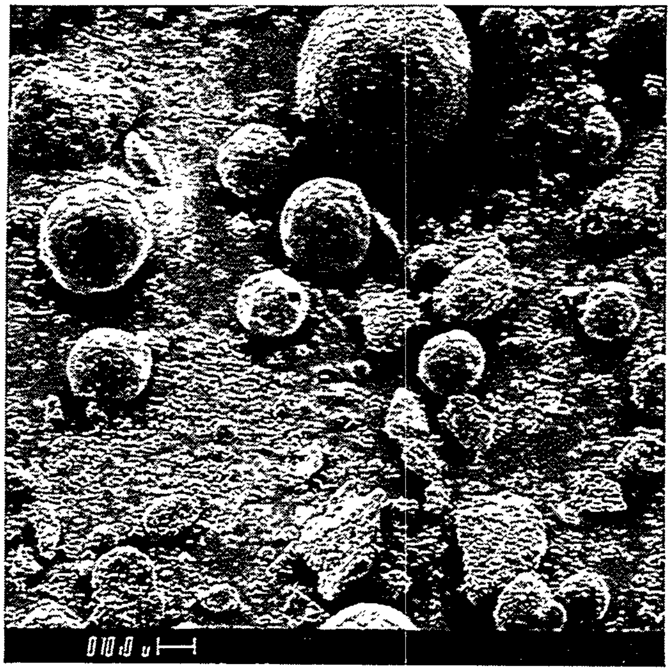

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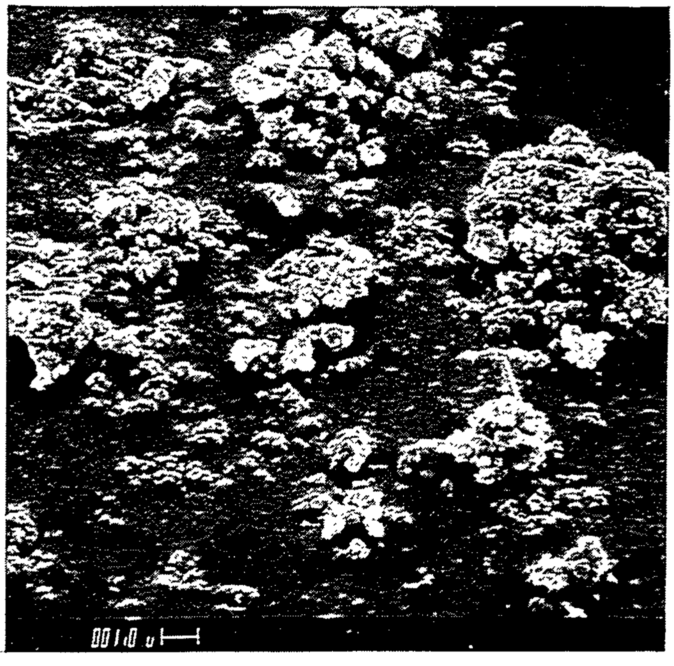

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Figure 1. SEM photographs of hopper ash from the UNDEERC TRDU (ID # 4176) taken at a) 100X, b) 500X, and c) 5000X.

Table 2
Chemical Analyses of UNDEERC TRDU and Karhula PCFB Ashes, % wt.

source / constituent ID #	UNDEERC TRDU 4176	Karhula PCFB 4182
Li_2O	0.01	0.01
Na_2O	0.63	0.87
K_2O	0.12	1.6
MgO	5.2	0.72
CaO	11.2	17.4
Fe_2O_3	1.9	11.0
Al_2O_3	22.9	12.6
SiO_2	54.8	34.4
TiO_2	0.8	0.6
P_2O_5	0.55	0.10
SO_3	0.96	19.8
LOI	4.3	0.22
soluble $SO_4^{=}$	--	23.8
Equilibrium pH*	--	7.4

* Equilibrium pH is dimensionless.

Table 3
Physical Analyses of UNDEERC TRDU and Karhula PCFB Ashes

source / quantity ID #	UNDEERC TRDU 4176	Karhula PCFB 4182
specific surface area, m^2/g	51	1.2
Stokes' MMD, µm	60	9.9
uncompacted bulk porosity, %	72	81
drag-equivalent diameter, µm	2.77	2.22
specific gas-flow resistance, in H_2O·min·ft/lb	2.8	1.5
tensile strength, N/m^2	2.8	3.8
true particle density, g/cm^3	2.60	2.83
filter cake nodule porosity, %		75

Because of the coarse size of this ash, we used our Bahco aerodynamic classifier to measure its size distribution. This procedure physically separates the ash into nine portions, depending on particle size. All nine fractions were about the same color gray, except the coarsest fraction (> 26 µm), which contained a fairly high percentage of large black particles. The size distribution of this TRDU ash is presented in Figure 2.

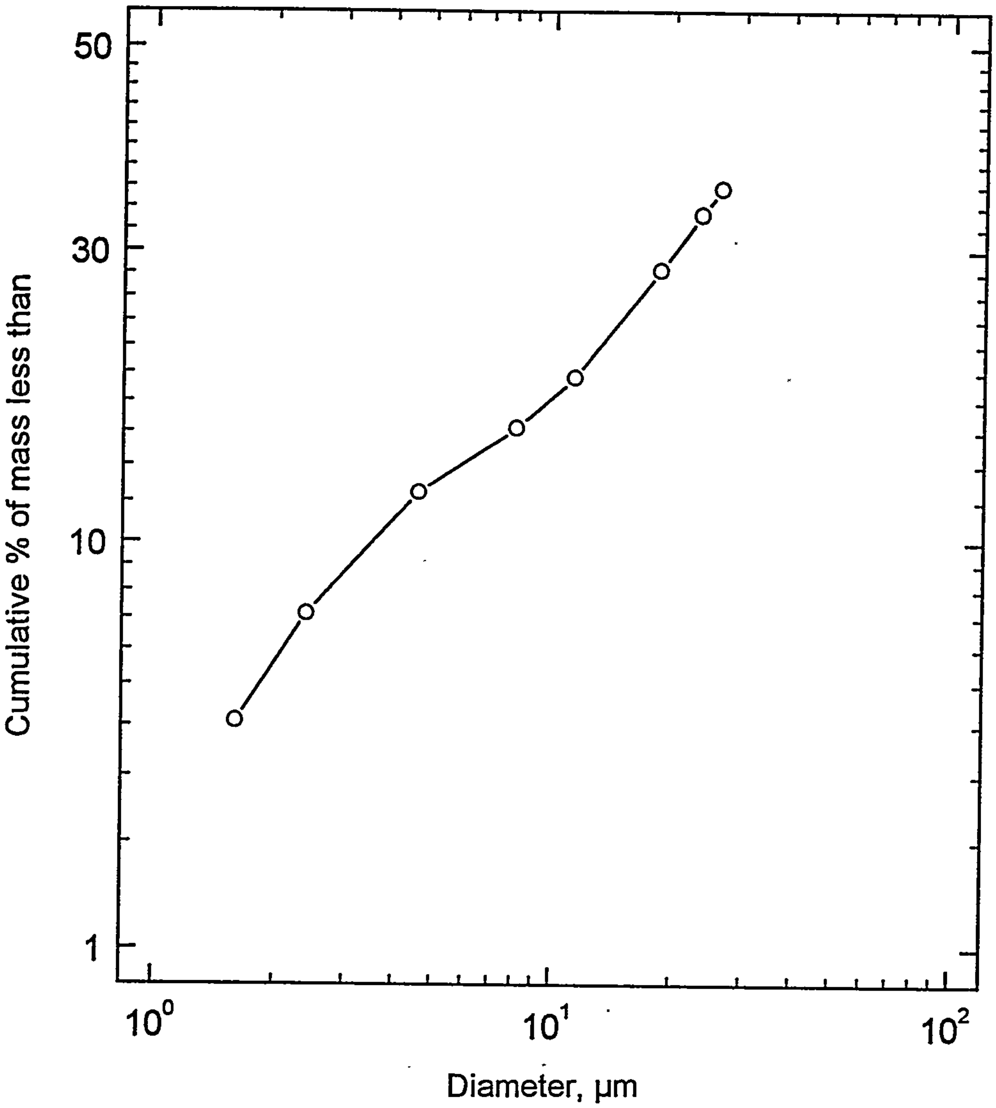


Figure 2. Cumulative size distribution of hopper ash from the UNDEERC TRDU (ID # 4176) measured with a Bahco aerodynamic classifier.

The specific surface area of this ash is quite high, although ashes with coarse size distributions generally have relatively low specific surface areas. The uncompacted bulk porosity and tensile strength of this ash indicate it is free flowing and has little cohesive strength, as would be expected for such a coarse size distribution. As with most other ashes with coarse size distributions, this ash exhibited a large drag-equivalent diameter and a low specific gas-flow resistance.

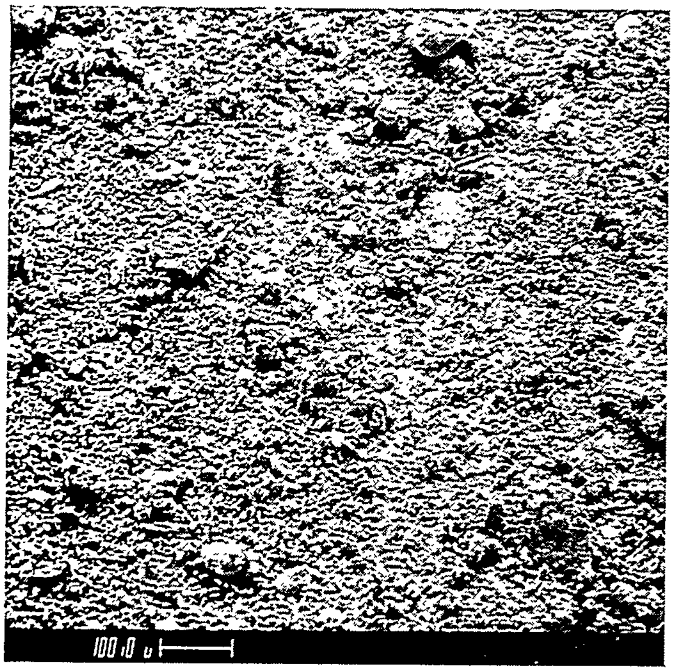
After we completed our analyses of this ash, we received limited information describing the conditions under which it was produced. Apparently the material in the TRDU standpipe overflowed causing some relatively large particles to enter the filter vessel. Although these particles may not have reached the filter cake, they did eventually collect in the filter hopper from which the ash sample we analyzed was obtained. The SEM photographs in Figure 1 clearly show large, spherical particles present in this sample. Because the presence of a high proportion of relatively large particles in an ash sample will significantly alter its bulk characteristics, the results of our analyses of the UNDEERC TRDU ash sample (ID # 4176) probably do not accurately reflect the characteristics of TRDU filter cake ash. We have requested more representative TRDU ash samples from Michael Swanson of UNDEERC who is in charge of TRDU operation.

ANALYSES OF KARHULA PCFB ASH

We received six ash samples from the Karhula PCFB facility which are briefly described in Table 1. We provided Kumar Sellakumar of Ahlstrom Pyropower with sample information forms for these samples to be completed and returned to us. We selected a filter cake ash sample taken from the surface of a candle in the middle plenum (ID # 4182) for detailed analysis. This sample contained loose ash and remnants of filter cake nodules. Our chemical and physical analyses of this sample are summarized in Tables 2 and 3. The SEM photographs of this filter cake ash sample presented in Figure 3 illustrate the irregular shapes associated with PFBC processes.

Figure 4 presents an SEM photograph of a fresh fracture surface of a filter cake nodule taken sample # 4182. This photograph indicates that the nodules found in the Karhula filter are concretions composed of discrete fine particles almost completely embedded in pervasive amorphous masses which apparently form in the filter vessel after the particles are initially collected. The appearance of the Karhula filter cake nodule in Figure 4 is very similar to the appearance of nodules we removed from the Tidd APF. This similarity, in combination with similar ash chemistries and flue gas environments, leads us to believe that eutectic formation, as we have described in prior reports summarizing our observations from the Tidd APF, is also responsible for nodule formation at Karhula.

All three filter cake ash samples we received from Karhula contained fragments of nodular filter cake. We used one of the methods we developed for measuring the overall pore volume of a nodule to characterize the nodule fragments in sample # 4182. Our on-site experience at the Tidd PFBC has shown that the porosity of the most recently deposited



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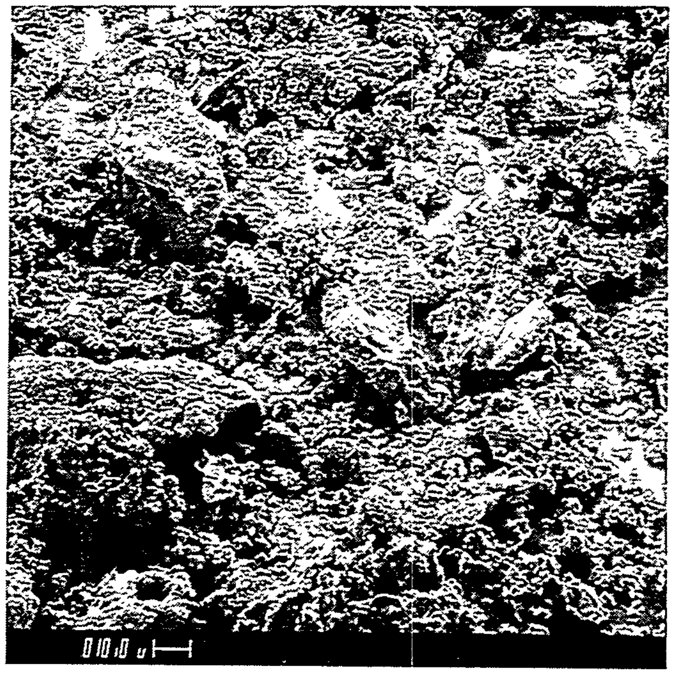

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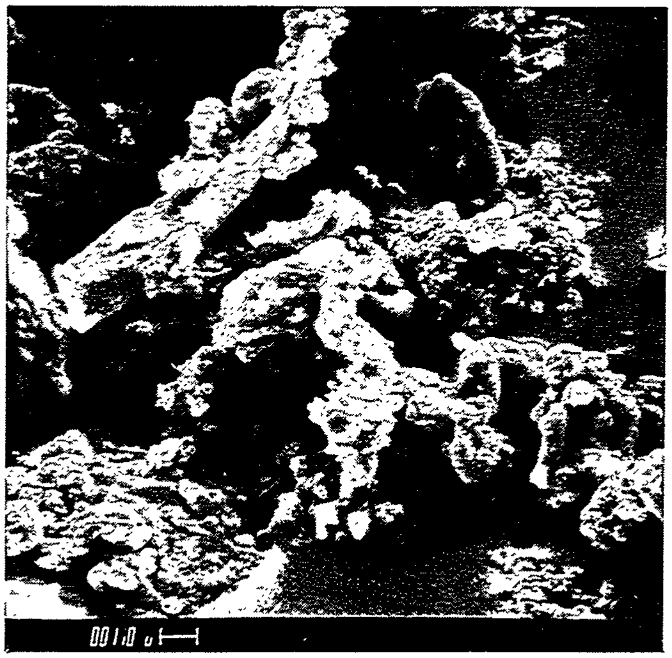

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Figure 3. SEM photographs of filter cake ash from the Karhula PCFB (ID # 4182) taken at a) 100X, b) 500X, and c) 5000X.

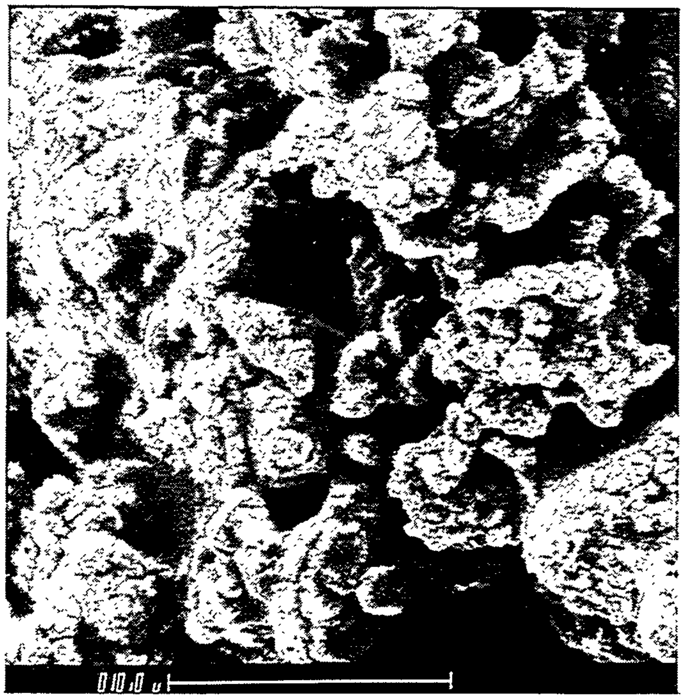

Figure 4. SEM photograph of a fresh fracture surface of a Karhula filter cake nodule (ID # 4182).

portions of filter cake is significantly greater than the porosity of older portions of the cake. It is very likely that during the shipment of the filter cake ash and nodule samples from Karhula to our laboratories, the fluffiest, most recently deposited parts of the cake were shaken or rubbed off of the cake nodules. Therefore, the reported filter cake porosity value of 75 % represents a lower bound for the actual overall filter cake porosity.

The specific surface area of this ash is lower and its mass median diameter is larger than other Karhula filter cake ashes we have characterized. These physical characteristics cause this ash sample to be a bit more free flowing, as evidenced by its uncompacted bulk porosity, than other Karhula filter cake ashes in the HGCU data base. The calculation of specific gas-flow resistance is strongly dependent on the porosity of the filter cake (which is assumed to equal the uncompacted bulk porosity when actual measurements of intact filter cakes are not possible). Therefore, even though the drag-equivalent diameter of ID # 4182 is comparable to other Karhula filter cake ashes, its specific gas-flow resistance is about 30 % lower than values measured for those other ashes. We will attempt to correlate the characteristics of this filter cake ash with filter performance at Karhula when information becomes available describing recent filter operation.

PERFORMANCE ESTIMATES FOR DOE/METC MGCR HOPPER ASH

Based on the measurements we performed on the DOE/METC gasifier residue sample (ID # 4170) we characterized in our last two quarterly reports, we have calculated some quantities relating to the type of filter performance that might be expected during filtration of this type of gasifier residue. The goals of the calculations we performed this quarter were to estimate the rate of filter cake and pressure drop accumulation for a ceramic candle type barrier filter collecting a gasifier residue like that produced by the DOE/METC Advanced Gasification and Hot Gas Cleanup Facility. We have assumed that the inlet particulate loading to the barrier filter is 4000 ppmw and the temperature and pressure in the filter vessel are 1150 °F and 20 atmospheres, respectively. We have also assumed that the filter is sized to handle a flow of 10 acfm/ft^2 (or 10 ft/min) of active filter area, and that the viscosity of the gas being filtered equals the viscosity of air at equivalent conditions. As we presented in our two most recent quarterly reports, the specific gas-flow resistance of this gasifier residue is 18 in. $H_2O/[(acfm/ft^2)\cdot(lb/ft^2)]$ for a filter cake having a porosity of 97 %. (The units for specific gas-flow resistance have been expanded to clarify the calculations presented below.) We have assumed a filter cake porosity of 97 % because this is the uncompacted bulk porosity of this material. The true density of the residue particles is 2.87 g/cm^3.

An inlet loading of 4000 ppmw at 20 atmospheres and 1150 °F is approximately equal to 0.0019 lb/acf. Therefore at a face velocity of 10 acfm/ft^2 it will take about 53 minutes to deposit 1.0 lb of residue on 1.0 ft^2 of active filter area. (The effect of the cylindrical geometry of the filter elements is considered later in this discussion.) For a filter cake with a porosity of 97 %, the volume of 1.0 lb of filter cake is about 320 in^3. On a standard candle with an outer diameter of 60 mm, 1.0 lb of this filter cake will accumulate in 53 minutes and will cover 19.4 in. of the length of the candle with a cake thickness of 1.4 in.

The pressure drop that would be accumulated over the 53 minutes it would take to build the cake described above can be calculated from the specific gas-flow resistance of the residue, the actual face velocity of the gas, the areal density of the filter cake, and the viscosity of the gas at 20 atm and 1150 °F. Therefore the specific gas-flow resistance of the residue (18 in. $H_2O/[(acfm/ft^2)\cdot(lb/ft^2)]$) must be multiplied by 10 to account for the face velocity, 1.0 to adjust for the areal density (which was defined for this example to be equal to 1.0 lb/ft^2), and 2.11 to adjust for the increase in gas viscosity. (The viscosity of air increases from 184 poise at the laboratory conditions under which the specific gas-flow resistance was measured, to 389 poise at 20 atm and 1150 °F.) Therefore, in the absence of any cake removal, and under the assumed conditions described above, the pressure drop across the filter will increase by 380 in. H_2O during a 53 minute filtering cycle. It is important to note that this pressure drop could increase many fold if the filter cake compacts to a porosity of near or below 90 % due to filtering pressure drop.

In a recent visit to METC, we had the opportunity to examine the filter cake that remained on the candles used during the most recent run performed at the DOE/METC Advanced Gasification and Hot Gas Cleanup Facility. This filter cake should correspond directly to sample # 4170, which we have characterized thoroughly in our laboratory. Although the cake remaining on the candle surface was patchy and thin, we were able to make a rough measurement of its porosity. The value we obtained (86 %) must be interpreted as a lower bound for the actual porosity of the filter cake during operation, since handling and storage of the used candles appeared to have possibly compressed much of the cake on the candles after they were removed from the filter vessel. Based on the calculations presented above, a filter cake porosity of 86 % would probably present severe pressure drop penalties for this gasifier residue.

OVERVIEW OF FACTORS CONTRIBUTING TO FILTER SYSTEM FAILURE

As part of the poster we prepared for the 1996 DOE/METC Contractor's Conference, we constructed a diagram illustrating the various factors that we believe can contribute to filter system failure at barrier filters collecting PFBC ashes. This diagram is presented in Figure 5. Most of the relationships shown in this diagram are based on our four on-site observations of the Tidd APF when it was opened for inspection and/or refitting. The diagram is not meant to infer that all of these factors are present in every PFBC/barrier filter application. It is meant to illustrate how filter design, PFBC ash characteristics, and the properties of filter materials can combine to create significant problems in a ceramic candle-based barrier filter vessel.

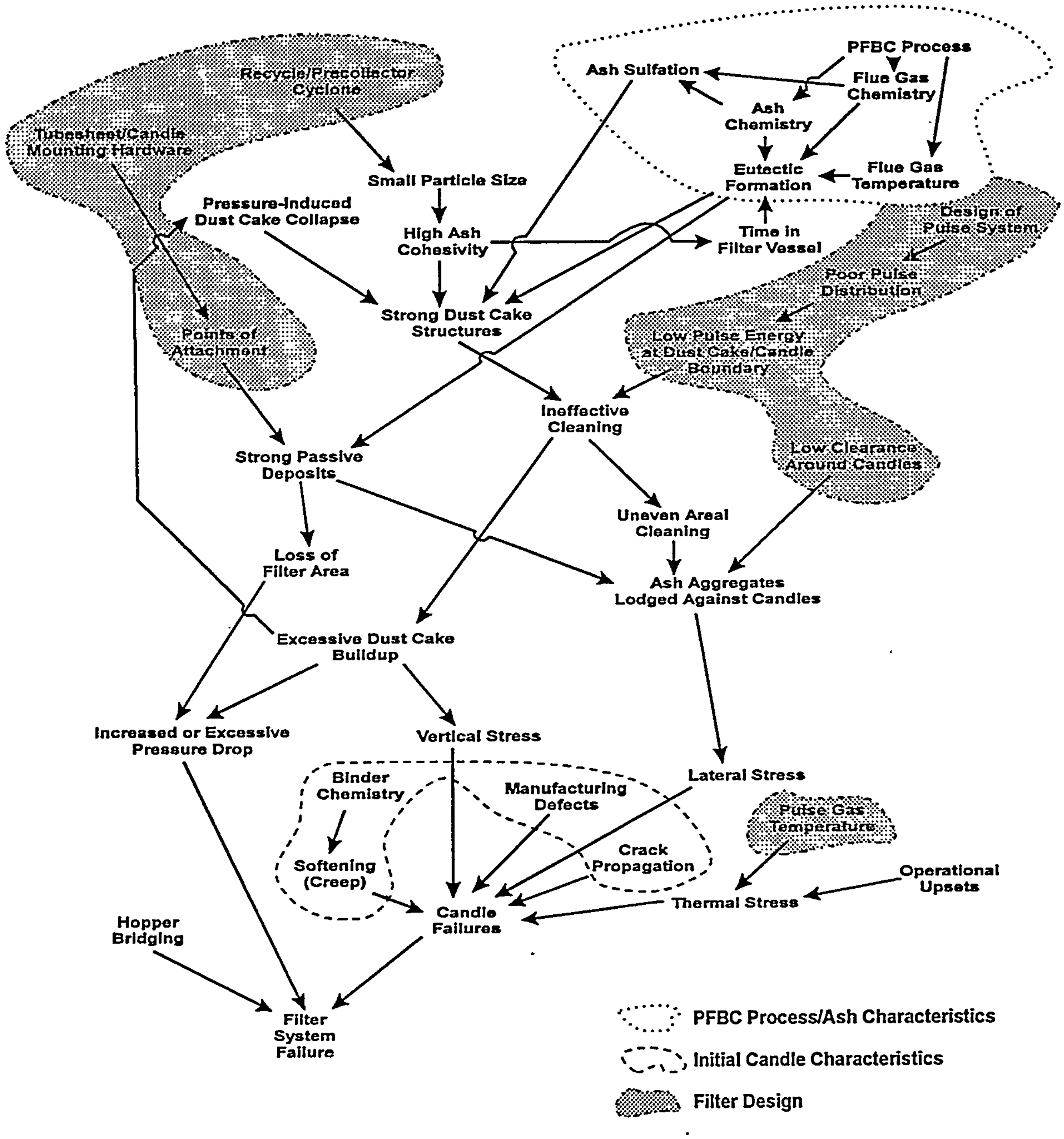

Figure 5. Diagram illustrating the various factors in a ceramic candle barrier filter collecting PFBC ash that can combine to cause filter failures.

TASK 2 RESEARCH ACTIVITIES

Hoop tensile testing of Schumacher FT20 and Refractron 326 candles removed from Karhula after ~540 hours in service was completed. Average results for used and as-manufactured materials are summarized in Table 4.

Table 4
Hoop Tensile Test Results for Schumacher FT20 and Refractron 326 Candles

Material	Service	Number of observations	Average hoop tensile strength, psi	Standard deviation, psi	COV, %
Schumacher FT20	540 hours	9	1530	38	2
Schumacher FT20	as-manuf.	9	1690	68	4
Refractron 326	540 hours	9	1430	88	6
Refractron 326	as-manuf.	6	2130	167	8

These results show a hoop tensile strength decrease of ~9 % for Schumacher FT20 and ~33 % for Refractron 326 after ~540 hours in service at Karhula.

Tensile creep testing of Schumacher FT20 and Refractron 326 is in progress. Results obtained so far for Schumacher FT20 indicate an average secondary creep rate of 6.5×10^{-9} in/in/sec at 1600 °F and a stress level of 500 psi. Schumacher FT20 specimen Creep-Ax-7 was tested at 1700 °F and 500 psi after being tested at 1600 °F for 110 hours. This specimen broke after 18 hours at 1700 °F. One Refractron 326 specimen was tested at 1700 °F and 500 psi and a secondary creep rate of 8.6 x 10-9 in/in/sec was obtained. Additional creep testing is in progress.

Microstructural evaluation of as-manufactured and post-service Refractron 326 and Schumacher FT20 are required so that material models can be developed to evaluate the creep response measured and the property degradation measured in the material from Karhula. Microstructural evaluation is currently in progress.

FUTURE WORK

Plans for the next quarter include continued construction of the HGCU data base and entry of additional data, photographs, and text. We have solicited the help of personnel from Schumacher to obtain samples and operating information from various European gasification facilities with which they are involved. We hope to receive information describing the most recent operation of the Karhula PCFB to correlate with the laboratory data presented in this report. We hope to receive additional samples from UNDEERC's TRDU test facility. We will also be presenting an overview of the HGCU data base and our laboratory characterizations of various ashes and particulate residues at the Thirteenth Annual International Pittsburgh Coal Conference to be held in September of this year. Microstructural evaluation of as-manufactured and post-service Refractron 326 and Schumacher FT20 are currently in progress. Creep testing of these two materials will continue.

PARTICULATE HOT GAS STREAM CLEANUP TECHNICAL ISSUES

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Approved by

Duane H. Pontius, Director   Particulate Sciences Department