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Particulate Hot Gas Stream Cleanup Technical Issues

Quarterly Report
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EXECUTIVE SUMMARY

This is the thirteenth quarterly report describing the 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 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 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 characterizations of additional ash samples from Pressurized Fluidized-Bed Combustion (PFBC) facilities to the HGCU data base. Task 1 plans for the next quarter include characterization of samples collected during a site visit on January 20 to the Department of Energy / Southern Company Services Power Systems Development Facility (PSDF). Further work on the HGCU data base is also planned.

Task 2 work during the past quarter included creep testing of a Coors P-100A-1 specimen machined from Candle FC-007 after 1166 hours in-service at the Karhula Pressurized Circulating Fluid Bed (PCFB) facility. Samples are currently in preparation for microstructural evaluations of Coors P-100A-1.Sixteen cordierite rings manufactured by Specific Surfaces were received for testing. Three of the specimens were exposed to the PFBC environment at the PSDF. These specimens are currently being machined for testing.

INTRODUCTION

This is the thirteenth 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 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 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, 317 particulate samples from thirteen 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 base. Many of the samples in the data base 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 format 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.

Problems with the durability of the filter elements are being addressed by the development and evaluation of elements constructed from alternative ceramic materials. Hoop and axial tensile tests, thermal expansion, compression, and creep evaluations of these materials at temperatures up to 1800 °F have been performed in order to understand the thermal and mechanical behavior of the various types of ceramic materials used in hot gas filtration. Nondestructive testing methods performed on filter specimens include density and ultrasonic velocity. Task 2 work during the past quarter included creep testing of a Coors P-100A-1 specimen machined from Candle FC-007 after 1166 hours in-service at Karhula. Samples are currently in preparation for microstructural evaluations of Coors P-100A-1. Sixteen cordierite rings manufactured by Specific Surfaces were received for testing. Three of the specimens were exposed to the PFBC environment at the PSDF. These specimens are currently being machined for testing.

OBJECTIVES

Task 1 has two primary objectives. The first is to generate a readily accessible data base of the key characteristics of particulate samples collected from operating advanced particle filters. The second objective is to relate these measured 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 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

A site visit was made on November 5 to the PSDF to characterize the condition of the filter and collect ash samples for analysis. Other efforts during the past quarter included analyses of ash samples collected from Karhula during the last quarter of 1997. The samples from these facilities that were analyzed during the past quarter and are discussed in this report are briefly described in Table 1.

Table 1
Particulate Samples Obtained and Characterized During the Past Quarter

ID #	Source	Brief description
4262	PSDF	bottom plenum filter cake (11-5-97)
4268	PSDF	flat roof over top plenum (11-5-97)
4276	Karhula	ash from silo after test run
4277	Karhula	top plenum filter cake ash

The majority of the physical analyses performed on the samples listed in Table 1 are summarized in Table 2. Chemical analyses performed on the samples listed in Table 1 are presented in Table 3. Additional measurements performed on these samples are discussed in the following sections.

Table 2
Physical Characteristics of PSDF and Karhula Samples

source	PSDF	PSDF	Karhula	Karhula
quantity ID #	4262	4268	4276	4277
specific surface area, m^2/g	5.3	--	1.6	2.2
Stokes' D_{16}, µm	1.2	--	--	--
Stokes' D_{50}, µm	3.8	--	--	--
Stokes' D_{84}, µm	13	--	--	--
GSD of size distribution	3.2	--	--	--
Stokes' D_{50}, µm (Bahco classifier)	--	--	15	7.8
uncompacted bulk porosity, %	87	--	75	82
nodule porosity, %	--	80.5	--	--
drag-equivalent diameter, µm	1.49	--	5.01	2.72
specific gas-flow resistance, in H_2O·min·ft/lb*	2.9	--	2.2	2.4
specific gas-flow resistance, in H_2O·min·ft/lb**	11	--	--	--
tensile strength, N/m^2	> 16	--	19	3.8
true particle density, g/cm^3	2.78	--	2.73	2.76

* calculated for an assumed filter cake porosity equal to the uncompacted bulk porosity
** calculated for the porosity of a nodule removed from the PSDF on November 5 (80.5 %)

Table 3
Chemical Composition of PSDF and Karhula Ashes, % wt.*

source ID # location particle size range	PSDF 4262 filter cake all	Karhula 4276 hopper < 45 µm	Karhula 4276 hopper > 45 µm	Karhula 4276 hopper all	Karhula 4277 filter cake all	Karhula 4277 filter cake < 45 µm
Li_2O	--	0.02	0.02	0.02	0.02	0.02
Na_2O	0.06	0.70	0.52	0.64	0.61	0.78
K_2O	1.76	1.4	1.5	1.4	1.6	1.6
MgO	8.00	0.87	0.87	0.87	0.88	0.87
CaO	9.98	23.9	24.3	24.0	21.3	21.0
Fe_2O_3	4.78	8.1	6.2	7.5	8.1	8.4
Al_2O_3	21.93	16.8	16.0	16.5	16.9	17.1
SiO_2	43.37	32.0	35.4	33.1	31.5	31.7
TiO_2	1.13	1.3	0.83	1.1	1.2	1.2
P_2O_5	0.51	0.18	0.15	0.17	0.18	0.19
SO_3	6.34	13.1	14.2	13.5	15.7	15.9
SrO	0.19	--	--	--	--	--
BaO	0.09	--	--	--	--	--
LOI	1.94	0.74	0.70	0.73	0.62	0.62
soluble $SO_4^=$	11.6	16.7	16.0	16.5	19.6	19.4
Equilibrium pH	7.3	--	--	--	10.18	--

* Equilibrium pH is dimensionless

SITE VISIT TO THE PSDF AND ANALYSES OF PSDF ASHES

A site visit was made on November 5 to the PSDF to characterize the condition of the filter and collect ash samples for analysis. Videotape and still pictures were made of all major subjects. In general, the candles were covered with thin, uneven cakes. Core samples (for areal density determinations) were obtained from 3 candles in the top plenum and 2 candles in the bottom plenum. For each of these candles, measurements were made about 10 inches below the tubesheet and 10 inches above the bottom of the candle. These areal density measurements are summarized in Table 4.

Table 4
PSDF Filter Cake Areal Density Measurements (November 5, 1997)

location	areal density, g/cm^2
top plenum	0.024
bottom plenum	0.023
near top of candles	0.019
near bottom of candles	0.028
overall average	0.023

The thickest cake nodule observed on candles in the top plenum was about 2 mm thick. On the bottom plenum a filter cake nodule was found that was about 2.5 mm thick. Filter cake thickness was measured at several points with the traversing transverse laser gauge. The data from these measurements are summarized in Table 5 but are probably not too meaningful because the cakes were very thin and had very irregular thickness.

Table 5
PSDF Filter Cake Thickness Measurements (November 5, 1997)

location	thickness, mm
top plenum (~10 inches below the top of the candle)	0.84
top plenum (~10 inches below the top of the candle)	1.40
top plenum (~10 inches above the bottom of the candle)	0.51
top plenum (~10 inches above the bottom of the candle)	0.64

The measured size distribution of PSDF filter cake ash (ID # 4262) is presented in Figure 1. The MMD of this sample (3.9 μm) is significantly smaller than the MMD's measured for various ashes collected from the PSDF in July 1997 (about 7 to 10 μm). A measured increase in the specific surface area of the filter cake from July (2.9 m^2/g) to November (5.3 m^2/g) agrees with changes in the size distributions of filter cake ashes between these two site visits. SEM photomicrographs of the November 1997 filter cake ash sample (ID # 4262) are shown in Figure 2.

During examination of the filter cakes on November 5, One nodule (from sample # 4268) was large enough and strong enough for a porosity determination to be performed with the ethanol impregnation method. The value of porosity measured for this nodule taken from the flat roof over the top plenum (80.5%) was used in one of the two calculations of specific gas-flow resistance presented in Table 2. The strong effect that filter cake porosity has on specific gas-flow resistance is evident in the two values calculated for ID # 4262 (2.9 and 11 in $H_2O\cdot min\cdot ft/lb$). The actual porosity of the filter cakes formed in the PSDF should be more precisely determined if thicker filter cakes are present when the filter vessel is opened for inspection and sampling. Additionally, as PSDF operation continues, the range of filter cake characteristics induced by differences in various process parameters may become better defined as more samples from distinct periods of operation become available.

ANALYSES OF KARHULA ASHES

Mr. Reijo Kuivalainen of Foster Wheeler Energia OY provided several samples from the Westinghouse filter assembly following a test conducted with Lakeland coal and sorbent during the last quarter of 1997 of the Foster Wheeler CPFBC Facility at Karhula. Of these ash samples, the two samples listed in Table 1 were selected for detailed analyses. The results of most of these analyses are presented in Tables 2 and 3. Differential size distribution data measured for Karhula hopper ash (ID # 4276) and filter cake ash (ID # 4277) as measured with a Bahco Classifier are presented in Figure 3. SEM photomicrographs of sample # 4277 are presented in Figure 4.

Because the Karhula samples that were provided included filter cake ashes and a corresponding hopper ash, analyses were performed to determine specific physical and chemical differences between samples obtained from these two locations. Measurements of

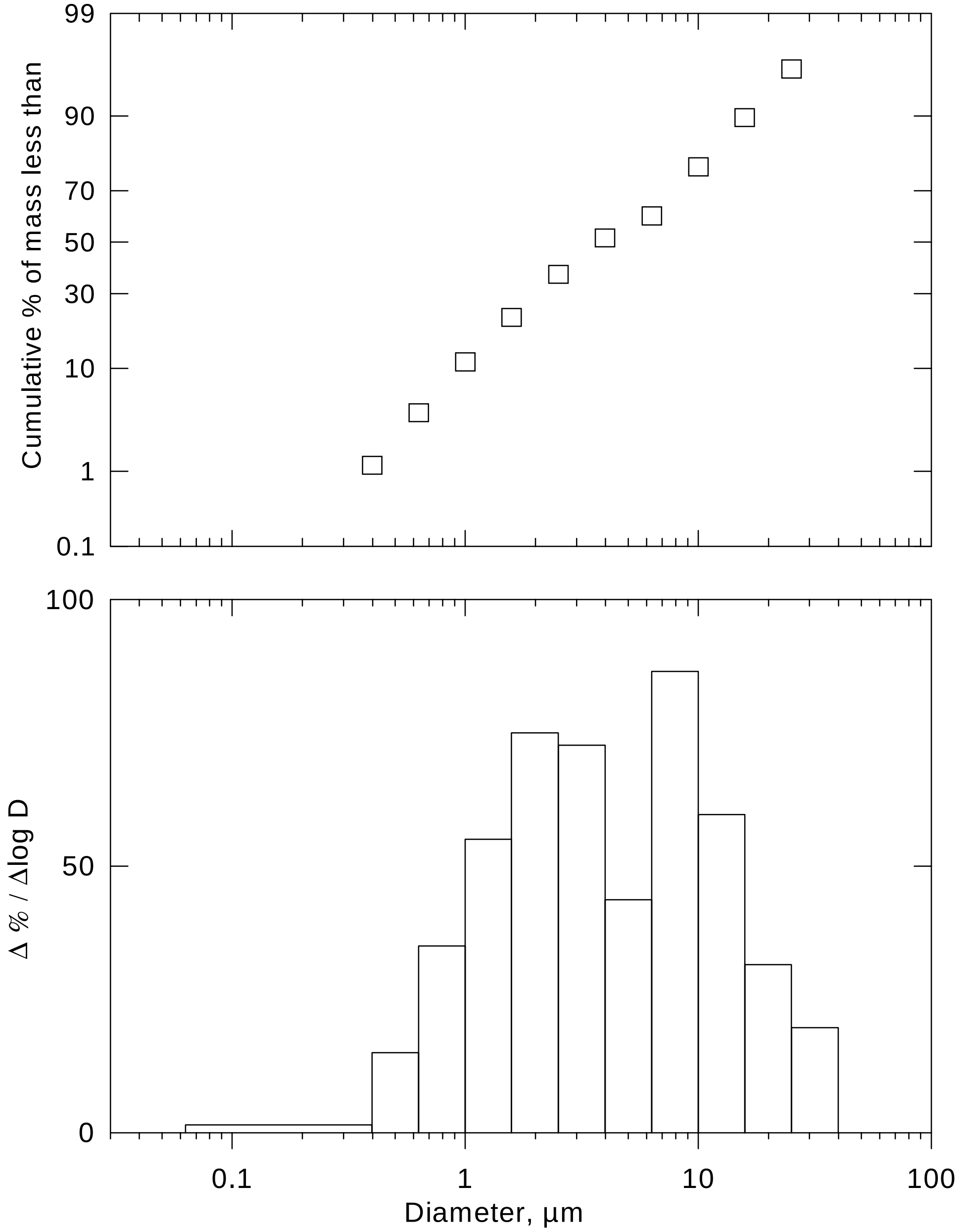
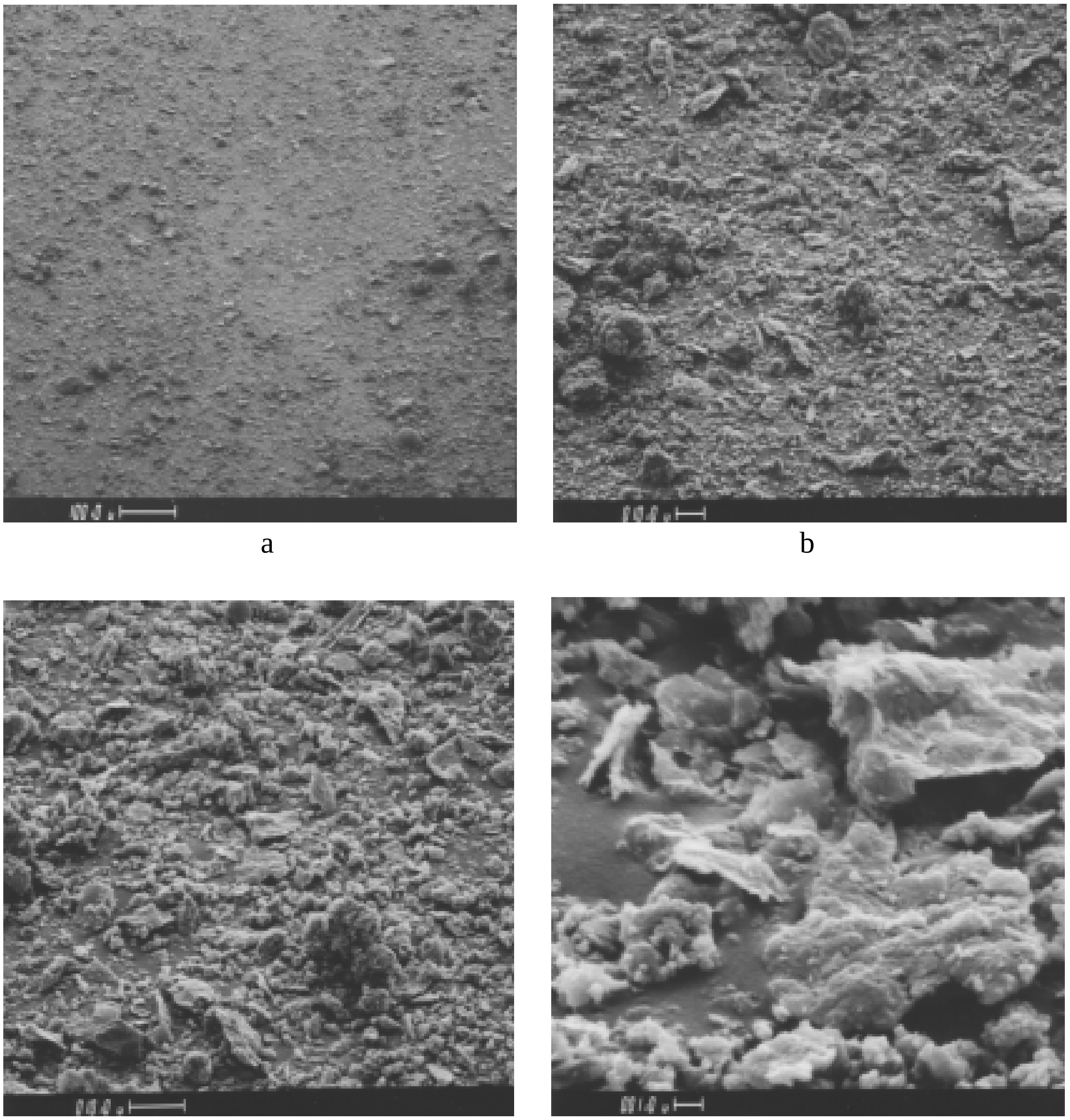


Figure 1. Cumulative and differential size distribution data measured for PSDF filter cake ash (ID # 4262) measured with a Shimadzu SA-CP4 Centrifugal Particle Size Analyzer. The Stokes' D_{16} of this distribution is 1.2 µm, its D_{50} is 3.8 µm, its D_{84} is 13 µm, and its geometric standard deviation is 3.8. (These size distribution data include the assumption that the sample contains no particles smaller than 0.063 µm.)



a

b

c

d

Figure 2. Representative scanning electron micrographs of PSDF filter cake ash (ID # 4262) taken at a) 100X, b) 500X, c) 1000X, and d) 5000X.

size distribution, specific surface area, uncompacted bulk porosity, specific gas-flow resistance (with associated drag-equivalent diameter), and tensile strength (see Table 2) all indicate that these two ashes have significantly different physical characteristics. The ash particles entering the filter vessel are apparently segregated based on their physical characteristics. The mechanism that is most likely responsible for this segregation is the selective continued entrainment of ash particles as determined by their individual settling velocities. As the gas entering the vessel slows to the filtering face velocity, previously entrained ash particles with higher settling velocities tend to divert from the flow paths of the flue gas (to the surface of the filter cake). Their trajectories instead become governed by the force of gravity, and they settle into the hopper before they ever reach the filter cake. Finer ash particles with low enough settling velocities continue to be entrained in the gas up until they impact on the surface of the filter cake. The same mechanism of selective continued entrainment would also apply to ash particles ejected from the filter cake during cleaning pulses. Individual ash particles, or agglomerates of ash particles with small enough settling velocities will reentrain in the flue gas and be recollected on the filter cake. Particles and agglomerates of particles with sufficiently high settling velocities will permanently leave the filter cake and settle into the hopper. The degree to which the Karhula hopper ash contains large particles not found in the Karhula filter cake ash can be observed in the data presented in Figure 3.

The hopper ash (ID # 4276) and the filter cake ash (ID # 4277) exhibited similar values of specific gas-flow resistance, despite their other physical differences. The reason for this similarity is the offsetting effect that filter cake porosity and specific surface area have on each other. Coarser particles, like those in the Karhula hopper sample, exhibit lower bulk cohesivity and would form filter cakes with lower porosities. In general, however, coarser particles also exhibit lower specific surface areas (as can be seen in Table 2). Pressure loss across a filter cake is incurred as the flue gas follows a tortuous path through the cake and past the surfaces of the particles. Because Karhula filter cake ash has more specific surface area than the corresponding hopper ash, the beneficial effect of the greater porosity of the filter cake is offset by the higher specific surface area of the filter cake ash particles.

Analyses were also performed to determine the extent to which the physical segregation of particles discussed above affected the chemical composition of the hopper and filter cake ashes. In general, the chemical composition of the hopper and filter cake ashes are basically independent of particle size. The chemical species in the hopper ash that most deviate from this trend are Na_2O, Fe_2O_3, TiO_2, and P_2O_5, which are enriched in the finer ash particles, and SiO_2, which is enriched in the coarser ash particles. For the filter cake ash, the only chemical species found to be size dependent was Na_2O, which, like the hopper ash, was enriched in the finer particles. In a comparison of the chemistry of the hopper and filter cake ashes, the main difference was in their sulfur contents. As has been previously reported under this project and by other researchers, sulfur was enriched in the filter cake ash relative to the hopper ash, most likely by the additional scrubbing of SO_2 from the flue gas by unreacted sorbent in the filter cake.

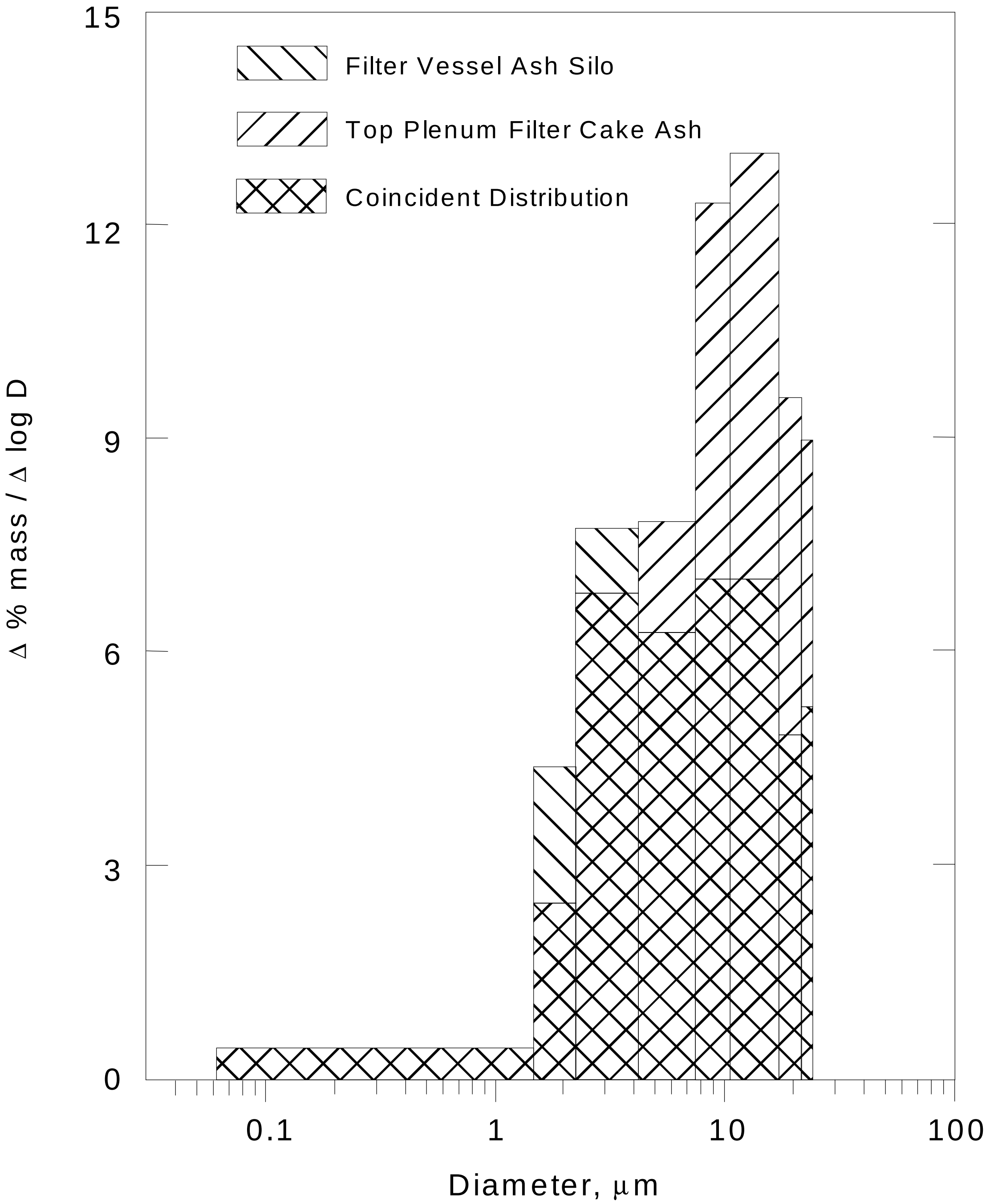


Figure 3. Differential size distribution data measured for Karhula hopper ash (ID # 4276) and filter cake ash (ID # 4277) measured with a Bahco Classifier. (These size distribution data include the assumption that the sample contains no particles smaller than 0.063 µm.)

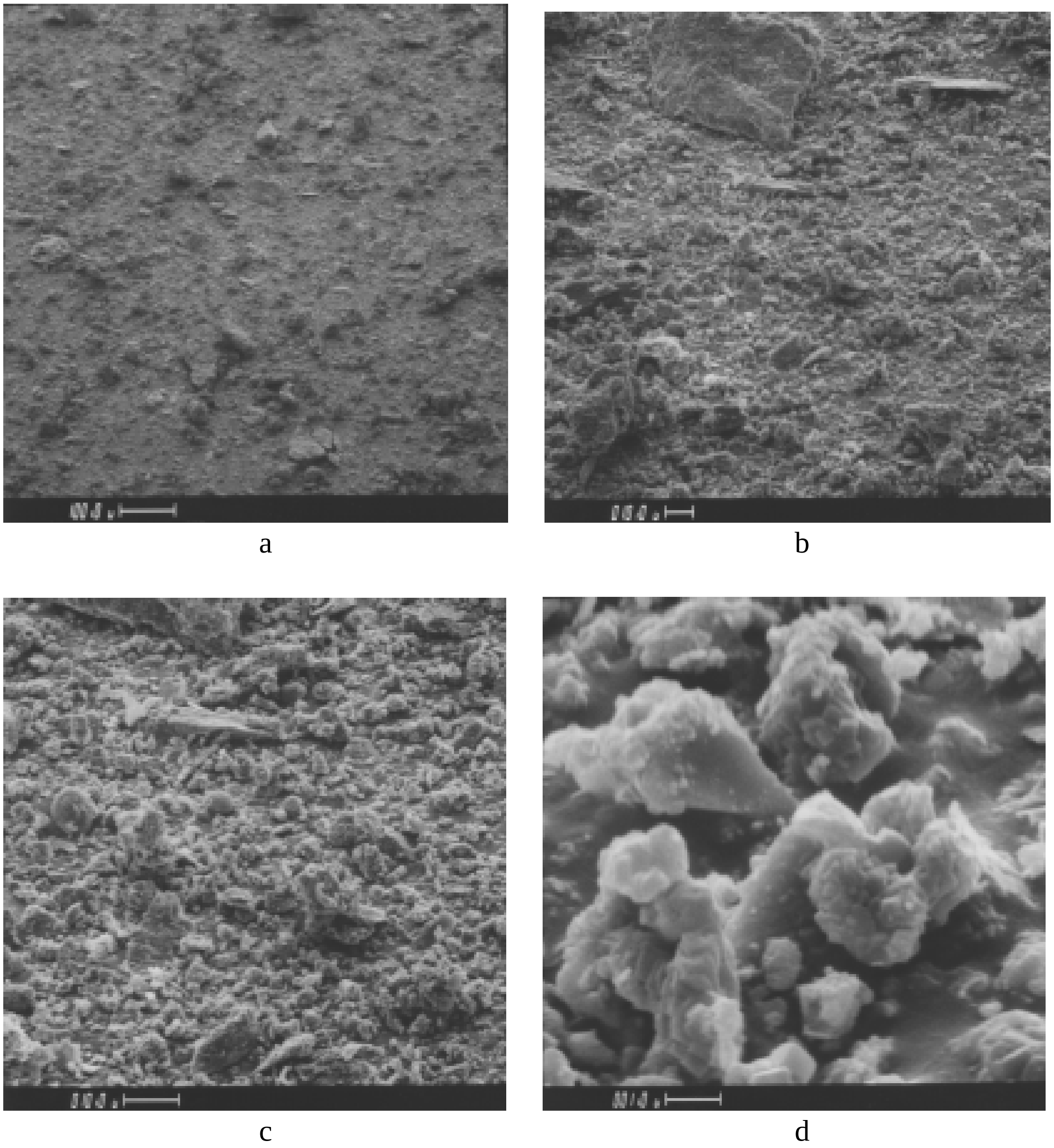


Figure 4. Representative scanning electron micrographs of Karhula filter cake ash (ID # 4277) taken at a) 100X, b) 500X, c) 1000X, and d) 10,000X.

TASK 2 FILTER MATERIAL CHARACTERIZATION

Creep testing was completed on one Coors P-100A-1 specimen machined from Candle FC-007 after 1166 hours in-service at Karhula. This creep test was conducted at 1600 °F, 200 psi. A summary letter describing the results of this creep test will be provided soon. Microstructural evaluations of Coors P-100A-1, as-manufactured and after PFBC service, are required. Samples are currently in preparation for these evaluations.

Sixteen cordierite rings manufactured by Specific Surfaces were recently received for testing. The received rings were nominally 1.55” I.D. x 2.35” O.D. x 1.00” high. Three of the specimens were exposed to the PFBC environment at the PSDF. The specimens exposed at this facility did not serve as filters but were exposed to the thermal and chemical environments in the PCD. These specimens are currently being machined to the correct height and will be tested after machining is complete.

FUTURE WORK

Efforts under Task 1 during the next quarter will include analyses of ashes collected during a site visit to the PSDF on January 20, 1998. Development of the HGCU data base will also continue during the next quarter. Under Task 2 in the upcoming quarter, microstructural examination Coors P-100A-1 after service in the PFBC environment will be conducted. Hoop tensile testing of cordierite manufactured by Specific Surfaces, as-manufactured and after PFBC service, will be conducted. Also in this upcoming quarter, an analysis of the properties measured to date on all candidate candle filters and the observations of these materials in service in PFBC applications will begin. The objective of this analysis is to assess the capabilities and limitations of the current hot gas filter materials and evaluate material property requirements for successful future use.

PARTICULATE HOT GAS STREAM CLEANUP TECHNICAL ISSUES

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Must be completed by the contractor.