DOE/MC/31160 -- 5115
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Particulate Hot Gas Stream Cleanup Technical Issues

Quarterly Report
April - June 1995

August 1995

Work Performed Under Contract No.: DE-AC21-94MC31160

For
U.S. Department of Energy
Office of Fossil Energy
Morgantown Energy Technology Center
Morgantown, West Virginia

By
Southern Research Institute
Birmingham, Alabama

DISCLAIMER

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EXECUTIVE SUMMARY

This is the third in a series of quarterly reports 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 aspects of filter operation that are apparently linked to the 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. Task 2 concerns testing and failure analysis of ceramic filters.

Activities performed during this reporting period include participation in the Review Meeting *Advanced Coal-Fired Power Systems '95*, which was held in June in Morgantown, WV, a site visit to the Tidd Plant to collect ash samples and document the condition of the filter vessel, and laboratory analyses of these Tidd ash samples. Plans for the next quarter include analyses of ashes obtained from General Electric's gasification facility in Schenectady, NY. We also plan to complete the design of the high-temperature test device described in our last quarterly report. This device will measure the uncompacted bulk porosity of aggregates of ash formed at temperatures commonly encountered in operating APFs. We will also continue work on the interactive data base of HGCU ash characteristics.

Mechanical and thermal testing of Dupont/Lanxide PRD-66, Dupont composite, 3M composite, and IF and P Fibrosics is continuing. Thermal expansion values of the PRD-66, 3M composite, and IF and P Fibrosics have been measured at 1500 °F. Testing of new Refractron and Schumacher candle filter materials is in progress. No creep was detected for the Refractron specimens after ~150 hours at 1550 °F and creep testing is proceeding at 1600 °F. Machined Schumacher creep specimens will be tested following completion of the Refractron tests.

INTRODUCTION

This is the third quarterly report describing the activities performed under Contract No. DE-AC21-94MC31160. Our analyses of HGCU ashes and descriptions of filter performance are designed to address the problems with filter operation linked to the characteristics of the collected ash. Some of the problems observed at the Tidd and Karhula 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. Task 2 of this contract includes characterization of new and used filter elements. In addition to these problems related to the characteristics of PFBC ashes, our previous laboratory characterizations of gasifier and carbonizer ashes have shown that these ashes also have characteristics that might negatively affect filtration.

To identify which ash characteristics can lead to problems with filtration, we have assembled over 220 ash samples from ten 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 and Karhula. 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.

Much of the work planned for Task 1 builds directly on work performed under a prior contract (No. DE-AC21-89MC26239). Two visits to the 70 MWe Pressurized Fluidized-Bed Combustion (PFBC) Facility at the Tidd Plant were made during this prior contract to collect samples and observe and record the condition of the APF. Two topical reports covering the collection and analyses of HGCU ashes were also issued under Contract No. DE-AC21-89MC26239. The first report, *Assessment of Ash Characteristics from Gas Stream Cleanup Facilities*, covered samples obtained and analyzed between October 1992 and September 1993. The second report, *Updated Assessment of Ash Characteristics from Gas Stream Cleanup Facilities*, is still in draft form, and covers samples obtained and analyzed between October 1993 and August 1994. Because operating experiences with HGCU facilities continue to accumulate, Task 1 of this current contract continues many of the same types of analyses described in these two topical reports.

OBJECTIVES

This task 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).

TASK 1 RESEARCH ACTIVITIES

SITE VISIT TO TIDD

The APF was opened for the last time on May 11, 1995 after the final shutdown of the Tidd Demonstration Plant. During this last operating period, the cyclone upstream of the APF was entirely bypassed in order to get as many relatively large particles into the filter vessel as possible. In general, the filter assembly was quite clean. The only significant deposits of ash were on the underside of the tube sheets, much like the deposits found in these locations during prior inspections. Most of the broken candle filter elements were of a particular experimental design. The vast majority of the other filter elements were intact. We collected twenty separate ash samples from the filter assembly during this inspection. These samples are listed and described in Table 1.

ANALYSIS OF TIDD ASH SAMPLES

Size distributions of three of the ashes listed in Table 1 were measured with a combination of sieving and sedigraphic analyses. Prior to derating and eventually bypassing the cyclone upstream of the APF, the ash particles collected at various places in the filter vessel in 1993 and 1994 were very fine, with MMD's around 3 to 5 μm. As efforts to increase the size of the ash entering the APF by derating the cyclone progressed, somewhat larger MMD's were observed. However, the major changes in the size distributions of the ashes collected in the APF did not occur until the last period of operation, during which the cyclone was completely bypassed.

Sieve analyses were performed on ashes collected in October 1994, after derated cyclone operation, and May 1995, after operation with the cyclone completely bypassed. The sieving process we used separated a portion of the original ashes into three size fractions: particle diameters > 45 μm, particle diameters > 15 μm, but smaller than 45 μm, and ash particles with diameters < 15 μm. (The sieving process caused this last, smallest fraction to be discarded along with the isopropanol used to wash the particles through the 45 μm and 15 μm sieves.) The data in Table 2, which include sieve data measured for Tidd ashes obtained in October 1994, show that bypassing the cyclone between October 1994 and May 1995 significantly increased the size distributions of ash collected in the APF.

Table 1. Ash Samples Collected at Tidd on May 11, 1995

ID #	Location	Description/Comments
4142	TP, A, TS	
4143	TP, A, FC	central section of candle (nodules 0.25" thick)
4144	TP, C, FC	clumps about 0.4" thick; bottom of viropore candles
4145	TP, C, FC	
4146	TP, B, FC	
4147	TP, C, CSC/AS	from junction between CSC and AS
4148	TP, C, AS	
4149	TP, A, CSC	flaky deposits from CSC
4150	TP, A, TS	flaky deposits from outer edge of tube sheet
4151	MP, A, TS	very fluffy deposits
4152	MP, A, FC	0.06" thick, brushes off easily
4153	MP, A, CSC	flaky deposits from CSC
4154	MP, A, AS	
4155	MP, A, CSC/AS	from junction between CSC and AS
4156	MP, C, FC	
4157	BP, A, TS	not as fluffy as ID # 4151
4158*	BP, A, FC	bottom section fairly thin, upper portion knobby
4159	BP, B, FC	knobby deposits up to 0.4" thick
4160*	BP, A, FC	from inner (3rd and 4th from outside) rings of candles
4161	BP, C, FC	fresh cake fluffy, older portion firmly attached to candle

* Portions of these samples were sent to Dr. T.K. Chiang at DOE/METC.

TP = Top Plenum   A = Array A   CSC = Center Support Column
MP = Middle Plenum   B = Array B   AS = Ash Shed
BP = Bottom Plenum   C = Array C   FC = Filter Cake Ash from Candle Surface
TS = Underside of Tube sheet (region of attachment of candles)

Table 2
Sieve Analyses of Tidd Ashes

ID #, date	% weight		
	diam > 45 μm	45 μm > diam > 15 μm	diam < 15 μm
ID # 4097, Oct. 1994	4.03	17.3	78.6
ID # 4114, Oct. 1994	0.64	3.84	95.5
ID # 4142, May 1995	28.9	50.7	20.4
ID # 4144, May 1995	30.1	34.3	35.6
ID # 4151, May 1995	44.8	33.3	21.9

We also performed sedigraphic analyses on the five samples listed in Table 2. The results of these sedigraphic analyses are limited to particles smaller than 40 μm, and are presented in Table 3.

Table 3
MMDs of Ashes Collected at Tidd in October, 1994 and May, 1995

ID #, date	size range measured, μm	Stokes' MMD, μm
ID # 4097, October, 1994	1.0 - 40	7.0
ID # 4114, October, 1994	1.0 - 40	3.6
ID # 4142, May 1995	0.4 - 40	17
ID # 4144, May 1995	0.6 - 40	11
ID # 4151, May 1995	0.6 - 40	16

In Figure 1, the size distribution data for these five samples obtained with by sieving have been combined with size distribution data obtained with our sedigraph. These data clearly show the increase in particle size induced by bypassing the cyclone.

The ash sample we collected from the tube sheet in the middle plenum (ID # 4151, MP,TS) behaved much more like a free flowing powder than the other samples collected on the same site visit. Based on our sieving measurements, this free flowing ash exhibited the largest particle size distribution of those ashes that were analyzed. When this ash was removed from the tube sheet, it was loose and fluffy, unlike the other ash samples that were consolidated into nodules and deposits. This type of difference is common with fine powders. Powders normally become more free flowing as their particle size distribution becomes coarser.

Chemical analyses of this free flowing ash and one of the nodular ashes (ID # 4144, TP, FC) were performed to determine if differences other than particle size might account for the tendency of the coarser ash to behave like a free flowing powder. Chemical analyses of these two ashes and the size-separated portions generated from them during sieving are summarized in Table 4.

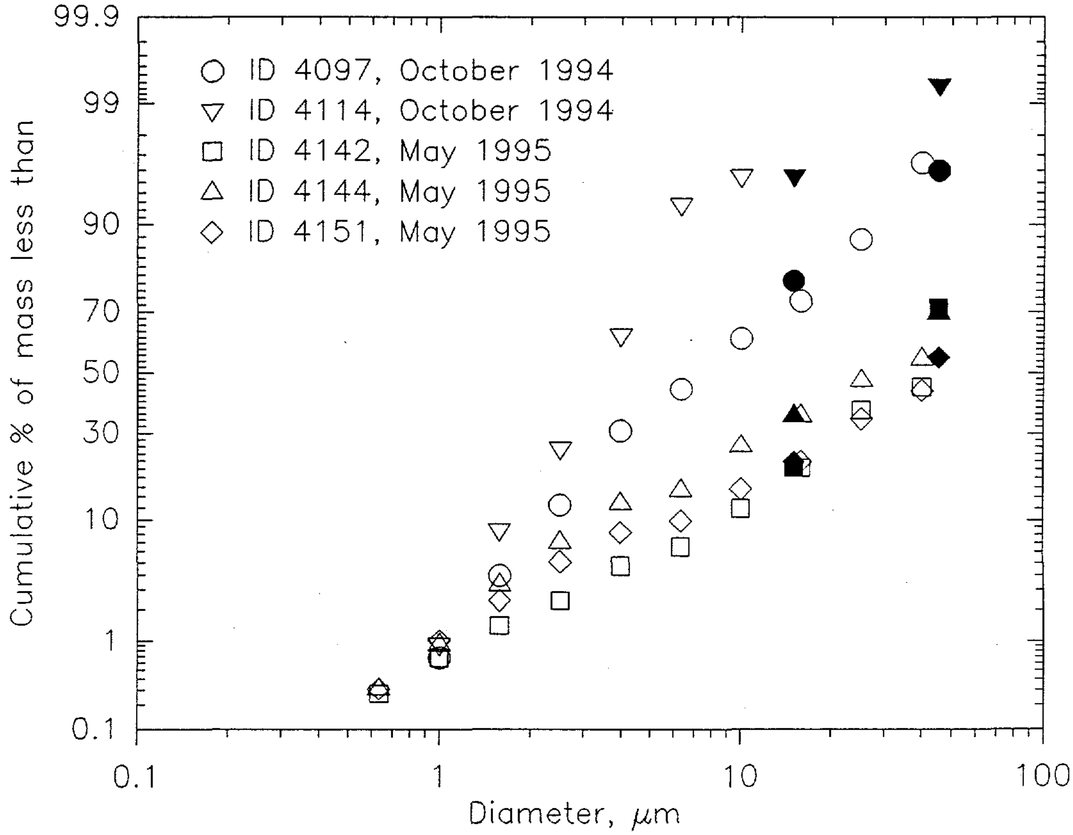


Figure 1. Cumulative size distribution data measured with a sedigraph and sieves for five ashes collected from the Tidd APF. The hollow data points represent sedigraphically obtained Stokes' diameters. The filled data points represent data obtained in the sieve analyses.

Table 4
Chemical Analyses of Tidd Ashes Collected in May, 1995, % wt.

constituent	nodular ash (ID # 4144)			free flowing ash (ID # 4151)		
	all diameters	d > 45 μm	15 μm < d, d < 45 μm	all diameters	d > 45 μm	15 μm < d, d < 45 μm
Li_2O	0.01	0.01	0.01	0.01	0.01	0.01
Na_2O	0.29	0.16	0.23	0.27	0.13	0.20
K_2O	1.3	0.83	1.3	1.2	0.77	1.1
MgO	8.3	11.3	8.5	13.2	16.5	13.5
CaO	14.1	18.1	15.0	20.5	24.7	21.0
Fe_2O_3	7.1	4.8	7.5	8.0	6.2	9.9
Al_2O_3	11.7	7.5	10.8	10.8	7.2	9.5
SiO_2	26.1	17.6	26.0	25.1	19.6	25.6
TiO_2	1.2	0.41	0.46	1.1	0.33	0.49
P_2O_5	0.15	0.12	0.14	0.14	0.10	0.14
SO_3	30.1	38.5	28.5	19.5	22.9	17.4
LOI	13.5	19.6	15.3	1.5	2.4	1.1
soluble $SO_4^=$	29.7	36.0	29.8	22.4	26.3	20.6

These analyses demonstrated that the larger ash particles in both samples (ID # 4144 and ID # 4151) contain more Mg, Ca, SO_3, and have higher LOI values than the smaller particles. The smaller particles are richer in Al ,Na, K, Fe, Ti, and Si. These results suggest that the larger ash particles are derived mainly from the sorbent used in the combustion process, while the smaller particles are derived mainly from the coal. This is supported by comparisons of the concentrations of calcium and magnesium in the two parent samples and their corresponding size fractions. The coarser ash (ID # 4151) contains about 50 % more calcium and magnesium than the finer filter cake ash. These differences suggest that the sorbent-derived ash particles are somewhat larger than the coal-derived ash particles in these samples.

Another interesting difference in ID # 4144 and ID # 4151 is evident in the acid/base ratios as defined in Table 5.

Table 5
Acid/Base Ratios in Tidd Ashes

ID #, location	SO_3/(Ca+Mg)	soluble $SO_4^=$/(Ca+Mg)
4144, TP, FC	1.3	1.3
4151, MP, TS	0.58	0.66

We believe the differences in these ratios is due to the exposure of the filter cake ash (ID # 4144) to much more flue gas than the ash obtained from the passive deposit under the tube sheet (ID # 4151). As flue gas is filtered through the filter cake, additional SO_2 in the flue gas is captured by calcium and/or magnesium still remaining in the ash. Although unreacted sorbent present in the passive ash deposits can still react with SO_2 in the flue gas, the reaction is diminished because the flue gas in direct contact with the ash particles in the deposit is exchanged or refreshed relatively slowly. We attribute the higher LOI of the filter cake ash to the presence of the increased amounts of sulfur captured by chemical adsorption in the filter cake. The comparisons discussed above suggest that it is possible that the increased flowability of the tube sheet ash (ID # 4151, MP, TS) may be partially due to chemical differences as well as differences in size distribution.

We also performed a variety of physical analyses of several of the ashes we collected during our recent site visit to Tidd. The results of these analyses are presented in Table 6.

Table 6
Physical Analyses of Tidd Ashes Collected in May, 1995

ID #, location	nodule porosity, %	uncompacted bulk porosity, %	specific surface area, m^2/g
ID # 4142, TP, TS	73.2	80	--
ID # 4144, TP, FC	69.5	84	--
ID # 4151, MP, TS	--	81	--
ID # 4144A, TP, FC	--	--	6.9
ID # 4144B, TP, FC	--	--	5.1
ID # 4151A, MP, TS	--	--	1.7
ID # 4151B, MP, TS	--	--	1.8

ID # 4144A: diameter $> 45\ \mu m$
ID # 4144B: $15\ \mu m <$ diameter $< 45\ \mu m$
ID # 4151A: diameter $> 45\ \mu m$
ID # 4151B: $15\ \mu m <$ diameter $< 45\ \mu m$

We also produced SEM photographs of six of the seven ashes listed in Table 6. These photographs are presented in Figures 2 through 7. The relative coarseness of ID # 4151, as indicated by the sieve analyses, is also evident in these SEM photographs (Figure 2 compared with Figure 5), and in the values of specific surface area data measured for samples 4144A and 4151A.

The optimum solution to the problems in the Tidd APF caused by the ash aggregates that have been consolidated and strengthened by pervasive eutectic formation is the removal of these aggregates from the APF before the eutectics have had enough time to develop. A large measure of success has been achieved by bypassing the cyclone upstream of the APF. This increases the size distribution of the particles forming the various ash deposits (filter

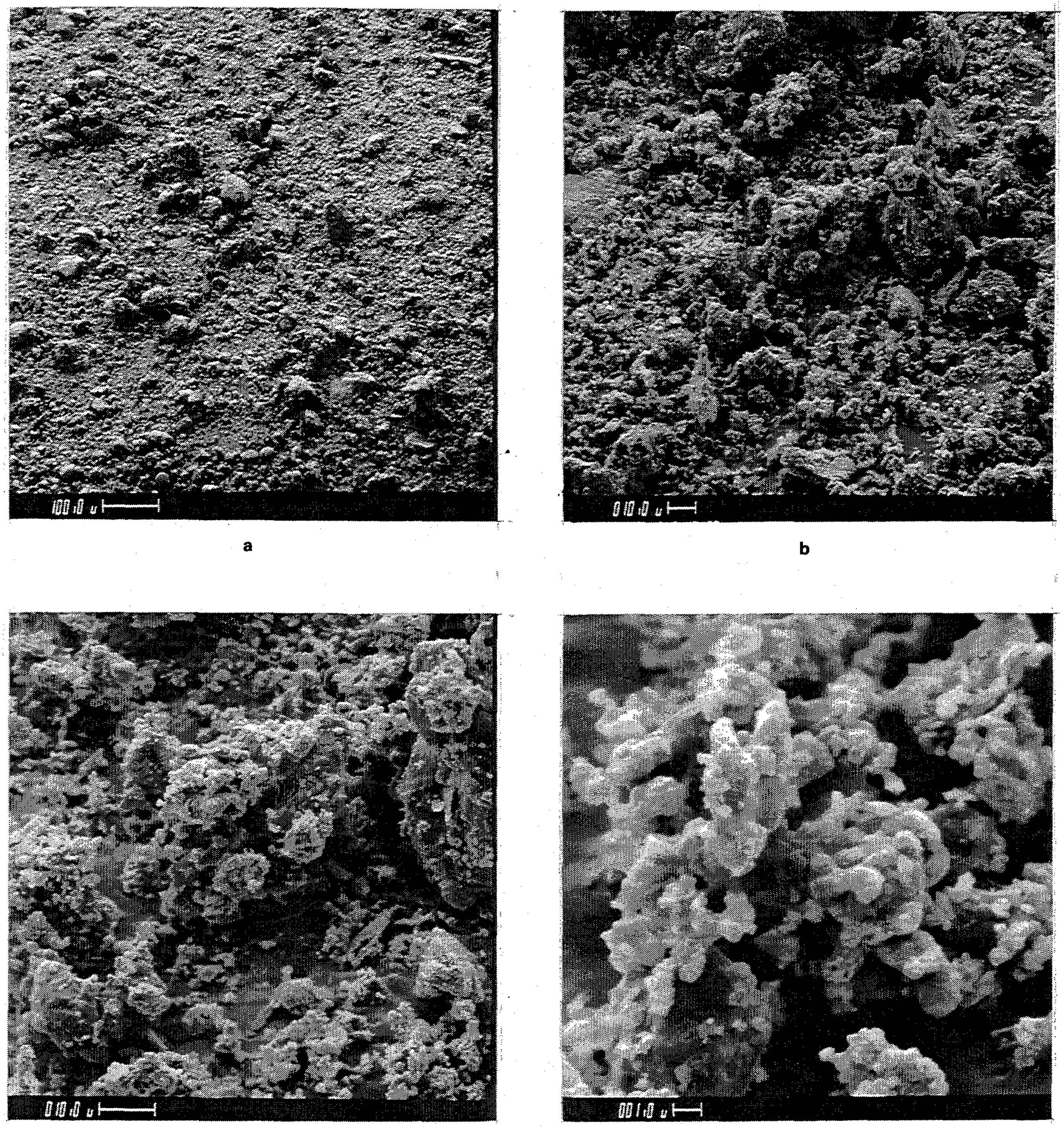
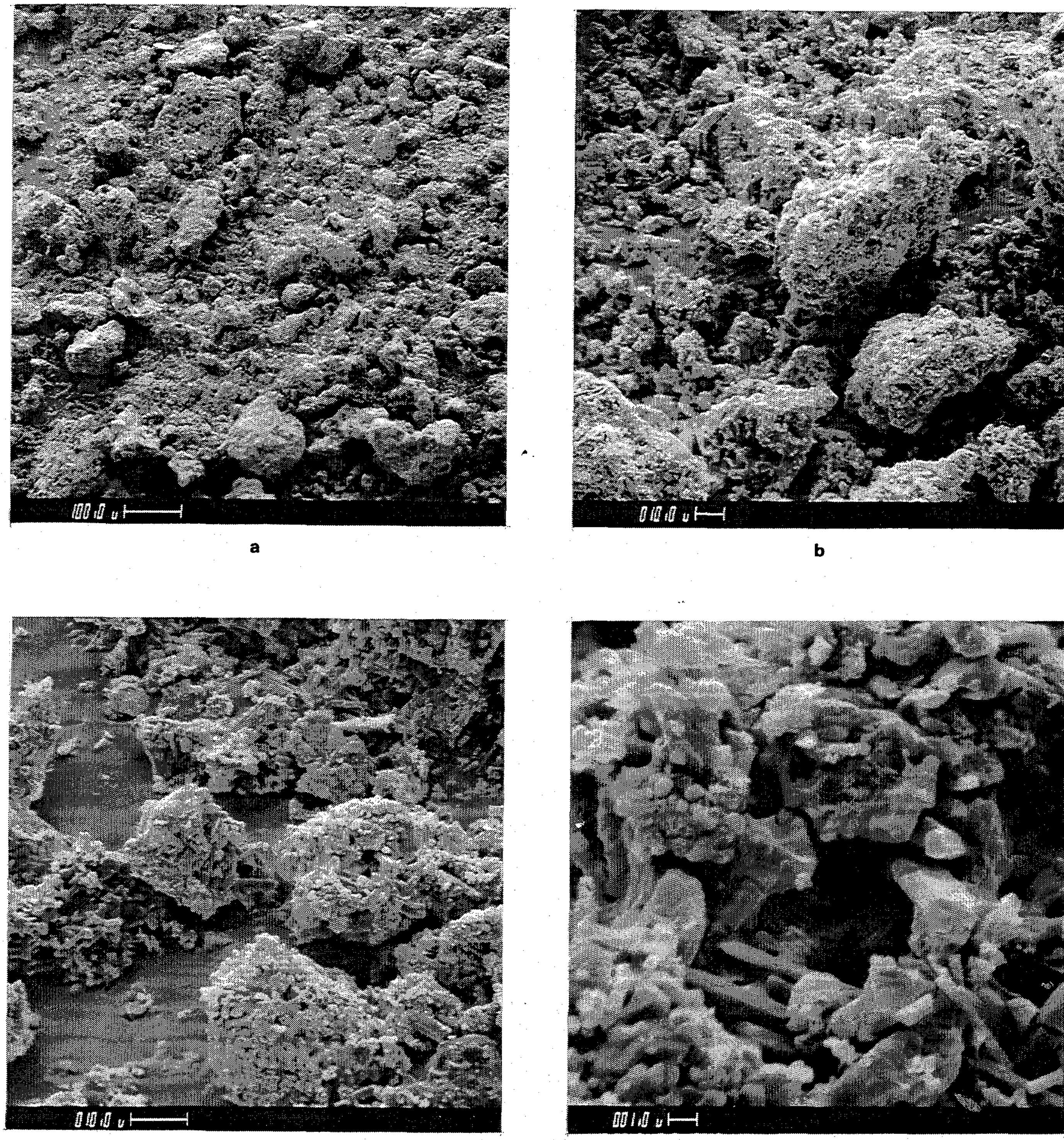


Figure 2. Representative SEM photographs of filter cake ash (ID # 4144) removed from the top plenum at Tidd on May 11, 1995 taken at a) 100X, b) 500X, c) 1000X, and d) 5000X.



a b c d

Figure 3. Representative SEM photographs of filter cake ash (ID # 4144A) removed from the top plenum at Tidd on May 11, 1995 taken at a) 100X, b) 500X, c) 1000X, and d) 5000X. This ash sample contains only particles with diameters greater than 45 μm.

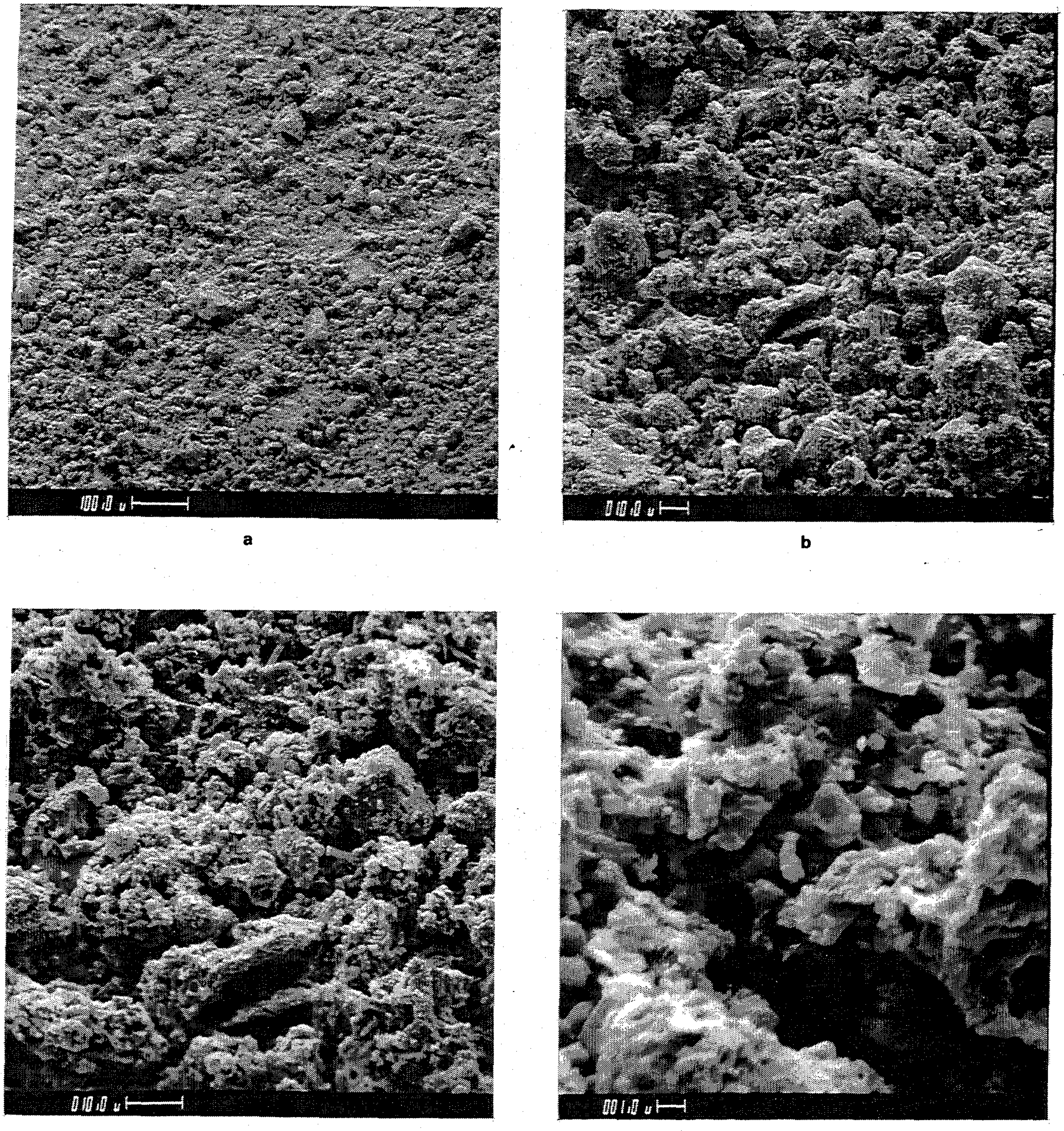


Figure 4. Representative SEM photographs of filter cake ash (ID # 4144B) removed from Tidd on May 11, 1995 taken at a) 100X, b) 500X, c) 1000X, and d) 5000X. This ash sample contains only particles with diameters less than 45 μm and greater than 15 μm.

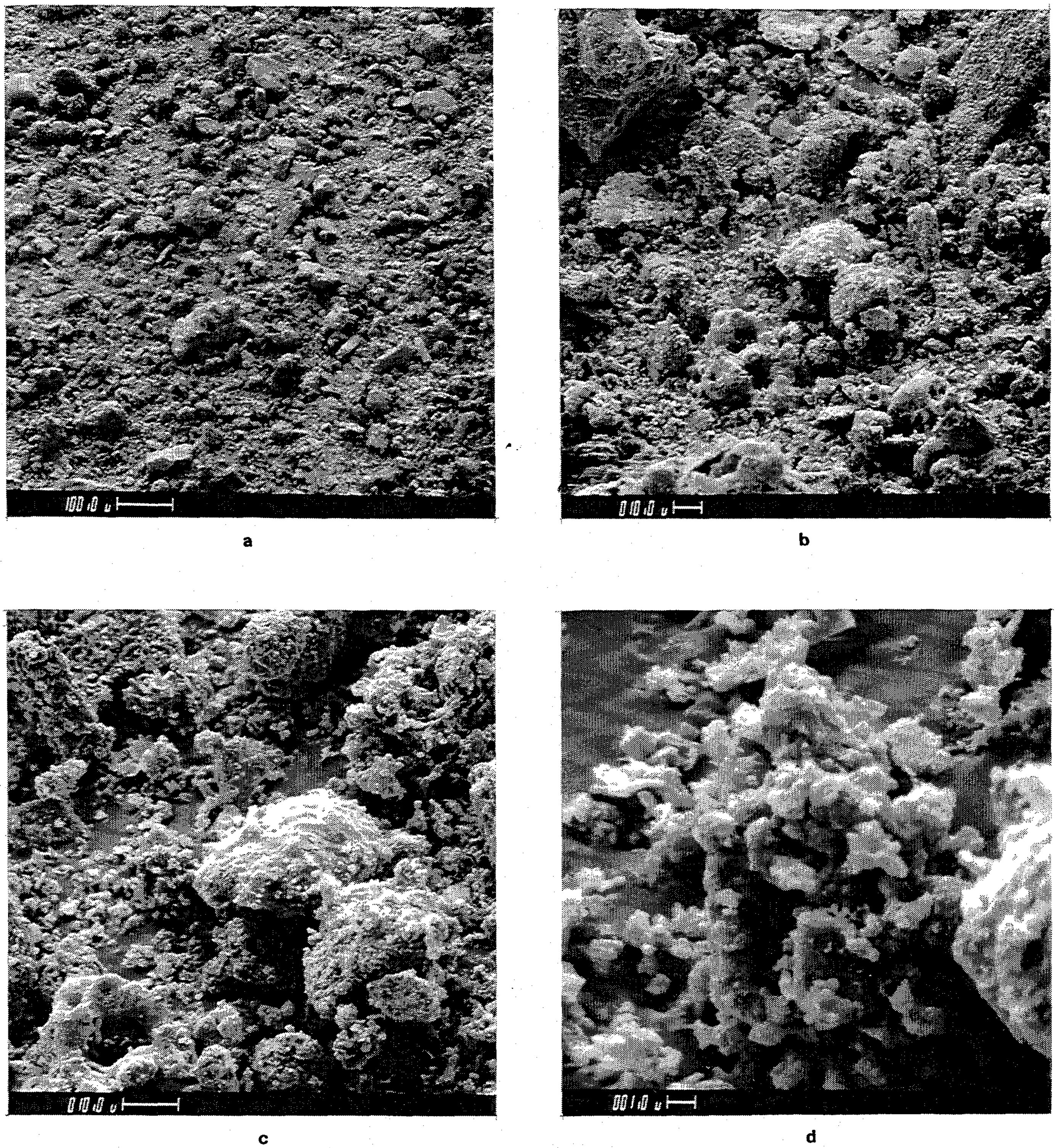
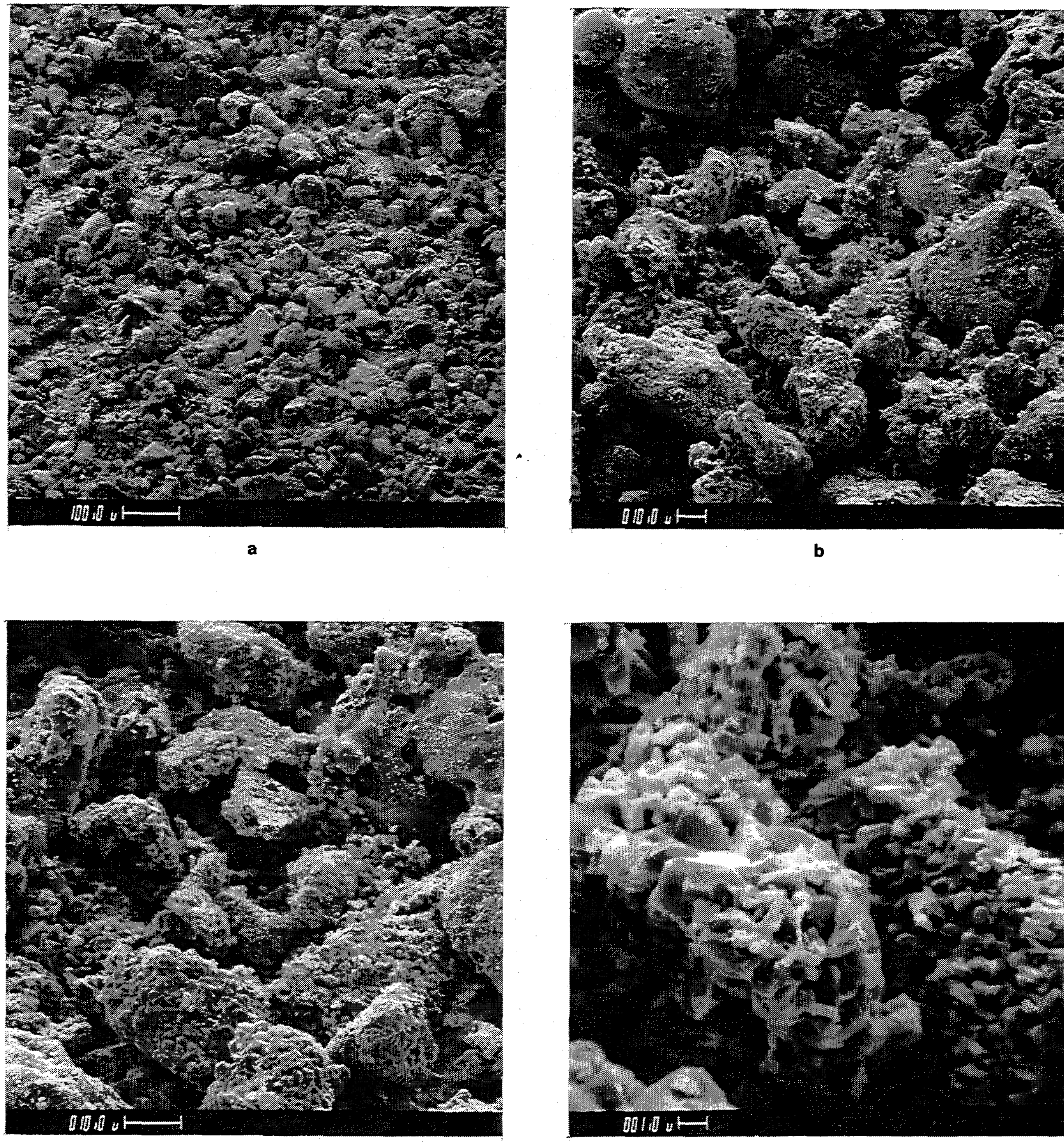


Figure 5. Representative SEM photographs of filter cake ash (ID # 4151) removed from the top plenum at Tidd on May 11, 1995 taken at a) 100X, b) 500X, c) 1000X, and d) 5000X.



a b c d

Figure 6. Representative SEM photographs of filter cake ash (ID # 4151A) removed from the top plenum at Tidd on May 11, 1995 taken at a) 100X, b) 500X, c) 1000X, and d) 5000X. This ash sample contains only particles with diameters greater than 45 μm.

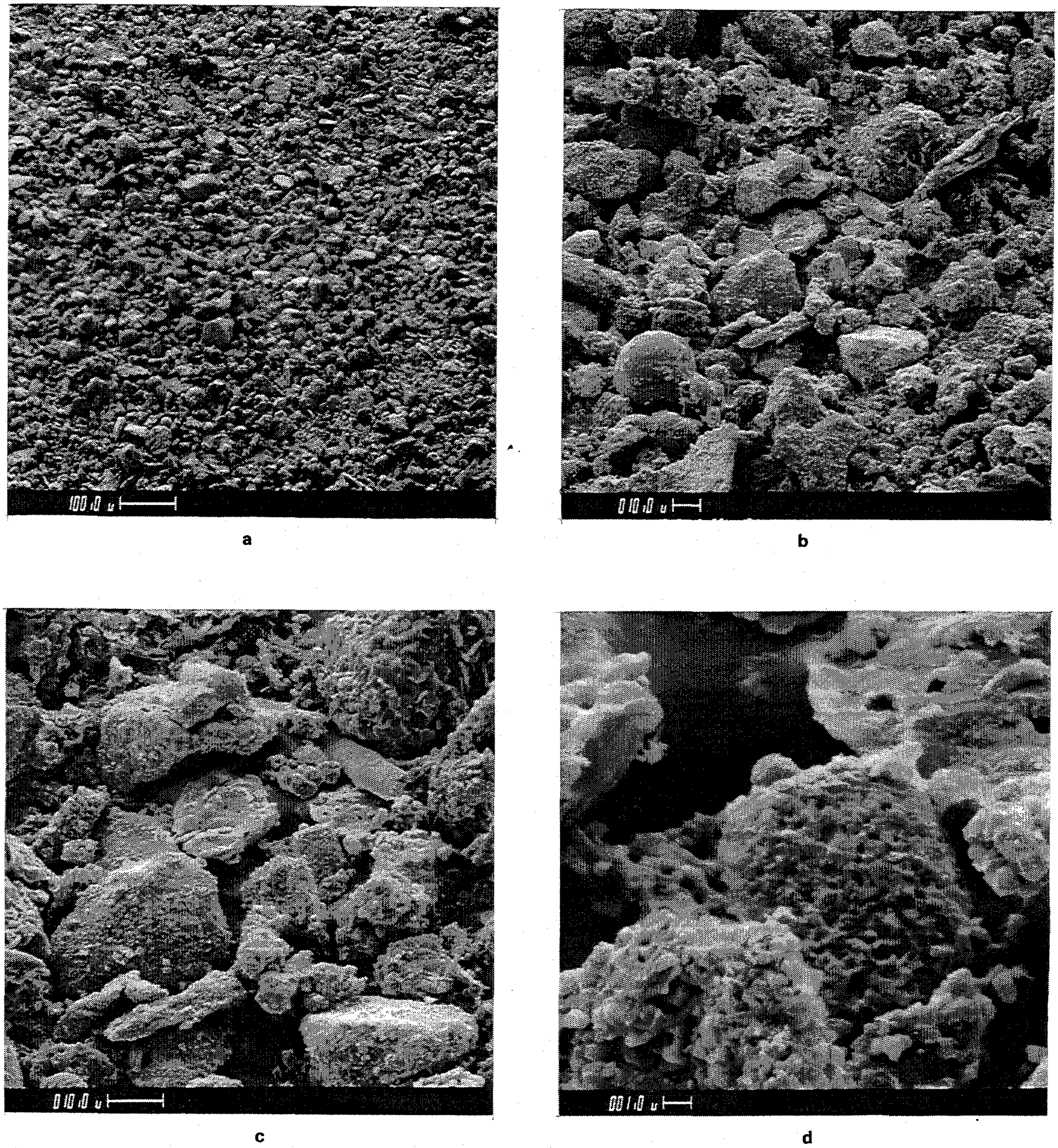


Figure 7. Representative SEM photographs of filter cake ash (ID # 4151B) removed from Tidd on May 11, 1995 taken at a) 100X, b) 500X, c) 1000X, and d) 5000X. This ash sample contains only particles with diameters less than 45 μm and greater than 15 μm.

cakes and passive deposits), thereby decreasing their inherent cohesivity. Although particle size is probably the primary reason for these decreases in cohesivity, the somewhat different chemical composition of these larger particles may also influence cohesivity. These agglomerates of lower cohesivity do not have sufficient strength to remain in the APF long enough to undergo consolidation. Gravity and/or vibration cause them to fall off the surface on which they initially formed.

TASK 2 RESEARCH ACTIVITIES

Mechanical and thermal testing of Dupont/Lanxide PRD-66, Dupont composite, 3M composite, and IF and P Fibrosics is continuing. A test matrix for these materials is shown in Table 7. Hoop tensile strengths for PRD-66, Dupont composite, and 3M composite and axial compressive results for IF and P Fibrosics have already been reported. Thermal expansion values measured at 1500 °F are presented in Table 8.

Table 7
Preliminary Screening Test Matrix for Dupont PRD-66, Dupont Composite, 3M Composite, and IF and P Fibrosics Materials

Material	Test/Property	Orientation	Temperature, °F 70 1700
PRD-66, Dupont composite, and 3M composite	tension	hoop	5*
PRD-66, Dupont composite, and 3M composite	tension after 100 hr. heat soak at 1700 °F	hoop	5*
PRD-66, Dupont composite, and 3M composite	thermal expansion	hoop	2------------------->
IF and P Fibrosics	compression	axial	3
IF and P Fibrosics	thermal expansion	axial	2------------------->

* Three replicates for 3M composite

Table 8
Measured Thermal Expansion Values

Material	Orientation	Thermal Expansion at 1500 °F
IF and P	axial	0.0047 in./in.
PRD-66	diametral	0.0033 in./in.
3M composite	diametral	0.0025 in./in.

Testing of new Refractron and Schumacher candle filter materials is also in progress. A test matrix for these materials is shown in Table 9. Cutting plans are shown in Figures 8 through 11. Hoop tensile results are shown in Table 10 for Refractron and Table 11 for Schumacher. Creep testing of Refractron is now in progress. No creep was detected after ~150 hours at 1550 °F and creep testing is proceeding at 1600 °F. Schumacher creep specimens have been machined and testing of these specimens will follow testing of Refractron.

Table 9
Test Matrix for New Schumacher and Refractron Candle Filters

Material	Test/Property	Orientation	Temperature, °F		
			70	1550	1700
Refractron	tensile	hoop	6		
Refractron	tensile creep	axial		4--------------	>
Schumacher	tensile	hoop	3		
Schumacher	tensile creep	axial		4--------------	>

Table 10
Hoop Tensile Results for New Refractron Candle Filter Material

Candle identification	Specimen number	Test temperature, °F	Specimen ID, in.	Specimen OD, in.	Ultimate tensile strength, psi
2-469	Tn-hoop-1	70	1.54	2.39	2000
2-469	Tn-hoop-2	70	1.54	2.38	2100
2-469	Tn-hoop-3	70	1.54	2.38	1980
4-471	Tn-hoop-4	70	1.54	2.38	2190
4-471	Tn-hoop-5	70	1.55	2.38	2470
4-471	Tn-hoop-6	70	1.54	2.38	2040

Average ultimate tensile strength = 2130 psi, standard deviation = 183 psi, COV = 8.6%.

Table 11
Hoop Tensile Results for New Schumacher Candle Filter Material

Candle identification	Specimen number	Test temperature, °F	Specimen ID, in.	Specimen OD, in.	Ultimate tensile strength, psi
S199/315E PT-20	Tn-hoop-1	70	1.54	2.37	1740
S199/315E PT-20	Tn-hoop-2	70	1.54	2.37	1720
S199/315E PT-20	Tn-hoop-3	70	1.53	2.37	1620

Average ultimate tensile strength = 1690 psi, standard deviation = 64 psi, COV = 3.8%.

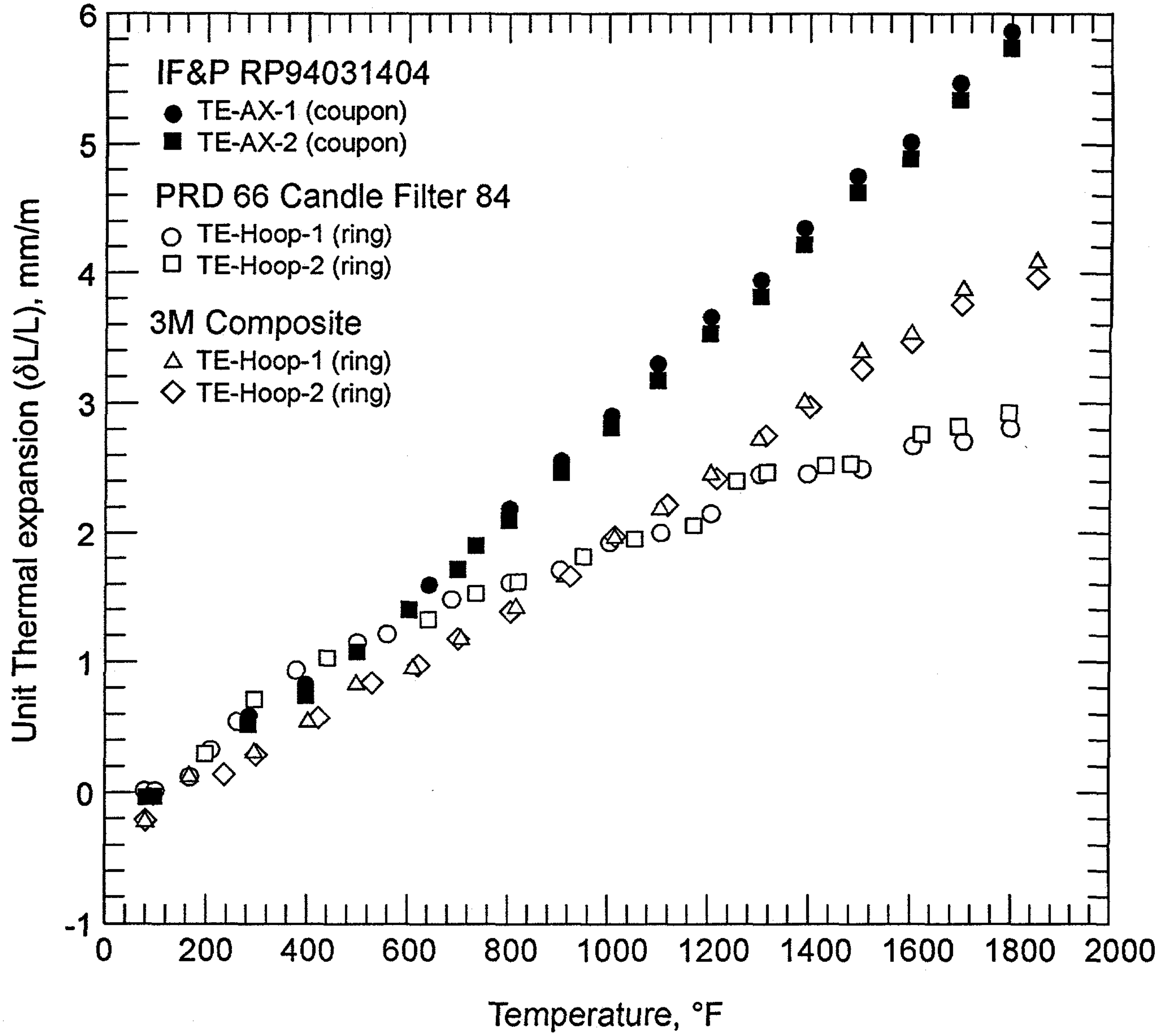
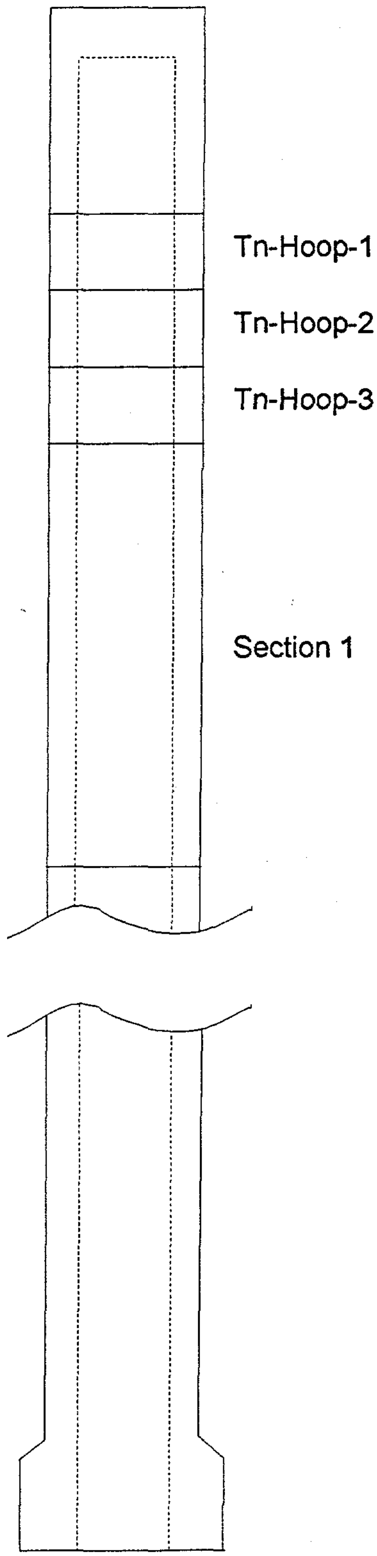
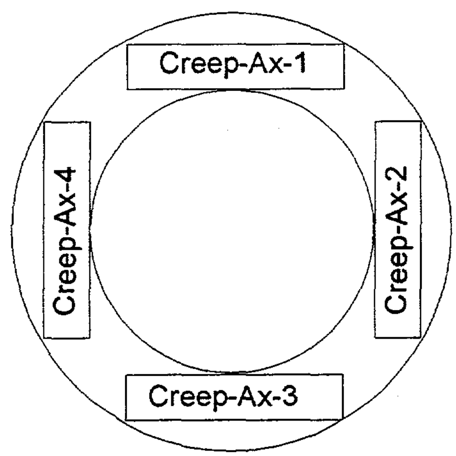


Figure 8. Thermal expansion of IF and P Fibrosics, Dupont PRD-66, and 3M composite materials.





Cutting Plan for Section 1

Figure 9. Cutting plan for Refractron candle filter 2-469.

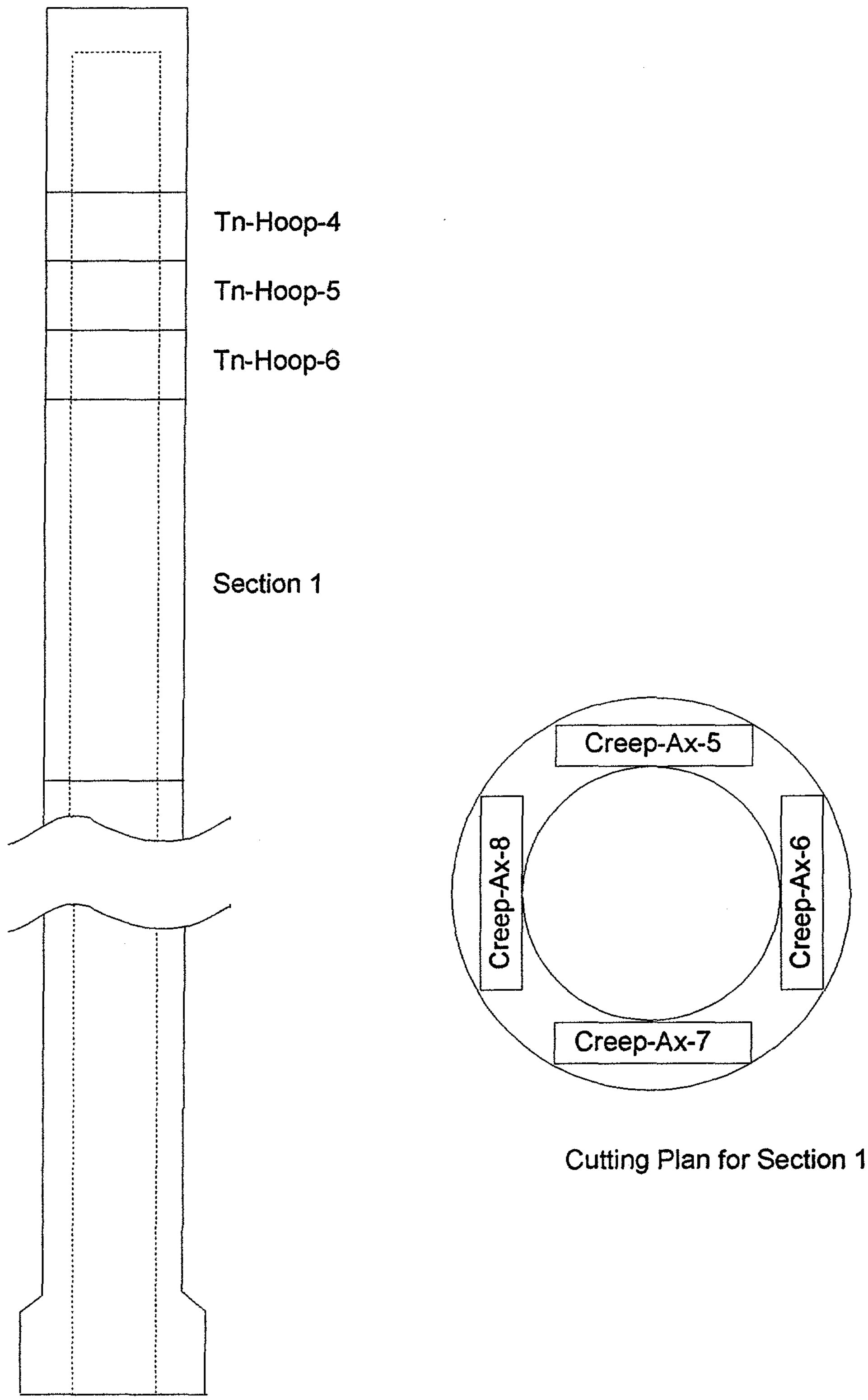
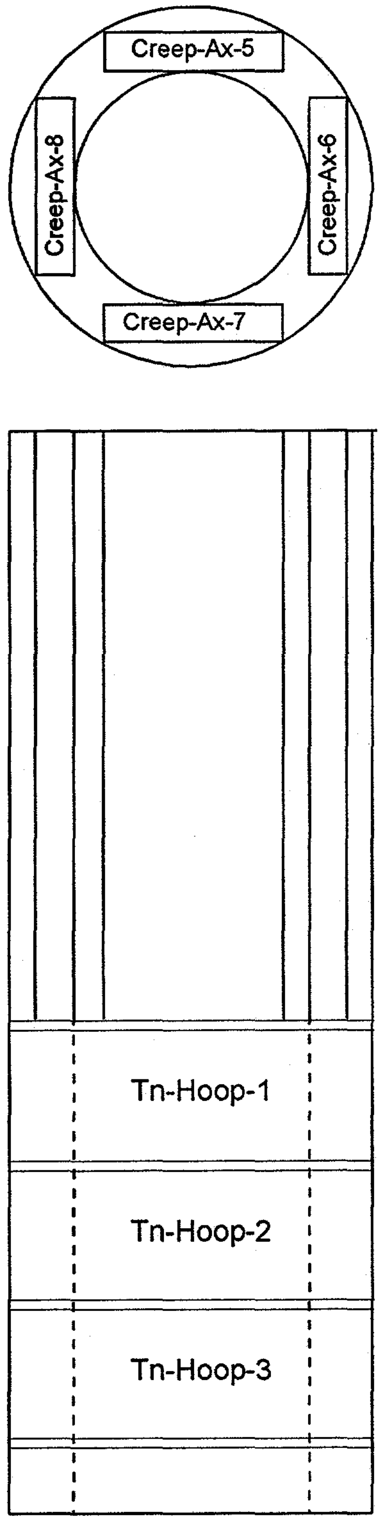


Figure 10. Cutting plan for Refractron candle filter 4-471.



Final Specimen Dimensions (Inches):

Creep-Ax—0.250 x 0.9995 x 7.00

Tn-Hoop—as received i.d.x as received o.d. x 1.000

Figure 11. Cutting plan for Schumacher candle filter S199/315E.

FUTURE WORK

Plans for the next quarter include completion of the design of the uncompacted bulk porosity test device described in our second quarterly report, continued work on the interactive data base of HGCU ash characteristics, and characterization of samples obtained from the General Electric gasification facility in Schenectady, NY. Creep testing of Refractron and Schumacher candle filter materials will continue.

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PARTICULATE HOT GAS STREAM CLEANUP
TECHNICAL ISSUES

DOE