DOE/MC/31160 -- 5224
(DE96004499)

Particulate Hot Gas Stream Cleanup Technical Issues

Quarterly Report
October - December 1995

March 1996

Work Performed Under Contract No.: DE-FC21-94MC31160

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

By
Southern Research Institute
2000 Ninth Avenue South
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EXECUTIVE SUMMARY

This is the fifth 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 measured the size distribution of a sample of ash from the METC MGCR (modular gas cleanup rig) and of samples of gasifier ashes from tests carried out by Texaco and KRW between 1988 and 1991. These measurements complement data we have obtained describing the specific surface area, uncompacted bulk porosity, specific gas flow resistance, drag-equivalent diameter, and tensile strength of these samples. We have also characterized the response of the METC MGCR ash to compacting forces. We have determined that normal filtering pressure drops may be sufficient to reduce the porosity of a filter cake formed from the MGCR gasification ash from about 95 % down to about 90 %. Our permeability data indicate that this degree of consolidation would increase filtering pressure losses by a factor of four.

We have analyzed the MGCR ash to determine if it contains fullerenes that might affect its cohesivity or tensile strength. We recently selected Microsoft Access® as the software that we will use to construct the interactive data base of HGCU ash characteristics. We are structuring the HGCU data base to accept the types of data and information that we will include in it.

Task 2 efforts during the past quarter focused on mechanical and thermal testing of new Schumacher candle filter material. Creep evaluations of the New Schumacher material will continue by increasing temperature and/or stress levels. We also have machined specimens of two compositions of alumina-mullite material received from Blash Precision Ceramics in preparation for tensile tests which will begin next quarter.

INTRODUCTION

This is the fifth 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 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. These problems have been attributed to ash characteristics, durability of the ceramic filter elements, and specific limitations of the filter design. In addition to these 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 235 ash samples from eleven 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.

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, and Schumacher 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 performed additional analyses on a sample of ash from the MGCR facility located at DOE/METC. The sample was provided by Richard Dennis of DOE/METC who requested a full analysis of this sample. We also measured the size distributions of gasifier ashes from tests carried out by Texaco and KRW between 1988 and 1991. These additional analyses supplemented our measurements of specific surface area, uncompacted bulk porosity, specific gas flow resistance, drag-equivalent diameter, and tensile strength. The samples we studied during the past quarter are described in Table 1.

Table 1
Gasifier Ash Samples from the HGCU Data Base Characterized during the Past Quarter

ID #	Source	Brief description
2550	KRW	fluidized bed gasification char (82 % carbon)
2556	KRW	TP-037-9: C-110 outlet composite
2557	KRW	TP-037-9: C-115 gasifier outlet composite
2558	KRW	TP-037-9: C-120 outlet composite
2559	KRW	TP-037-9: SC 41 hopper composite
2560	KRW	TP-037-9: C-121 hopper (4/25/88)
2561	KRW	TP-037-9: C-121 hopper (5/1/88)
2562	KRW	TP-037-9: C-121 hopper (4/28/88)
2678	Texaco M.R.L.	run L8902-04 filter vessel ash pot solids
4170	DOE/METC	modular gas cleanup rig pilot-scale gasifier

The results of our mineral analysis of the DOE/METC MGCR gasifier ash sample (ID # 4170) were presented in our last quarterly report. Like other gasifier ashes, the most distinctive chemical characteristic of this sample was its high value of loss-on-ignition

(LOI) due to the high carbon content remaining in the ash. The LOI of the DOE/METC MGCR gasifier ash was measured to be 35.9 % by wt. Based on literature describing the identification of chemical compounds in carbonaceous soot, we performed some analyses to determine if a significant proportion of the carbon in this gasifier ash might be present as fullerene molecules. We believed it might be possible for fullerenes to form an ultrafine powder that would serve as a physical conditioning agent for the primary ash particles. Ultrafine powders, such as fumed silica, can be used to increase the flowability of powders having size distributions similar to fly ashes. However, analysis of the DOE/METC MGCR ash with a mass spectrometer failed to identify any significant quantity of C_{60}, which is the most common of the fullerenes.

We also used our sedigraph to measure the size distributions of the ten gasification ashes listed in Table 1. These data are presented in Figures 1 through 10. The mass median diameter data we obtained in these measurements are summarized with the results of prior standard physical analyses of these gasification ashes in Tables 2 and 3.

Table 2
Physical Characteristics of KRW Gasification Ashes*

quantity ID #	2550	2556	2557	2558	2559	2560	2561	2562
specific surface area, m^2/g	278	112	108	184	135	218	381	293
Stokes' MMD, µm	0.22	16	17	1.4	16	3.1	0.38	0.36
uncompacted bulk porosity, %	94	93	93	95	92	95	95	96
drag-equivalent diameter, µm	0.14	0.99	0.93	0.28	0.95	0.26	0.26	0.25
specific gas flow resistance, in H_2O·min·ft/lb	45	1.3	1.5	6.4	2.0	7.4	7.8	4.5
tensile strength, N/m^2	3.5	0.3	0.3	2.5	0.3	3.0	1.8	1.1
true particle density, g/cm^3	2.17	2.11	2.08	2.14	2.18	2.12	2.12	2.17

* Quantities measured prior to the most recent reporting quarter are shaded.

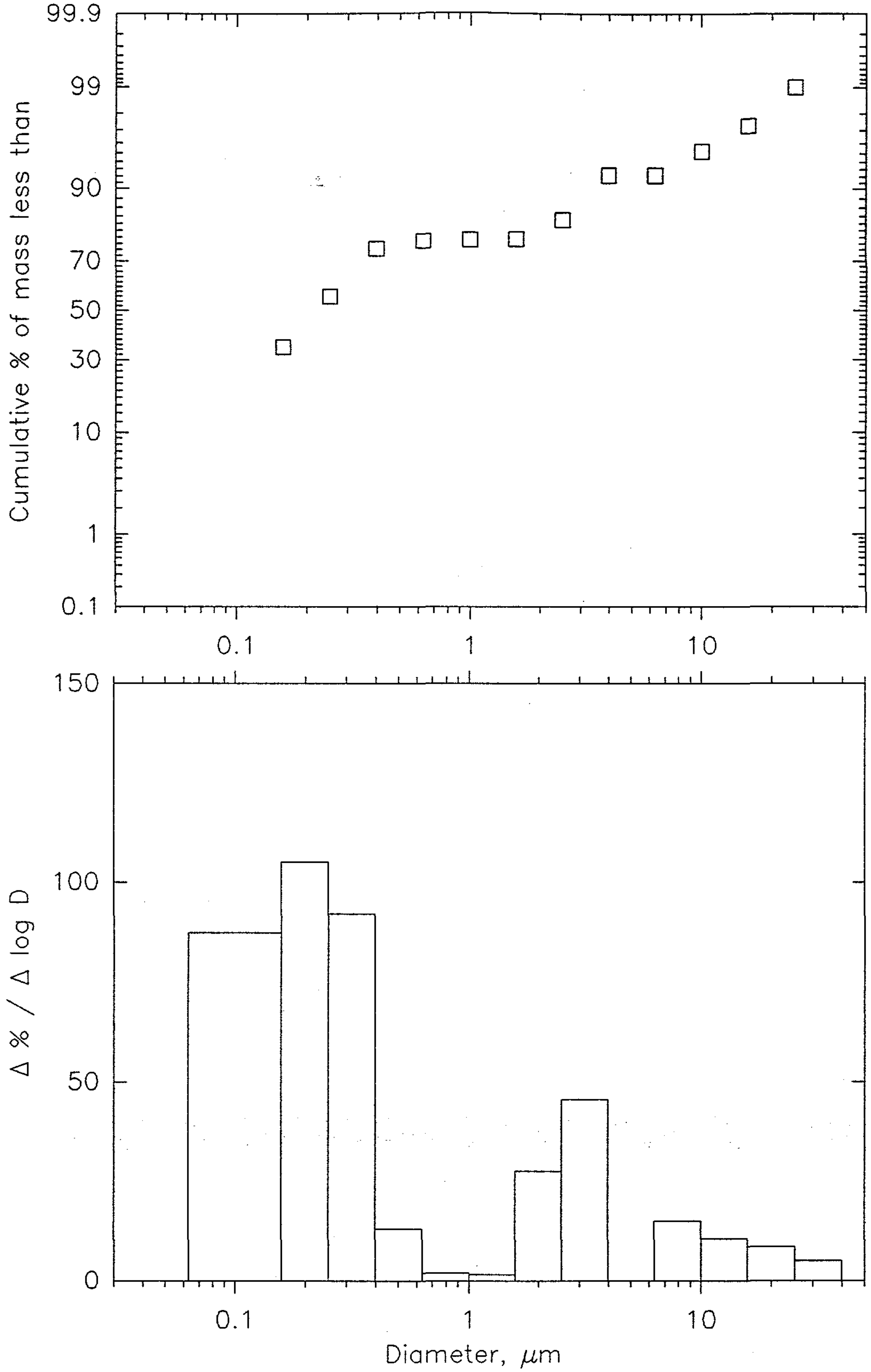

Figure 1. Cumulative and differential size distribution data measured for KRW fluidized bed gasification char (ID # 2550) measured with a Shimadzu SA-CP4 Centrifugal Particle Size Analyzer. The Stokes MMD of this distribution is 0.22 μm, and its geometric standard deviation is 5.2. (This size distribution data includes the assumption that the char contains no particles smaller than 0.063 μm.)

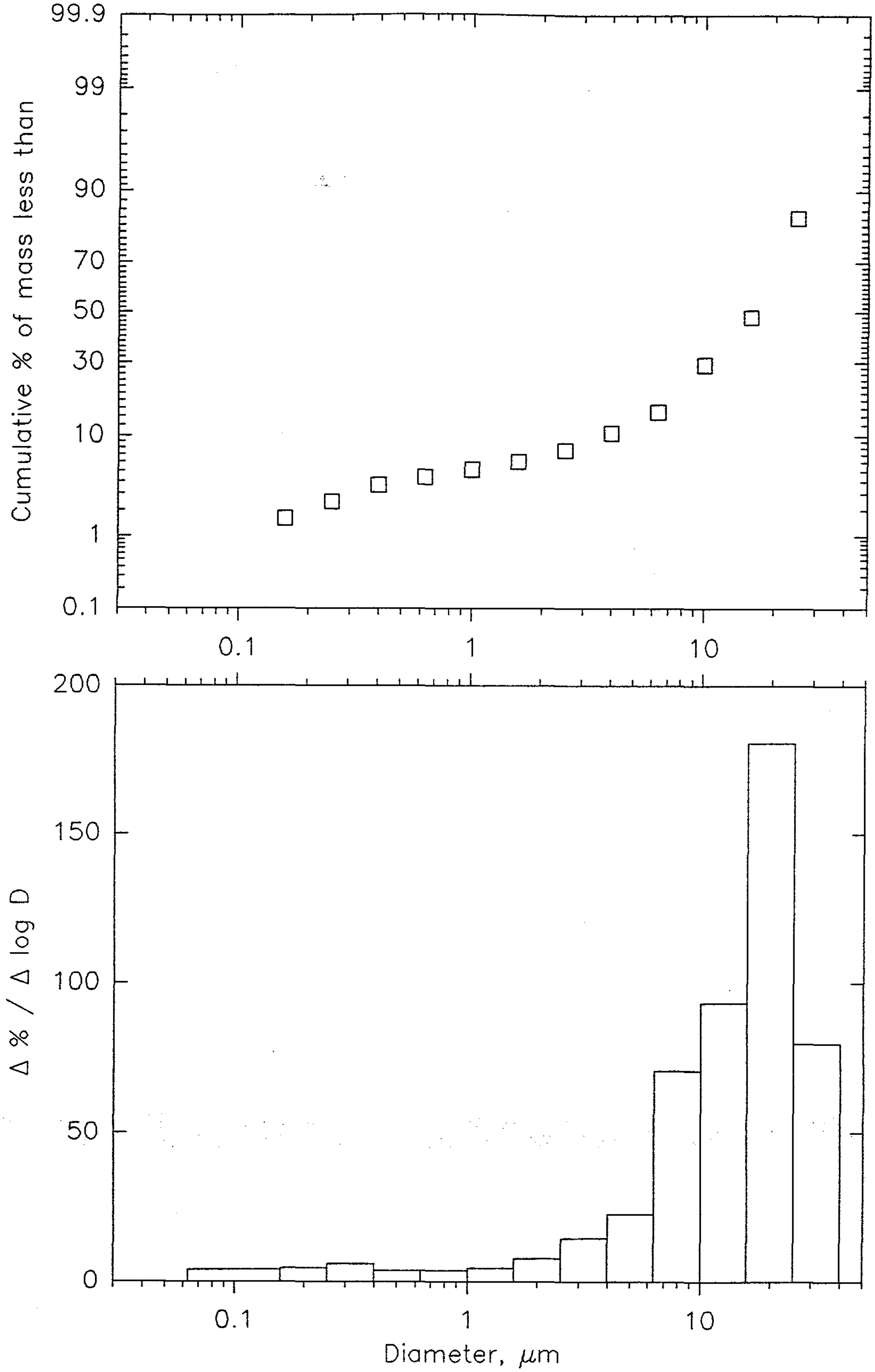

Figure 2. Cumulative and differential size distribution data measured for KRW C-110 outlet composite gasification char (ID # 2556) measured with a Shimadzu SA-CP4 Centrifugal Particle Size Analyzer. The Stokes MMD of this distribution is 16 μm, and its geometric standard deviation is 2.0. (This size distribution data includes the assumption that the char contains no particles smaller than 0.063 μm.)

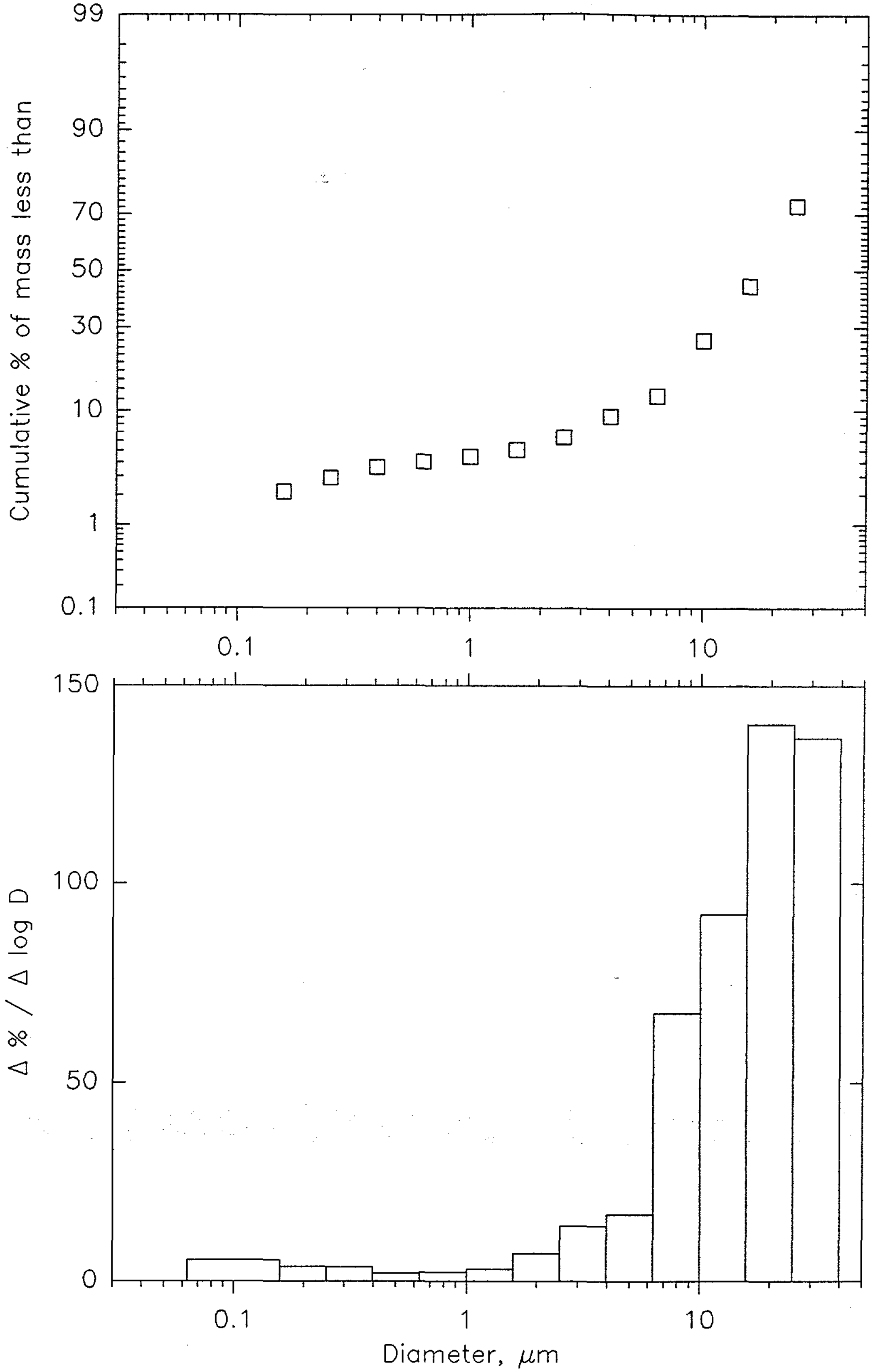

Figure 3. Cumulative and differential size distribution data measured for KRW C-115 outlet composite gasification char (ID # 2557) measured with a Shimadzu SA-CP4 Centrifugal Particle Size Analyzer. The Stokes MMD of this distribution is 17 μm, and its geometric standard deviation is 2.1. (This size distribution data includes the assumption that the char contains no particles smaller than 0.063 μm.)

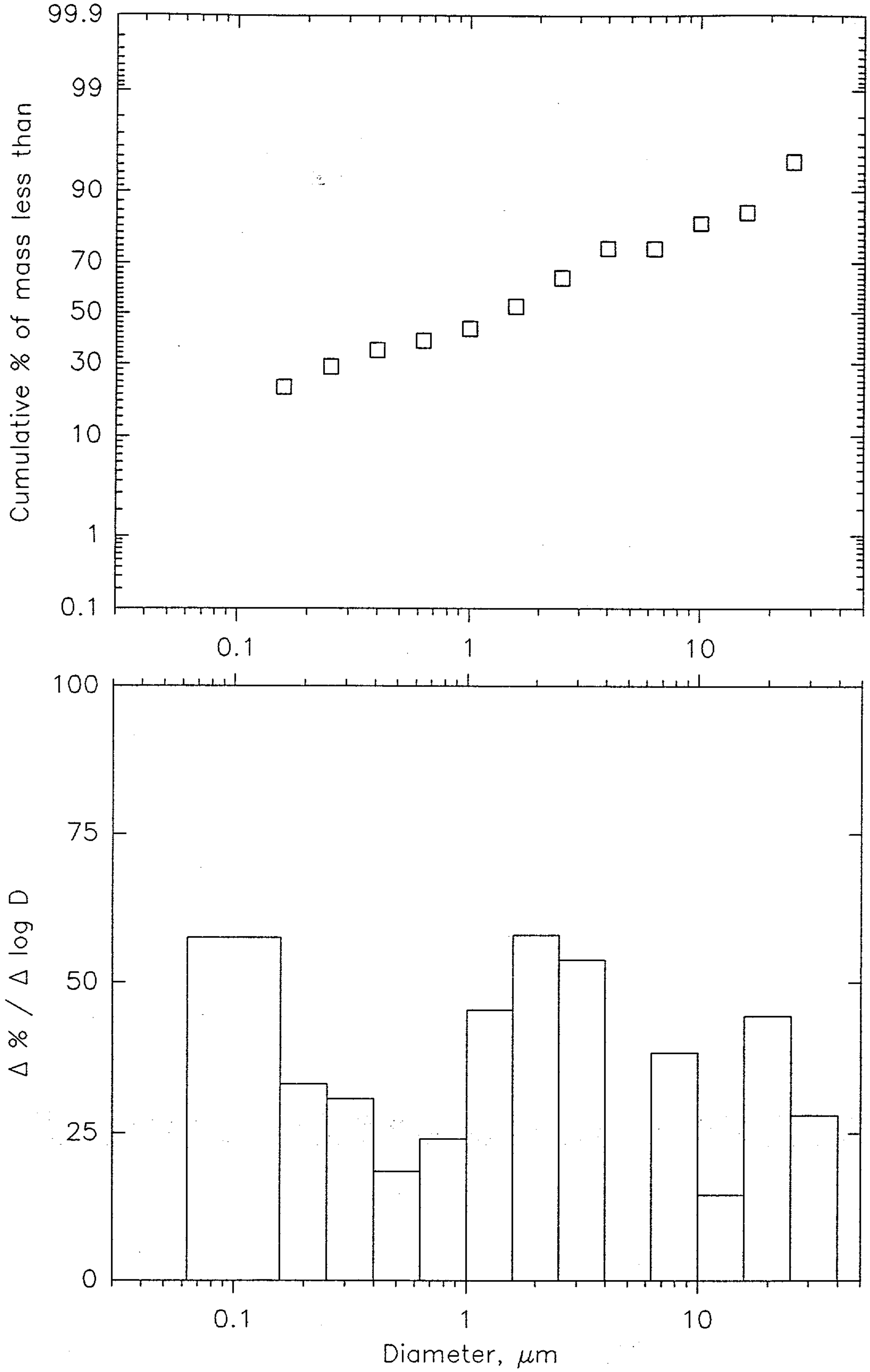

Figure 4. Cumulative and differential size distribution data measured for KRW C-120 outlet composite gasification char (ID # 2558) measured with a Shimadzu SA-CP4 Centrifugal Particle Size Analyzer. The Stokes MMD of this distribution is 1.4 μm, and its geometric standard deviation is 10. (This size distribution data includes the assumption that the char contains no particles smaller than 0.063 μm.)

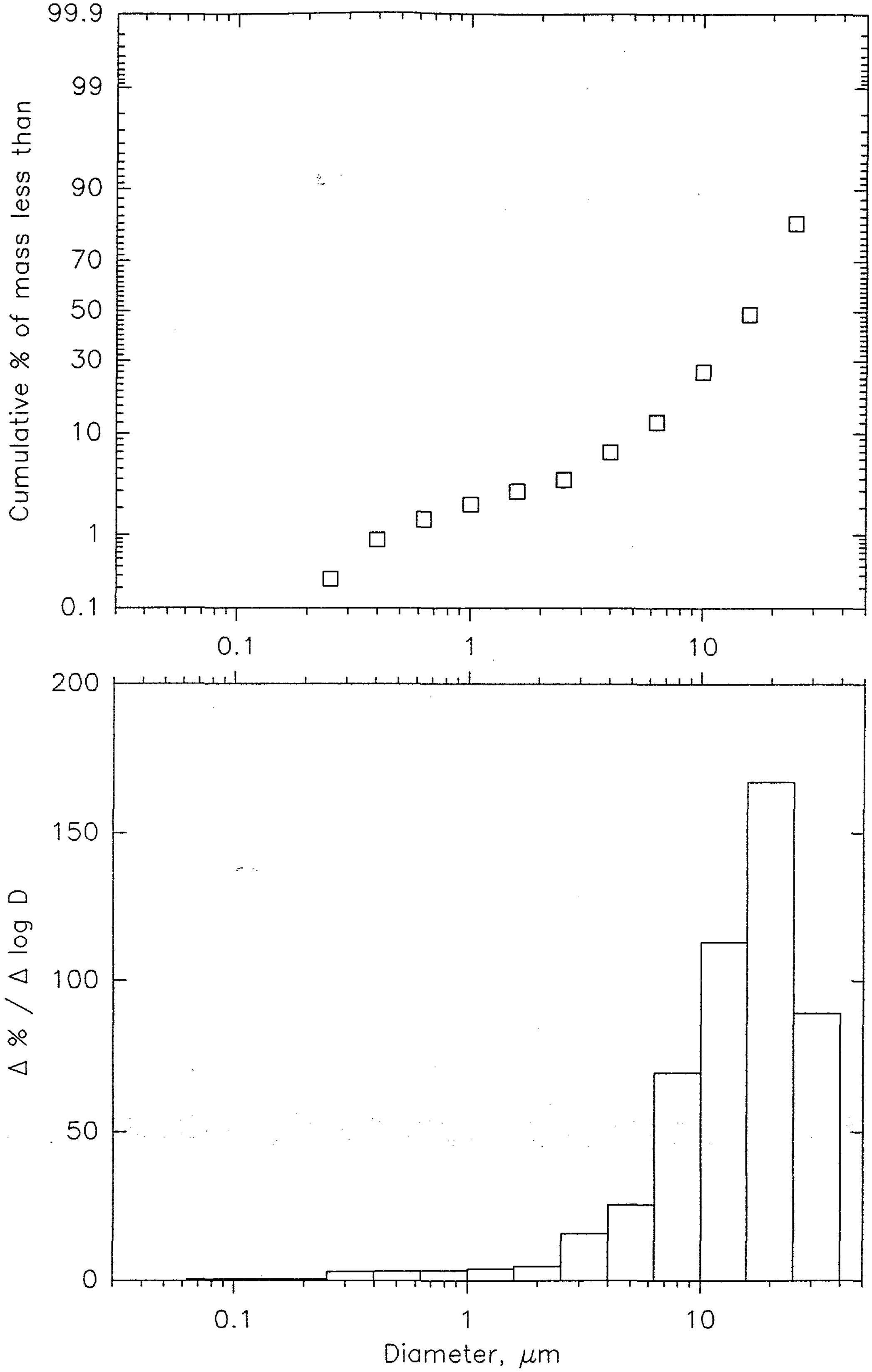

Figure 5. Cumulative and differential size distribution data measured for KRW SC 41 hopper composite gasification char (ID # 2559) measured with a Shimadzu SA-CP4 Centrifugal Particle Size Analyzer. The Stokes MMD of this distribution is 16 μm, and its geometric standard deviation is 1.9. (This size distribution data includes the assumption that the char contains no particles smaller than 0.063 μm.)

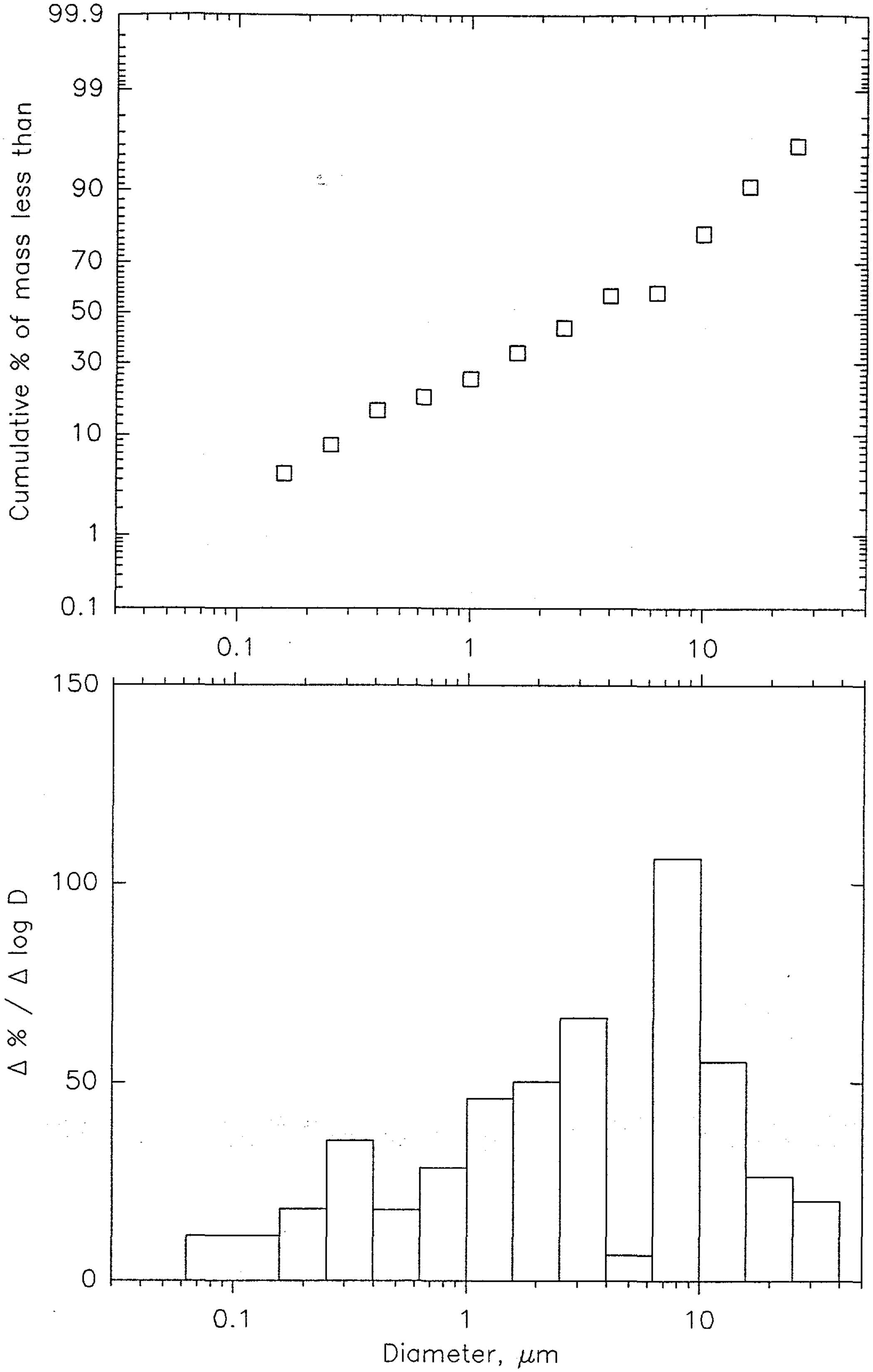

Figure 6. Cumulative and differential size distribution data measured for KRW C-121 hopper (4/25/88) gasification char (ID # 2560) measured with a Shimadzu SA-CP4 Centrifugal Particle Size Analyzer. The Stokes MMD of this distribution is 3.1 μm, and its geometric standard deviation is 5.2. (This size distribution data includes the assumption that the char contains no particles smaller than 0.063 μm.)

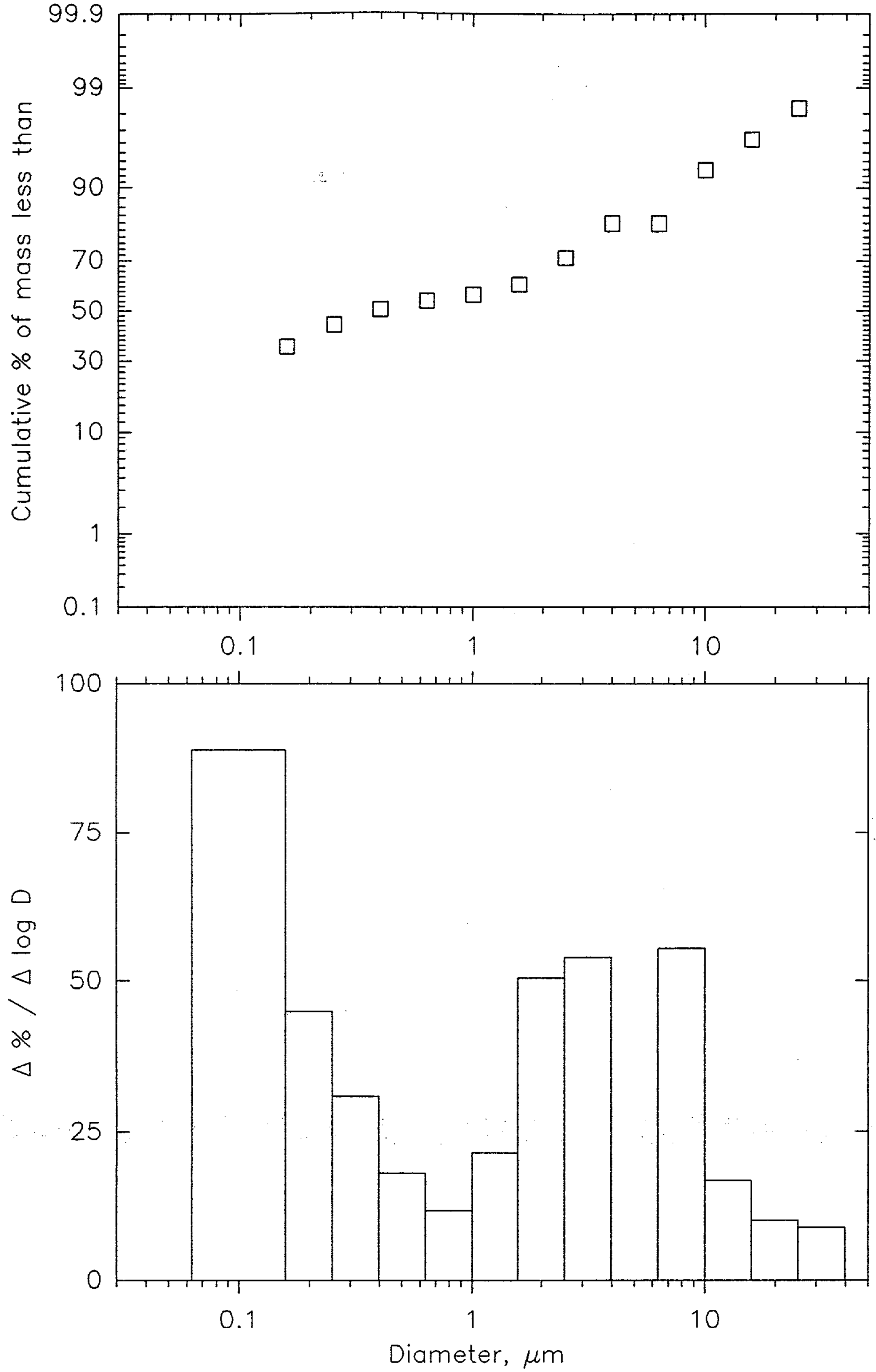

Figure 7. Cumulative and differential size distribution data measured for KRW C-121 hopper (5/1/88) gasification char (ID # 2561) measured with a Shimadzu SA-CP4 Centrifugal Particle Size Analyzer. The Stokes MMD of this distribution is 0.38 μm, and its geometric standard deviation is 8.5. (This size distribution data includes the assumption that the char contains no particles smaller than 0.063 μm.)

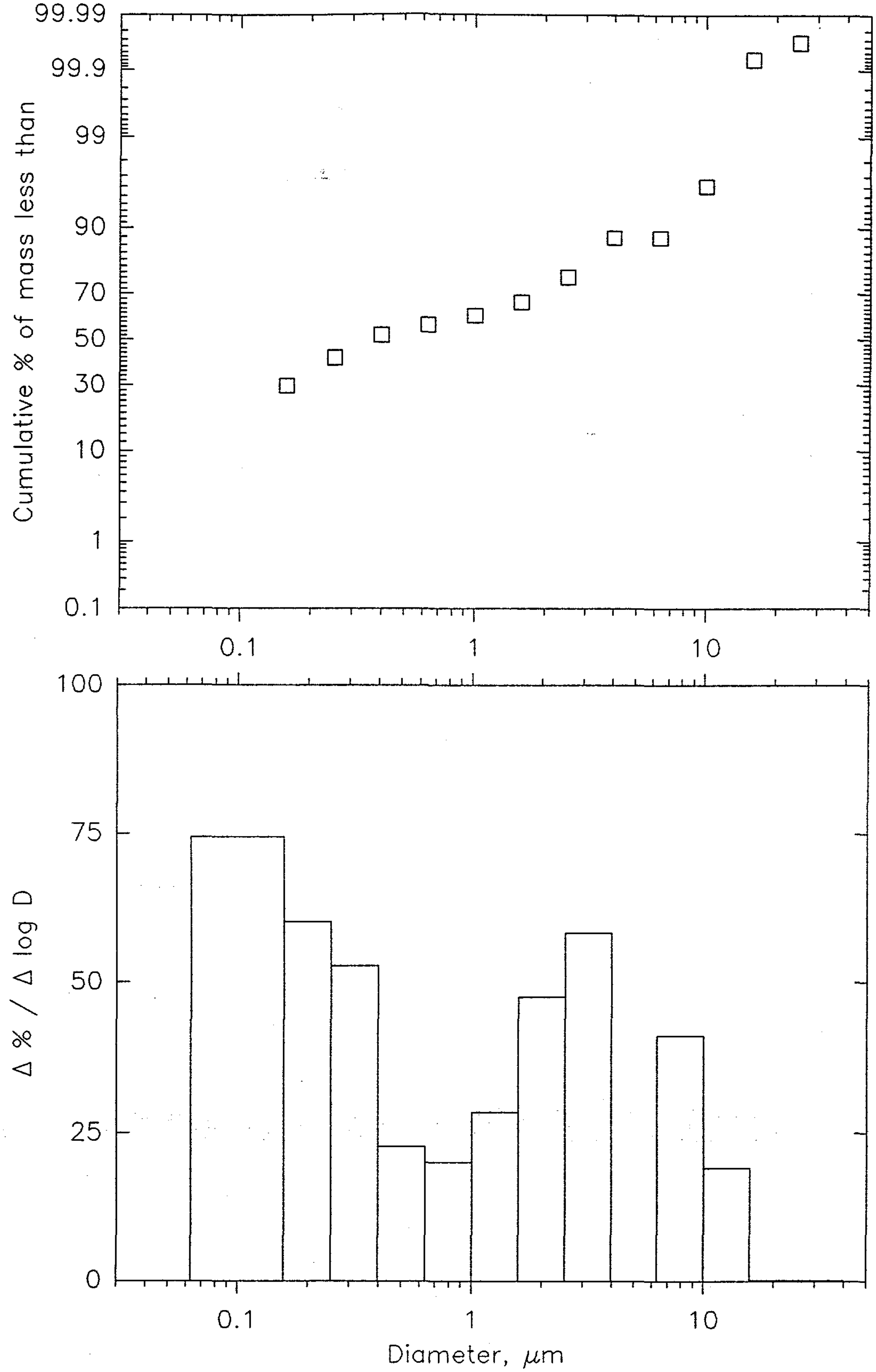

Figure 8. Cumulative and differential size distribution data measured for KRW C-121 hopper (4/28/88) gasification char (ID # 2562) measured with a Shimadzu SA-CP4 Centrifugal Particle Size Analyzer. The Stokes MMD of this distribution is 0.36 μm, and its geometric standard deviation is 5.8. (This size distribution data includes the assumption that the char contains no particles smaller than 0.063 μm.)

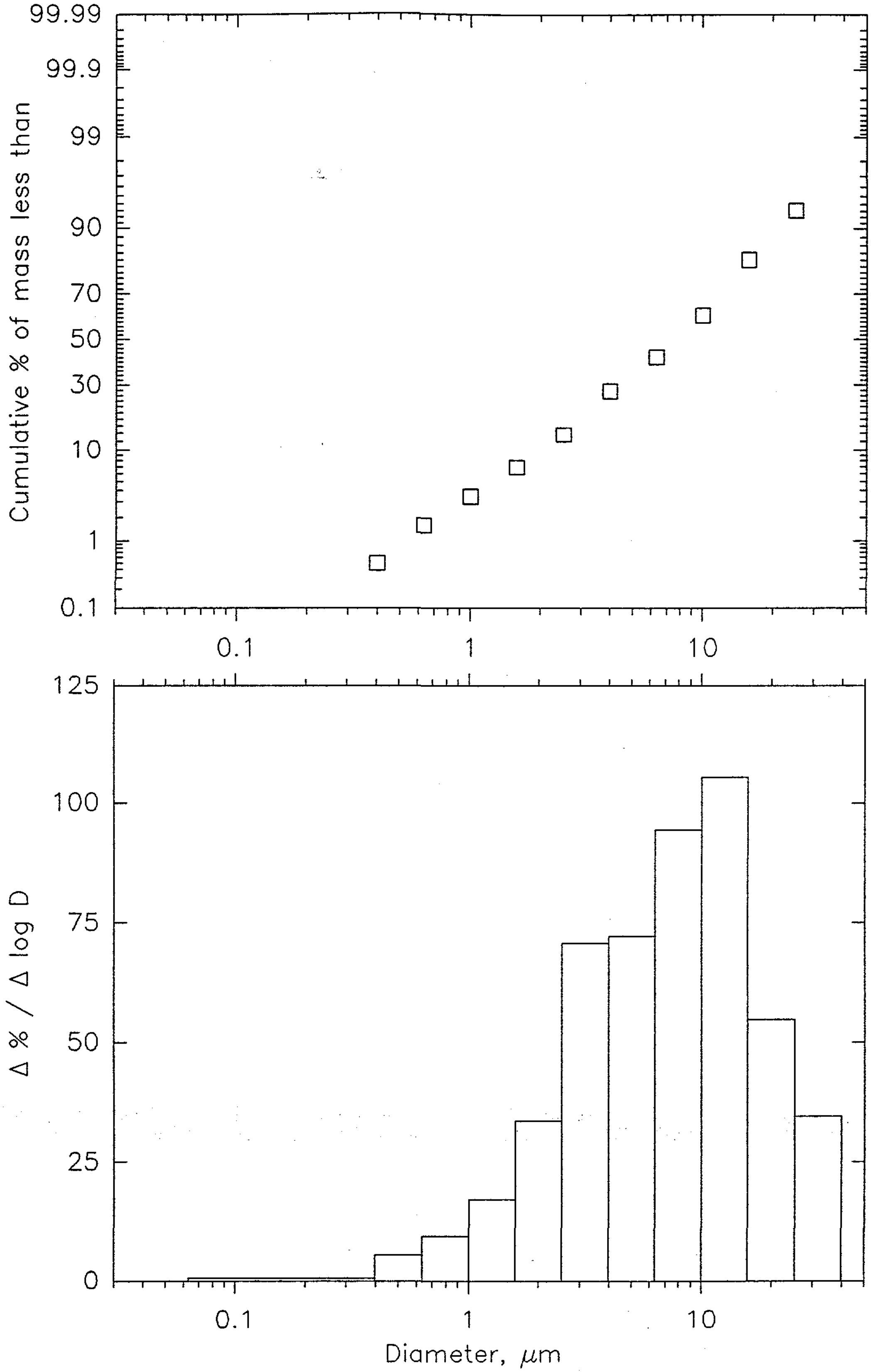

Figure 9. Cumulative and differential size distribution data measured for ash from the Texaco Montebello Research Laboratory gasifier (ID # 2678) measured with a Shimadzu SA-CP4 Centrifugal Particle Size Analyzer. The Stokes MMD of this distribution is 7.6 μm, and its geometric standard deviation is 2.5. (This size distribution data includes the assumption that the ash contains no particles smaller than 0.063 μm.)

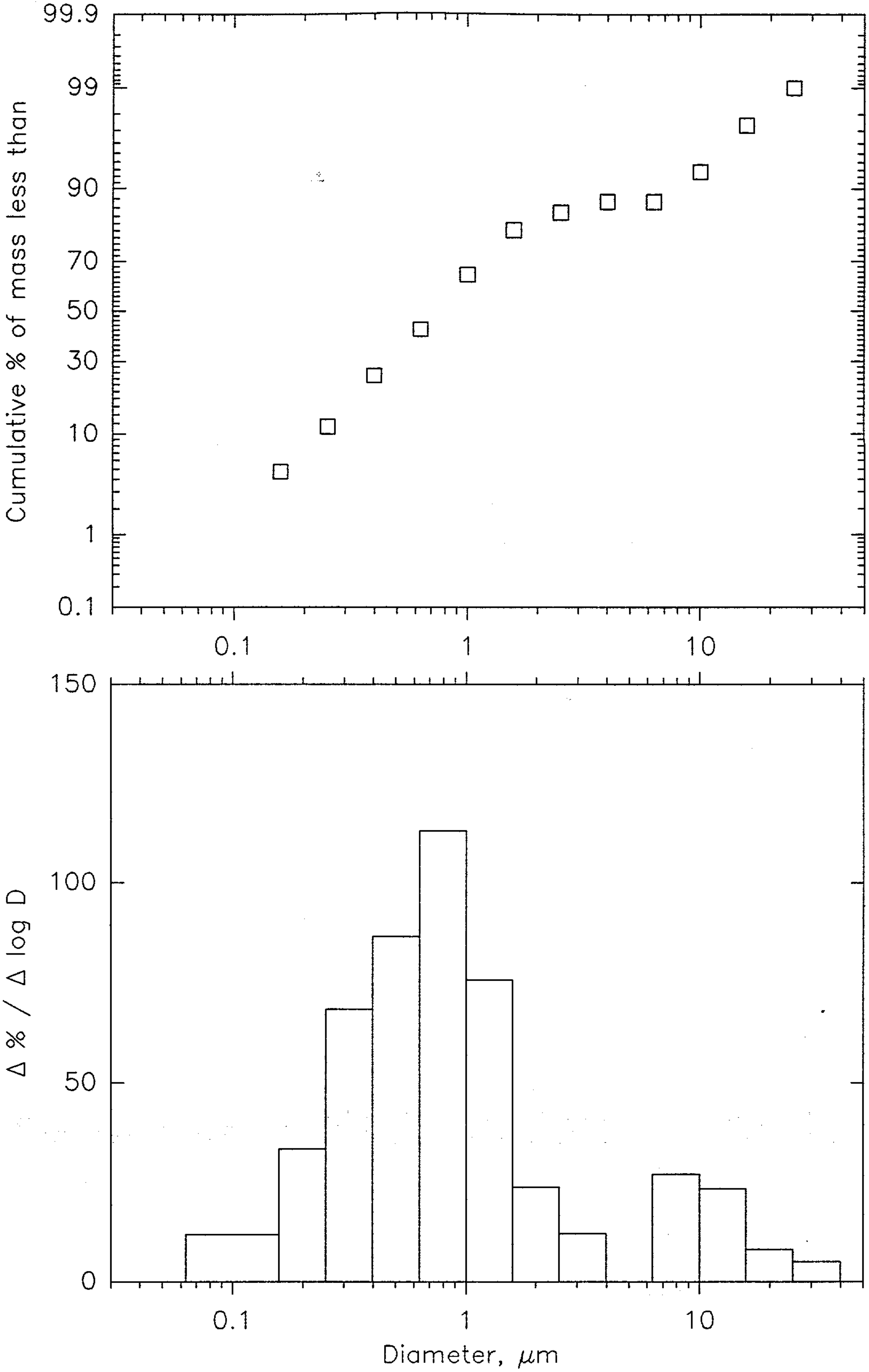

Figure 10. Cumulative and differential size distribution data measured for ash from the DOE/METC gasifier (ID # 4170) measured with a Shimadzu SA-CP4 Centrifugal Particle Size Analyzer. The Stokes MMD of this distribution is 0.74 μm, and its geometric standard deviation is 2.8. (This size distribution data includes the assumption that the ash contains no particles smaller than 0.063 μm.)

Table 3
Physical Characteristics of Texaco and DOE/METC Gasification Ashes*

quantity ID #	2678	4170
specific surface area, m^2/g	88	140
Stokes' MMD, μm	7.6	0.74
uncompacted bulk porosity, %	92	94
drag-equivalent diameter, μm	1.16	0.08
specific gas flow resistance, in H_2O·min·ft/lb	1.1	101
tensile strength, N/m^2	0.6	0.6
true particle density, g/cm^3	2.62	2.87

* Quantities measured prior to the most recent reporting quarter are shaded.

In our last quarterly report we presented some representative SEM photographs of the DOE/METC MGCR ash particles and size distribution data measured with a laser-based device being evaluated for use at the Power Systems Development Facility. Based on both of these observations, the fineness of the particle size distribution of this ash was readily apparent. The data measured with this laser-based device, which we reported in our last quarterly report, showed a distinctly bimodal size distribution with a median diameter of 1.8 μm. Because of the appearance of the particles in ID # 4170 in the SEM photographs we produced (and presented in our last quarterly report), we were skeptical that the distribution measured by this device accurately represented the actual size distribution of this sample. We believe that the process used to prepare and deagglomerate the sample prior to its characterization by laser-based light scattering either failed to fully separate the primary particles or caused the primary particles to agglomerate. Since we had recently repaired the sedigraph that we normally use to measure the size distributions of ash samples, we were able to verify the true size distribution of the DOE/METC MGCR gasification ash. The size distribution data we measured for this ash, shown in Figure 10, demonstrate that the earlier data obtained with the laser-based light scattering device reflected the presence of a large proportion of agglomerates. The true median diameter of this ash, as we suspected from examination of the earlier data, is around 0.74 μm. These experiences during the measurement of the size distribution of ID # 4170 highlight some of the difficulties that are often encountered with samples containing a large proportion of ultrafine particles.

We also characterized the response of the METC MGCR ash to compacting forces. The compaction data, which we measured at room temperature, are presented in Figure 11. The data show that a filtering pressure drop of 3.4 psi (94 in. H_2O) may be sufficient to reduce the porosity of a filter cake formed from the MGCR gasification ash from about 95 % down to about 90 %. In Figure 12 we have used our permeability model to plot the characteristic permeability of this ash as a function of filter cake porosity. These data

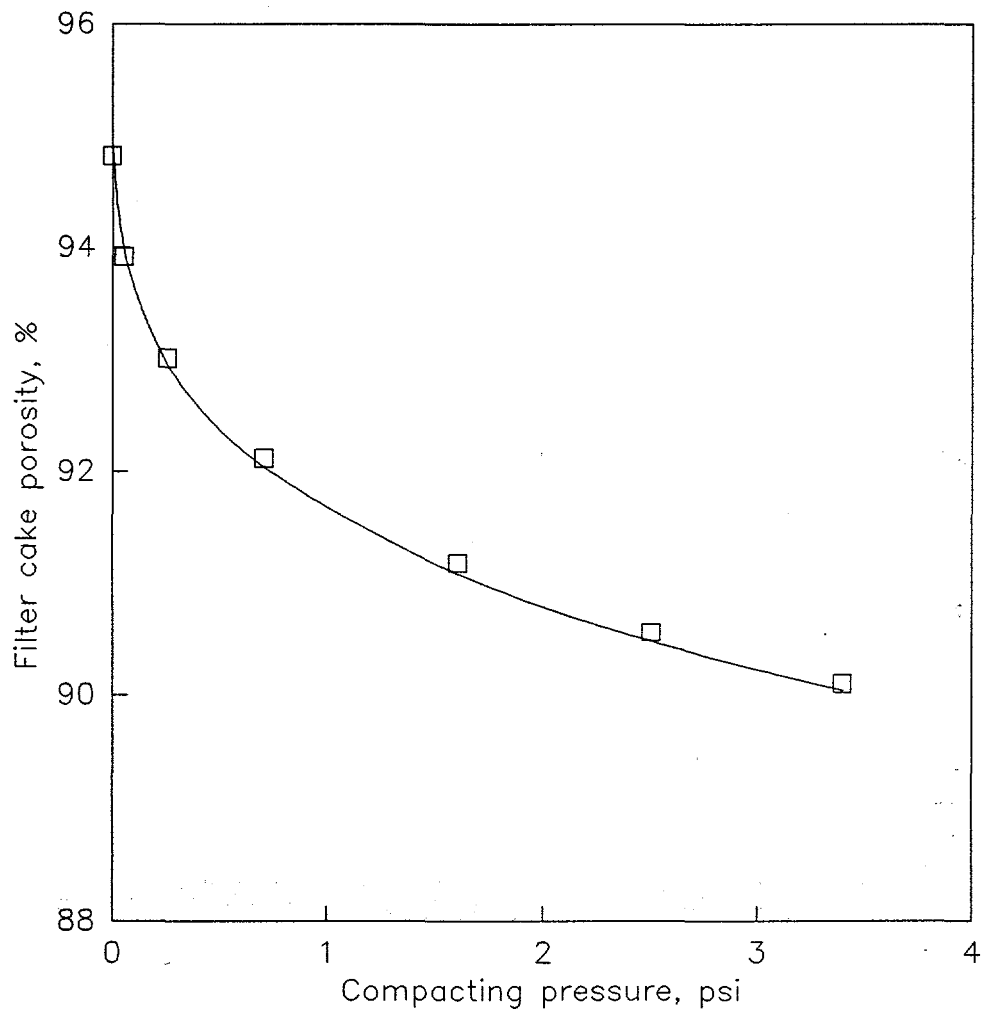

Figure 11. Data measured for the METC MGCR gasification ash (ID # 4170) showing the dependence of cake porosity on mechanical pressure applied across the cake.

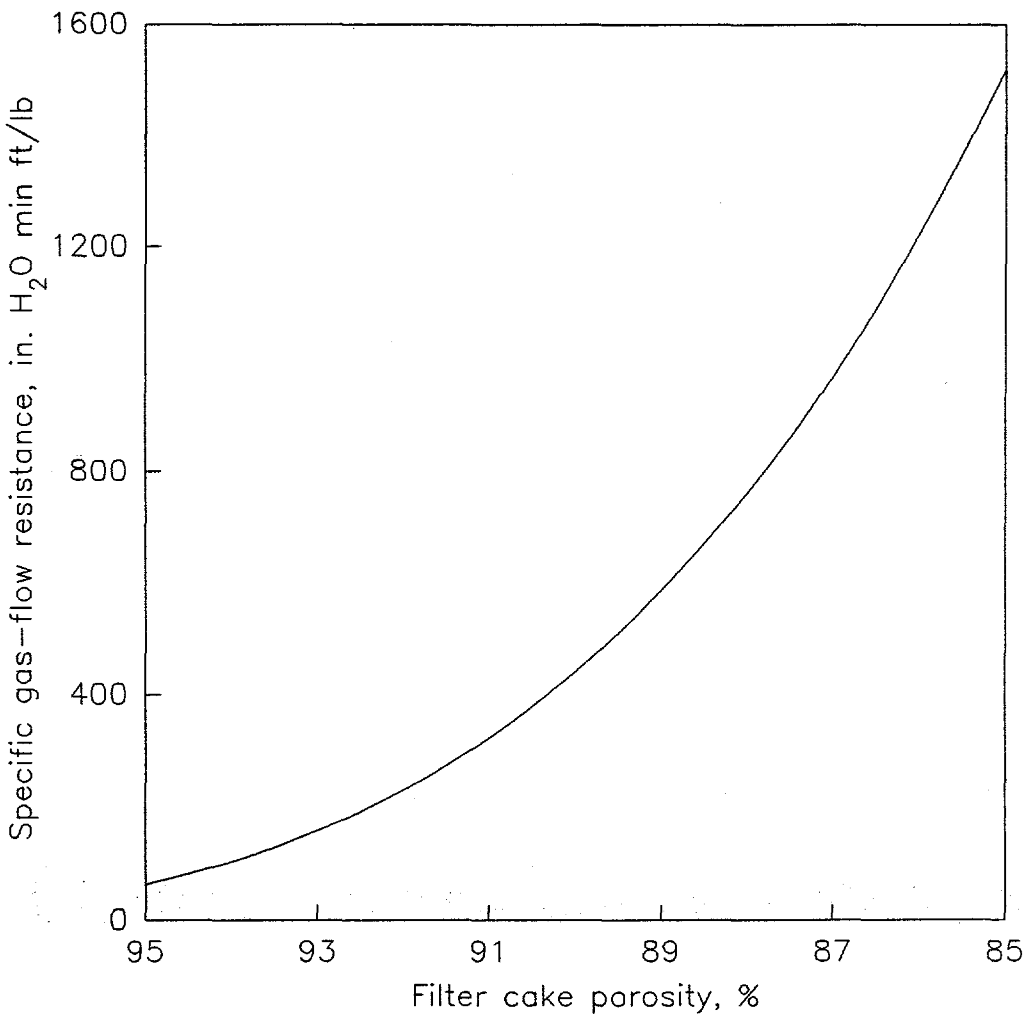

Figure 12. Data calculated with the permeability model for the METC MGCR gasification ash (ID # 4170) showing the strong dependence of relative gas-flow resistance on filter cake porosity.

indicate that the degree of filter cake consolidation that may be induced by normal filtering pressure drops of around 3 to 4 psi may increase filtering pressure losses by a factor of four. This is especially detrimental since the specific gas-flow resistance of this ash is extremely high (5 to 10 times the characteristic values measured for Tidd PFBC ashes) even when the filter cake is uncompacted. These ash characteristics ultimately lead to high pressure losses and/or very frequent cleaning. We did note that although the METC MGCR ash did show an apparent increase in strength as it was compacted, the ash cake compacted with a pressure of 3.4 psi still had comparatively low strength.

TASK 2 RESEARCH ACTIVITIES

Mechanical and thermal testing of the new Schumacher candle filter material was conducted during the past quarter. The test matrix used to evaluate the material is shown in Table 4.

Table 4
Test Matrix used to Evaluate Schumacher Candle Filter Material

Test Type	Orientation	RT	1600°F	1700°F	1800°F
Tensile	Hoop	9			
	Axial	4	4	4	4
Tensile Creep	Axial		4	4	
Thermal Expansion	Hoop	2----------------------------------			
	Axial	2----------------------------------			
Microstructure					

The necessary specimens to perform the matrix were removed from filters according to the cutting plans given in Figures 13 and 14. Hoop and axial tensile results for the new Schumacher material are given in Tables 5 and 6. The average axial strength at room temperature was 606 psi; the average hoop strength was 1692 psi. These strengths were significantly lower than that of earlier material which had an average room temperature hoop strength of 2280 psi and an average axial strength of 1120 psi. A plot of tensile strength versus temperature is given in Figure 15.

Creep evaluations are in progress. At 500 psi, no creep was detected after ~140 hours at 1500°F and, as shown in Figure 16, very little creep was observed after ~165 hours at 1600°F. However, at 1700°F, the specimen failed after ~7 hours. Tests will continue by increasing temperature and/or stress levels. Axial thermal expansion is given in Figure 17. Note the "jump" in expansion at ~400°F. This is probably indicative of the new binder that was incorporated into this material.

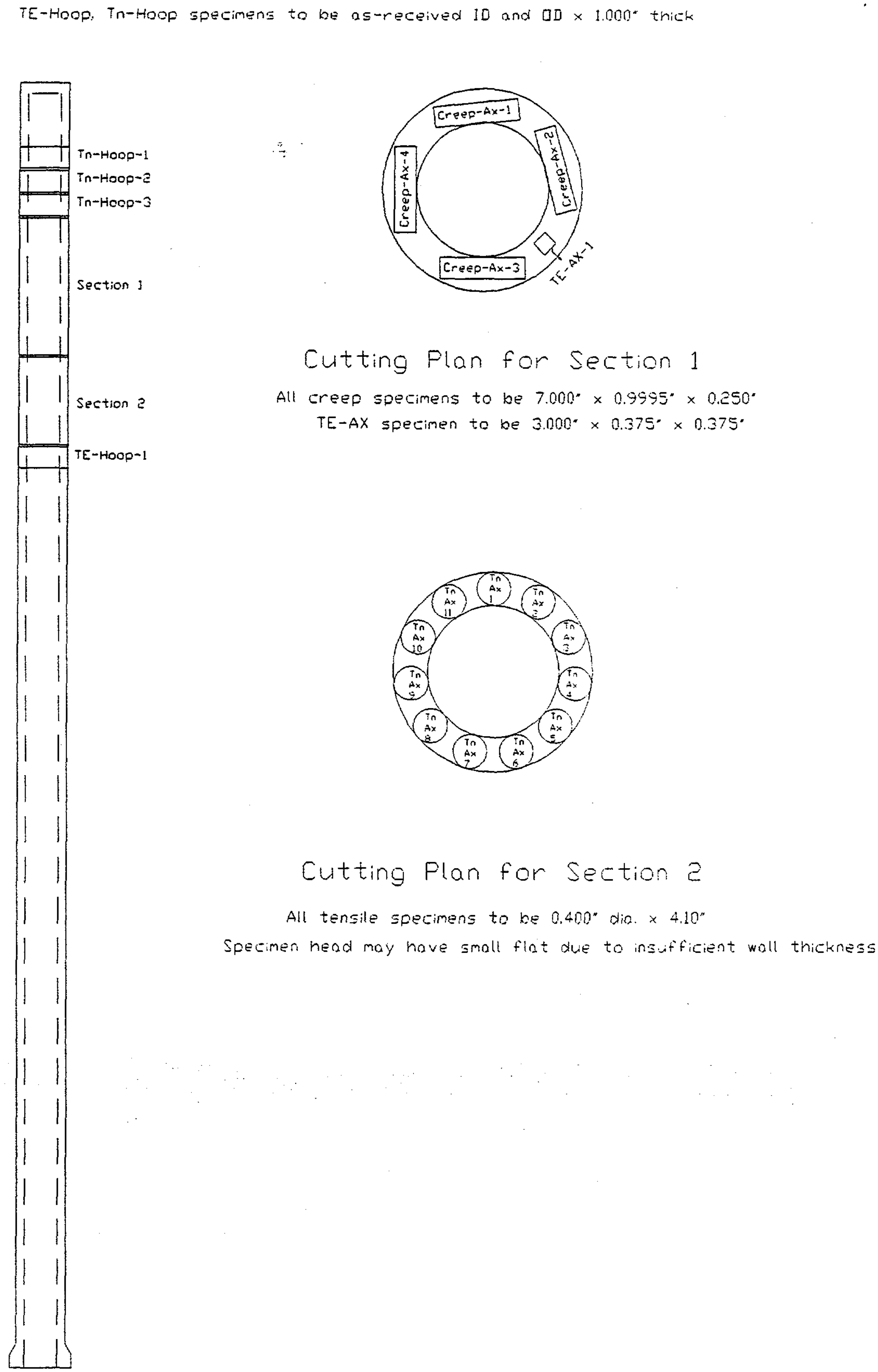

Figure 13. Cutting plan for Schumacher candle 344E-295.

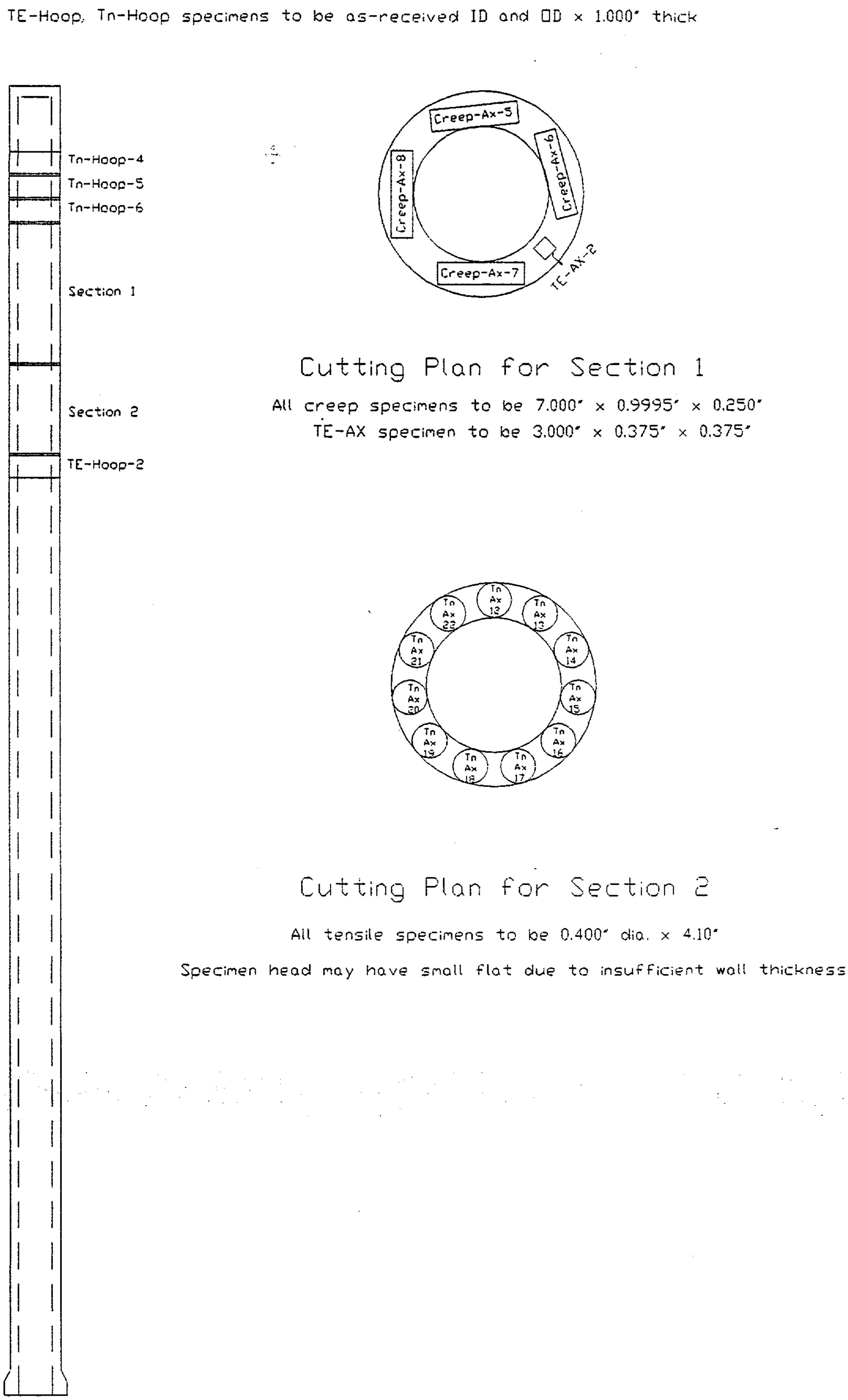

Figure 14. Cutting plan for Schumacher candle 344E-309.

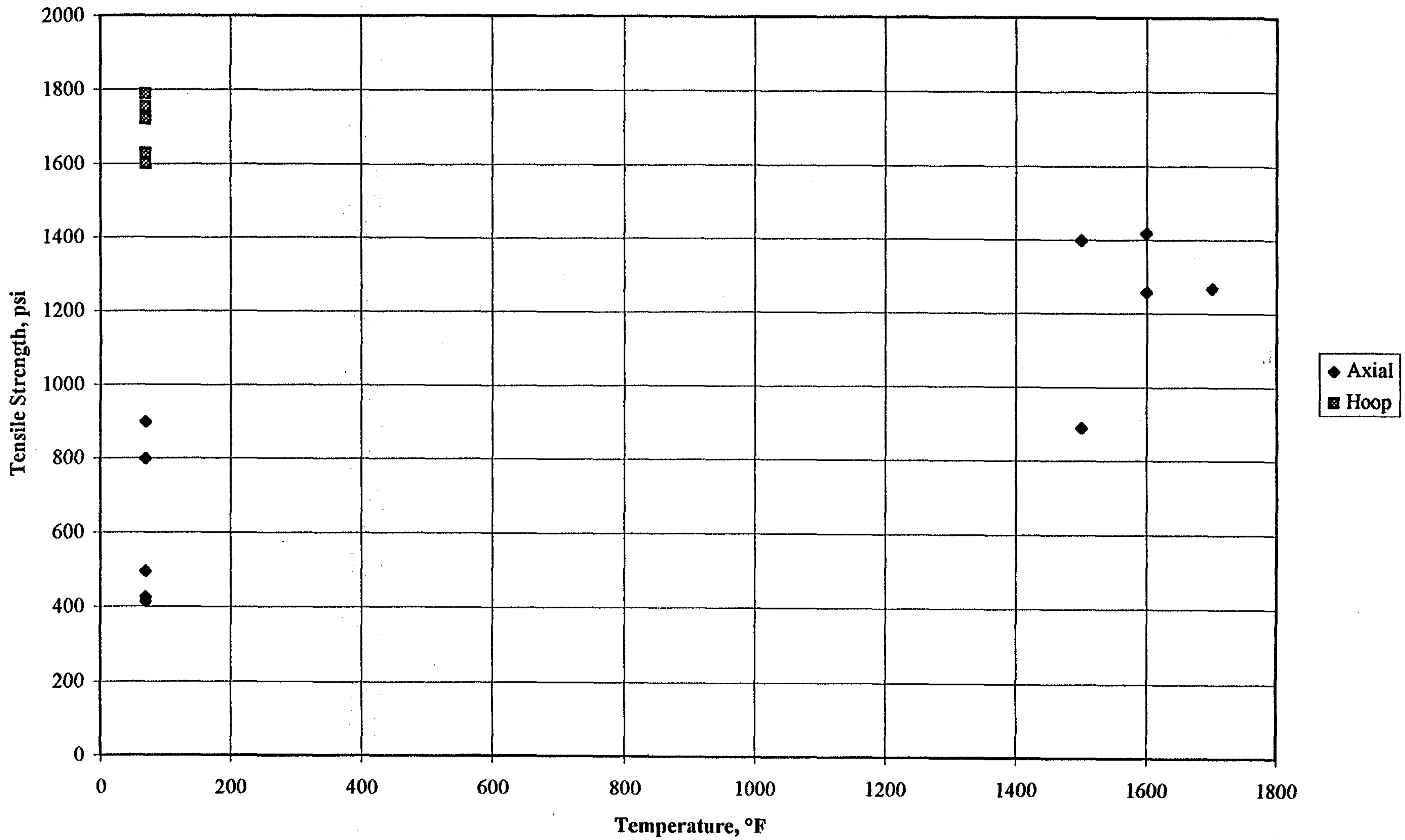

Figure 15. Tensile strength vs. temperature for new Schumacher material.

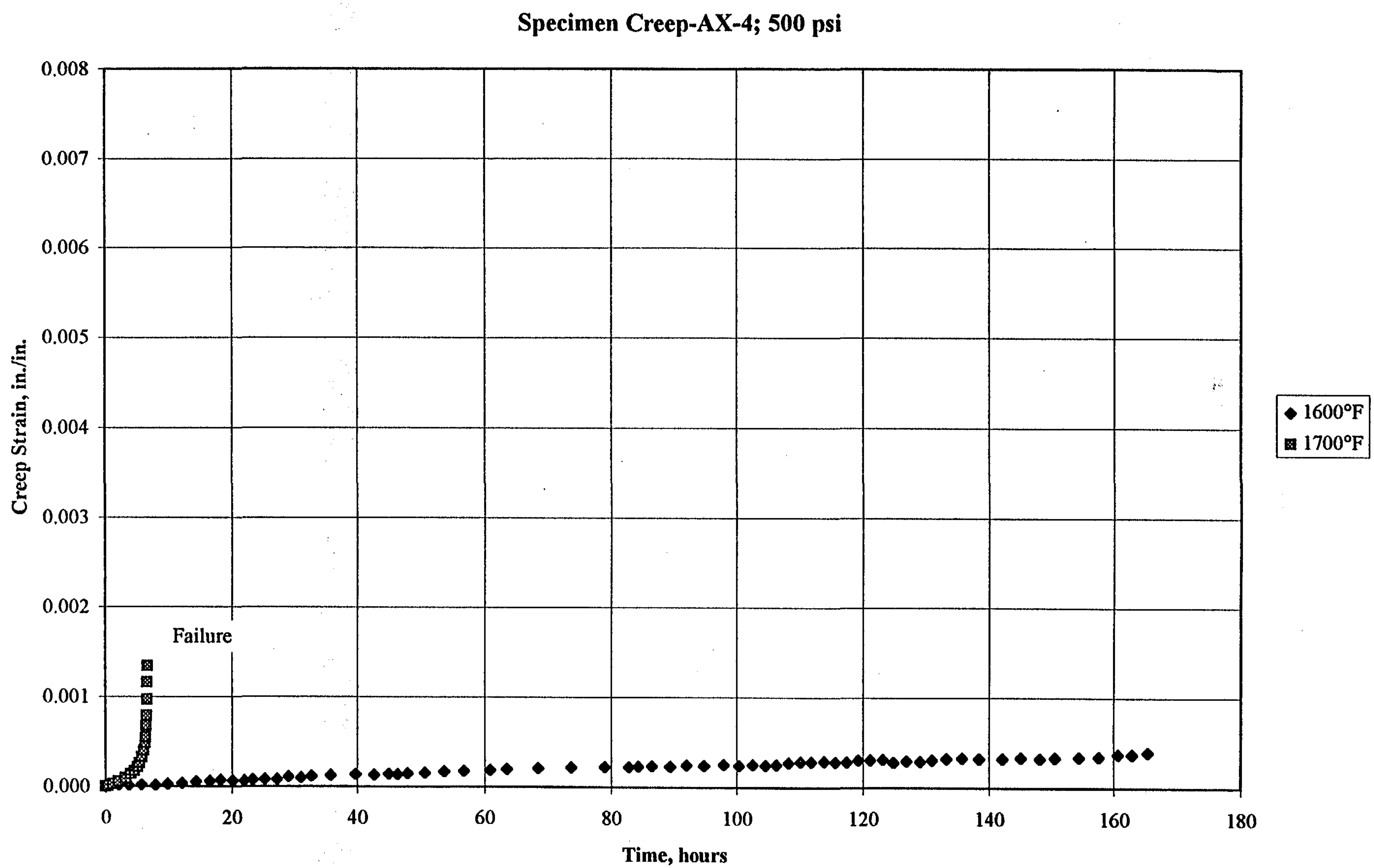

Figure 16. Creep strain vs. time for new Schumacher material.

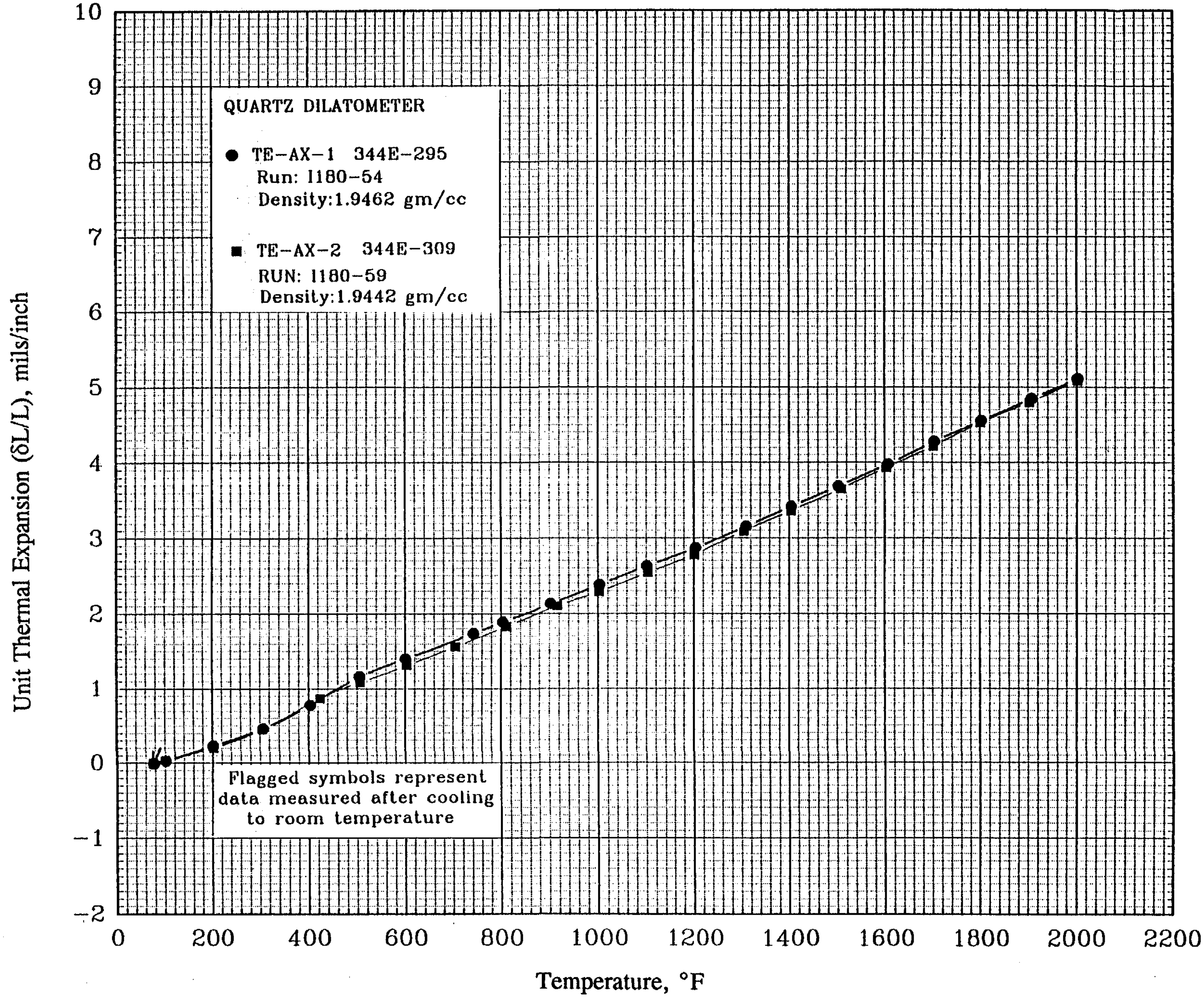

Figure 17. Axial thermal expansion of Schumacher candle coupons.

Two different compositions of alumina-mullite material were received from Blash Precision Ceramics. Even with the utmost care, specimens were difficult to machine without breakage, usually an indication of low tensile strength. The material also contained regions of hard "beads" that would tear out during machining.

Table 5
Tensile Properties of New Schumacher Material (axial measurements)

Candle	Specimen #	Temp., °F	Ultimate strength, psi	Modulus, Msi	Strain-to-failure, in./in.
344E-295	TN-AX-3	70	426	3.22	0.00015
344E-309	TN-AX-12	70	798	4.28	0.00020
344E-295	TN-AX-7	70	414	3.70	0.00011
344E-309	TN-AX-17	70	496	5.00	0.00010
344E-309	TN-AX-21	70	898	3.92	0.00023
Average			606	4.02	0.00016
344E-295	TN-AX-2	1500	1398	2.20	0.00064
344E-309	TN-AX-18	1500	888	2.32	0.00038
Average			1143	2.26	0.00051
344E-309	TN-AX-14	1600	1258	--	--
344E-295	TN-AX-6	1600	1418	2.25	0.00073
Average			1338	2.25	0.00073
344E-295	TN-AX-4	1700	1268	1.77	0.0013

Note: All other specimens broken in assembly

Table 6
Tensile Properties of New Schumacher Material (hoop measurements)

Candle	Specimen #	Temp., °F	ID, inches	OD, inches	Ultimate 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
344E-295	TN-Hoop-1	70	1.54	2.38	1600
344E-295	TN-Hoop-2	70	1.54	2.38	1628
344E-295	TN-Hoop-3	70	1.54	2.38	1628
344E-309	TN-Hoop-4	70	1.53	2.38	1750
344E-309	TN-Hoop-5	70	1.53	2.38	1754
344E-309	TN-Hoop-6	70	1.53	2.38	1788
Average					1692

FUTURE WORK

Plans for the next quarter include continued formatting of the HGCU data base and entry of data, photographs, and text. We also expect to receive a suite of ash samples from the Ahlstrom 10 MWt Pressurized Fluidized Circulating Fluid Bed facility located in Karhula, Finland. The analyses we will perform on these samples will depend on their particular histories.

Creep evaluations of the New Schumacher material will continue by increasing temperature and/or stress levels. Tensile tests of two different compositions of alumina-mullite material received from Blash Precision Ceramics will commence during the next quarter.

PARTICULATE HOT GAS STREAM CLEANUP TECHNICAL ISSUES

QUARTERLY REPORT

October 1995 - December 1995

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Contract No. DE-AC21-94MC31160

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Approved by

Duane H. Pontius, Director   Particulate Sciences Department