Contract No. DE-AC21-94MC31160

PARTICULATE HOT GAS STREAM CLEANUP TECHNICAL ISSUES

QUARTERLY REPORT

July 1996 - September 1996

Prepared for

UNITED STATES DEPARTMENT OF ENERGY
Morgantown Energy Technology Center
Post Office Box 880, 3610 Collins Ferry Road
Morgantown, West Virginia 26505

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for

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Contract No. DE-AC21-94MC31160
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EXECUTIVE SUMMARY

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

Under Task 1 during the past quarter, additional analyses were performed on ashes from the Ahlstrom 10 MWt Pressurized Fluidized Circulating Fluid Bed (PCFB) facility located at Karhula, Finland. Work continued on the HGCU data base being constructed in Microsoft Access®. A variety of information has been entered into the data base, including numerical values, short or long text entries, and photographs. Detailed design of a bench top device for high-temperature measurement of ash permeability has also begun. In addition to these activities, a paper was prepared and a poster was presented summarizing recent work performed under this contract at the 1996 DOE/METC Contractor's Conference. A presentation was also given corresponding to the manuscript entitled *Particle Characteristics and High-Temperature Filtration* that was prepared for publication in the Proceedings of the Thirteenth Annual International Pittsburgh Coal Conference held this September in Pittsburgh, PA. Arrangements have been made to be present at the DOE/METC Modular Gas Cleanup Rig (MGCR) at the conclusion of the next run of the DOE/METC air blown Fluid Bed Gasifier (FBG). This visit will include on-site sampling to collect and characterize the filter cakes collected during FGB operation.

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 eighth quarterly report describing the activities performed under Contract No. DE-AC21-94MC31160. Task 1 of this contract concerns analyses of HGCU ashes and descriptions of filter performance that are designed to address the problems with filter operation linked to the characteristics of the collected ash. Task 2 of this contract includes characterization of new and used filter elements. Some of the problems observed at PFBC facilities include excessive filtering pressure drop, the formation of large, tenacious ash deposits within the filter vessel, and bent or broken candle filter elements. These problems have been attributed to ash characteristics, durability of the ceramic filter elements, and specific limitations of the filter design. In addition to the problems related to the characteristics of PFBC ashes, laboratory characterizations of gasifier and carbonizer ashes have shown that these ashes also have characteristics that might negatively affect filtration. Problems with the durability of the filter elements are being addressed by the development and evaluation of elements constructed from alternative ceramic materials.

To identify which ash characteristics can lead to problems with filtration, 242 ash samples from twelve facilities involved in METC's HGCU program have been assembled. Many of these ashes have been analyzed with a variety of laboratory tests. Physical attributes of the particles that have been examined include size distribution, specific surface area, particle morphology, and bulk ash cohesivity and permeability. A range of chemical analyses on these 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 have also been performed. The data obtained in these studies is being assembled into an interactive data base which will help the manufacturers and operators of high-temperature barrier filters tailor their designs and operations to the specific characteristics of the 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, hoop and axial tensile tests, thermal expansion, compression, and creep evaluations of these materials at temperatures up to 1800 °F have been performed. Nondestructive testing methods performed on filter specimens include density and ultrasonic velocity. To date various characteristics of Dupont/Lanxide PRD-66, Dupont composite, 3M composite, IF&P Fibrosics, Refractron, Schumacher, and Blasch ceramic materials have been evaluated.

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

venues prescribed by 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 ASSESSMENT OF ASH CHARACTERISTICS

Primary efforts during the past quarter were directed toward detailed design of the high-temperature permeability tester and continued development and data entry for the HGCU data base which is being constructed in Microsoft Access®. Limited analyses were performed on two of the Karhula ash samples that were received this June. In earlier quarterly reports a schematic diagram of the test column of the high-temperature permeability tester was presented. Before engineering drawings are produced that will be used for fabrication of this device and the high temperature electrostatic tensiometer, the design details will be discussed with the DOE/METC Project Manager.

As data was added to the HGCU data base, it was determined that the original structure set up in Microsoft Access® needed to be modified. The modified structure is presented in Figure 1 which summarizes the relationships between the tables that will hold the data and information entered in the HGCU data base. When the data base is complete, this display and the structure it represents will not be visible to the end user.

An SEM photograph of a fresh fracture surface of a Karhula filter cake nodule from the suite of six samples received last June was presented previously. In this picture the nodules appear as concretions composed of discrete fine particles almost completely embedded in pervasive amorphous masses. As has been discussed in prior reports, these amorphous masses apparently form in the filter vessel after the particles are initially collected. The two primary mechanisms that may be responsible for the formation of these masses, eutectic melts and sulfation, have also been discussed in prior reports. By comparing the relative concentrations of sulfate ions in hopper and filter cake nodule ashes from Karhula, the suite of samples recently received from Karhula allow another evaluation of the sulfation mechanism. When the concentration of soluble sulfate in the Karhula hopper ash was measured (25.6 % by wt.), it was found that it was somewhat greater than the concentration in a filter cake nodule from Karhula (21.6 % by wt.). However, the consolidated nodule would have to contain significantly more soluble sulfate than the hopper ash to attribute the consolidation of the nodule solely to sulfation. Therefore, as has been postulated for the Tidd PFBC, these data support the hypothesis that eutectic formation is also responsible for nodule formation at Karhula.

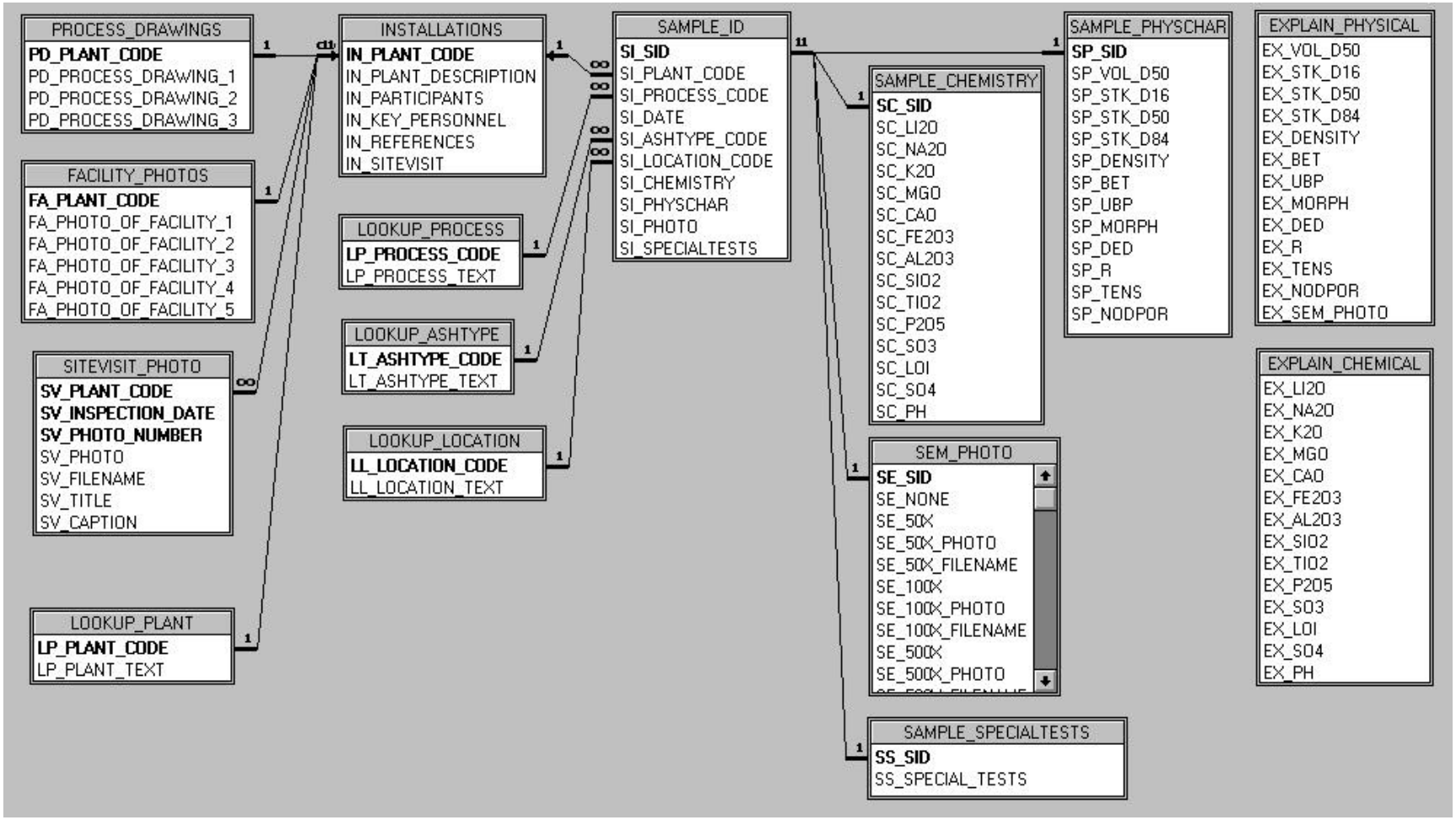

Figure 1. This diagram shows the relationships between the tables that will hold the data and information we enter in the HGCU data base. When the data base is complete, this display and the structure it represents will not be visible to the end user.

TASK 2 FILTER MATERIAL CHARACTERIZATION

Profilometry measurements were carried out on one Refractron 326 candle filter, one Schumacher FT20 candle filter, and one Coors alumina mullite candle filter retrieved from Karhula in June, 1996 after approximately 540 hours in service. The maximum bow measured on the candle filters is given in Table 1.

Table 1
Profilometry Measurements of Candle Filters used at Karhula

Material	Maximum Bow (in.)
Refractron 326	0.084
Schumacher	0.044
Coors	0.065

Creep testing has been performed on as-manufactured Refractron 326 specimen Creep-ax-5 at 1600 °F, 1700 °F, and 1800 °F with a tensile load of 500 psi. The creep test was run continuously for 164 hr at 1600 °F, 236 hr at 1700 °F, and 140 hr at 1800 °F for a total duration of 540 hr. The results of these measurements are summarized in Table 2.

Table 2
Creep Measurements for As-Manufactured Refractron 326 Material

Temperature, °F	Tensile load, psi	Duration, hr	Initial creep rate, in/in/hr	Secondary creep rate, in/in/hr
1600	500	164	2.6×10^{-8}	8.0×10^{-10}
1700	500	236	4.7×10^{-9}	3.4×10^{-9}
1800	500	140	3.4×10^{-8}	not obtained

Creep testing of Schumacher FT20 is in progress. Additional Refractron 326 specimens have been machined for tensile testing in the critical temperature range of 1500 °F to
1600 ºF.

FUTURE WORK

Plans for the next quarter include continued construction of the HGCU data base and entry of additional data, photographs, and text. Detailed design of the high-temperature permeability tester and electrostatic tensiometer will also continue. Contacts have been made with Herman Research Pty Ltd. (HRL) of Melbourne, Australia, the Coal Technology Development Division in Gloucester, UK, and VTT Energy in Finland about obtaining particulate samples from the various gasification/high-temperature filtration facilities operated by these organizations. Several samples from HRL gasification studies of three Australian brown coals are expected to be received in the near future. Information will hopefully be obtained describing the most recent operation of the Karhula PCFB to correlate with the laboratory data measured for samples from this facility. Additional samples from UNDEERC's TRDU test facility have been requested. Creep testing of Schumacher FT20 will continue. Tensile testing in the critical temperature range of 1500 °F to 1600 ºF will begin on recently machined Additional Refractron 326 specimens.

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