Ruth Ann Yongue Principal Presenter Senior Engineer rayongue@southernco.com (205) 670-5088 Xaiofeng Guan xguan@southernco.com Principal Engineer (205) 670-5891

Southern Company Services, Power Systems Development Facility Fax (205) 670-5843 P.O. Box 1069, Wilsonville, AL 35186

Robert S. Dahlin Senior Engineer x2dahlin@southernco.com (205) 670-5068 E. Carl Landham Senior Engineer x2landha@southernco.com (205) 670-5990

Southern Research Institute, Power Systems Development Facility Fax (205) 670-5843 P.O. Box 1069, Wilsonville, AL 35186

Update on Hot Gas Filtration Testing at the Power Systems Development Facility

32nd International Technical Conference on Coal Utilization & Fuel Systems June 10 – 15, 2007

Keywords: hot gas filtration, coal gasification, filter elements, integrated gasification combined cycle (IGCC)

Introduction

Hot gas filter system and component testing for future application in Integrated Gasification Combined Cycle (IGCC) power plants is an area of major focus at the Power Systems Development Facility (PSDF). The PSDF is an engineering scale demonstration of key features of advanced coal-fired power systems, including a KBR (formerly Kellogg, Brown, & Root) Transport Gasifier and a Siemens Particulate Control Device (PCD), which are designed at sufficient size to provide data for commercial scale-up. The objective of the PCD is to clean the syngas of particulate so that it can be utilized in a gas turbine or fuel cell. As of May, 2007, the Transport Gasifier train, including the Siemens PCD, had been operated for more than 10,000 hours, providing valuable operational data for future commercial scale IGCC power plants.

Recent testing at the PSDF related to hot gas filtration has focused on several areas key to achieving high filter system efficiency and availability. These areas include demonstration of reliable filter element and failsafe performance and optimization of filter system operations. Significant improvements in performance and reliability have been realized, and operational understanding has been gained through on-going testing.

This paper will discuss the hot gas filtration testing in recent years of gasification operation at the PSDF.

Project Description

The KBR Transport Gasifier which operates at the PSDF is a circulating fluidized bed reactor designed to operate at higher circulation rates and riser densities than conventional circulating bed units. The higher circulation rates result in higher throughput, better mixing, and higher mass and heat transfer rates. Since the gasifier uses a dry feed system and does not slag the ash, it is well-suited for high moisture fuels such as subbituminous and lignite coals, but can also process some higher-rank coals. The gasifier can be operated in both air-blown and oxygen-blown mode. An update on Transport Gasifier operations is discussed in a companion paper in these proceedings (Leonard, 2007).

Virtually all the particulate from the syngas exiting the gasifier is removed by the downstream PCD. The PSDF's most extensively tested PCD, a Siemens hot gas filter vessel using candle-type filters, is shown in Figures 1 and 2 below. This PCD utilizes a tube sheet holding up to 91 filter elements which are attached to one of two plenums. Process gas flows into the vessel through a tangential entrance, around a shroud, and through the filter elements into the plenums. High pressure gas is used to pulse clean the elements periodically to remove the accumulated solids and control the pressure drop across the tube sheet. A failsafe device is located on the clean side of each element and is designed to stop solids leakage in the event of a filter failure by acting as a back-up filter. Maintaining a sufficiently low solids outlet loading and tube sheet pressure drop are the major operational objectives of the filter system.



Figure 1. Filter Vessel Internals.

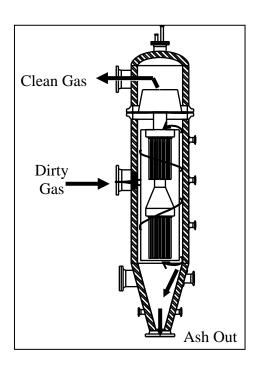


Figure 2. Schematic of Filter Vessel.

Operational History

System commissioning and initial test campaigns were performed in combustion mode from 1996 to 1999, and gasification operation began in late 1999. Four gasification commissioning tests, each lasting nominally 250 hours, were completed by early 2001. Since then, 17 test campaigns, each nominally 250 to 1,500 hours in duration, have been completed. The most extensively tested fuel has been Powder River Basin subbituminous coal, although several bituminous and lignite coals have also been tested. PCD operation during combustion mode operation has been previously described (Davidson, 1999), as has operation in early gasification testing (Martin, 2002). Table 1 below lists typical PCD operating conditions during recent testing.

Table 1. Typical Filter System Operating Conditions in Gasification Operation.	
Dustcake Drag, inH ₂ O/(ft/min)/(lb/ft ²)	100
Normalized to Room Temperature	
Temperature, °F (°C)	750 (400)
Pressure, psig (bar)	200 (14)
Face Velocity, ft/min (cm/s)	3.5 (1.8)
Baseline ΔP , inH ₂ O (bar)	80 (0.2)
ΔP Rise Rate, inH ₂ O/min (bar/min)	10 (0.025)
Pulse Cycle Time, min	5
Pulse Duration, sec	0.2
Pulse Pressure, psig (bar)	450 (31)
Inlet Loading, ppmw	13,000
Particle Mass Mean Diameter, µm	15

Filter element and failsafe materials must be compatible with these operating conditions in the corrosive gasification environment and must be able to withstand system upsets. As discussed in previous papers (Martin et al., 2005; Guan et al., 2005), many different types of filter elements have been tested at the PSDF, including monolithic ceramic, ceramic composite, sintered metal powder, and sintered metal fiber. The ceramic elements were primarily used in early combustion-mode tests at temperatures around 1400°F (760°C). When the filter operating temperature was reduced to around 750 to 850°F (400 to 450°C) for gasification mode operation, it was possible to use the more durable metal elements. In gasification tests to date, the most experience has been achieved with iron aluminide (FEAL) sintered metal powder elements and Haynes HR-160 sintered metal fiber elements.

Results

Operational concerns of early gasification testing were addressed, and the PCD has been consistently reliable and available during recent testing, demonstrating collection efficiencies greater than 99.999%. Challenges such as ash level control and upset conditions were successfully addressed, and the primary focus has become demonstrating long-term filter element and failsafe performance.

Evaluation of Filter Elements

A central part of the PSDF hot gas filter test program is the evaluation of filter elements in terms of collection efficiency and corrosion resistance. With respect to the collection efficiency, the primary concern is whether the outlet particle loading and particle size distribution will satisfy commercial turbine specifications. With respect to corrosion resistance, the primary concern is that the elements provide a service life of at least one or two years of continuous operation without an unacceptable loss of strength or buildup of pressure drop.

Particulate Collection Efficiency

Experience with the iron aluminide sintered powder elements and with the HR-160 sintered fiber elements have shown that both types of elements are capable of providing excellent particulate collection performance. During normal operations, the combination of these filter elements and their downstream failsafe devices have been able to routinely achieve outlet particle loadings of <0.1 ppmw. (The *in situ* sampling method employed to characterize filter performance is described in detail in Dahlin, et al., 1998). Even during the initial period of filter "seasoning" outlet particulate loadings are generally well below the turbine specification of 2.4 ppmw (for a turbine inlet flow to fuel ratio of 4) (GE Power Systems, 2002) as shown in Figure 3.

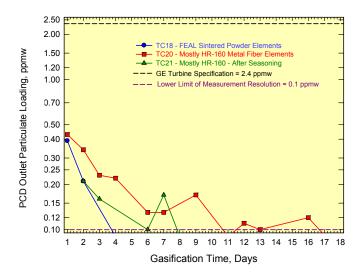


Figure 3. Comparison of Initial Particulate Collection of Sintered Metal Powder and Sintered Metal Fiber Elements.

While turbine specifications on total particle loading can be easily met with either type of filter element, meeting turbine specifications for large particles (> $10 \mu m$) is a concern. Particles larger than $10 \mu m$ have been observed on the outlet sampling filters, with a smaller number of these large particles seen when the sintered metal powder elements are used exclusively. It should be noted that some of these large particles may result from

gasket leakage or re-entrainment of contaminants from the piping downstream of the filter elements.

One of the key differences between the sintered metal powder and sintered metal fiber elements can be seen in the morphologies and size of the openings in the media (See Figure 4.). The metal fiber elements typically have some larger openings as a result of the random deposition of the fibers. The fiber cross sections are also much smoother than the angular grains of sintered powder, thereby providing a less tortuous path for migration of particles through the filter medium. Ordinarily, these differences in filter structure do not affect the collection efficiency, since most of the filtration is performed by the dustcake. However, these differences in morphology and opening size become important immediately after a backpulse, when the dustcake is altered.

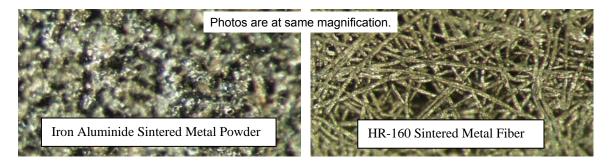


Figure 4. Photomicrographs of Filter Element Surfaces

Real-time particulate monitoring has confirmed that most of the particle emissions occur immediately after backpulses. Backpulsing of the metal fiber elements appears to produce more particle penetration than backpulsing of the sintered metal powder elements.

Because of the strict limits on large particles imposed by turbine vendors, there is clearly a need to accurately quantify the concentration of large particles penetrating through the filter vessel. The quantification of these large particles is very difficult to do from filter samples because of the limited resolution of optical microscopes and because some of the particles penetrate into the filter matrix and are no longer visible during microscopic examination of the filters. More research is needed to completely understand the factors that affect particulate penetration through the filter vessel and how they relate to compliance with the turbine specifications.

Corrosion Resistance

Corrosion resistance is an important consideration in the selection of filter elements. As mentioned earlier, current testing at the PSDF is focused on two types of metal filter elements: iron aluminide sintered metal powder and HR-160 sintered metal fiber. In general, the iron aluminide sintered metal powder elements offer superior particle collection, but they have shown signs of corrosion. The original batch of HR-160 sintered metal fiber elements have not yet shown any signs of significant corrosion

(although they have been tested for less time), but they allow more particle penetration, including particles that appear to be larger than the turbine limit (> $10 \mu m$). Large particle penetration through the sintered fiber elements may be mitigated by reducing fiber size, although this must be balanced with pressure drop and corrosion concerns. Cold flow testing of finer fiber media at the PSDF showed significant improvement in particulate collection efficiency. Batch testing of HR-160 elements with finer fiber media will be conducted at the PSDF in the near future to evaluate their corrosion resistance and particulate collection performance under real operating conditions.

Examination of iron aluminide filter elements tested at the PSDF has shown signs of a considerable degree of sulfidation and plugging in certain areas of the filter cross section. Figure 5 shows an example of this where both plugged and open areas can be seen in a filter cross section that has 7,158 exposure hours in gasification.

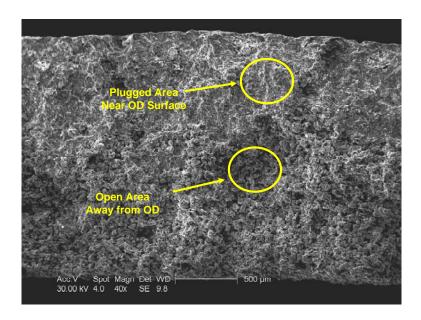


Figure 5. Cross Section of Iron Aluminide Filter Element Exposed for 7,158 Hours in Gasification.

Energy-dispersive X-ray (EDX) analyses of the plugged and open areas are shown in Figure 6. The comparison of the two areas suggests that sulfidation is responsible for the plugging. This same effect was observed in iron aluminide filter elements used at the Wabash River IGCC project (McKamey et al., 2002). However, it should be noted that the H₂S concentration in the syngas at the Wabash River facility was much higher than at the PSDF because of higher sulfur feedstock. Both the PSDF and the Wabash River facility results confirm that sulfidation and plugging can occur in iron aluminide elements at temperatures of about 750 to 800°F (400 to 425°C). Moreover, published data on iron aluminide confirm small but measurable rates of sulfidation (Judkins and Rao, 2000).

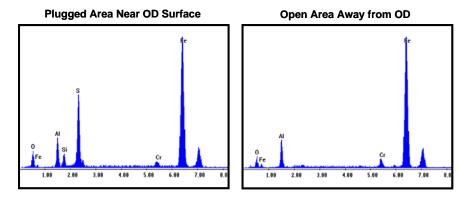


Figure 6. Energy-Dispersive X-Ray Analyses of Areas Shown in Figure 5.

During the manufacturing of iron aluminide filter elements, a pre-oxidation step is used to form a protective layer of alpha-alumina on the surface of the iron aluminide grains. If this layer is intact, it should prevent sulfidation and other forms of corrosion. However, compared to other alumina-forming alloys, the protective alumina layer on iron aluminide spalls more readily (Pint et al., 2001). Estimates of the expansion differential between the alumina and the underlying iron aluminide also show that cracking of the layer would be expected during startups when the filter element is heated from ambient temperature to 750°F.

Once the alumina layer is cracked, the iron in the underlying iron aluminide is susceptible to attack by H_2S , as well as other reactive gases such as steam. Actually, steam attack should proceed more rapidly than the attack by H_2S , since the partial pressure of steam in the PSDF syngas is two orders of magnitude higher than the partial pressure of H_2S . Moreover, steam is known to react very vigorously with finely divided iron. This may explain why the formation of iron oxide was observed on the PSDF filter elements as a precursor to the formation of iron sulfide.

Even though the steam attack is very rapid initially, it eventually forms an extensive layer of iron oxide that tends to be converted to iron sulfide because iron oxide reacts rapidly with H_2S at 750 to 800°F (400 to 425°C) (Milbourne and Huff, 1930; Tamhankar et al., 1981; and Danielewski et al., 1982). The reaction rate increases with increasing temperature up to a point where the reaction product (FeS) becomes thermodynamically unstable. At a temperature of 750°F, both the thermodynamics and the kinetics are favorable for formation of FeS by the reaction of Fe₂O₃ with H_2S .

To summarize, a postulated mechanism of the iron aluminide corrosion is:

- (1) The protective alumina layer is cracked by differential expansion during startup and other thermal transients.
- (2) The underlying iron is attacked by steam to form iron oxide.
- (3) Iron oxide is sulfidized to FeS by reaction with H_2S .

While the proposed mechanism has not been verified, each step of the mechanism seems to be supported by observations at the PSDF and by the published literature on iron aluminide behavior and sulfidation.

In standard flow tests performed on cleaned (pressure-washed) iron aluminide filter elements, a steady increase in the pressure drop even after cleaning has been observed. Some of this increase could be due to sulfidation. One way to investigate this possibility is to measure the filter pressure drop before and after descaling with a citric acid solution, which removes the iron oxide and iron sulfide scale. While this type of measurement has not been made on a filter element, it has been done with an iron aluminide failsafe, which is similar to the filter element except in porosity. The results obtained are illustrated in Figure 6, which shows how the pressure drop was reduced by cleaning in an ultrasonic cleaner and then by two consecutive descaling treatments using inhibited citric acid. The additional reduction in pressure drop is most probably attributable to the fact that both iron oxide and iron sulfide are very soluble in hot citric acid. The reduction could not be attributed to additional removal of ash, because no ash particles were found in the spent descaling solution. Therefore, it appears that, at least in the case of this failsafe, sulfidation/corrosion is a major contributor to the pressure drop increase. A similar test is needed on the iron aluminide filter element to determine whether this also holds true for the elements.

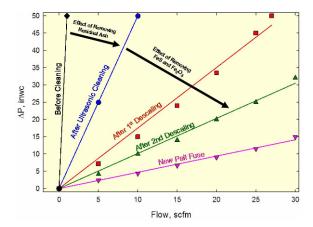


Figure 6. Effect of Cleaning and Descaling on Iron Aluminide Failsafe Pressure Drop

Although the tests with the failsafe descaling suggest that sulfidation can contribute to pressure drop buildup in iron aluminide failsafes, the evidence of this effect is less clear with the iron aluminide filter elements. With the filter elements, pressure washing with water generally eliminates most of the pressure drop buildup, but the remaining pressure drop is still somewhat higher than that of a virgin filter element. This indicates that the residual pressure drop is primarily attributable to particulate, possibly due to augmented retention of fine particulate on the rough element surface caused by corrosion. Also, it has been observed that the older iron aluminide elements tend to rapidly return to a high pressure drop when put back into service in the PCD.

Currently at the PSDF, several iron aluminide filter elements have achieved more than one year of cumulative exposure time, and a large number of others are approaching that time. However, more prolonged monitoring of the pressure drop buildup is needed to determine whether the elements can achieve a commercially viable service life with acceptable pressure drop based on the design criteria of a commercial PCD.

In addition to the pressure drop buildup, the effect of the corrosion on the strength of the elements is also of concern. To address this concern, selected iron aluminide elements have been removed from service after various periods of exposure, and measurements have been made of their axial and hoop tensile strengths. The results obtained to date are shown in Figure 7.

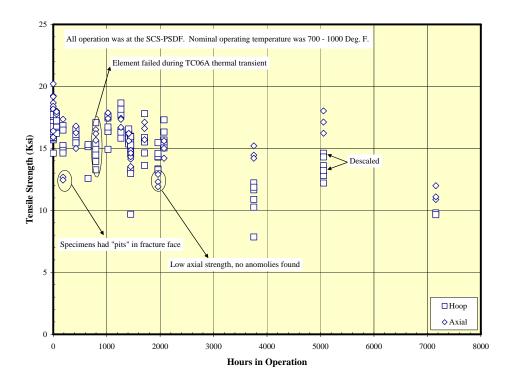


Figure 7. Axial and Hoop Tensile Strength of Iron Aluminide Elements Tested at Room Temperature

As shown in Figure 7, the latest measurements made after 7,158 hours of gasification exposure show a potential decline in strength. However, the strength is still sufficient for filter element structural integrity. As with the pressure drop buildup, more prolonged monitoring of the strength is needed to ensure that an acceptable commercial service life is achievable.

Failsafe Development

Because filter element failures during early test campaigns at the PSDF resulted in high particle penetration due to poor failsafe performance, a failsafe test program was initiated to develop and identify failsafe devices that would effectively prevent particle penetration

in the event of filter element failure. As part of the failsafe program, failsafe testing was designed to simulate the failures of single filter elements under various conditions causing small, moderate, and large amounts of particle loading to the failsafe. Since the implementation of the failsafe development program, six different types of failsafes have been used and tested at the PSDF: (1) a PSDF-designed failsafe (a metal platform and metal fiber media), (2) a Pall iron aluminide sintered powder fuse, (3) a Pall HR-160 metal fiber failsafe, (4) a Specific Surface ceramic honeycomb failsafe, (5) a CeraMem ceramic honeycomb failsafe, and (6) a Pall HR-160 reversed-media failsafe.

A detailed presentation of the failsafe test program and results obtained has been published previously (Howard, 2007). Because of the development program, reliable failsafes have been identified that effectively prevent particle penetration. With these failsafes, filter element failures have been undetectable during test campaigns.

Future Testing

Plans are underway to test a new backpulse system with a "coupled pulse pressure" or CPPTM (Pall Corporation) design to compare its performance against the jet pulse system currently used. The CPPTM system is designed to achieve high pulse intensity at lower pulse pressures than conventional systems. In conjunction with the CPPTM testing, a new filter system with a single-tiered, modular tube sheet design will be implemented. Compared to the non-modular tube sheet design currently installed, the modular design will allow greater flexibility in areas such as filter element and failsafe configurations and sizes.

Summary

Measurements of particle collection efficiency show that the iron aluminide sintered metal powder elements are better particle collectors than the HR-160 sintered metal fiber elements. While turbine specifications on the overall particle loading can be achieved with either type of element, there is more penetration of large (> 10 μm) particles with the metal fiber elements. This large-particle penetration is of concern, since some turbine specifications call for complete elimination of particles larger than 10 μm .

While the iron aluminide elements offer better particle collection, they are showing signs of a considerable degree of corrosion and sulfidation. The mechanism of the corrosion appears to involve cracking of the protective alumina layer, formation of an iron oxide scale, and sulfidation. The corrosion appears to be indirectly contributing to a steady increase in the baseline pressure drop across the iron aluminide elements. The mechanism is unknown, but it is theorized that the roughening of the filter element surface is effectively retaining fine particles, causing higher residual pressure drop.

In addition to the effect on pressure drop, there is some evidence that the corrosion has produced a slight decrease in the strength of the iron aluminide elements after 7,158 hours of exposure. However, the strength is still well above the mechanical requirement for the filter element structural integrity. Further monitoring of the pressure drop and strength is needed to determine whether a two-year service life is realistic.

Acknowledgments

The authors wish to acknowledge the contributions and support provided by various project managers: Ron Breault (Department of Energy), Neville Holt (Electric Power Research Institute), Nicola Salazar (KBR), and Ben Wiant (Siemens). We would also like to thank Ms. Cheri Moss of Southern Research Institute who conducted the SEM/EDX analysis of the filter elements. This material is based upon work supported by the Department of Energy (DOE) under award #DE-FC21-90MC25140. However, any opinions, findings, conclusions, or recommendations expressed herein are those of the authors and do not necessarily reflect the views of the DOE.

References

Danielewski, M., S. Mrowec, and A. Stoklosa. "Sulfidation of Iron at High Temperatures and Diffusion Kinetics in Ferrous Sulfide" *Oxidation of Metals*, Vol. 17, No. 1-2, pp. 77-97. 1982.

Dahlin, R.S., E.C. Landham, and H.L. Hendrix. "In-Situ Particulate Sampling and Characterization at the Power Systems Development Facility" *Aerosol Science and Technology* 29: 170-182. 1998.

Davidson, Matt D., R.S. Dahlin, E.N. Galloway, X. Guan, H.L. Hendrix, E.C. Landham, and P.T. Scarborough. *Power Systems Development Facility: High-Temperature, High-Pressure Filter System Operations in a Combustion Gas.* 15th International Conference on Fluidized-Bed Combustion. American Society of Mechanical Engineers. New York, New York. 1999.

GE Power Systems, Specification for Fuel Gases for Combustion in Heavy-Duty Gas Turbines, Publication No. GEI-41040G, Revised January, 2002.

Guan, X., B. Gardner, R.A. Martin, and J.D. Spain, *Demonstration of Hot-Gas Filtration in Advanced Coal Gasification System*, 6th International Symposium on Gas Cleaning at High Temperatures, Osaka, Japan. October 20-22, 2005.

Howard, Nikia, X. Guan, R.A. Martin, R.S. Dahlin, and E.C. Landham, *Evaluation of Failsafe Performance for Hot Gas Filtration*. Proceedings of GT2007 ASME Turbo Expo. Montreal, Canada. May 14-17, 2007.

Judkins, R.R. and U.S. Rao. "Fossil Energy Applications of Intermetallic Alloys" *Intermetallics*, Vol. 8, pp. 1347-1354. 2000.

Leonard, Roxann, R. Breault, B.F.Gardner, P. Vimalchand, *Update on Gasification Testing at the Power Systems Development Facility*. 32nd International Technical Conference on Coal Utilization & Fuel Systems. Clearwater, FL. June, 2007.

Martin, Ruth Ann, B.F. Gardner, X. Guan, and H.L. Hendrix, *Power Systems Development Facility: High Temperature, High Pressure Filtration in Gasification*

Operation. 5th International Symposium on Gas Cleaning at High Temperature. Morgantown, West Virginia. September, 2002.

Martin, Ruth Ann, J.D. Spain, B.F. Gardner, and X. Guan, *Characterization of Candle Filter Elements in Coal Gasification Operation*, American Filtration & Separation Society 2005 Annual Conference, Atlanta, Georgia. April 10-13, 2005.

McKamey, C.G., P.F. Tortorelli, E. Lara-Curzio, D. McCleary, J. Sawyer, and R.R. Judkins. *Characterization of Field-Exposed Iron Aluminide Hot-Gas Filter*. 5th International Symposium on Gas Cleaning at High Temperature. Morgantown, WV. September, 2002.

Milbourne, C.G. and W.J. Huff. "Humidity Effects in the Iron Oxide Process for the Removal of Hydrogen Sulfide from Gas." *Industrial and Engineering Chemistry*, Vol. 22, No. 11, pp. 1213-1224. 1930.

Pint, B.A., K.L. More, P.F. Tortorelli, W.D. Porter and I.G. Wright. "Optimizing the Imperfect Oxidation Performance of Iron Aluminides," *Materials Science Forum*, Vol. 369-372, pp. 411-418. 2001.

Spain, Jack D., *Characterization of Filter Elements for Service in a Coal Gasification Environment*. 5th International Symposium on Gas Cleaning at High Temperature. Morgantown, West Virginia. September, 2002.

Tamhankar, S.S., M. Hasatani, and C.Y. Wen. "Kinetic Studies on the Reactions Involved in the Hot-Gas Desulfurization Using a Regenerable Iron Oxide Sorbent" *Chemical Engineering Science*, Vol. 36, pp. 1181-1191. 1981.