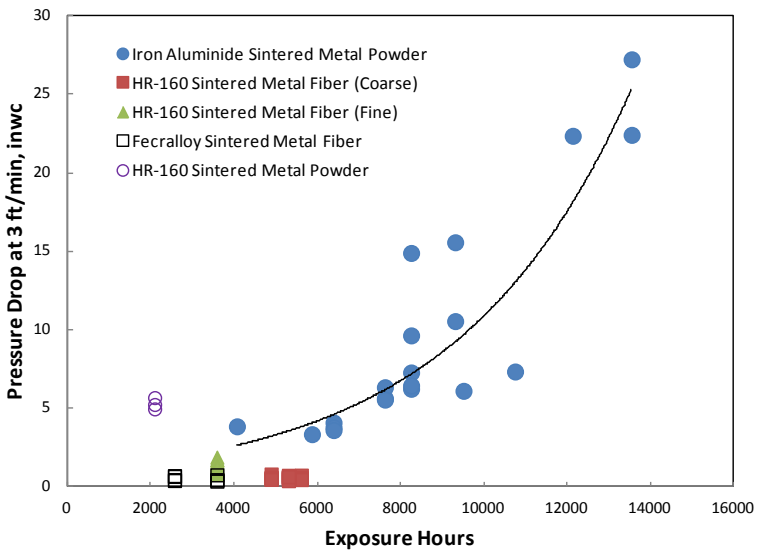
FILTER ELEMENT LIFETIME TESTING AT THE PSDF

Since 1999, the Power Systems Development Facility (PSDF) has been demonstrating efficient removal of gasification ash from syngas using candle filters [1], [2], [3], [4]. Extensive testing with ceramic, sintered metal powder, and sintered metal fiber elements has shown that the mass concentration of gasification ash can be reduced from 10,000-30,000 ppmw at the filter inlet to less than 0.1 ppmw at the filter outlet [4]. Experience at the PSDF has shown that ceramic filter elements are prone to crack and fail during thermal excursions associated with coal feeder trips and other system upsets. To avoid these failures, recent testing at the PSDF has focused on metal filter elements. The maximum accumulated syngas exposure for various element types through March 2013 is given in the table below.

Type of Element	Material	Exposure Hours
Sintered Metal Powder	Iron Aluminide	15,995
Sintered Metal Powder	HR-160	4,541
Sintered Metal Fiber	HR-160 (Coarse)	8,064
Sintered Metal Fiber	HR-160 (Fine)	6,036
Sintered Metal Fiber	Fecralloy	5,018
Sintered Ceramic Powder	Silicon Carbide	1,585
Monolithic Ceramic	Alumina	980

Thus far, some evidence of corrosion has been found in the HR-160 and Fecralloy elements, but there is no evidence that this corrosion is affecting filter performance or pressure drop across the filter media. However, a progressive corrosion and increase in pressure drop has been noted in the iron aluminide elements. The corrosion was first detected as reddish-brown spots of iron oxide after about 2,000 to 3,000 hours of syngas exposure. As the corrosion progressed, the spots of iron oxide merged to form a continuous scale after about 5,000 to 5,500 hours of exposure. With additional exposure, a black scale containing iron sulfide also appeared, and localized areas of sulfidation and plugging were noted in element cross sections. These effects have not resulted in any significant reduction in tensile strength, but a gradual increase in the clean element pressure drop has been noted as shown in the graph below. (It should be noted that the curve for the iron aluminide elements represents the effects of corrosion and sulfidation alone, since the ash layer was removed by air blowing and pressure washing, and microscopic examination of the element surface and element cross sections confirmed that there was no residual ash present.)



Filter element testing at the PSDF has been conducted during gasification runs with Powder River Basin (PRB) subbituminous coal, several types of North Dakota lignite, Hiawatha bituminous coal, Illinois-basin bituminous coal, and Alabama bituminous coals. After each test run, selected filter elements have been removed for detailed inspection under the optical microscope, flow testing to check for any change in pressure drop characteristics, and bubble testing to identify any media or weld problems. Scanning electron microscopy (SEM) with energy-dispersive x-ray (EDX) analysis has also been used for detailed examination of corrosion and ash plugging in selected filter element sections.

FILTER TEST PROCEDURES

Each filter element that is evaluated at the PSDF is subjected to a series of tests before it is installed in the hot-gas filter vessel. These "pre-qualification" tests include:

- Visual/microscopic inspection to confirm pore/fiber size and to detect any manufacturing defects or damage
- Bubble testing in pure isopropyl alcohol to detect any holes or cracks in the filter media or weld joints
- Flow testing to establish a clean baseline pressure drop for the element
- Measurement of particulate collection efficiency using resuspended gasification ash in a cold-flow filter vessel

After an element is pre-qualified, it is installed in the hot-gas filter vessel to evaluate how it performs in the filtration of char from coal-derived syngas. In these tests, the full syngas output from the KBR Transport Gasifier is passed through the hot-gas filter vessel, which is a Siemens-Westinghouse two-tier candle filter containing up to 91 filter elements. Typical conditions for these tests are given below.

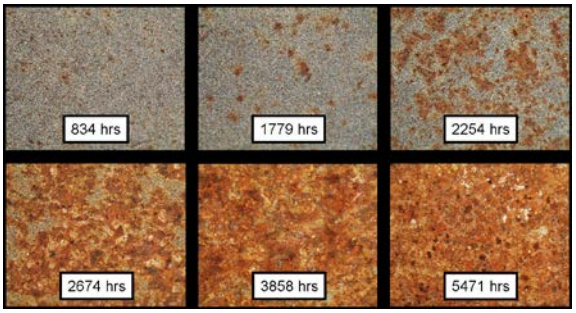
Range of Operating Conditions for Hot-Gas Filter Tests

Number of Filter Elements Installed	72 to 91
Typical Individual Test Duration, hours	250 to 750
Syngas Temperature, ºC (°F)	400 to 510 (750 to 950)
Syngas Pressure, kPa (psig)	965 to 1654 (140 to 240)
Face Velocity, cm/s (ft/min)	1.5 to 2.5 (3.0 to 5.0)
Baseline ΔP, kPa (inH_2O)	9.9 to 39.8 (40 to 160)
ΔP Rise Rate, kPa/min (inH_2O/min)	0.37 to 2.5 (1.5 to 10)
Back-Pulse Timer, min	5 to 20
Back-Pulse Duration, s	0.2
Back-Pulse Pressure (Above System Pressure), kPa (psi)	1723 to 2756 (250 to 400)
Inlet Particulate Loading, ppmw	10,000 to 30,000
Outlet Particulate Loading, ppmw	< 0.1 ppmw
Mean Inlet Particle Size, µm	12 to 20

To assess any change in pressure drop, filter elements that are removed from the hot-gas filter vessel after a gasification run are flow tested in a dirty condition and after cleaning by air blowing and after further cleaning by pressure washing with water. The pressure-washed elements are then bubble tested and examined visually and microscopically to assess the degree of corrosion and to detect any plugging or any other changes in the filter element surface.

CORROSION STUDIES

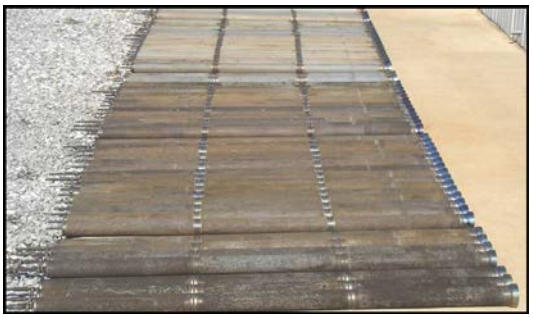
Filter elements exposed at the PSDF have shown varying degrees of corrosion depending on the materials of construction used in the element. A progressive corrosion has been observed in the iron aluminide sintered metal powder elements and failsafes, beginning with the formation of reddish-brown iron oxide spots at an exposure time of about 2,000 to 3,000 hours. The first stage of corrosion observed in the iron aluminide filter elements was the gradual formation of a reddish-brown iron oxide scale as shown in the photograph and micrographs below.



Iron oxide scale formation in iron aluminide elements and corresponding micrographs

To confirm the chemical composition of the scale and compare it to the composition of the original iron aluminide material, filter element specimens were examined by energy dispersive x-ray (EDX) analysis. The EDX results confirmed that the protective alumina surface layer had been compromised, exposing the underlying iron-rich material to attack. Studies at Oak Ridge National Laboratory [5] showed that, relative to other alumina-forming alloys, the alumina scale on iron aluminide spalls more readily, even with the addition of reactive elements such as zirconium and yttrium that are known to enhance scale adhesion. The ORNL studies showed that the fundamental problem is the relatively high coefficient of thermal expansion (CTE) of the iron aluminide compared to the CTE of alumina.

With longer syngas exposure times--beyond about 6,000 hours--we began to observe the formation of a black scale in addition to the reddish-brown iron oxide scale discussed previously. The transition from a reddish-brown iron oxide scale to a black scale can be seen in the layout of elements shown below.


Increasing presence of black scale on the older elements

The effect of sulfidation can be seen in cross sections of the older iron aluminide elements. Micrographs and EDX spectra confirm the presence of plugging associated with localized sulfidation. This finding was consistent with the observations of McKamey et al [6] who reported extensive sulfidation and plugging in non-pre-oxidized iron aluminide elements after 1,628 hours of use in the hot-gas filter system at the Wabash River gasification plant. Based on the PSDF observations and analysis by PSDF and ORNL, the mechanism of the iron aluminide corrosion appears to be as follows:

1. Protective alumina layer is damaged by differential expansion effects during startup and other thermal transients.
2. Underlying iron is attacked by steam to form iron oxide spots that gradually merge into an iron oxide scale.
3. The iron oxide scale is sulfidized by reaction with H_2S to form iron sulfide.

CONCLUSIONS

Based on experience at the PSDF and studies conducted by ORNL, it appears that the protective alumina layer on the iron aluminide is being compromised, allowing the underlying iron to be

attacked by steam and H_2S. The attack produces an iron oxide scale and a sulfidized scale on the filter surface, along with localized regions of sulfidation and plugging in the filter element cross section. While there is some evidence of a loss of material, tensile strength measurements have not shown a statistically significant loss in strength. However, we have observed a steady increase in the filter element pressure drop with increasing syngas exposure, and the pressure drop increase appears to be accelerating with increasing exposure time. Pressure drop measurements on elements with tightly bonded residual ash suggest that the residual ash has more effect on pressure drop as the filter surface becomes more corroded. This suggests that there is an interaction between the ash and the corroded filter surface that effectively alters the near surface residual cake formation. Additional testing will be required to determine the lifetime of the iron aluminide elements with acceptable pressure drop.

ACKNOWLEDGEMENTS

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