METAL FILTERS FOR PRESSURIZED FLUID BED COMBUSTION (PFBC) APPLICATIONS

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ABSTRACT

Advanced coal and biomass-based gas turbine power generation technologies (IGCC, PFBC, PCFBC, and Hipps) are currently under development and demonstration. Efforts at the Siemens Westinghouse Power Corporation (SWPC) have been focused on the development and demonstration of hot gas filter systems as an enabling technology for power generation. As part of the demonstration effort, SWPC has been actively involved in the development of advanced filter materials and component configuration, has participated in numerous surveillance programs characterizing the material properties and microstructure of field-tested filter elements, and has undertaken extended, accelerated filter life testing programs. This report reviews SWPC's material and component assessment efforts, identifying the performance, stability, and life of porous commercial metal, advanced alloy, and intermetallic filters under simulated, pressurized fluidized-bed combustion (PFBC) conditions.

LIST OF ACRONYMS

DOE	Department of Energy
EDX	Energy dispersive x-ray analysis
in-wg	Inches water gauge
PCFBC	Pressurized circulating fluidized-bed combustion
PFBC	Pressurized fluidized-bed combustion
NETL	National Energy Technology Laboratory
SEM	Scanning electron microscopy
STC	Science and Technology Center
SWPC	Siemens Westinghouse Power Corporation
USF	U.S. Filter/Fluid Dynamics

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EXECUTIVE SUMMARY

As a key component in advanced coal or biomass-based power applications, hot gas filtration systems protect the downstream gas turbine components from particle fouling and erosion, cleaning the process gas to meet emission requirements. When installed in either pressurized fluidized-bed combustion (PFBC) or integrated gasification combined cycle (IGCC) plants, lower downstream component costs are projected, in addition to improved energy efficiency, lower maintenance, and elimination of additional and expensive fuel or flue gas treatment systems. As a critical component, long-term performance, durability and life of the porous filter elements are essential to the successful operation of hot gas filtration systems in advanced combustion and gasification applications.

Porous metal filter media as Hastelloy X were used to construct particulate filtration elements during the late 1970's. Accelerated oxidation or corrosion, and loss of tensile strength and load bearing capability of the porous metal filter media resulted. As proposed PFBC system operating temperatures were increased to 870°C (1600°F), efforts were refocused through the early 1990's on the development of filter systems which utilized monolithic ceramic clay bonded silicon carbide candle filters. For operation in combustion gas environments, oxide-based, porous ceramic filter matrices were developed in order to achieve the chemical stability requirements for materials during long-term operation. Both oxide, as well as nonoxide porous ceramic filters were installed and operated in Siemens Westinghouse's Advanced Particulate Filtration systems at the American Electric Power PFBC Tidd Demonstration Plant in Brilliant, Ohio, and at the Foster Wheeler pressurized circulating fluidized-bed combustion (PCFBC) test facility in Karhula, Finland. Siemens Westinghouse conducted extensive filter material surveillance programs for candles that had been operated in each facility. Failure of the oxide-based candles was attributed to thermal failure of alumina/mullite matrix resulting during long-term pulse cycling of the elements, as well as to thermal shock of the matrix during plant thermal transient events. For the nonoxide-based clay bonded silicon carbide elements, elongation and failure of the candles resulted which lead to improvements and stabilization of the clay binder, while oxidation of the silicon carbide grains and volume expansion of the component were addressed through the inclusion of oxidation resistant grain and/or binder additives.

To provide a more 'ruggedized' filter system, emphasis in the mid-1990's was focused on the development and manufacture of advanced nonoxide- and oxide-based, porous second-generation, continuous fiber ceramic composite and filament wound filter elements which were projected to have significantly improved fracture toughness characteristics over that of the first-generation monolithic ceramic filter materials. When tested in Siemens Westinghouse filter systems, oxidation of the nonoxide-based elements lead to brittle failure of the candles, while debonding of external particulate filtration membranes, and failure initially occurred along seams and non-integral flanges and end caps of the advanced second-generation oxide-based candle filters.

Towards the end of the 1990's, advanced corrosion resistant porous metal and intermetallic filter media were considered as a potential low risk alternative for use in PFBC hot gas filtration systems, particularly when installed in second-generation PCFBC systems operating at temperatures of 650-750°C (1200-1400°F). Porous advanced metal and intermetallic filters were expected to be non-brittle (i.e., ductile), and exhibit improved thermal stress damage tolerance in comparison to ceramic filter elements. Advantages over ceramic candles also included eliminating the need for thermal regenerators, the ability to directly weld metal filters to support structures to form hermetic seals, eliminating the use of ceramic fiber dust seals, and the ability to form metals in a wide variety

of shapes. These advantages would permit more robust designs to be considered, improve packaging, lower system costs, and increase process operating reliability.

In September 1998, Siemens Westinghouse Power Corporation (SWPC) initiated effort on the Metal Filters for Pressurized Fluid Bed Combustion Applications program to develop and evaluate the use of porous, high temperature, sinter bonded fiber and powder metal media candle filters in PFBC and PCFBC systems. Advanced alloys selected for evaluation in this program included Haynes 230, Haynes 214, Haynes 188, Haynes 556, iron aluminide (FeAl), and Fecralloy (FeCrAl(Y)) porous metal media manufactured by U.S Filter/Fluid Dynamics (USF), Pall Advanced Separation Systems, Mott Corporation, Fairey Microfiltrex, and Technetics. Elements constructed from 310S, Inconel 600, and Hastelloy X were included as commercially available materials for comparison with the performance and stability of the advanced alloys and intermetallic media in this program.

Testing of composite candle filters constructed from joined metal media filter sections was conducted in Siemens Westinghouse's bench-scale PFBC particulate filtration system in Pittsburgh, PA. Composite candle filter elements were installed and operated in a 650°C (1200°F), 760°C (1400°F), and 840°C (1550°F), pressurized (1.103 kPa (147 psi)), simulated PFBC process gas environment containing 6% O₂, 7% CO₂, 73% N₂, 14% H₂O, and 200 ppm (max) SO₂, for periods of ~250, 500, and 1000 hours. Post-test characterization of each commercially available, advanced alloy metal media, and intermetallic filter media included determination of the residual process temperature strength, and an assessment of the microstructural and/or phase stability of the porous filter matrix. Testing was similarly conducted at 840°C (1550°F) in the absence of gas phase sulfur, and with the addition of 1 ppm gas phase alkali in the sulfur-laden simulated PFBC process gas environment.

Based on testing conducted in this program, porous sinter bonded FeAl and Fecralloy media are identified as candidate materials for construction of metal filter elements for installation and potential extended operation in high temperature, gas phase alkali-free, PFBC/PCFBC applications. In the presence of 1 ppmv gas phase alkali, accelerated oxidation, pore closure, crack formations through the surface oxide, and/or removal of filter media sections limit the use of all porous sinter bonded metal media filter elements during operation in an 840°C (1550°F) PFBC/PCFBC process gas environment.

1.0 INTRODUCTION

1.1 Program Overview and Filter Element Construction

In September 1998, Siemens Westinghouse Power Corporation (SWPC)¹ initiated effort on the Metal Filters for Pressurized Fluid Bed Combustion (PFBC) Applications program² to develop and evaluate the use of porous, high temperature, metal media candle filters in PFBC and pressurized circulating fluidized bed combustion (PCFBC) systems. In conjunction with U.S. Filter/Fluid Dynamics (USF),³ Pall Advanced Separation Systems, Mott Corporation, Fairey Microfiltrex,⁴ Technetics, and Ultramet, SWPC produced composite metal media filter elements (Figure 1) for exposure to simulated PFBC conditions in the SWPC Science and Technology Center (STC) test facilities in Pittsburgh, PA.

Advanced alloys selected for evaluation in this program included Haynes 230, Haynes 214, Haynes 188, Haynes 556, iron aluminide (FeAl), and Fecralloy (FeCrAl(Y)) porous metal media (Table 1). Elements constructed from 310S, Inconel 600, and Hastelloy X were included as commercially available materials for comparison in this program. Metal filter elements manufactured by USF consisted of ~25 μm diameter metal media fibers that were sinter bonded at high temperature, forming a ≤ 1 mm thick filtration mat layer (Table 2, Figure 2). The filtration media was subsequently inter-layered with wire mesh screens for structural support, wrapped around a perforated, densified metal tube, and longitudinally seam welded. Joiner end rings of 310S were welded to both ends of the ~20.3 cm long cylindrical USF filter media sections for assembly of multiple units into an ~1.5 m composite candle filter. Densified end caps and SWPC's fail-safe/regenerator-contained flanges were welded to the ends of the 1.5 m long cylindrical filter body [1].

Figure 1 — Metal filters for PFBC and PCFBC applications.

¹ Formerly Westinghouse Electric Corporation.

² DOE/NETL Contract No. DE-AC26-98FT40002.

³ Currently Pall Corporation.

⁴ Currently Microfiltrex, a member of the Porvair Filtration Group.

TABLE 1
DEVELOPMENTAL AND COMMERCIALY AVAILABLE METAL FILTER MEDIA

Supplier	U.S. Filter/ Dynamics	Pall Advanced Separation Systems	Mott Corporation	Fairey Microfiltrex	Technetics	Ultramet
Media Architecture	Fibers	Powder	Powder	Fibers	Fibers	Reticulated Foam (b)
310S	X	X				
Inconel 600	X		X			
Hastelloy X	X	X				
Haynes 214	X					
Haynes 556	X					
FeAl (Mod)	X	X (a)				
Fecralloy	X			X	X	
Haynes 188	X					
Haynes 230	X					
Ni						X

(a) 2% Cr.

(b) Sections were not included within the composite candle filter architecture, but exposed to simulated PFBC process gas at temperature in a flow-over fashion.

Similarly, the architecture of the Fairey Microfiltrex filter element sections consisted of a longitudinally seam welded, porous, fibrous, filtration mat and an external wire mesh support layer that were welded to an underlying perforated cylindrical metal structural support. In contrast, the Technetics filter element sections consisted of a porous, fibrous, metal filtration layer, and an external wire mesh support overlayer. Joiner end rings of 310S were welded to the Fairey Microfiltrex and Technetics filter element sections.

The Pall and Mott filter element sections consisted of ~25 μm metal particles (i.e., powder) that were sinter bonded into an ~2 mm and ~4 mm thick porous filter wall, respectively. In contrast to the seamless monolithic Pall 310S and Mott Inconel 600 filter media sections, the Pall Hastelloy X filter section contained a longitudinal seam weld. Joiner end rings of 310S were added to the cylindrical Pall and Mott filter sections, prior to assembly with the Fairey Microfiltrex and Technetics filter sections, forming an ~1.5 m composite candle. Fail-safe/regenerator-contained flanges and end caps were welded to the body of each composite candle filter.

1.2 Program Objectives

The objectives of this program were to evaluate the potential use of porous metal media for hot gas filter applications in PFBC systems. Siemens Westinghouse has worked with key industry metal media suppliers to develop suitable, corrosion-resistant materials representing both the metal powder, fiber, and wire laminate technologies. The application of metal media to PFBC systems requires materials to be capable of withstanding operating temperatures of 650°C to 900°C (1200°F to 1650°F) in coal combustion process streams that potentially contain gas phase sulfur and alkali.

TABLE 2
METAL FILTER ELEMENTS
— COMPONENT ARCHITECTURE AND MATERIALS OF CONSTRUCTION —

Supplier	Metal Media	Media Architecture	Outer Wire Mesh	Internal Structural Core	Longitudinal Weld	Weld Material	End Rings
U.S. Filter	310S	Fiber	Hast X/Hast X (a)	310S	Included	556 Wire	310S/310S**
	Inconel 600	Fiber	Hast X/Hast X (a)	310S	Included	556 Wire	310S/310S**
	Hastelloy X	Fiber	Hast X/Hast X (a)	310S	Included	556 Wire	310S/310S**
	Haynes 214	Fiber	Fecralloy/ Fecralloy or Hast X or Inconel (a)	310S	Included	556 Wire or Inconel	310S/310S**
	Haynes 556	Fiber	Hast X/Hast X (a)	310S	Included	556 Wire	310S/310S**
	FeAl (Mod)	Fiber	Hast X/Hast X (a)	310S	Included	TBD (Kanthal)	310S/310S**
	Fecralloy	Fiber	Fecralloy/ Fecralloy or Hast X (a)	310S	Included	Kanthal or 556 Wire or Hastelloy	310S/310S**
	Haynes 188	Fiber	Hast X/Hast X (a)	310S	Included	556	310S/310S**
	Haynes 230	Fiber	Hast X/Hast X (a)	310S	Included	556	310S/310S**
Pall	310S	Powder	None	None	None	309/310S	310S/310S **
	Hastelloy X	Powder	None	None	Included	Hastelloy C276	310S/ Hastelloy C276**
	Iron Aluminide	Powder	None	None	None	309/310S	310S/310S**
Mott	Inconel 600	Powder	None	None	None	Inconel 82	310S
Fairey Microfiltrex	CCTF™ Fecralloy	Fiber	Hastelloy C276	Hastelloy C276 316L Support Core	Included	Autogeneous ***	310S
Technetics	Fecralloy*	Fiber			Included	Hastelloy X	310S
Ultramet	Ni	Reticulated Foam	None	None	None	None	None

(a) Mesh embedded within filtration mat/Mesh layered between filtration mat and structural support core.

* Fe(22.5% Cr, 5% Al, 0.5% Y)

** Transition ring section added to join porous filter media to SWPC provided end rings.

*** Weld pool generated from parent metal.

As shown in Table 3, five test campaigns were conducted at SWPC STC for periods up to ~1,000 hours in duration, exposing six, 1.5 m, composite metal media candle filters to temperatures of 650°C (1200°F), 760°C (1400°F), and 840°C (1550°F), and system pressures of 1,013 kPa

Figure 2 — Porous, sinter bonded, fibrous, USF filter metal media. (a) Composite architecture; (b) Cross-sectioned filtration media wall; (c) External filtration surface; (d) Internal structural mesh surface.

TABLE 3
SIMULATED PFBC TEST CAMPAIGNS

Test Campaign	System Temperature	Segment No. 1	Segment No. 2	Segment No. 3
1	650°C (1200°F)	242 hrs	500 hrs	—
2	760°C (1400°F)	258 hrs	524.5 hrs	1016 hrs
3	815-840°C (1500-1550°F)	259 hrs	476 hrs	986 hrs
4	840°C (1550°F) (a)	253 hrs	546.5 hrs	1014 hrs
5	840°C (1550°F) (b)	225 hrs	496 hrs	—
6	840°C (1550°F) (c)	81 hrs Steady State	10,110 Pulse Cleaning Cycles Representing 5055 Operating Hours	17 Thermal Transients
	840°C (1550°F) (b)	282 hrs		

(a) Absence of SO₂/SO₃.

(b) Inclusion of SO₂/SO₃ and gas phase alkali.

(c) Particulate filtration and life testing.

(147 psia), under simulated PFBC process gas conditions containing 200 ppmv SO₂/SO₃ (Figure 3). Gas phase alkali was included in the 840°C (1550°F) simulated PFBC process gas environment in Test Campaign No. 5, at concentrations of 1 ppmv. Pre- and post-test characterization of the component, metal media, and welds included visual inspection, room temperature gas flow resistance measurements, microstructural analyses, and high temperature tensile strength testing. A final test campaign series was conducted in which 1.5 m and 2.0 m metal media candle filters were subjected to 840°C (1550°F) steady state particulate filtration, accelerated pulse cycling, and thermal transient testing. Fecralloy elements manufactured by Mott⁵ were included in Test Campaign No. 6. In a parallel effort at SWPC STC, 1.5 m Mott elements were exposed to the 840°C (1550°F) simulated PFBC process gas containing gas phase alkali and sulfur.

⁵ Formerly Bekaert Fibre Technology.

Figure 3 — Composite metal filter array used in SWPC STC PFBC simulator testing.

2.0 COMPONENT AND MATERIALS EVALUATION

2.1 Room Temperature Gas Flow Resistance Measurements

As shown in Figure 4, the as-manufactured, room temperature, gas flow resistance through the sinter bonded fibrous, USF, Fairey Microfiltrex, and Technetics metal media filter element sections was ≤ 2 in-wg at 10 ft/min.⁶ In contrast, the as-manufactured, room temperature, gas flow resistance through the sinter bonded metal powder, Pall and Mott filter media ranged from 4 to 10 in-wg at 10 ft/min. In terms of component architecture and relative gas flow resistance, the porous sinter bonded metal powder media is analogous to the thicker walled, oxide or nonoxide-based, monolithic, ceramic filter materials (i.e., Coors P-100A-1 alumina/mullite; Schumacher FT20; Pall 326; Blasch and Ensto mullite bonded alumina; etc.), while the porous sinter bonded fibrous metal media is analogous to the thinner walled, continuous fiber ceramic composites (CFCC; 3M oxide/nonoxide, McDermott, and Techniweave CFCCs), and filament wound porous ceramic filter matrix (DuPont⁷ PRD-66).

Figure 4 — Room temperature gas flow resistance measurements of as-manufactured, porous, metal filter media.

⁶ SWPC specifications: ≤ 10 in-wg at 10 ft/min.

⁷ Currently GE Power Systems.

2.2 Metal Filter Media and Component Integrity

Table 4 identifies the order of the filter element sections within the six composite candle filters that were used in this test program. After completion of 500 hours of 650°C (1200°F) simulated PFBC testing, all filter element sections remained intact (Table 5). As temperature was increased to 760°C (1400°F), circumferential failure of the Pall 310S filter element sections resulted along the metal joiner ring welds (Figure 5). When removed from the ash hopper, the brittle fractured surface of the powder metal media was observed.

At temperatures >650°C (>1200°F), debonding and removal of sections of the USF Haynes 556 media resulted, reducing the expected particulate collection efficiency of the porous filtration media. Similar failure of the Pall 310S and USF Haynes 556 media occurred at temperatures of 815-840°C (1500-1550°F).

In the presence of gas phase sulfur and alkali at 840°C (1550°F), densification of the outer surface of the USF Fecralloy, Haynes 230, 310S, and Inconel 600 filtration media resulted. In addition, failure of the Pall FeAl and 310S matrices, the USF Haynes 214, Haynes 188, Haynes 556, and Hastelloy X matrices, and Fairey Microfiltrex Fecralloy matrix occurred. Circumferential cracks near the metal joiner ring of the Mott Inconel 600 filter section were observed, and the integrity of the Technetics' longitudinal seam weld was questioned.

TABLE 4 COMPOSITE METAL FILTER ELEMENTS	
Flange	Flange
USF 214 USF 230 USF 556 USF 188 USF Fecralloy USF IN600 USF Hast X USF 310S	Pall FeAl Mott Inconel 600 Pall Hast X-1309H Pall 310S-329S100-11 Fairey CCTF™ Technetics Fecralloy
End Cap	End Cap

2.3 Mechanical Properties — Process Temperature Tensile Strength

With the exception of the Pall FeAl filter element sections, the length of all metal filter media sections was 13.5 cm (~5-5/16 in). The FeAl filter elements sections used in this program were Pall's standard 44.5 cm (15.5 in) length materials. After removal of the heat affect zone adjacent to the welds along the metal joiner rings, and longitudinal sectioning removing weld seams, if present, both the as-manufactured and simulated PFBC-exposed filter media (Test Campaign No. 1 through No. 6) were prepared for tensile strength testing. Standard tensile bars⁸

⁸ Overall dimensions: 12.4 cm (4.88 in) long x 0.64 cm (0.25 in) wide; Gauge section: 3.8 cm (1.5 in) long x 1.27 cm (0.5 in) wide.

TABLE 5
METAL FILTER MEDIA AND COMPONENT PHYSICAL INTEGRITY

Filter Media	Simulated PFBC Operating Conditions				
	650°C (1200°F)	760°C (1400°F)	815-840°C (1500-1550°F)	840°C (1550°F) — No SO ₂ —	840°C (1550°F) — SO ₂ & Na —
Pall FeAl	Intact	Intact	Intact	Intact	Failure at weld; Volume expansion; Raised Bubbles; Longitudinal and circumferential distortion within 225 hrs
USF Fecralloy	Transverse linear indications and through-cracks along longitudinal weld seam after 242 hr; Section removed and media was modified	Intact	Intact	Intact	Intact; Densified external surface (496 hrs)
USF Haynes 230	Intact	Intact	Intact	Intact	Intact; Densified external surface (496 hrs)
USF Haynes 214	Intact	Intact	Intact	Intact	Removal of the filtration media and outer mesh within 496 hrs
USF Haynes 188	Intact	Intact	Intact	Intact	Expansion and separation of filtration media radiating from the circumferentially welded metal joiner rings within 496 hrs (i.e., elephant foot formation); Longitudinal cracks within filter media
USF Haynes 556	Intact	Isolated removal of the outer filtration mat occurred within 1,016 hrs	Isolated removal of the outer filtration mat occurred within 259 hrs	Isolated removal of the outer filtration mat occurred within 253 hrs	Expansion and separation of filtration media radiating from the circumferentially welded metal joiner rings within 496 hrs (i.e., elephant foot formation); Longitudinal cracks within filter media
USF 310S	Intact	Intact	Intact	Intact	Intact; Densified external surface (496 hrs)
USF Inconel 600	Intact	Intact	Intact	Intact	Intact; Densified external surface (496 hrs)
USF Hastelloy X	Intact	Intact	Intact	Intact	Expansion and separation of filtration media radiating from the circumferentially welded metal joiner rings within 496 hrs (i.e., elephant foot formation); Longitudinal cracks within filter media; Embrittlement and cracking of the filtration media
Pall 310S	Intact	Circumferential failure at metal ring weld within 258 hrs	Circumferential failure at metal ring weld within 259 hrs	Circumferential failure at metal ring weld within 253 hrs	Circumferential failure at metal ring weld within 225 hrs
Pall Hastelloy X	Intact	Intact	Intact	Intact	Surface scaling (496 hrs)
Mott Inconel 600	Intact	Intact	Intact	Intact	Crack detected near circumferential weld to ring with 225 hrs
Fairey Microfiltrex Fecralloy	Intact	Intact	Intact	Intact	Failure of the external Hast C276 mesh; Localized removal of filtration mat within 225 hrs
Technetics Fecralloy	Intact	Intact	Intact	Intact	Continuity of the longitudinal weld in question (496 hrs)

Pall 310S

USF 556

Pall FeAl

Figure 5 — Failure of porous metal filter media during simulated PFBC operation.

USF 214

USF 556 (Top)
USF 188 (Bottom)

Pall Hastelloy X

Microfiltrex
Fecralloy

Mott Inconel 600

Technetics Fecralloy

Figure 5 (Cont'd.) — Failure of porous metal filter media during simulated PFBC operation.

were cut from each filtration media, and subjected to tensile pull testing at the respective simulated PFBC process operating temperatures. As-manufactured filter media was similarly subjected to tensile strength testing at 650°C (1200°F), 760°C (1400°F) and 840°C (1550°F) in static air in order to establish the initial strength of the porous filter media. Figures 6-9 identify the impact of simulated PFBC exposure on the residual tensile strength of the porous sinter bonded filter materials. In general, as the process operating temperature increases, the strength of the porous metal filter media decreases. In addition, as-manufactured and residual process temperature strength are shown to be dependent on the architecture of the filtration media (i.e., sinter bonded fibers vs powders; monolithic filter wall vs inclusion of mesh support layers), as well as the composition of the metal alloys or intermetallic substrates used to construct the porous filter element sections.

The porous, fibrous, nickel-based, USF filter media generally tended to initially experience an increase and subsequently a reduction in tensile strength during operation in the 650°C (1200°F) simulated PFBC environment. With the exception of the USF Fecralloy media, an increase in tensile strength for the USF iron-based alloys was observed at 650°C (1200°F). The 650°C (1200°F) tensile strength for the USF cobalt-based media remained relatively constant with time. As simulated PFBC operating temperatures were increased to 760-840°C (1400-1550°F), the tensile strength of the USF filter media decreased. Although dependent on the alloy or intermetallic composition, process temperature tensile strength generally stabilized with continued simulated PFBC exposure time. Typically the presence of gas phase sulfur in the 840°C (1550°F) PFBC process gas promoted a slight further reduction in metal media tensile strength in comparison to the strength of the various USF filter materials during exposure in a sulfur-free process gas environment. In contrast, significant reduction in metal media tensile strength resulted at 840°C (1550°F) when gas phase alkali and sulfur were present in the simulated PFBC process gas stream. Similar trends were observed for the Pall, Mott, Fairey Microfiltrex, and Technetics porous filter media.

Figure 6 — Process temperature tensile strength of the iron-based, intermetallic and advanced alloy, porous filter media after exposure to simulated PFBC operating conditions.

Figure 7 — Process temperature tensile strength of the iron-based, advanced and commercial alloy, porous filter media after exposure to simulated PFBC operating conditions.

Figure 8 — Process temperature tensile strength of the nickel-based, commercial alloy, porous filter media after exposure to simulated PFBC operating conditions.

Figure 9 — Process temperature tensile strength of the nickel and cobalt-based, advanced alloy, porous filter media after exposure to simulated PFBC operating conditions.

3.0 MICROSTRUCTURAL ANALYSIS

In addition to the visual observations and mechanical strength test results, post-test characterization of the various porous commercial, advanced alloy, and intermetallic filter media included scanning electron microscopy/energy dispersive x-ray analysis (SEM/EDX) of polished cross-sections of the candle filter sections exposed in Test Campaigns No. 1 through No. 5. The results of this effort follow.

3.1 Pall FeAl — 74.77% Fe, 22.85% Al, 2.38% Cr

3.1.1 Simulated PFBC Operation

The open porosity throughout the ~1.6 mm, cross-sectioned, sinter bonded FeAl wall was retained during exposure of the Pall filter element sections to 650-840°C (1200-1550°F) simulated PFBC operating conditions (Test Campaign No.1 — No. 5). An ~0.2 μm thick alumina-enriched surface oxide formed along the outer periphery of the sinter bonded FeAl particles after 500 hours of exposure at 650°C (1200°F) (Figure 10). The thickness of the alumina-enriched surface increased to ~0.5 μm after 986 hours of operation at 840°C (1550°F) (Figure 11). Negligible intragranular oxidation was observed, while limited oxidation was detected between adjacent sinter bonded FeAl particles.

Figure 10 — Microstructure of the porous Pall FeAl filter media after 500 hours of operation in the 650°C (1200°F) simulated PFBC process gas environment.

3.1.2 Impact of Gas Phase Alkali

After 225 hours of exposure in the 840°C (1550°F), sulfur and alkali-containing,⁹ simulated PFBC environment, failure of the Pall FeAl filter media occurred. Volume expansion of the FeAl filter matrix resulting in longitudinal, as well as circumferential distortion, and generation of raised bubble-like formations with matrix cracking were evident (Figure 12). The diameter of the bubble-like formations was nominally 25 mm, ranging between 10 and 40 mm. An iron-sulfur-oxide-enriched

⁹ A solution of sodium chloride was injected into the combustor gas passage, which subsequently was converted to sodium sulfate as a result of reaction with gas phase sulfur (SO_2/SO_3) in the simulated PFBC process gas stream.

Figure 11 — Microstructure of the porous Pall FeAl filter media after 258-1016 hours of operation in the 760°C (1400°F) and 840°C (1550°F) simulated PFBC process gas environment.

phase formed along the outer surface of the filter media. Sodium was also detected to be present. An ~0.2-0.3 mm “orange” band formed, defining the outer periphery of the raised bubble-like formations. The “orange” band consisted of iron oxide and sodium sulfate. Along the raised section of the bubble-like formations, ~20-30 μm sodium-sulfur-enriched crystals (i.e., sodium sulfate complex) were formed. Low concentrations of sulfur were present within the underlying iron oxide-enriched outer surface of the raised bubble-like formations.

When the Pall FeAl filter media was cross-sectioned, a continuous melt-like iron oxide phase was seen to have formed along the o.d. and i.d. surfaces of the filter wall, closing porosity, limiting direct passage of the process gas through the filter media (Figure 13). Similarly, the open porosity through the filter wall was reduced as a result of oxidation of the sinter bonded FeAl particles. An iron oxide (FeO)-enriched phase formed along the outer surface of the contained particles. Subsequently FeAl_2O_x and FeAlO_x were formed, surrounding the underlying particle core. Sulfur was detected to be present within both iron aluminate-enriched layers. The central core of the residual FeAl particles was either a densified structure consisting principally of the base metal iron, or an internally oxidized mottled structure consisting principally of iron oxide.

Figure 12 — Failure of the Pall FeAl filter element sections after 225 hours of exposure in the 840°C (1550°F) simulated PFBC process gas environment containing gas phase sulfur and alkali.

Figure 13 — Accelerated oxidation of the Pall FeAl filter media after 225 hours of exposure in the 840°C (1550°F) simulated PFBC process gas environment containing gas phase sulfur and alkali.

3.2 USF Fecralloy — Hoskins 875 fibers: Fe Bal; 22.5% Cr, 5.5% Al, 0.5% Si, 0.1% C

3.2.1 Simulated PFBC Operation

During the initial 242 hours of operation in the 650°C (1200°F) simulated PFBC process gas environment, an ~0.1 µm thick alumina-enriched phase formed along the outer surface of the sinter bonded Fecralloy fibers in the USF filtration media. After 1016 hours of operation in the 760°C (1400°F) simulated PFBC environment, the thickness of the alumina-enriched layer increased to ~0.2-0.5 µm. After 476 hours of operation in the 840°C (1550°F) simulated PFBC process gas environment, a discontinuous ~1-2 µm thick, alumina-iron oxide-chromia layer formed along the outer surface of the Fecralloy fibers (Figure 14). Although surface oxidation of the Fecralloy fibers occurred, the open porosity of the USF filtration media was retained. During simulated PFBC testing, internal oxidation of the Fecralloy fibers was not observed. Negligible oxidation resulted along the fibrous sinter bond interface.

3.2.2 Impact of Gas Phase Alkali

In contrast, the open porosity of the USF Fecralloy filter media was limitedly retained after 225 hours of operation in the 840°C (1550°F) simulated PFBC process gas environment that contained both gas phase sulfur and alkali (Figure 15). Alumina-enriched, ~2-3 µm, needle-like whisker formations resulted along the outer surface of the Fecralloy fibers and Fecralloy structural support mesh. Internal oxidation was not observed throughout the sinter bonded USF Fecralloy fibers.

After 496 hours of operation in the 840°C (1550°F) simulated PFBC process gas environment, the microstructure of the USF Fecralloy fibrous filtration mat was no longer retained due to oxidation and coalescence of the originally porous, fibrous, metal media. Gas flow permeability through the oxidized filtration mat was expected to be severely restricted. Surface and subsurface void formations frequently resulted along the periphery of the USF Fecralloy structural support mesh after 496 hours of operation in the gas phase sulfur and alkali-containing, simulated PFBC process gas environment. Limited sulfation was seen to have resulted within chromia-enriched phases that were embedded within the coalesced USF Fecralloy fibrous media.

3.3 USF Haynes 230 — 57% Ni, 22% Cr, 14% W, 5% Co, 3% Fe, 0.1% C, 0.4% Si, 0.5% Mn, 0.02% La, 0.015% B, 2% Mo, 0.3% Al

3.3.1 Simulated PFBC Operation

The open porosity of the USF Haynes 230 was retained after 500 hours of operation in the 650°C (1200°F) simulated PFBC process gas environment (Figure 16). An ~2.5-3.0 µm thick oxygen-chromium-nickel-enriched layer formed along the surface of the sinter bonded Haynes 230 fibers. Internal oxidation of the fibers was initiated.

Oxidation similarly resulted along the outer surface of the sinter bonded Haynes 230 fibers in the USF filtration media after 258 hours of operation in the 760°C (1400°F) simulated PFBC process gas environment. The open porosity of the filtration media appeared to have been retained during the initial 258 hours of exposure, but may have been limited after 524.5 and 1016 hours of operation. With time, an ~3-5.5 µm thick oxide scale formed along the outer surface of the Haynes 230 fibers in the USF filtration media.

Figure 14 — Morphology of the USF Fecralloy filter media after exposure to 650-840°C (1200-1550°F) simulated PFBC operating conditions.

Figure 15 — Morphology of the USF Fecralloy filtration media after exposure to 840°C (1550°F) simulated PFBC operating conditions containing gas phase sulfur and alkali.

Figure 16 — Morphology of the USF Haynes 230 filter media after exposure to 650-840°C (1200-1550°F) simulated PFBC operating conditions.

At 760°C (1400°F), an external nickel oxide layer initially formed along the outer surface of the Haynes 230 fibers. Subsurface layers were enriched with chromia and minor concentrations of nickel, and a nickel chromate spinel. After 524.5 hours, iron was seen to be present within a thin band that formed between the external nickel oxide and chromia-enriched layers. Along the i.d. or pulse cycled surface of the USF Haynes 230 filter element, a thicker (i.e., ~2 µm) oxygen-nickel-iron-enriched layer formed along the outer surface of the USF Haynes 230 fibers. As the simulated PFBC exposure time was extended to 1016 hours, nickel oxide was identified as the primary phase along the outer surface of the Haynes 230 fibers, with minor concentrations of nickel present within an underlying chromia-enriched subsurface layer.

After 259 hours of operation in the 815°C (1500°F) simulated PFBC process gas environment, oxidation similarly resulted within the USF Haynes 230 filtration media. The open porosity of the filtration media appeared to have been retained during the initial 259 hours of operation, but may have been limited after 986 hours of exposure in the 815-840°C (1500-1550°F) simulated PFBC environment. With time, an ~1.5-6 µm thick oxide scale formed along the outer surface of the 815-840°C (1500-1550°F), simulated PFBC-exposed, Haynes 230 fibers in the USF filtration media. A continuous, ~10-30 µm thick, iron-nickel oxide spinel or nickel oxide layer formed along the o.d. surface of the Haynes 230 filtration media.

Microstructural characterization of the Haynes 230 fibers within the USF filtration media indicated that the fibers were encapsulated with an external chromia-enriched layer, and a subsurface chrome-nickel oxide layer after 259 hours of exposure in the 815°C (1500°F) simulated PFBC environment. After 476 hours of exposure at 815-840°C (1500-1550°F), a nickel oxide or nickel-iron oxide layer, and a subsurface chromia layer formed along the outer surface of the Haynes 230 fibers. After 986 hours of exposure, the outer surface of the oxide was enriched with either a chromium-iron-nickel spinel or a nickel-chromate complex. Chromia was present as the primary subsurface oxide phase.

Void formations were evident along the oxide/base metal interface of the USF Haynes 230 fibers. With continued exposure time at 760°C (1400°F) and 815-840°C (1500-1550°F), this became more pronounced. Intragranular oxidation within Haynes 230 fibers, oxidation between sinter bonded Haynes 230 fibers, as well as oxidation at the Haynes 230 fiber/Hastelloy X structural support mesh interface resulted during exposure of the USF filtration media in the 760°C (1400°F) and 815-840°C (1500-1550°F) simulated PFBC process gas environment.

A Hastelloy X structural support mesh was used in the manufacture of the USF Haynes 230 filtration media. An ~3-6 µm thick oxide scale formed along the outer surface of the 760°C (1400°F), simulated PFBC-exposed, Hastelloy X structural support mesh. Between 258 and 524.5 hours of simulated PFBC exposure, the outer surface of the oxide layer was enriched with oxygen-nickel-iron. Subsurface layers consisted primarily of oxygen-chromium-nickel, and subsequently chromia. After 1016 hours of simulated PFBC operation, the composition of the oxide layer was seen to be enriched with nickel oxide along the outer surface of the scale, followed by an oxygen-nickel-iron-enriched layer, and subsequently a chromia-enriched layer.

In the 815-840°C (1500-1550°F), simulated PFBC-exposed, USF Haynes 230 filter media, an ~1-10 µm thick oxide layer formed along the outer surface of the Hastelloy X, structural support mesh. Within the initial 259 hours of operation, the oxide layer consisted of chrome-nickel spinel. After 476 and 986 hours of exposure, the external chrome-nickel spinel became enriched with iron, while a chromia-enriched subsurface oxide formed.

3.3.2 Impact of Gas Phase Alkali

In the presence of gas phase alkali, oxidation resulted along the outer surface of the Haynes 230 fibers after 225 hours of operation in the 840°C (1550°F) simulated PFBC process gas environment, limiting the open porosity of the filtration media (Figure 17). Internal oxidation and numerous voids were observed along the cross-sectioned surface of the Haynes 230 fibers. With continued exposure (i.e., 496 hours of operation), extensive oxidation was seen to have resulted along the o.d. surface of the filter element sections, as well as within the filtration mat, severely limiting the open porosity of the USF Haynes 230 filter media.

The thickness of the oxide layer that formed along the outside surface of the Haynes 230 fibers tended to increase with continued operation in the 840°C (1550°F) simulated PFBC environment (i.e., ~1-25 µm after 225 hours; ~25-50 µm after 496 hours). The outer surface layer of the oxide after both 225 and 496 hours of operation was enriched with nickel and oxygen. Subsurface oxides consisted of oxygen-chromium-nickel-iron phases.

The thickness of the oxide layer that formed along the outer surface of the Hastelloy X structural support mesh in the USF Haynes 230 filter media tended to remain constant after 225 and 496 hours of exposure in the 840°C (1550°F), alkali-laden, simulated PFBC process gas environment (i.e., ~4.25 µm and ~5 µm, respectively). The composition of the outer surface of the oxide was enriched with oxygen-nickel-chromium. Within the 496 hour-exposed, Hastelloy X structural support mesh, a subsurface chromia layer was identified.

3.4 USF Haynes 214 — 75% Ni, 16% Cr, 3% Fe, 0.05% C, 0.2% Si, 0.5% Mn, 0.01% B, 4.5% Al, 0.1% Zr, 0.01% Y

3.4.1 Simulated PFBC Operation

Limited oxidation resulted within the USF Haynes 214 filter media after 242-500 hours of exposure to the 650°C (1200°F) simulated PFBC operating condition (Figure 18). An <0.5 µm thick scale formed along the external surface of the Haynes 214 fibers that was enriched with nickel oxide. As the thickness of the oxide scale increased to 0.75-1.5 µm, a chromium-iron oxide-enriched external surface scale formed. After extended exposure (i.e., 500 hours), the chromium-iron oxide phase tended to result as the primary phase throughout the <0.5-1.5 µm scale that formed along the outer surface of the Haynes 214 fibers.

Pitting along the external surface of the oxidized Haynes 214 fibers was frequently observed after 500 hours of operation in the 650°C (1200°F) simulated PFBC environment. Similarly, sulfur was observed in isolated areas within the external surface oxide, as well as within the internal inclusions of the Haynes 214 matrix. Void formations along the sinter bond interface that formed between adjacent Haynes 214 fibers were frequently detected, particularly after 500 hours of simulated PFBC testing.

After 258-1016 hours of operation in the 760°C (1400°F) simulated PFBC process gas environment, the open porosity remained within the USF Haynes 214 filtration media. An external oxide layer formed along the outer surface of the simulated PFBC-exposed Haynes 214 fibers, and whisker-like void formations were clearly evident along the internal periphery of the cross-sectioned Haynes 214 fibers (Figure 19). Internal oxidation of the Haynes 214 fibers was also observed.

Figure 17 — Morphology of the USF Haynes 230 filtration media after exposure to 840°C (1550°F) simulated PFBC operating conditions containing gas phase sulfur and alkali.

Figure 18 — Morphology of the USF Haynes 214 filtration media after 500 hours of operation in the 650°C (1200°F) simulated PFBC process gas environment.

Figure 19 — Morphology of the USF Haynes 214 filtration media after 258 hours of operation in the 760°C (1400°F) simulated PFBC process gas environment.

After 258 hours of operation in the 760°C (1400°F) simulated PFBC process gas environment, an ~1.9 μm thick oxide layer formed along the outer surface of the cross-sectioned USF Haynes 214 fibers. The external oxide surface contained O-Cr-Ni, with an underlying O-Ni-Cr-enriched subsurface layer. After 524.5 hours of operation in the 760°C (1400°F) simulated PFBC environment, chromia-enrichment was observed along external surface of the ~1.5 μm thick oxide layer. The underlying subsurface was O-Ni-Al-Cr-enriched. After 1016 hours of operation, the ~2.1-3.1 μm thick oxide consisted of an external chromia-enriched layer and an underlying O-Ni-Cr-enriched layer. An O-Cr-Ni phase was identified within the oxidized islands that penetrated into the base metal substrate (Figure 20).

Figure 20 — Micrograph montage illustrating the morphology of the 760°C (1400°F) USF Haynes 214 filtration media after 1016 hours of operation in the simulated PFBC process gas environment.

Although the open porosity of the USF Haynes 214 filter matrix appeared to be retained after 259 hours of operation in the 815°C (1500°F) simulated PFBC process gas environment, internal oxidation and void formations within the sinter bonded fibers were evident. Separation of the ~2.2-2.9 μm thick surface oxide from the underlying base metal substrate was observed (Figure 21). The surface oxide consisted of an external nickel oxide layer, with subsurface layers of O-Cr-Ni, and chromia.

After 476 hours of operation in the 815-840°C (1500-1550°F) simulated PFBC environment, the thickness of the external surface layer ranged between 5 and 10 μm . The oxide was identified to consist of an external O-Ni(Fe) spinel, with underlying O-Ni-Fe-Cr, and chromia-enriched layers. In contrast, after 986 hours of operation in the 815-840°C (1500-1550°F) simulated PFBC environment, the thickness of the chromia-enriched external surface layer ranged between 3 and 8 μm . Pore closure within the Haynes 214 filtration media appeared to have occurred primarily as a result of the extensive oxidation that resulted after 986 hours of operation in the simulated PFBC process gas environment.

In the absence of gas phase sulfur in the 840°C (1550°F) simulated PFBC process gas environment, pore closure of the Haynes 214 filtration media, and complete oxidation through the cross-sectioned USF Haynes 214 fibers similarly resulted with extended exposure time (i.e., 1014 hours; Figure 22). The composition of the external surface oxide consisted of a chromia-enriched

Figure 21 — Morphology of the USF Haynes 214 filtration media after operation in the 815-840°C (1500-1550°F) simulated PFBC process gas environment.

layer after 253 hours, an external nickel oxide with an O-Cr-Ni subsurface layer after 546.5 hours, and an O-Cr(Ni-Fe) external surface with a subsurface O-Ni-Cr-Fe formation after 1014 hours of simulated PFBC operation.

Figure 22 — Morphology of the USF Haynes 214 filtration media after 1014 hours of operation in the 840°C (1550°F), sulfur-free, simulated PFBC process gas environment.

3.4.2 Impact of Gas Phase Alkali

Extensive oxidation of the sinter bonded Haynes 214 fibers resulted during operation of the USF filtration media in the 815°C (1550°F) simulated PFBC process gas environment that contained gas phase sulfur and alkali. Pore closure within the Haynes 214 filtration media resulted, leading to restriction of gas flow through the filter wall. As shown in Figure 23, complete oxidation through the entire cross-sectioned Haynes 214 fibers resulted after 496 hours of operation.

An ~7-10 µm thick oxide layer was identified to have formed along the outer surface of discrete Haynes 214 fibers after 225 hours of operation in the 840°C (1550°F), simulated PFBC operating environment. The composition of the oxide layer that formed along the surface of the Haynes 214 fibers consisted of an external O-Cr-Ni-Fe layer, with underlying subsurface chromia and O-Ni-Fe-Cr layers, and a S-Cr-enriched phase at the oxide/base metal interface. After 496 hours, the oxide consisted of an external nickel oxide layer, with O-Fe-Ni and O-Cr(Ni)-enriched subsurface layers.

Surface oxidation of the embedded, structural support, Hastelloy X mesh resulted in the formation of an ~2 µm thick oxide after 225 hours of operation in the 840°C (1550°F) simulated PFBC process gas environment that contained gas phase alkali and sulfur. The composition of the oxide layer included an external O-Cr(Ni) layer, and an underlying chromia-enriched subsurface layer. After 496 hours of operation in the 840°C (1550°F) process gas environment, an O-Fe-Ni(Cr)-enriched external layer, and a chromia-enriched subsurface layer formed along the surface of the cross-sectioned Hastelloy X mesh.

Figure 23 — Morphology of the extensively oxidized USF Haynes 214 fibers after 496 hours of operation in the 840°C (1550°F) simulated PFBC process gas environment that contained gas phase sulfur and alkali.

3.5 USF Haynes 188 — Co Bal; 21-23% Cr, 20-24% Ni, 13-15% W, 3% Fe, 0.05-0.15% C, 0.2-0.5% Si, 1.25% Mn, 0.03-0.12% La, 0.015% B

3.5.1 Simulated PFBC Operation

The open porosity of the USF Haynes 188 filter media was retained throughout the filter wall after 242 and 500 hours of exposure in the 650°C (1200°F) simulated PFBC environment (Figure 24). Oxidation of the USF Haynes 188 fibers, however, resulted. Typically two distinct oxide layers were observed along the surface of each material. An ~1-1.6 μm thick oxide-enriched layer formed along the surface of the cobalt-based USF Haynes 188 fibers after 242 hours of exposure in the 650°C (1200°F) simulated PFBC operating environment. The external oxide surface was enriched with either cobalt oxide or cobalt chromate, while generally an underlying chromia-enriched layer was formed adjacent to the residual base metal. With extended exposure to the 650°C (1200°F) simulated PFBC operating conditions, the thickness of the USF Haynes 188 surface oxide layer increased, ranging between ~1.3-2.3 μm. Varying with location, the composition of the external surface of the multi-layered oxide consisted of a cobalt chromate or a chromium-iron-oxide-enriched phase. The underlying oxide layer was determined to consist principally of chromia.

Pit and void formations were evident along the periphery of the USF Haynes 188 fibers, as well as along the sinter bond that resulted between adjacent fibers, after 242-500 hours of operation of the filter media in the 650°C (1200°F) simulated PFBC process gas environment. Crack formations were frequently evident through the discrete external oxide layer that formed along the surface of the USF Haynes 188 fibers. In localized areas, removal of the external oxide layer resulted, leading to discontinuities and exposure of the subsurface oxide scale. Particularly along the o.d. surface of the filter element, debonding of the multi-layered oxide from the USF Haynes 188 base metal substrate was observed. Along the depleted or affected zone of the base metal,

Figure 24 — Morphology of the USF Haynes 188 filtration media after 500 hours of operation in the 650°C (1200°F) simulated PFBC process gas environment.

oxide formations intermittently penetrated to depths of ~2-6 µm into the surface of the Haynes 188 fibers.

After 258-1016 hours of operation in the 760°C (1400°F) simulated PFBC process gas environment, limited oxidation of the porous USF Haynes 188 metal media resulted. The open porosity throughout the filter wall was retained, thus permitting gas flow through the porous filter media.

The thickness of the oxide ranged between ~2.4 to ~8 µm (Figure 25). After 258 hours of operation, an external O-Fe-Co-Cr spinel formed. After 524.5 hours, an external O-Co layer formed, with an underlying subsurface O-Cr(Co) formation. After 1016 hours, the composition of the external surface oxide was determined to include O-Co(Ni-Cr), with the formation of a subsurface O-Cr-Co layer. Under all conditions, a chromia-enriched layer resulted at the base metal substrate interface.

Crack formations through the USF Haynes 188 external oxide layer, separation and/or removal of the oxide from the underlying base metal substrate, internal oxidation within the USF Haynes 188 fiber penetrating along grain boundaries to depths of ~4 µm into the base metal, and oxidation along the sinter bonds between adjacent Haynes 188 fibers were observed during operation of the USF filtration media in the 760°C (1400°F) simulated PFBC process gas environment.

Figure 25 — Impact of PFBC operating temperature and time on the morphology of the USF Haynes 188 filtration media.

External and internal surface oxidation resulted along and within the sinter bonded USF Haynes 188 fibers after 259-986 hours of operation in the 815-840°C (1500-1550°F) simulated PFBC process gas environment. Void formations within the base metal media and oxidation along the grain boundaries contained within the USF Haynes 188 fibers were observed. The thickness of the oxide layer that formed along the outer surface of the USF Haynes 188 fibers ranged from ~4 μm to 7 μm . The external surface oxide layer consisted of O-Cr-Co(Fe). An O-Cr-Co-enriched subsurface layer was also detected. Within the internally oxidized areas of the USF Haynes 188 fibers, chromia-enrichment was identified.

Oxidation of the USF Haynes 188 fibers resulted after 253-1014 hours of operation in the 840°C (1550°F) simulated PFBC process gas environment that did not contain gas phase sulfur. The thickness of the oxide ranged between ~2-4 μm . With time, the extent of oxidation tended to increase (i.e., ≥ 546.5 hours) within the interior of the cross-sectioned Haynes 188 fibers (Figure 26). Oxidation resulted along the grain boundaries, penetrating to depths of ~6 μm . The external surface oxide consisted primarily of chromia.

Cracks were typically observed through the external surface of the oxide layer after 253 hours of operation in the 840°C (1550°F) simulated PFBC process gas environment. Sections of the oxide were also seen to have been removed from the base metal substrate. Where sinter bonded fibers were adjacent to each other, an O-Ni-Co-Cr-Fe layer formed along the external surface of the fiber. An underlying chromia-enriched layer was also present.

Figure 26 — Micrographs illustrating extensive external and internal oxidation that resulted along the surface and throughout the 840°C (1550°F), sulfur-free, simulated PFBC-exposed, USF Haynes 188 filtration media.

3.5.2 Impact of Gas Phase Alkali

Extensive oxidation was observed in localized area of the USF Haynes 188 filtration media after 225-496 hours of operation in the 840°C (1550°F) simulated PFBC process gas environment that contained gas phase sulfur and alkali (Figure 27). An encapsulating oxide layer appeared to

have resulted along the o.d. surface of the USF Haynes 188 filter element section. The resulting oxide layer was identified by EDX analysis to be primarily nickel oxide enriched with cobalt.

Although oxidation of the USF Haynes 188 fibers resulted, extensive sections of the external surface oxide layer were removed after 225 hours of operation in the 840°C (1550°F) simulated PFBC process gas environment (Figure 28). Where present, the external surface oxide was ~2.4 μm thick, and consisted primarily of chromia.

Figure 27 — USF Haynes 188 filtration media after operation in the 840°C (1550°F) simulated PFBC process gas environment containing gas phase sulfur and alkali.

Figure 28 — Extensive oxidation, as well as removal of the oxide formation along the external surface of the USF Haynes 188 fibers after 225 hours of operation in the 840°C (1550°F), gas phase sulfur and alkali-containing, simulated PFBC process gas environment.

3.6 USF Haynes 556 — Fe Bal; 22% Cr, 20% Ni, 18% Co, 2.5% W, 0.1% C, 0.02% La, 3% Mo, 0.02% Zr, 0.6% Ta

3.6.1 Simulated PFBC Operation

Voids were seen to be present not only along the sinter bonds that formed between adjacent fibers in the as-manufactured USF Haynes 556 filter matrix, but were also present along the periphery of the individual USF Haynes 556 fibers. Similar void formations were evident after exposure of the USF Haynes 556 filtration media to 242 and 500 hours of operation under 650°C (1200°F) simulated PFBC process conditions.

Although the relative open porosity was retained within the USF Haynes 556 filtration media after 242 and 500 hours of operation under the 650°C (1200°F) simulated PFBC process conditions, surface oxidation of the Haynes 556 fibers resulted. An ~1-6 µm thick iron-chromium- or iron-enriched surface oxide was formed along the periphery of the Haynes 556 fibers after 242 hours of simulated PFBC operation (Figure 29). With extended exposure, an ~1 µm chromia-enriched layer developed between the external oxide scale and the base metal substrate. The thickness of the multi-layered oxide ranged between ~1-10 µm along the surface of the Haynes 556 fibers after 500 hours of operation.

Figure 29 — Morphology of the USF Haynes 556 filtration media after operation in the 650°C (1200°F) simulated PFBC process gas environment.

Extensive external oxidation resulted along the outer surface of the sinter bonded USF Haynes 556 fibers after 258-1016 hours of operation in the 760°C (1400°F) simulated PFBC process gas environment. The thickness of the external surface oxide appeared to increase with extended operating time:

- Filtration o.d. surface: ~4.6-6.6 µm after 258 hours; ~7.5-10 µm after 524.5 hours; ~8.5-12.5 µm after 1016 hours.
- Filtration i.d. surface: ~6-10 µm after 258 hours; ~10-11.5 µm after 524.4 hours.

Cracks within the external surface oxide formation, and separation of the external oxide layers were observed.

The composition of the external surface oxide consisted of O-Fe(Co-Ni), with underlying subsurface chromia-enriched layers. With time, the concentration of Co-Ni increased within the iron oxide-enriched outer surface layer. Low concentrations of sulfur were identified within the chromia-enriched layers, particularly along the oxide/base metal interface, as well as within the internal oxide formations that resulted along the cross-sectioned surface of the 258 hour, 760°C (1400°F), simulated PFBC-exposed, USF Haynes 556 fibers (Figure 30).

Figure 30 — Impact of PFBC operating temperature and time on the morphology of the USF Haynes 556 filtration media.

When the USF Haynes 556 filtration media experienced 259 hours of operation in the 815°C (1500°F) simulated PFBC process gas environment, extensive oxidation of the metal media fibers resulted, leading to pore closure and localized restriction of gas flow through the filtration media (Figure 30). After 986 hours of operation in the 815-840°C (1500-1550°F) simulated PFBC process gas environment, extensive external surface oxidation of Haynes 556 fibers, complete internal oxidation throughout the entire cross-section fibers, void formations and separation of the oxide from the residual base metal, and oxidation along the sinter bonds between adjacent Haynes 556 fibers were observed.

The thickness of the oxide layer that formed along the outer surface of discrete Haynes 556 fibers ranged between ~6-14.8 μm . Spinel enriched with O-Fe-Cr(Ni-Co) were the primary phases identified along the outer surface of the encapsulating oxide layer. Subsurface O-Cr-Co(Fe) and O-Cr-Co phases were identified.

Extensive oxidation similarly resulted along the external surface, as well as within the interior of the 253-1014 hour, 840°C (1550°F), sulfur-free, simulated PFBC-exposed, USF Haynes 556 fibers. Along the outer surface of discrete Haynes 556 fibers, the thickness of the oxide layer formation ranged between ~8-16 μm . The composition of the oxide consisted primarily of an iron-enriched, Co-Ni, or Ni-Cr spinel external surface layer, with subsurface chromia-enriched, Fe-Ni or Co-Ni spinels. Separation of the external oxide layer from subsurface oxides resulted along the O-Cr-Ni(Co) interface. Along the i.d. or pulse cycled surface of the filtration media, only the underlying chromia-enriched layer remained along the outer surface of the USF Haynes 556 fibers.

After 1014 hours of 840°C (1550°F), sulfur-free, simulated PFBC operation, concentric rings of oxide were seen to form along the outer surface of the cross-sectioned USF Haynes 556 fibers (Figure 31). Complete oxidation throughout the USF Haynes 556 fibers was clearly observed.

Figure 31 — Morphology of the USF Haynes 556 filtration media after 1014 hours of operation in the sulfur-free, 840°C (1550°F), simulated PFBC process gas environment.

3.6.2 Impact of Gas Phase Alkali

Extensive oxidation similarly resulted along the external surface, as well as within the interior of the cross-sectioned, sinter bonded, USF Haynes 556 fibers after 225-496 hours of operation in the 840°C (1550°F) simulated PFBC process gas environment that contained gas phase sulfur and alkali. Oxidation of the sinter bonds between adjacent Haynes 556 fibers also occurred. Although after 496 hours of operation the structural outline of the original USF Haynes 556 fibers was identifiable, complete oxidation of the USF Haynes 556 fibers resulted (Figure 32).

An ~8-13 μm oxide layer formed along the outer surface of discrete USF Haynes 556 fibers after 225-496 hours of operation in the 840°C (1550°F), gas phase sulfur and alkali-containing, simulated PFBC process gas environment. After 225 hours, an O-Fe-Co(Ni)-enriched external surface oxide formed. After 496 hours of operation in the 840°C (1550°F) simulated PFBC environment containing gas phase sulfur and alkali, an O-Co-Cr-Fe external surface spinel formed. Chromia-enriched subsurface layers were generally present.

An amorphous sulfur-enriched phase (i.e., chrome sulfide) was identified within the cross-sectioned Haynes 556 base metal after 496 hours of operation of the USF filtration media in the 840°C (1550°F), gas phase sulfur and alkali-containing, simulated PFBC process gas environment.

Crack formations were seen to have formed within the external oxide that encapsulated the USF Haynes 556 fibers after 496 hours of operation. Separation of the external oxide layer from the residual base metal of the USF Haynes 556 fibers was also observed.

Figure 32 — Extensive oxidation of the USF Haynes 556 filtration media after operation in the 840°C (1550°F), gas phase sulfur and alkali-containing, simulated PFBC process gas environment.

3.7 USF 310S — Fe Bal; 25% Cr, 20.5% Ni, 1.6% Mn, 0.75% Mo, 0.5% Si, 0.05% C

3.7.1 Simulated PFBC Operation

Exposure of the porous USF 310S filter matrix at 650°C (1200°F) to the simulated PFBC process operating environment resulted in oxidation of the 310S filtration mat fibers, leading to coalescence of adjacent fibers that ultimately limited the open porosity of the filtration mat layer (Figure 33). Along the surface of the USF 310S fibers, an ~1 µm thick iron oxide external scale, and an ~1 µm subsurface oxide layer enriched with chromium, iron, and nickel formed after 242 hours of operation. The thickness of both external and subsurface oxides increased to ~3-5 µm after 500 hours of exposure. Typically cracks extending through the oxide scale formations were observed.

Extensive oxidation of the USF sinter bonded 310S fibers resulted after 258-1016 hours of operation in the 760°C (1400°F) simulated PFBC process gas environment (Figure 34). Typically internal oxidation within the sinter bonded 310S fibers was also observed. Localized crack

Figure 33 — Microstructure of the USF 310S filtration media after operation in the 650°C (1200°F) simulated PFBC process gas environment.

Figure 34 — Microstructure of the USF 310S filtration media after 1016 hours of operation in the 760°C (1400°F) simulated PFBC process gas environment.

formations along the external surface oxide layer were also evident along the i.d. or pulse cycled surface of the filter element. Pore closure were observed in localized areas particularly along the outer surface of the USF 310S filter wall after 1016 hours of operation in the 760°C (1400°F) simulated PFBC process gas environment.

The composition of the oxide layer that encapsulated the sinter bonded USF 310S fibers consisted of an iron oxide/spinel-enriched external phase, with an underlying chromia-enriched phase formed at the base metal interface. The thickness of the oxide layer ranged from ~3 μm after 258 hours, to ~6.6-13.3 μm after 524.5 hours, to ~3.5 μm after 1016 hours along the outer surface of the sinter bonded 310S fibers.

Extensive external and internal oxidation resulted along/within the USF sinter bonded 310S fibers after 259-986 hours of operation in the 815-840°C (1500-1550°F) simulated PFBC process gas environment (Figure 35). Localized areas of pore closure were observed along the outer surface of the USF filter wall, particularly after 986 hours of operation. The composition of the oxide layer that encapsulated the 310S sinter bonded fibers consisted of an external iron-nickel spinel with an underlying chromia-enriched layer.

Figure 35 — Morphology of the cross-sectioned, 986 hour, 815-840°C (1500-1550°F), simulated PFBC-exposed, USF 310S filtration media.

In the absence of gas phase sulfur, extensive oxidation of the USF 310S fibers resulted after 253-1014 hours of 840°C (1550°F) simulated PFBC operation (Figure 36). Pore closure of the originally porous filter media was observed, reducing gas flow permeability through the filter wall.

The composition of the oxide layers that resulted along the sinter bonded USF 310S fibers initially consisted of an iron-nickel spinel that was enriched with chromium, and an underlying chromia-iron layer. With time, the composition of the oxide consisted of an external iron-chrome oxide layer, with underlying iron-chromium-nickel oxide, chromium-iron oxide, and nickel-iron-chromium oxide layers.

Figure 36 — Morphology of the USF 310S filtration media after operation in the sulfur-free, 840°C (1550°F), simulated PFBC process gas environment.

3.7.2 Impact of Gas Phase Alkali

Extensive external, as well as internal oxidation of the USF 310S fibers resulted during 225-496 hours of operation in the 840°C (1550°F) simulated PFBC process gas environment that contained gas phase sulfur and alkali (Figure 37). Pore closure of the filtration media was observed, restricting gas flow through the filter wall.

The composition of the oxide that resulted along the external surface of the USF 310S particles consisted of an iron-nickel spinel, with underlying iron oxide and iron-nickel/chromium-enriched subsurface layers.

Along the i.d. or pulse cycled surface of the USF 310S filtration media, crack formations were observed within the external oxide formation, between adjacent sinter bonded 310S particles, as well as along/within the oxide layer that encapsulated the Hastelloy X structural support mesh.

Figure 37 — Morphology of the USF 310S filtration media after operation in the 840°C (1550°F) simulated PFBC process gas environment that contained gas phase sulfur and alkali.

3.8 USF Inconel 600 — Ni Bal; 15.5% Cr, 8% Fe, 0.3% Mn, 0.2% Si, 0.2% Al, 0.2% Ti

3.8.1 Simulated PFBC Operation

At 650°C (1200°F) under simulated PFBC process gas conditions, the Inconel 600 fibers in the USF filtration media underwent extensive oxidation forming an ~8-11 μm thick, multi-layered oxide scale along the outer surface of the fibers in the metal media (Figure 38). As a result of oxidation, the porosity of the filter media was reduced, limiting gas flow permeability through the matrix. The thickness of the oxide layers was comparable after 242 and 500 hours of exposure of the Inconel 600 filter media in the 650°C (1200°F) simulated PFBC process gas environment.

After 242 and 500 hours of exposure, the composition of the densified external, ~2 μm thick, oxide layer that formed along the outer surface of the Inconel 600 fibers consisted of an oxygen-nickel-iron- or oxygen-nickel-enriched phase. Sulfur appeared to be present within the nickel oxide layer, forming a mottled or filamentous structure. A mottled, ~8 μm thick, subsurface layer formed that was enriched with a nickel chromate phase.

Frequently along the o.d. surface of the filtration media, removal of the oxide from the Inconel 600 fibers was observed. In contrast, retention of the oxide scale resulted through the filtration media (i.e., beneath the first fiber layer), encapsulating the 650°C (1200°F), simulated PFBC-exposed, Inconel 600 fibers.

Figure 38 — Morphology of the USF Inconel 600 filtration media after 500 hours of operation in the 650°C (1200°F) simulated PFBC process gas environment.

An oxide layer formed along the outer surface of the sinter bonded USF Inconel 600 fibers after 258-1060 hours of operation in the 760°C (1400°F) simulated PFBC process gas environment. The thickness of the oxide layer was determined to be ~1.5 µm after 258 hours, and ~7.5 µm after 1016 hours of operation (Figure 39). Crack formations in the external oxide layer that encapsulated the Inconel 600 fibers were intermittently seen to result. With time, the extent of internal oxidation within the cross-sectioned, sinter bonded, USF Inconel 600 fibers tended to increase.

The composition of the oxide layer appeared to change with continued operating time. After 258 hours of operation in the 760°C (1400°F) simulated PFBC process gas environment, the oxide layer consisted of a chromia/nickel oxide-enriched, or chrome-nickel spinel. Alternately along the continuous amorphous oxide layer that formed along the outer surface of the filter element, the oxide was principally iron/nickel oxide-enriched, with an underlying chromia layer along the base metal interface. After 1016 hours, nickel oxide was detected within the oxidized Inconel 600 fibers that formed a continuous mat. An underlying iron-nickel oxide spinel, and subsequently nickel chromate phase formed.

In localized areas, a continuous oxide layer appeared to have been formed along the outer surface of the filter element, leading to pore closure, restricting gas flow through the filter media.

Figure 39 — Morphology of the USF Inconel 600 filtration media after operation in the 760°C (1400°F) simulated PFBC process gas environment.

Typically a multi-layered oxide formation encapsulated the outer surface of the cross-sectioned, sinter bonded, Inconel 600 fibers after 259 to 986 hours of operation of the USF filtration media in the 815-840°C (1500-1550°F) simulated PFBC process gas environment (Figure 40). The thickness of the Inconel 600 surface oxidation layer remained generally constant as a function of filter operating time: ~7.6 µm after 259 hours; ~4-7.2 µm after 476 hours; and ~8-10 µm after 986 hours.

The composition of the external surface oxide layer consisted of nickel oxide. Subsurface layers included O-Ni-Fe, O-Ni, O-Ni-Cr, and O-Cr-Ni. Crack formations were seen to intermittently result within the external Inconel 600 surface oxide. Internal oxidation of the cross-sectioned, sinter bonded, Inconel 600 fibers was also observed.

Figure 40 — Microstructure of the USF Inconel 600 filtration media after 986 hours of operation in the 815-840°C (1500-1550°F) simulated PFBC process gas environment.

In the absence of gas phase sulfur within the 840°C (1550°F) simulated PFBC process gas environment, a continuous amorphous oxide layer formed along the o.d. surface of the 1014 hour, 840°C (1550°F), simulated PFBC-exposed, USF filtration media (Figure 41), restricting gas flow through the filter wall. In addition, extensive oxidation resulted along the outer surface of the sinter bonded Inconel 600 fibers after 253-1014 hours of operation. The thickness of the oxide layer that resulted along the external surface of the cross-sectioned, sinter bonded, USF Inconel 600 fibers tended to slightly increase with extended simulated PFBC operating time: ~6.25 µm after 253 hours; ~7.5 µm after 546.5 hours; ~8.3-10 µm after 1014 hours.

The composition of the oxide layer that formed along the external surface of the sinter bonded USF Inconel 600 fibers remained relatively constant during 1014 hours of operation. A nickel/nickel oxide-enriched phase formed along the external surface of the sinter bonded Inconel 600 fibers. Subsurface nickel chromate and chromia-enriched layers were subsequently identified. Crack formations within, and separation of the external oxide phase from underlying subsurface oxides, as well as void formations through the cross-sectioned Inconel 600 fibers were evident.

3.8.2 Impact of Gas Phase Alkali

Extensive oxidation of the sinter bonded Inconel 600 fibers resulted after 225 to 496 hours of operation of the USF filtration media in the 840°C (1550°F) simulated PFBC process gas environment that contained gas phase alkali and sulfur (Figure 42). A continuous, amorphous, nickel/nickel oxide-enriched layer formed along the o.d. surface of the USF Inconel 600 filter element sections. Localized pore closure resulted, restricting gas flow through the filter media. Void formations were observed along the cross-sectioned surface of the simulated PFBC-exposed, Inconel 600 fibers.

Along discrete sinter bonded Inconel 600 fibers, crack formations were observed through the ~6.7 µm thick, multi-phase, external, surface oxide. The oxide consisted of an external O-Ni/Fe-

Figure 41 — Morphology of the USF Inconel 600 filtration media after 1014 hours of operation in the sulfur-free simulated PFBC process gas environment.

Figure 42 — Microstructure of the USF Inconel 600 filtration media after operation in the 840°C (1550°F) simulated PFBC process gas environment that contained gas phase sulfur and alkali.

enriched surface layer. Subsurface nickel/nickel oxide, O-Ni-Cr/Fe, and O-Ni-Cr layers were identified. A chromia-enriched layer was detected along the base metal interface.

3.9 USF Hastelloy X — 47% Ni, 22% Cr, 19% Fe, 9% Mo, 1.7% Co, 0.6% W, 0.5% Mn, 0.3% Si, 0.08% C

3.9.1 Simulated PFBC Operation

The open porosity of the 650°C (1200°F), simulated PFBC-exposed, USF Hastelloy X filtration media was retained after 242 and 500 hours of operation (Figure 43). An oxide scale was seen to have formed along the external surface of the sinter bonded Hastelloy X fibers. The thickness of the oxide scale ranged between ~0.6 and 4 µm after 242 and 500 hours of operation, respectively. The composition of the oxide varied through the thickness of the scale. Typically along the external surface, a nickel-iron-chromium oxide-enriched phase was formed. Enrichment of chromium in the form of a nickel chromate phase tended to result within the subsurface oxide layer.

Figure 43 — Morphology of the USF Hastelloy X sinter bonded filtration media after 242 and 500 hours of operation in the 650°C (1200°F) simulated PFBC process gas environment.

External surface and internal oxidation of the sinter bonded Hastelloy X fibers resulted after 258-1016 hours of operation of the USF Hastelloy X filtration media in the 760°C (1400°F) simulated PFBC process gas environment (Figure 44). Localized areas of pore closure were observed within the sinter bonded Hastelloy X filtration media.

The composition of the oxide layer that formed along the surface of the sinter bonded USF Hastelloy X fibers consisted of an external O-Fe-Ni-Cr spinel, and an underlying chromia-enriched subsurface layer. The thickness of the oxide layer ranged from ~3.75 to 6.6 µm. Crack formations were detected along the external oxide layer that encapsulated the sinter bonded Hastelloy X fibers that were present along the i.d. or pulse cycled surface of the USF Hastelloy X filtration media.

Figure 44 — Morphology of the USF Hastelloy X filtration media after 258-1016 hours of operation in the 760°C (1400°F) simulated PFBC process gas environment. The lower right micrograph illustrates the thickness of the various oxide layers that resulted after 1016 hours.

Extensive oxidation of the sinter bonded Hastelloy X fibers resulted after 259-986 hours of operation of the USF Hastelloy X filter element sections in the 815-840°C (1500-1550°F) simulated PFBC process gas environment. Pore closure resulted in localized areas along the outer surface of the USF Hastelloy X filtration media, particularly after 986 hours of operation.

The thickness of the external oxide layer ranged from ~1.6 to 4.3 µm (Figure 45). The thickness of the oxide layer appeared to minimally increase with extended operating time: ~1.6-3.6 µm after 259 hours; ~2.4-3.3 µm after 476 hours; ~3.1-4.3 µm after 986 hours. The composition of the outer surface of the oxide layer that encapsulated the sinter bonded Hastelloy X fibers tended to vary with time. After 259 hours, an O-Cr-Ni-Fe spinel formed. After 476 hours, an O-Fe-Ni/Cr-rich spinel resulted. After 986 hours, an O-Ni-Fe-Cr phase was observed. Irrespective of the operating time, a chromia-enriched subsurface layer was detected.

Internal oxidation also resulted throughout the cross-sectioned, sinter bonded, USF Hastelloy X fibers. Crack formations were also observed throughout the external oxide layer that encapsulated the sinter bonded, Hastelloy X fibers along the outer surface, as well as i.d. or pulse cycled surface of the USF filtration media.

Figure 45 — Morphology of the sinter bonded USF Hastelloy X fibers as a result of operation in the 815-840°C (1500-1550°F) simulated PFBC process gas environment.

Similarly, extensive external surface oxidation, as well as internal oxidation of the sinter bonded Hastelloy X fibers contained within the USF filtration media resulted after 253 to 1014 hours of operation in the 840°C (1550°F), sulfur-free, simulated PFBC process gas environment. Pore closure resulted along the oxidized outer surface of the USF Hastelloy X filtration media after 546.5 to 1014 hours of operation (Figure 46).

The thickness of the oxidized layer that formed along discrete (i.e., isolated) Hastelloy X fibers was ~2.7-4.2 μm . Spinel formations (O-Cr-Ni-Fe; O-Fe-Ni-Cr) resulted along the external surface of the oxidized Hastelloy X fibers, while an underlying chromia-enriched subsurface layer formed.

Cracks were identified within the oxide layer that formed along the external surface of the Hastelloy X fibers that were present along the i.d. or pulse cycled surface of the 546.5 hour, 840°C (1550°F), simulated PFBC-exposed, USF filtration media.

Figure 46 — Microstructure of the USF Hastelloy X filtration media after operation in the sulfur-free simulated PFBC process gas environment.

3.9.2 Impact of Gas Phase Alkali

Extensive external, as well as internal oxidation of the sinter bonded Hastelloy X fibers resulted after 225-496 hours of operation of the USF filtration media in the 840°C (1550°F) simulated PFBC process gas environment that contained gas phase sulfur and alkali (Figure 47). Pore closure was observed which restricted gas flow through the filter media.

Along the outer surface of discrete Hastelloy X fibers, an ~1.8-2.5 µm thick oxide layer was seen to have formed after 225 hours of operation. The composition of the outer surface of the oxide consisted of an O-Ni-Cr-Fe spinel, and an underlying chromia-enriched subsurface oxide. Low concentrations of sulfur were detected within the subsurface oxide formation.

Figure 47 — Morphology of the USF Hastelloy X filtration media after operation in the 840°C (1550°F) simulated PFBC process gas environment that contained gas phase sulfur and alkali.

3.10 Pall 310S — Fe Bal; 25% Cr, 20.5% Ni, 1.6% Mn, 0.75% Mo, 0.5% Si, 0.05% C

3.10.1 Simulated PFBC Operation

Based on microstructural analyses, a more porous matrix appeared to exist along the outer and inner surfaces of the as-manufactured Pall 310S sinter powder metal filtration media in contrast to a more densified center section. The open porosity of the Pall 310 filter matrix appeared to have been retained after 242 and 500 hours of operation in the 650°C (1200°F) simulated PFBC operating environment (Figure 48).

After 242 hours of operation, an ~1 μm chromium-iron-oxide-enriched scale formed along the surface of the sinter bonded Pall 310S particles. With continued exposure, the thickness of the oxide scale increased to ~4-7 μm . Crack formations were frequently detected within the oxide scale, with spallation resulting, exposing areas of the base metal media. The composition of the ~4-7 μm oxide scale was typically enriched with chromia and/or iron chromate.

Internal oxidation was evident along the surface of the 500 hour, 650°C (1200°F), simulated PFBC-exposed, Pall 310S sinter bonded particles, as well as oxidation along the sinter bonds that remained between adjacent particles within the filtration media.

Figure 48 — Morphology of the Pall 310S filtration media after operation in the 650°C (1200°F) simulated PFBC process gas environment.

The open porosity of the sinter bonded powder, Pall 310S filter media was retained throughout the ~3 mm thick cross-sectioned filter wall after 258 hours of operation in the simulated 760°C (1400°F) PFBC environment. High temperature oxidation of the filter media lead to both internal oxidation of the sinter bonded 310S particles, and the formation of an ~6 μm thick oxide layer along the outer surface of the 310S Pall particles (Figure 49). The external surface of the oxide layer contained iron oxide, and subsequently iron chromate. Frequently the external oxide layer was separated from the subsurface O-Ni-Cr or O-Cr-Ni-Fe spinel. Chromia typically formed at the oxide/metal substrate interface.

Figure 49 — Morphology of the Pall 310S filtration media after 258 hours of operation in the 760°C (1400°F) simulated PFBC process gas environment.

Failure of the Pall 310S filter media typically resulted with extended 760°C (1400°F) simulated PFBC operation, as well as at higher simulated PFBC operating temperatures. The fresh fractured surfaces initiating at the weld and traversing the filtration wall were similar to the fractures resulting for monolithic ceramic filter matrices under thermal fatigue or shock conditions (Figure 50). As a result of failure of the Pall 310S filtration media, additional characterization was not undertaken, since use of this matrix at higher operating temperatures was designated as not feasible.

3.10.2 Impact of Gas Phase Alkali

In the presence of gas phase sulfur and alkali, densification resulting from oxidation of the porous Pall 310S filter media resulted along the o.d. and i.d. surfaces of the 840°C (1550°F), simulated PFBC-exposed, ~3 μm thick filter wall (Figure 51). Densification of the Pall 310S media extended ~1 mm along both surfaces of the cross-sectioned filter wall. As a result, pore closure of the filter media occurred, restricting gas flow through the filter element. In contrast, open porosity was seen to exist along an ~1 mm thick area along the mid-section of the cross-sectioned Pall 310S filter element.

The thickness of the external oxide layer that formed along the o.d. surface of the 840°C (1550°F), gas phase sulfur and alkali-laden, PFBC-exposed, Pall 310S filter element ranged

between ~ 6.6 to ~ 8.3 μm (Figure 52). Two distinct oxides layers were present. The external oxide layer consisted principally of iron oxide, with an O-Ni-Fe spinel formed at the base metal interface.

Extensive external oxidation similarly resulted along the sinter bonded particles that were present along the i.d. or pulse cycled surface of the 840°C (1550°F), gas phase sulfur and alkali-laden, PFBC-exposed Pall 310S filter element (Figure 53). The composition of the external surface of the oxide layer consisted principally of iron oxide, with an underlying chromia formation. In addition to the extensive external surface oxidation that resulted along the o.d. and i.d. surfaces of the Pall 310S filter element, internal oxidation of the sinter bonded 310S particles also occurred.

Where the open porosity existed along the mid-section of the 840°C (1550°F), gas phase sulfur and alkali-laden, PFBC-exposed, Pall 310S filter element, chromia-enriched whisker-like features were seen to extend from the surface of the sinter boned 310S particles (Figure 54). An underlying chromia layer was seen to form at the base metal interface.

Figure 50 — Fracture characteristics of the Pall 310S filtration media at $\geq 650^{\circ}\text{C}$ ($\geq 1400^{\circ}\text{F}$) simulated PFBC operating temperatures.

Figure 51 — Morphology of the Pall 310S filtration media after 225 hours of operation in the 840°C (1550°F), gas phase sulfur and alkali-containing, simulated PFBC process gas environment (o.d. surface).

Figure 52 — Higher magnification micrographs illustrating the thickness and morphology of the oxide layer that formed along the outer surface of the 225 hour, 840°C (1550°F), gas phase sulfur and alkali-containing, simulated PFBC-exposed, sinter bonded, Pall 310S particles (o.d. surface).

Figure 53 — Higher magnification micrographs illustrating the extensive surface and internal oxidation that resulted along and within the sinter bonded Pall 310S particles after 225 hours of operation in the 840°C (1550°F), gas phase sulfur and alkali-containing, simulated PFBC process gas environment.

Figure 54 — Morphology of the cross-sectioned, sinter bonded, Pall 310S particles along the mid-section of the 225 hour, 840°C (1550°F), gas phase sulfur and alkali-containing, simulated PFBC-exposed, filter wall.

3.11 Pall Hastelloy X — 47% Ni, 22% Cr, 19% Fe, 9% Mo, 1.7% Co, 0.6% W, 0.5% Mn, 0.3% Si, 0.08% C

3.11.1 Simulated PFBC Operation

The open porosity of the Pall Hastelloy X filter media was retained after 242 hours of operation in the 650°C (1200°F) simulated PFBC process gas environment. Under these conditions an ~4 µm thick scale intermittently formed along the external surface of the Pall Hastelloy X sinter bonded particles (Figure 55). The external scale contained numerous crack formations and was enriched a nickel-chromium-iron oxide layer. A thinner, more continuous, subsurface oxide was also detected which was principally enriched with a nickel chromium-containing phase.

Limited or negligible oxidation typically resulted throughout the porous Pall Hastelloy X filter media after 500 hours of exposure at 650°C (1200°F) in the simulated PFBC process gas environment.

Figure 55 — Morphology of the Pall Hastelloy X filtration media after 242 hours of operation in the 650°C (1200°F) simulated PFBC process gas environment.

The open porosity of the Pall Hastelloy X filter media was retained after 258, 524.5, and 1016 hours of operation in the 760°C (1400°F) simulated PFBC process gas environment. After 258 hours of exposure, an ~6 µm thick oxide layer formed along the outer surface of the sinter bonded Pall Hastelloy X particles that were present along the o.d. surface of the filter element (Figure 56). The composition of the external ~5 µm thick oxide layer consisted primarily of an O-Ni-Fe spinel. Directly beneath this layer was an ~1 µm thick chromia-enriched layer. In contrast, an oxide layer intermittently was seen to have formed along the surface of the sinter bonded Hastelloy X particles that were present along the mid-section of the 258 hour, 760°C (1400°F),

(a)

(b)

(c)

Figure 56 — Microstructure of the Pall Hastelloy X filtration media after 258 hours of operation in the 760°C (1400°F) simulated PFBC process gas environment. (a) o.d. surface; (b) Mid-section; (c) Pulse cycled or i.d. surface.

simulated PFBC-exposed, Pall filter media. Along the i.d. or pulse cycled surface, an $\sim 4.3\text{--}5.7\text{ }\mu\text{m}$ thick oxide layer formed along the surface of the Hastelloy X sinter bonded particles. The composition of the external surface was nickel oxide, with an underlying O-Fe-Ni spinel phase, and subsequently an $\sim 1.4\text{ }\mu\text{m}$ thick chromia-enriched phase formed adjacent to the base metal interface.

After 524.5 hours of exposure in the 760°C (1400°F) simulated PFBC process gas environment, surface oxidation of the sinter bonded Pall Hastelloy X particles resulted. Internal oxidation and pitting of the underlying base metal directly below the surface oxide formation, and oxidation along the sinter bonded grain (particle) boundaries were evident (Figure 57). The composition of the oxide formation that resulted along the sinter bonded Hastelloy X particles that were present along the o.d. surface of the filter element consisted of an O-Ni-Fe spinel phase, and an underlying chromia-enriched layer. Along the i.d. or pulse cycled surface of the 524.5 hour, 760°C (1400°F), simulated PFBC-exposed, Pall Hastelloy X particles, the composition of the outer surface oxide layer included O-Cr-Ni, with a chromia-enriched layer formed along the base metal interface.

Figure 57 — Micrograph illustrating the morphology of the Pall Hastelloy X filter media after 524.5 hours of 760°C (1400°F) simulated PFBC operation.

After 1016 hours of exposure in the 760°C (1400°F) simulated PFBC environment, surface oxidation, as well as internal oxidation along the sinter bonds between adjacent Pall Hastelloy X particles was evident (Figure 58). The composition of the $\sim 2.85\text{-}5\text{ }\mu\text{m}$ thick oxide layer that formed along the outer surface of the Pall Hastelloy X sinter bonded particles that were present along the o.d. surface of the filter element consisted of an external O-Cr-Ni layer, and an underlying chromia-enriched layer. Along the i.d. or pulse cycled surface of the filter element, an $\sim 3.5\text{-}8.75\text{ }\mu\text{m}$ thick oxide layer was seen to have formed along the outer surface of the sinter bonded Pall Hastelloy X particles. Two distinct oxide layers were observed. The composition of the external oxide layer consisted of O-Ni-Fe-Cr, while an underlying chromia-enriched layer formed.

(a)

(b)

Figure 58 — Morphology of the Pall Hastelloy X filter media along the (a) o.d. surface and (b) i.d. or pulse cycled surface of the filter element after 1016 hours of operation in the 760°C (1400°F) simulated PFBC process gas environment.

Although the open porosity of the Pall Hastelloy X filter media appeared to be retained after 259, 476, and 986 hours of exposure in the 815-840°C (1550-1550°F) simulated PFBC process gas environment, the thickness of the oxide layer that formed along the outer surface of the Pall Hastelloy sinter bonded particles tended to increase with extended operating time: 259 hrs: ~2.4 μm on the o.d. vs ~4 μm on the i.d.; 476 hrs: ~8-10 μm on the o.d.; 986 hrs: ~9 μm on the o.d. vs ~5-9 μm on the i.d. (Figure 59).

Also with increased exposure time, the apparent extent of oxidation at the sinter bonded particle interface, and oxidation of the residual metal substrate directly below the external surface oxidation layer appeared to increase. Typically the composition of the external surface oxidation layer consisted of an O-Ni-Fe spinel along the outer surface of the oxide after 259 hours of simulated PFBC operation. With extended simulated PFBC exposure, the composition of the external surface oxide tended to form an O-Fe-Ni spinel. Irrespective of the exposure time period, a chromia-enriched layer formed at the oxide/base metal interface.

Figure 59 — Oxide formation along the outer surface of the Pall Hastelloy X sinter bonded particles after 259 and 986 hours of operation in the 815-840°C (1500-1550°F) simulated PFBC process gas environment.

Although open porosity existed within the Pall Hastelloy X filter media, oxidation of the sinter bonded metal particles appeared to be enhanced with extended exposure of the filter element in the 840°C (1550°F), sulfur-free, simulated PFBC process gas environment. After 253 hours of operation the thickness of the oxide layer that formed along the surface of the Pall Hastelloy X particles ranged from 2-4 μm . What remained along the surface of the Hastelloy X particles that were present along the o.d. surface of the filter element was a chromia-enriched layer. Within the mid-section of the cross-sectioned filter wall, an outer layer of O-Ni-Fe spinel, and an underlying chromia-enriched layer resulted. Two distinct and separated oxide layers formed along the surface of the Hastelloy X particles that were present along the i.d. or pulse cycled surface of the simulated PFBC-exposed, Pall filter element.

After 546.5 hours of exposure in the 840°C (1550°F) simulated PFBC process gas environment, an ~2.6-3.4 μm thick, chromia-enriched layer formed along the surface of the Pall Hastelloy X particles that were located along the o.d. surface of the filter element. The oxide layer that remained along the surface of the Pall Hastelloy X particles that were present along the i.d. or

pulse cycled surface of the filter element contained cracks, implying that removal of the oxide layer formation may have occurred.

With continued exposure in the simulated PFBC environment (i.e., 1014 hours), an ~16-20 μm thick oxide layer formed along the surface of the Pall Hastelloy X sinter bonded particles that were present along the o.d. surface of the filter element (Figure 60). The composition of the outer oxide layer consisted of O-Fe-Ni, with a subsurface chromia-enriched layer. Significant crack formations were evident along the residual ~3 μm thick oxide layer that remained along the external surface of the particles that were present along the i.d. or pulse cycled surface of the Pall Hastelloy X filter element.

Internal oxidation along the base metal interface, as well as along the sinter bonds that formed between adjacent particles were clearly observed after 253 hours of exposure in the 840°C (1550°F) simulated PFBC process gas environment. Internal oxidation of the Pall Hastelloy X particles was more pronounced with extended simulated PFBC exposure time.

Based on the results generated during simulated PFBC testing, it appeared that pulse cycling reduced the adherence or integrity of the oxide formation to remain attached to the Pall Hastelloy X base metal substrate (Figure 61).

Figure 60 — Morphology of the Pall Hastelloy X filter media after 1014 hours of operation in the 840°C (1550°F) simulated PFBC process gas environment.

3.11.2 Impact of Gas Phase Alkali

The open porosity of the Pall Hastelloy X sinter bonded particle matrix was generally retained after 225 and 496 hours of exposure to the 840°C (1550°F) simulated PFBC process gas environment that contained gas phase sulfur and alkali. Surface oxidation of the contained particles, oxidation of the base metal at the oxide/metal interface, and oxidation along the sinter bonds between adjacent Hastelloy X particles were evident.

Figure 61 — Micrographs illustrating the formation of cracks and separation of the oxide layers along the outer surface of the sinter bonded Pall Hastelloy X particles after 253, 546.5, and 1014 hours of exposure in a sulfur-free simulated PFBC process gas environment.

Typically the oxide formation that resulted along the surface of the sinter bonded Hastelloy X particles existed as a continuous bi-layer structure. The composition of the external oxide surface was enriched with an O-Fe-Ni spinel (~2.5-8.8 µm thick after 225 hours; ~6.5-8.5 µm thick after 498 hours). An underlying chromia-enriched layer formed along the base metal interface (~1.8-5 µm after 225 hours; ~1.6-4 µm after 496 hours).

Near the center of the cross-sectioned filter wall, and particularly along the i.d. or pulse cycled surface of the filter element, crack formations were readily evident through the thickness of the residual oxide formation. Where present, the thickness of the residual oxide formation was estimated to be ~2-5 µm, and was identified to be chromia-enriched. Removal of an external oxide layer which separated from the chromia-enriched surface was expected to have occurred after 496 hours of operation along the pulse cycled surface of the 840°C (1550°F), gas phase sulfur and alkali-containing, simulated PFBC-exposed, Pall Hastelloy X filter media.

3.12 Mott Inconel 600 — Ni Bal, 15.5% Cr, 8% Fe, 0.3% Mn, 0.2% Si, 0.2% Al, 0.2% Ti

3.12.1 Simulated PFBC Operation

In contrast to the U.S. Filter/Fluid Dynamics (USF) Inconel 600 filter media, the ~3.5 mm thick monolithic Mott filter wall consisted of sinter bonded ~50 µm x ~100 µm particles. A more open or porous structure was formed along both o.d. and i.d. surfaces of the filter matrix, with densification resulting along the mid-section of the filtration media. The open porosity of the Mott Inconel 600 filtration media appeared to have been retained after 242 and 500 hours of operation in the 650°C (1200°F) simulated PFBC process gas environment (Figure 62).

After 242 hours of exposure at 650°C (1200°F) in the simulated PFBC operating environment, the Mott Inconel 600 filter media underwent oxidation leading to the formation of an external ~3-3.5 µm thick nickel chromate iron-enriched scale, and an underlying ~4.5 µm thick nickel oxide or nickel chromate-enriched layer, along the o.d. surface of the filter element (Figure 63). The thickness of the oxide coating was not uniformly distributed around the individual sinter bonded Inconel 600 particles, but appeared to preferentially result along the surface that was

directly contacted with the simulated PFBC process gas stream. Along the densely packed mid-section of the Mott Inconel 600 filter wall, limited oxidation was apparent. Along the i.d. surface of the Mott filter media, an $\sim 3.2\text{ }\mu\text{m}$ thick oxide scale was observed to have formed intermittently along the surface of the Inconel 600 sinter bonded particles.

Although intermittently identified, the thickness of the oxide scale which formed along the outer surface of the 500 hour, 650°C (1200°F) simulated PFBC-exposed, sinter bonded, Inconel 600 particles ranged from $\sim 1\text{-}2\text{ }\mu\text{m}$ along the o.d. surface, to $\sim 1\text{ }\mu\text{m}$ along the denser mid-section, to $\sim 2\text{-}4\text{ }\mu\text{m}$ along the i.d. surface of the filter element.

Figure 62 — Morphology of the as-manufactured Mott Inconel 600 filtration media.

Although oxidation occurred throughout the Mott Inconel 600 filter media after 258-1016 hours of operation in the 760°C (1400°F) simulated PFBC process gas environment, open porosity was retained throughout the cross-sectioned, monolithic, sinter bonded particle, filter wall. Typically the oxide layer that formed along the outer surface of the Mott Inconel 600 sinter bonded particles consisted of an external nickel oxide layer, an underlying nickel chromate layer, and a chromia-enriched layer adjacent to the base metal substrate (Figure 64).

Crack formations and debonding of localized areas of the oxide were observed within the Mott Inconel 600 filter media after 258-1016 hours of operation. Where retained, the thickness of the oxide was $\sim 3.3\text{ }\mu\text{m}$ after 258 hours of simulated PFBC exposure; $\sim 6\text{ }\mu\text{m}$ after 524.5 hours of simulated PFBC exposure; and between 4 and $11.7\text{ }\mu\text{m}$ after 1016 hours of simulated PFBC exposure. Internal oxidation and oxidation along the sinter bonds between adjacent particles within the Mott Inconel 600 filter media were also evident.

The open porosity of the Mott Inconel 600 filter media similarly was retained after 986 hours of operation in the $815\text{-}840^{\circ}\text{C}$ ($1500\text{-}1550^{\circ}\text{F}$) simulated PFBC process gas environment. Typically oxidation resulted along the outer surface of the sinter bonded Inconel 600 particles. More extensive oxidation of the filtration media appeared to have resulted along the outer and i.d. or pulse cycled surfaces of the filter element. General trends indicative of oxide growth along the external surface of the sinter bonded Inconel 600 particles as a function of time were not apparent.

Figure 63 — Morphology of the Mott Inconel 600 filtration media after 242 and 500 hours of operation in the 650°C (1200°F) simulated PFBC process gas environment.

Figure 64 — Morphology of the Mott Inconel 600 filtration media after 258 and 1016 hours of operation in the 760°C (1400°F) simulated PFBC process gas environment.

The thickness of the oxide ranged between ~ 8.3 and $\sim 16.6 \mu\text{m}$ after 259 hours of exposure at 815°C (1500°F); between ~ 1.6 and $\sim 7.5 \mu\text{m}$ after 476 hours of exposure at 815 - 840°C (1500 - 1550°F); and between ~ 4.6 and $10 \mu\text{m}$ after 986 hours of exposure at 815 - 840°C (1500 - 1550°F) (Figure 65). Crack formations within the external oxide layer, as well as internal oxidation within the residual base metal of the sinter bonded particles were observed.

Typically nickel oxide resulted along the outer surface of the oxide layer. Beneath this layer, a nickel chromate or O-Fe-Ni- phase formed. A subsurface chromia-enriched layer was also observed. At the base metal/oxide interface, nickel oxide was seen to be present within the 986 hour, 815 - 840°C (1500 - 1550°F), simulated PFBC-exposed, Mott Inconel 600 filter media.

Figure 65 — Morphology of the Mott Inconel 600 filtration media after 259 and 986 hours of operation in the 815 - 840°C (1500 - 1550°F) simulated PFBC process gas environment.

Extensive oxidation resulted along the outer surface of the sinter bonded Inconel 600 particles after 253-1014 hours of operation in the 840°C (1550°F), sulfur-free, simulated PFBC process gas environment. With the exception of localized areas, the open porosity of the filter media was retained.

An internal oxidation zone within the sinter bonded Inconel 600 particles was typically present in localized areas beneath the multi-phase external surface oxide formation (Figure 66). From the external oxide surface to the base metal interface, the composition of the oxide consisted of

- Nickel/low chromium oxide, nickel oxide, nickel chromate spinel, and chromia after 253 hours of operation.

- Nickel chromate, O-Ni-Fe, O-Ni-Cr, and chromia after 546.5 hours of operation.
- Nickel oxide, O-Ni(Fe), O-Cr-Ni, and chromia after 1014 hours of operation.

Along the outer surface of the cross-sectioned filter element, the thickness of the external oxide layer was ~ 7.2 - $17.2\ \mu\text{m}$. Internal oxidation within the sinter bonded particles penetrated to depths of ~ 4.8 - $7.2\ \mu\text{m}$. Along the i.d. or pulse cycled surface of the Mott Inconel 600 filter element, the thickness of the external oxide was $\sim 8.8\ \mu\text{m}$. The thickness of the internal oxidation zone ranged between ~ 12 - $14\ \mu\text{m}$ within the sinter bonded Inconel 600 particles.

Along the i.d. or pulse cycled surface of the Inconel 600 filter media, internal oxidation within the sinter bonded particles, as well as along sinter bonds between adjacent particles were more frequently observed than along the mid-section or outer surface of the simulated PFBC-exposed Mott Inconel 600 filter element.

Figure 66 — Morphology of the Mott Inconel 600 filtration media after 253 and 1014 hours of operation in the 840°C (1550°F), sulfur-free, simulated PFBC process gas environment.

3.12.2 Impact of Gas Phase Alkali

The open porosity of the Mott sinter bonded Inconel 600 filtration media appeared to generally be retained after 225-496 hours of exposure in the 840°C (1550°F) simulated PFBC process gas environment that contained gas phase sulfur and alkali (Figure 67). Where pore closure tended to occur, primarily along the outer surface of the filter element, internal oxidation of the base metal media was observed. After 225 hours in the 840°C (1550°F) simulated PFBC process gas environment, internal oxidation penetrated to depths of $\sim 2\ \mu\text{m}$ into the base metal substrate. After 496 hours, depth of penetration of the internal oxide reached $\sim 10\ \mu\text{m}$.

After 496 hours of operation in the 840°C (1550°F) gas phase sulfur and alkali-containing simulated PFBC process gas environment, chromia-enriched dendritic formations were observed to extend from the surface of the Mott Inconel 600 sinter bonded particles that were present along the mid-section of the filter wall (Figure 68). Beneath the dendritic formations, an ~6 μm thick Cr-Ni-O phase formed.

Along the i.d. or pulse cycled surface of the filter media, crack formations within and localized removal of the oxide layer were evident. In contrast to the outer surface of the filter media, internal oxidation of the Mott Inconel 600 sinter bonded particles was not observed along the i.d. or pulse cycled surface of the Mott Inconel 600 filter media after 225-496 hours of operation.

Figure 67 — Morphology of the Mott Inconel 600 filtration media after 225 and 496 hours of operation in the 840°C (1550°F) simulated PFBC process gas environment that contained gas phase sulfur and alkali

Figure 68 — Micrographs illustrating the morphology of the cross-sectioned Mott Inconel 600 media along the mid-section of the filter wall after 496 hours of exposure in the 840°C (1550°F) simulated PFBC process gas environment containing gas phase sulfur and alkali.

3.13 Fairey Microfiltrex Fecralloy

3.13.1 Simulated PFBC Operation

The as-manufactured Fairey Microfiltrex CCTF™ filter matrix consisted of a filtration mat layer that was surrounded by an open wire mesh screen along its o.d. surface, and was structurally supported along its i.d. surface via a perforated metal cylindrical section. When sectioned, the porous, fibrous, metal media filtration mat was easily removed. As shown in Figure 69, the ~0.5 mm thick Fecralloy filtration media consisted of a porous, sinter bonded, fibrous outer area, and a more densified, underlying, porous filtration area. The cross-sectional diameter of the Fairey Microfiltrex CCTF™ fibers contained in the outer surface filtration mat layer was ~17-30 μm , while the cross-sectioned diameter of the underlying filtration mat fibers (i.e., mid-section to i.d.) was ~8-10 μm .

Figure 69 — Morphology of the as-manufactured Fairey Microfiltrex filtration media.

The open porosity of the Fairey Microfiltrex Fecralloy CCTF™ filtration matrix was retained after 242 and 500 hours of operation in the 650°C (1200°F) simulated PFBC environment. Enrichment of aluminum along the external surface of the filtration mat fibers tended to occur (Figure 70). Oxygen was also detected along the external surface of the filtration mat fibers, implying the formation of alumina or a precursor alumina phase.

In contrast to the filtration media that was used for testing in the 650°C (1200°F) simulated PFBC environment, the outer surface of the Fairey Microfiltrex media that was subjected to the 760°C (1400°F) simulated PFBC environment consisted of the denser layer of ~9-12 μm (cross-sectional diameter) sinter bonded Fecralloy fibers. Along the inner surface of the filtration media, the more open, ~15-30 μm (cross-sectional diameter) sinter bonded fibrous matrix was used.

Although the open porosity of the Fairey Microfiltrex filtration media was retained after 258-1016 hours of operation in the 760°C (1400°F) simulated PFBC process gas environment, both fiber-containing layers experienced surface oxidation (Figure 71). Initially an iron-oxide-enriched, minor chromium-aluminum layer formed along the surface of the finer fibers in the outer surface layer of the Fairey Microfiltrex filtration media. With time, the formation of an underlying iron oxide/alumina-enriched subsurface layer was initiated. After 1016 hours of exposure in the 760°C (1400°F) simulated PFBC environment, the adherent, encapsulating oxide layer consisted solely of

(a)

(b)

Figure 70 — Morphology of the sinter bonded Fairey Microfiltrex Fecralloy fibers after 500 hours of operation in the 650°C (1200°F) simulated PFBC process gas environment. (a) Outer surface fibers; (b) Inner surface fibers.

Figure 71 — Microstructure of the Fairey Microfiltrex Fecralloy fibers after 1016 hours of operation in the 760°C (1400°F) simulated PFBC process gas environment.

iron oxide/alumina (i.e., iron aluminate). The thickness of the resulting oxide tended to be constant as a function of time (i.e., $\sim 0.7\ \mu\text{m}$).

In contrast, the composition of the oxide layer that formed along the outer surface of the coarser fibers contained within the inner section of the Fairey Microfiltrex filtration media was initially iron oxide/alumina (i.e., iron aluminate). With continued exposure time, the concentration of iron decreased, while the concentration of aluminum increased. The thickness of the oxide formed along the surface of the coarser fibers ranged from $\sim 0.3\text{--}0.7\ \mu\text{m}$, tending to increase with extended exposure time. Notably, the thickness of the oxide along the surface of the coarse fibers tended to be less than that formed along the surface of the finer fibers contained along the outer section of the Fairey Microfiltrex filtration mat.

The open porosity of the Fairey Microfiltrex Fecralloy filtration media was retained after 259-986 hours of operation in the $815\text{--}840^\circ\text{C}$ ($1500\text{--}1550^\circ\text{F}$) simulated PFBC process gas environment. Both fine and coarse fibers experienced surface oxidation (Figure 72). Along the outer surface of the finer fibers, an $\sim 0.3\text{--}0.5\ \mu\text{m}$ thick oxide layer resulted. The encapsulating layer principally contained oxides of iron and aluminum. After 259 hours of operation in the 815°C (1500°F) simulated PFBC environment, the concentration of iron-to-aluminum was equivalent. With extended time in the 840°C (1550°F) simulated PFBC environment, the concentration of alumina exceeded that of iron (oxide).

Along the outer surface of the coarser fibers, an $\sim 0.2\text{--}0.6\ \mu\text{m}$ thick oxide layer resulted. Once again, the encapsulating layer principally contained oxides of iron and aluminum. A slightly higher ratio of aluminum-to-iron was identified in the oxide layer after 259 hours of exposure in the 815°C (1500°F) simulated PFBC environment. With extended time at 840°C (1550°F), alumina was seen to form as the primary phase.

The open porosity of the Microfiltrex filtration media was similarly retained after 253-1014 hours of operation in the 840°C (1550°F), sulfur-free, simulated PFBC process gas environment. Along the outer surface of the finer fibers, an $\sim 0.2\text{--}0.5\ \mu\text{m}$ thick oxide layer resulted. The encapsulating layer principally contained oxides of iron and aluminum. After 253 hours of operation, the concentration of iron-to-aluminum was nearly equivalent. With extended time, the concentration of alumina exceeded that of iron (oxide).

Along the outer surface of the coarser fibers, an $\sim 0.2\text{--}1.5\ \mu\text{m}$ thick oxide layer resulted. Once again, the encapsulating layer principally consisted of alumina and iron oxide.

3.13.2 Impact of Gas Phase Alkali

In the presence of gas phase sulfur and alkali, oxidation and densification of the Fairey Microfiltrex fine Fecralloy fibers resulted after 225 hours of operation in the 840°C (1550°F) simulated PFBC process gas environment. Due to extensive oxidation and densification along the outer surface of the filtration mat, gas flow permeability was restricted through the filtration media (Figure 73). Iron oxide phases primarily resulted along the surface of the extensively oxidized, fine Fecralloy fibers. Within the interior of the filtration mat where fine fibers did not experience densification and/or agglomeration, an iron oxide-enriched/alumina layer formed, encapsulating the outer surface of the contained fine Fecralloy fibers.

In contrast, an alumina-enriched/iron oxide layer formed along the outer surface of the coarse Fecralloy fibers after 225 hours of exposure in the 840°C (1550°F) simulated PFBC operating environment (Figure 74).

Figure 72 — Microstructure of the Fairey Microfiltrex Fecralloy fibers after 986 hours of operation in the 815-840°C (1500-1550°F) simulated PFBC process gas environment.

Figure 73 — Densification of the outer surface of the Fairey Microfiltrex filter element section that contained the fine Fecralloy fibers after 225 hours of operation in the 840°C (1550°F), sulfur and alkali-containing, simulated PFBC process gas environment.

Figure 74 — Morphology of the coarse Fecralloy fibers contained within the Fairey Microfiltrex filtration media after 225 hours of operation in the 840°C (1550°F) simulated PFBC process gas environment that contained gas phase sulfur and alkali.

3.14 Technetics Fecralloy

3.14.1 Simulated PFBC Operation

The cross-sectioned surface of the ~1.9 mm, as-manufactured, Technetics Feltmetal Fecralloy, porous filter matrix is shown in Figure 75. The as-manufactured Technetics filter matrix was seen to consist of irregularly shaped (i.e., shaved) metal media fibers that were sinter bonded together, and which were structurally supported by either an external coarse or fine Fecralloy mesh screen, as well as internally embedded mesh screen layers. From the micrographs presented in Figure 75, a denser layer of fibers appeared to be present along the i.d. wall of the filtration mat layer in comparison to areas where fibers were embedded within the structural support mesh screens along the o.d. surface of the filter element. The structural integrity of the Technetics Feltmetal filtration media resulted from the inclusion of the multi-layered mesh screens. This is in contrast to the U.S.Filter-Fluid Dynamics (USF) filter media which incorporated a thick-walled perforated metal cylindrical insert along the i.d. surface of the filter element to provide enhanced structural support and strength, or the Fairey Microfiltrex filter media which utilized an outer mesh, as well as an interior, thick-walled, perforated metal architecture.

The open porosity of the Technetics Feltmetal Fecralloy filter matrix was retained after 500 hours of exposure to the 650°C (1200°F) simulated PFBC process gas environment. The composition of the Technetics Fecralloy filtration mat fibers appeared to be comparable to that of the exterior and interior-embedded, structural support mesh screen, thus making the Feltmetal filter matrix a compositionally homogeneous filter body.

The formation of a detectable oxide-enriched layer along the o.d. surface of the filtration mat fibers or structural support mesh screens was not observed during the initial 242 hours of exposure of the Technetics Feltmetal Fecralloy filter to the 650°C (1200°F) simulated PFBC process gas environment. With continued exposure, enrichment of aluminum was detected along the o.d. surface of the filtration mat fibers, as well as the structural support mesh.

Figure 75 — Morphology of the as-manufactured Technetics Fecralloy filter media.

The open porosity of the ~1.6 mm thick, sinter bonded, fibrous, Technetics filtration media was retained after 1016 hours of exposure in the 760°C (1400°F) simulated PFBC process gas environment. Limited oxidation resulted along the surface of the shaved, sinter bonded, Fecralloy fibers. As a function of time, the thickness of the external oxide layer increased from 0.13 μm after 258 hours of operation to 0.5-0.8 μm after 1016 hours of operation (Figure 76). With time, the composition of the oxide changed from initially being enriched with an iron oxide complex, to that of an iron-alumina-oxygen-enriched phase (i.e., iron aluminate).

Limited oxidation resulted along the outer surface of the embedded Technetics Fecralloy structural support mesh. The thickness of the oxide layer ranged from 0.2 to 0.6 μm . Compositionally, the oxide layer was determined to be enriched with oxygen, iron, and aluminum (i.e., iron aluminate).

Similarly the open porosity of the porous, sinter bonded, Technetics Fecralloy filtration media was retained after 986 hours of operation in the 815-840°C (1500-1550°F) simulated PFBC process gas environment. The thickness of the oxide layer that formed along the outer surface of the Fecralloy fibers and embedded mesh increased with extended exposure time (~0.5-1.0 μm , and ~0.66-2.2 μm , respectively; Figure 77). The composition of the oxide layer that formed along the outer surface of the Fecralloy fibers consisted of alumina or alumina/iron oxide (iron aluminate) phases, while the oxide layer that formed along the outer surface of the embedded mesh consisted principally of alumina with minor contributions of iron oxide.

The open porosity of the porous, sinter bonded, Technetics Fecralloy filtration media was similarly retained after 546.5 hours of operation in the 840°C (1550°F), gas phase sulfur-free, simulated PFBC process gas environment.

Figure 76 — High magnification micrograph illustrating the thickness and morphology of the oxide layer that formed along the outer surface of the cross-sectioned Fecralloy fibers contained within the 1016 hour, 760°C (1400°F), simulated PFBC-exposed, Technetics filtration media.

Figure 77 — Microstructure of the Technetics Fecralloy filtration media as a result of operation in the 815-840°C (1500-1550°F) simulated PFBC process gas environment.

The thickness of the oxide layer that formed along the outer surface of the Technetics Fecralloy fibers and embedded mesh generally remained relatively constant with extended exposure ($\sim 0.2\text{--}0.4\text{ }\mu\text{m}$, and $\sim 0.13\text{ }\mu\text{m}$, respectively). In the gas phase sulfur-free environment, the thickness of the oxide layer that resulted along the surface of the Fecralloy fibers and embedded mesh was less than that which resulted during exposure of the Technetics filtration media to the sulfur-containing, simulated PFBC process gas environment.

The composition of the oxide layer that formed along the outer surface of the Fecralloy fibers consisted initially of an iron/aluminum oxide phase (i.e., iron aluminate). With time, the oxide tended to be enriched with alumina. The composition of the oxide layer that formed along the outer surface of the embedded mesh consisted initially of an iron oxide complex. With time, the composition of the oxide layer consisted of an iron/aluminum oxide phase (i.e., iron aluminate).

3.14.2 Impact of Gas Phase Alkali

Extensive surface and internal oxidation of the Fecralloy fibers, and surface oxidation of the embedded mesh, resulted along the outer surface, and approximately through one-half of the cross-sectioned Technetics filtration media as a result of operation in the 840°C (1550°F) simulated PFBC process gas environment that contained gas phase sulfur and alkali (Figure 78). Oxidation of the Fecralloy fibers limited the open porosity of the filter matrix, restricting flow of the process gas through the filter wall.

Where extensive oxidation resulted within the Technetics filter media, an iron oxide phase primarily formed along the outer surface of the Fecralloy fibers (Figure 79). Internal oxidation of the base metal principally lead to the formation of a chromia/iron oxide-enriched matrix. In contrast limited oxidation of the Fecralloy fibers resulted along the inner or pulse cycled surface of the Technetics filter media (Figure 80).

An aluminum/iron oxide-enriched phase initially formed along the outer surface of the extensively oxidized embedded mesh. With time, the composition of the surface oxide contained either alumina or chromia (Figure 81).

Figure 78 — Morphology of the Technetics Fecralloy filtration media after 225 hours of operation in the 840°C (1550°F) simulated PFBC process gas environment containing gas phase sulfur and alkali.

Figure 79 — Higher magnification micrograph illustrating the extensive surface and internal oxidation of the Fecralloy fibers that were present along the outer surface of the Technetics filtration media after 496 hours of operation in the 840°C (1550°F) simulated PFBC process gas environment that contained gas phase sulfur and alkali.

Figure 80 — Micrographs illustrating the morphology and thickness of the oxide formation that resulted along the outer surface of the Technetics Fecralloy fibers that were located near the i.d. or pulse cycled surface of the 496 hour, 840°C (1550°F), simulated PFBC-exposed, filter media.

Figure 81 — Higher magnification micrographs illustrating extensive oxidation of the Fecralloy fibers and embedded mesh that were present along the outer surface of the Technetics filtration media after 496 hours of operation in the 840°C (1550°F), gas phase sulfur and alkali-containing, simulated PFBC process gas environment.

4.0 PARTICULATE FILTRATION

During conduct of the final test campaign (Test Campaign No. 6), metal filter elements were subjected to

- 81 hours of 840°C (1550°F), steady state, particulate filtration
- 10,111 accelerated pulse cleaning cycles, representing 5055 hours of operating life
- 17 thermal transients

in order to demonstrate not only particulate filtration collection capability, but also the stability of each metal matrix architecture to withstand extended life under accelerated pulse cycling and extreme thermal transient conditions. In Test Campaign No. 6, Siemens Westinghouse Power Corporation (SWPC) introduced an additional sinter bonded, fibrous, Fecralloy filter element manufactured by Bekaert/Mott into the test program. The Bekaert/Mott filter element consisted of a Fecralloy felt mat that was wrapped over a cylindrical, open mesh metal grid, structural support. The felt mat was joined by a longitudinal seam weld to form the outer surface of the filter body. Joiner end rings and a closed end cap were welded onto the cylindrical filter element section. As with all metal filter elements, SWPC's fail-safe/regenerator was welded to the filter body, forming the flange.

As shown in Figure 82 and Table 6, 1.5 m and 2.0 m Bekaert/Mott Fecralloy and Pall FeAl filter elements, and 1.5 m Microfiltrex Fecralloy and U.S Filter/Fluid Dynamics (USF) 316L¹⁰ filter elements were included in the Test Campaign No. 6 filter array. Ceramic candle filters were additionally installed within the filter array,¹¹ thus subjecting both filter element types to similar simulated PFBC operating conditions.

Figure 82 — Filter element array for demonstration of particulate removal capabilities and thermal stability of 1.5 m and 2.0 m candle filter elements.

¹⁰ USF commercial grade filter media architecture. SWPC does not support 316L for extended use in high temperature PFBC filter applications due to its lack of oxidative stability.

¹¹ Testing conducted under SWPC STC's Filter Component Assessment program (DOE/NETL Contract No. DE-AC21-94MC31147).

TABLE 6
CANDLE FILTER ARRAY — STEADY STATE, ACCELERATED PULSE CYCLING AND
THERMAL TRANSIENT TESTING^(a)

Array Position	Filter Element	Length, m	Steady State Testing	Accelerated Pulse Cycle Testing	Thermal Transient Testing
1	DuPont PRD-66 <i>C736</i>	1.5	Intact; Planned Removed after Steady State Testing	—	—
2	Techniweave CFCC <i>IP3488-AITF2-03</i>	1.5	Intact; Planned Removed after Steady State Testing	—	—
3	Blasch <i>#3</i>	1.5	Intact; Planned Removed after Steady State Testing	—	—
4	Ferro SiC <i>#2</i>	1.5	Intact; Planned Removed after Steady State Testing	—	—
5	Bekaert/Mott Fecralloy <i>SW030122 006</i>	1.5	Intact; Retained for Continued Testing	Wrinkling of the Filtration Media; Gap Formed along the Longitudinal Seam Observed after Completion of Accelerated Pulse Cycle Testing; Element Retained within the Array for Thermal Transient Testing	Intact; Canted/Bowed; Wrinkling of the Filtration Media; Gap along the Longitudinal Seam Observed; Cross-Sectioned for Microstructural Analysis
6	Microfiltrex Fecralloy <i>ICC108618</i>	1.5	Intact; Retained for Continued Testing	Wrinkling of the Filtration Media at the Metal Joiner Ring Observed after Completion of Accelerated Pulse Cycle Testing; Retained within the Array for Thermal Transient Testing	Intact; Canted/Bowed; Wrinkling of the Filtration Media at the Metal Joiner Ring; Cross-Sectioned for Microstructural Analysis
7	DuPont PRD-66 <i>C-638</i>	1.5	Intact; Retained for Continued Testing	Intact; Retained for Thermal Transient Testing	Intact
8	Techniweave CFCC <i>IP3488 AITF 1-03</i>	1.5	Intact; Retained for Continued Testing	Intact; Retained for Thermal Transient Testing	Intact
9	Blasch <i>#9</i>	1.5	Intact; Retained for Continued Testing	Intact; Retained for Thermal Transient Testing	Broken on Removal from the Filter Array
10	Ferro SiC <i>#1</i>	1.5	Intact; Retained for Continued Testing	Intact; Retained for Thermal Transient Testing	Intact
11	Pall FeAl <i>#11</i>	1.5	Intact; Retained for Continued Testing	Intact; Retained for Thermal Transient Testing	Intact; Cross-Sectioned for Microstructural Analysis
12	USF 316L* <i>D215 #6</i>	1.5	Intact; Retained for Continued Testing	Intact; Retained for Thermal Transient Testing	Intact; Cross-Sectioned for Microstructural Analysis
13	Ferro SiC <i>#2</i>	2.0	Intact; Retained for Continued Testing	Intact; Retained for Thermal Transient Testing	Intact
14	Bekaert/Mott Fecralloy <i>SW03 122 001</i>	2.0	Element Removed after Steady State Testing; Hole through the Filtration Media; Wrinkling of the Filtration Media; Gaps/Openings along the Longitudinal Seam Weld	—	—
15	—	—	—	—	—
16	—	—	—	—	—
17	—	—	—	—	—
18	DuPont PRD-66 <i>C-738</i>	1.5	Intact; Retained for Continued Testing	Intact; Retained for Thermal Transient Testing	Intact
19	Pall FeAl <i>#16</i>	2.0	Intact; Retained for Continued Testing	Intact; Retained for Thermal Transient Testing	Intact

* Commercial Grade Media.

- (a) Steady State Testing: 81 hr 6 min; 14 pulse cleaning cycles delivered to the filter array.
Accelerated Pulse Cycle Testing: 419 hr 46 min; 10,110 pulse cleaning cycles delivered to the filter array.
Thermal Transient Testing: 87 hr 45 min; 17 thermal transients conducted.

After 81 hours of steady state testing, the filtration media along the outer surface of the 2.0 m Bekaert/Mott filter element was seen to have “wrinkled” or buckled (Figure 83). A hole was also seen to have formed through the filter media wall. The 2.0 m Bekaert/Mott element was removed from the filter array prior to continued testing.

Figure 83 — Failure of the 2.0 m Bekaert/Mott candle filter after 81 hrs of high temperature, steady state, PFBC testing.

Wrinkling of the 1.5 m Bekaert/Mott and 1.5 m Microfiltrex filter media (Figure 84), and hole formations through the Bekaert/Mott filter body and along the longitudinal seam weld were also identified after completion of the accelerated pulse cycle segment of Test Campaign No. 6. In

Figure 84 — Wrinkled appearance of the Bekaert/Mott and Microfiltrex filtration media after completion of the simulated PFBC accelerated pulse cycle test campaign.

contrast, the Pall FeAl and USF 316L filter elements were visually observed to have remained intact during steady state and accelerated pulse cycle testing.

In a parallel effort at SWPC STC, a 1.5 m Bekaert/Mott element was exposed to the 840°C (1550°F) simulated PFBC process gas environment containing gas phase sulfur and alkali (Table 7). Once again, “wrinkling” of the Bekaert/Mott filtration media resulted along the area adjacent to the metal joiner ring.

TABLE 7 CANDLE FILTER ARRAY — HIGH TEMPERATURE CORROSION TESTING	
Position	Test Segment No. 1 (281 Hrs)
1	McDermott SCS — #1262 SCS
2	DuPont PRD-66 — C741 New
3	Techniweave CFCC — IP3488 AITF 4-03
4	Blasch — #4
5	Ferro SiC — #3
6	Bekaert/Mott Fecralloy — SW03 122 005

Since subsequent testing of the filter array identified in Table 6 included exposure to a series of seventeen thermal transients representing rapid system temperature decreases experienced during plant shutdown resulting from turbine trip events (Figure 85), the failed Bekaert/Mott and Microfiltrex elements were not removed from the filter array. After conducting the simulated thermal transient sequence, the filter array was slow cooled, and the vessel was open for inspection of the filter elements. The Bekaert/Mott and Microfiltrex elements were seen to be canted primarily below the welded mid-body metal joiner rings (Figure 86). In contrast, the 1.5 m and 2.0 m Pall FeAl and 1.5 m USF 316L filter elements were intact, without any apparent distortion to the metal architecture.

Since a particulate loading of ~100-500 ppm was measured in the isokinetic outlet gas samples taken during thermal transient testing, the 1.5 m Pall FeAl, USF 316L, Microfiltrex Fecralloy, and Bekaert/Mott Fecralloy filter elements were cross-sectioned in order to determine whether ash penetrated through the various porous metal, and to demonstrate the overall effective particulate removal capability of each metal filter media. As shown in Figure 87, ash fines were present along the i.d or pulse cycled surface of the Bekaert/Mott Fecralloy filter element. This primarily resulted from the wrinkled and gap formations that were observed along the longitudinal seam weld which permitted fines to pass directly from the dirty to the clean gas side of the filter array. A trace of ash was observed along the i.d. or pulse cycled surface of the Microfiltrex filter element, while visible evidence of ash was not observed along the i.d. surface of the Pall FeAl or USF 316L filter elements.

Scanning electron micrographs presented in Figure 88, indicate that although extensive oxidation of the commercial USF 316L filter media resulted under 840°C (1550°F) simulated PFBC operating conditions, both the USF 316L and Pall FeAl filter media prevented fines penetration through the porous sinter bonded metal media architecture. In contrast, ash fines were limitedly detected along the o.d. and i.d. surfaces of the Microfiltrex and Bekaert/Mott Fecralloy

Number of Transients Conducted	Temperature Decrease during Transient Event			Temperature Increase during System Reheat		
	840°C (1550°F) to 600°C (1112°F), min	ΔT (°F)/Initial 1 min	ΔT (°F)/Initial 5 min	600°C (1112°F) to 840°C (1550°F), min	ΔT (°F)/Initial 1 min	ΔT (°F)/Initial 5 min
2	~65	-50	-172	~130	+45	+180
5	~35-40	-68	-280	~125	+49	+164
10	~24	-86	-338	~88	+82	+259

Figure 85 — Temperature profile during thermal transient testing.

metal filter media. Although passage of fines through the Bekaert/Mott and Microfiltrex Fecralloy filter wall was not expected to have occurred during conduct of the steady state, accelerated pulse cycling, thermal transient test campaign, failure of the felt mat architecture and seam welds, and the extent of open porosity for capture and retention of ash fines require additional development prior to commercial use of these filter elements. In terms of chemical stability, limited oxidation was seen to have resulted along the sinter bonded particles contained within the Pall FeAl filter element, and within the sinter bonded fibrous Bekaert/Mott and Microfiltrex Fecralloy filter elements.

Figure 86 — Filter elements at the conclusion of steady state, accelerated pulse cycling, and thermal transient testing. From left to right: 2.0 m Pall FeAl, 1.5 m USF 316L, 1.5 m Pall FeAl, 1.5 m Bekaert/Mott Fecralloy, and 1.5 m Microfiltrex Fecralloy metal media filter elements; 1.5 m Ferro SiC, 1.5 m Techniweave CFCC, two 1.5 m DuPont PRD-66, and 2.0 m Ferro SiC ceramic filter elements.

Figure 87 — Photograph illustrating the absence of ash fines along the i.d. or pulse cycled surface of the Pall FeAl, USF 316L, and Microfiltrex Fecralloy filter elements, but minor presence of ash along the i.d. or pulse cycled surface of the Bekaert/Mott Fecralloy filter element at the conclusion of the steady state, accelerated pulse cycle, and thermal transient test campaign. From left to right: Pall FeAl, USF 316L, Microfiltrex Fecralloy, and Bekaert/Mott Fecralloy.

Figure 88 — Scanning electron micrographs illustrating limited penetration of ash fines into the Bekaert/Mott and Microfiltrex Fecralloy filtration media.

5.0 SUMMARY AND CONCLUSIONS

In order to assess the potential viability of metal filter elements for use in pressurized fluidized-bed combustion (PFBC) or pressurized circulating fluidized-bed combustion (PCFBC) systems, the integrity of the filter component architecture, filtration media strength, as well as high temperature corrosion or accelerated oxidative stability, and retention of open porosity through the sinter bonded fibers or particles need to be addressed. Appendix A summarizes the details described in this report, identifying the stability, response, and recommendation for PFBC use of the ten intermetallic, advanced alloy and commercial metal media used to manufacture the U.S. Filter/Fluid Dynamics (USF), Pall, Mott, Fairey Microfiltrex, and Technetics filter elements. Table 8 provides an overview summary, identifying the recommended potential use of the various metal filter elements for extended operation in 1,013 kPa (147 psia), 650°C (1200°F), 760°C (1400°F), and 840°C (1550°F), PFBC systems containing 200 ppmv SO₂/SO₃, in the presence or absence of 1 ppm gas phase alkali.

TABLE 8
RECOMMENDED USE OF METAL FILTER ELEMENTS IN PFBC APPLICATIONS

Filter Media	PFBC Process Operating Conditions				
	650°C (1200°F)	760°C (1400°F)	815-840°C (1500-1550°F)	840°C (1550°F) — No SO ₂ —	840°C (1550°F) — SO ₂ & Na —
Pall FeAl	Yes	Yes	Yes	Yes	No
Microfiltrex Fecralloy	Yes	Yes	Yes	Yes	No
Technetics Fecralloy	Yes	Yes	Yes	Yes	No
USF Fecralloy	Yes	Yes	Yes	Yes	No
USF Haynes 230	Short-term*	Short-term	Short-term	Short-term	No
USF Haynes 214	Short-term	Short-term	Short-term	Short-term	No
USF Haynes 188	Short-term	Short-term	Short-term	No	No
USF Haynes 566	Short-term	No	No	No	No
USF Hastelloy X	Yes	Short-term	Short-term	No	No
Pall Hastelloy X	Yes	Yes	Short-term	Short-term	No
USF Inconel 600	Short-term	Short-term	Short-term	Short-term	No
Mott Inconel 600	Yes	Yes	Short-term	Short-term	No
USF 310S	Short-term	No	No	No	No
Pall 310S	Short-term	Short-term	No	No	No

* 500-1000 hrs.

In the presence of 1 ppmv gas phase alkali, accelerated oxidation, pore closure, crack formations through the surface oxide formation, and/or removal of filter media sections limit the use of all porous sinter bonded metal media filter elements during operation in an 840°C (1550°F) PFBC process gas environment.

Typically the Pall iron aluminide and USF, Fairey Microfiltrex, and Technetics Fecralloy media are identified to be oxidatively stable under gas phase alkali-free, 650°C (1200°F), 760°C

(1400°F), and 815-840°C (1500-1550°F) PFBC process operating conditions. Extended exposure at 840°C (1550°F) beyond 1000 hours of simulated PFBC testing will be required to demonstrate both continued oxidative stability, and potential commercial use of these materials. Both the Fairey Microfiltrex and Technetics elements, however, require structural modifications to be made to the filter architecture (i.e., absence of seam welds and mid-body joiner rings) to assure component integrity during extended commercial use in hot gas filtration applications. Similarly closure of the open porosity along the o.d. and i.d. surfaces of both filter elements through the application of a thin less porous membrane is recommended to prevent capture and retention of ash fines within the sinter bonded fibrous architecture which could ultimately lead to blinding and restricted gas flow through the porous filter media.

As shown in Table 8, the USF Haynes 230, Haynes 214, and Haynes 188 filter media are identified for short-term use in a 650°C (1200°F) PFBC process gas environment. Concern is expressed for use of these materials in higher temperature PFBC applications. As a result of the physical instability of the filtration mat, as well as extensive oxidation at high temperature, initial or short-term use at 650°C (1200°F) is identified for the USF Haynes 556 filter media.

Sinter bonded Hastelloy X fibers or particles used to manufacture the USF and Pall filter elements exhibited temperature stability at 650°C (1200°F) during the initial 500 hours of simulated PFBC testing. Oxidation and localized pore closure of the Hastelloy X filter media limits extended use of both filter elements at PFBC temperatures $\geq 760^{\circ}\text{C}$ ($\geq 1400^{\circ}\text{F}$).

When sinter bonded as a fibrous matrix, the USF Inconel 600 filtration media exhibited only short-term stability in the 650-840°C (1200-1550°F) simulated PFBC process gas environment. In contrast, the heavier, thicker walled, sinter bonded particle, Mott Inconel 600 filter element was identified for use at 650-760°C (1200-1400°F), with only short-term use at 815-840°C (1500-1550°F).

Both the USF and Pall 310S filtration media exhibited short-term stability in the 650°C (1200°F) simulated PFBC process operating environment. Fracture of the Pall 310S metal matrix resulted with extended operation at temperatures $\geq 760^{\circ}\text{C}$ ($\geq 1400^{\circ}\text{F}$), and extensive oxidation and pore closure of the USF 310S filtration media resulted at 760-840°C (1400-1550°F). Neither the Pall nor USF 310S filtration media is recommended for extended use in the PFBC process gas environment.

In general, limited sulfur sorption into the various porous metal media was observed during conduct of the simulated PFBC test program. Retention of the sinter bonds between adjoining fibers, particles, or contained structural support mesh layers was observed during the initial ~1000 hours of simulated PFBC testing. Crack formations and removal of the oxide layer(s) that encapsulated the Haynes 188, Hastelloy X, Inconel 600, and 310S sinter bonded fibers and particles were identified particularly along the i.d. or pulse cycled surface of the USF, Pall, and Mott filter elements.

In summary, from the series of ten commercial metal, advanced alloy, and intermetallic materials assessed during conduct of this program, porous sinter bonded FeAl and FeCrAlloy are identified as candidate materials for construction of metal filter elements for installation and potential extended operation in high temperature, gas phase alkali-free, PFBC applications.

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7.0 REFERENCES

1. Filter Assembly for Metallic and Intermetallic Tube Filters, SWPC, US Patent 6,273,925, 8/14/01.

APPENDIX A

SUMMARY OF METAL FILTER MEDIA STABILITY UNDER SIMULATED PFBC OPERATING CONDITIONS

Pall FeAl	Simulated PFBC Operating Conditions				
	650°C (1200°F)	760°C (1400°F)	815-840°C (1500-1550°F)	840°C (1550°F) — No SO ₂ —	840°C (1550°F) — SO ₂ & Na —
	242 and 500 Hrs	258, 524.5, and 1016 Hrs	259, 476, and 986 Hrs	253, 546.5, and 1014 Hrs	225 and 496 Hrs
Physical Integrity	Intact	Intact	Intact	Intact	Failure at weld; Volume expansion; Raised bubbles; Longitudinal and circumferential distortion within 225 hrs
Open Porosity*	Retained	Retained	Retained	Retained	Internal pore closure after 225 hrs; Formation of a densified ~20-60 µm FeO o.d/i.d. layer
Surface Oxidation*	~0.2 µm alumina-enriched after 500 hrs	~0.5-0.6 µm alumina-enriched after 1016 hrs	~0.2-0.3 µm alumina-enriched after 986 hrs	~0.5 µm alumina-enriched after 1014 hrs	Extensive oxidation after 225 hrs
Internal Oxidation*	Not observed	Not observed	Not observed	Not observed	Extensive oxidation after 225 hrs
Recommendation for PFBC Use	Yes	Yes	Yes	Yes	No

* Based on SEM/EDX analyses.

Microfiltrex Fecralloy	Simulated PFBC Operating Conditions				
	650°C (1200°F)	760°C (1400°F)	815-840°C (1500-1550°F)	840°C (1550°F) — No SO ₂ —	840°C (1550°F) — SO ₂ & Na —
	242 and 500 Hrs	258, 524.5, and 1016 Hrs	259, 476, and 986 Hrs	253, 546.5, and 1014 Hrs	225 and 496 Hrs
Physical Integrity	Intact	Intact	Intact	Intact	Failure of the external Hast C276 mesh; Localized removal of filtration mat within 225 hrs
Open Porosity*	Retained	Retained	Retained	Retained	Extensive pore closure along the external surface of the filter element
Surface Oxidation*	Limited surface oxidation of the ~9-12 µm outer surface fibers, and ~15-30 µm inner surface fibers Enrichment of aluminium and oxygen along the external surface of the fibers after 500 hrs	Limited surface oxidation of the ~9-12 µm outer surface fibers, and ~15-30 µm inner surface fibers Fine fibers: ~0.7 µm after 258-1016 hrs; Initially FeO/Cr-Al formed; Subsurface FeO/AlO formed with time. After 1016 hr, iron aluminate resulted Coarse fibers: ~0.3-0.7 µm oxide formed, increasing with extended time; Initially FeO/AlO resulted; With time the concentration of Fe decreased while Al increased	Limited surface oxidation Fine fibers: ~0.3-0.5 µm oxide Fe-Al-O after 259 hrs Al-Fe-O after 986 hrs Coarse fibers: ~0.2-0.6 µm Fe-Al-O after 259 hrs Al-Fe-O after 986 hrs	Limits surface oxidation Fine fibers: ~0.2-0.5 µm Fe-Al-O after 253 hrs Al-Fe-O after 1-14 hrs Coarse fibers: ~0.2-1.5 µm Al-Fe-O	Extensive oxidation and densification along the outer surface of the filtration mat; Iron oxide phases resulted As isolated fine fibers: Fe-Al-O Coarse fibers: Alumina, FeO
Internal Oxidation*	Not observed	Not observed	Not observed	Not observed	Extensive internal oxidation within fibers present along outer filtration mat; Absence of internal oxidation within discrete internal fibers
Recommendation for PFBC Use	Yes	Yes	Yes	Yes	No

* Based on SEM/EDX analyses.

Technetics Fecralloy	Simulated PFBC Operating Conditions				
	650°C (1200°F) 242 and 500 Hrs	760°C (1400°F) 258, 524.5, and 1016 Hrs	815-840°C (1500-1550°F) 259, 476, and 986 Hrs	840°C (1550°F) — No SO ₂ — 253, 546.5, and 1014 Hrs	840°C (1550°F) — SO ₂ & Na — 225 and 496 Hrs
Physical Integrity	Intact	Intact	Intact	Intact	Continuity of the longitudinal weld in question
Open Porosity*	Retained	Retained	Retained	Retained	Extensive pore closure along the external surface of the filter element
Surface Oxidation*	Limited Aluminum enriched along the external surface of the fibers after 500 hrs	Limited Shaved fibers: ~0.13 µm Fe-O after 258 hrs ~0.5-0.8 µm Fe-Al-O after 1016 hrs Support fibers: ~0.2-0.6 µm Fe-Al-O	Surface oxidation Shaved fibers: ~0.5-1.0 µm Alumina, Al-Fe-O Support fibers: ~0.66-2.2 µm Alumina, minor FeO	Surface oxidation Shaved fibers: ~0.2-0.4 µm Fe-Al-O after 253 hrs Alumina after 546.5 hrs Support fibers: ~0.13 µm Fe-O after 253 hrs Fe-Al-O after 546.5 hrs	Extensive surface oxidation; Volume expansion of fibers; Penetration into ~1/2 of the filtration metal thickness Shaved fibers: Fe-O resulted along the outer surface Cr-Fe-O resulted within the base metal Support fibers: Al/Fe-O after 225 hrs Alumina or chromia after 496 hrs
Internal Oxidation*	Not observed	Not observed	Not observed	Not observed	Extensive internal oxidation within fibers present along outer filtration mat; Absence of internal oxidation within discrete internal fibers
Recommendation for PFBC Use	Yes	Yes	Yes	Yes	No

* Based on SEM/EDX analyses.

USF Fecralloy Hoskins 875 fibers: Fe Bal; 22.5% Cr, 5.5% Al, 0.5% Si, 0.1% C	Simulated PFBC Operating Conditions				
	650°C (1200°F) 242 and 500 Hrs	760°C (1400°F) 258, 524.5, and 1016 Hrs	815-840°C (1500-1550°F) 259, 476, and 986 Hrs	840°C (1550°F) — No SO ₂ — 253, 546.5, and 1014 Hrs	840°C (1550°F) — SO ₂ & Na — 225 and 496 Hrs
Physical Integrity	Transverse linear indications and through-cracks along longitudinal weld seam after 242 hrs; Section removed and media was modified	Intact	Intact	Intact	Intact; Densified external surface (496 hrs)
Open Porosity*	Retained	Retained	Retained	Retained	Limited retention after 225 hrs; Pore closure after 496 hrs
Surface Oxidation*	~0.1 µm alumina-enriched after 500 hrs	~0.2-0.5 µm alumina-enriched after 1016 hrs	~1-2 µm discontinuous alumina, iron oxide, chromia layer after 986 hrs	<1.1 µm discontinuous alumina, iron oxide, chromia layer after 1014 hrs	~2-3 µm alumina needle-like whiskers after 225 hrs; Extensive oxidation after 496 hrs
Internal Oxidation*	Not observed	Not observed	Not observed	Not observed	Extensive oxidation after 496 hrs
Recommendation for PFBC Use	Yes	Yes	Yes	Yes	No

* Based on SEM/EDX analyses.

USF Haynes 230 57% Ni, 22% Cr, 14% W, 5% Co, 3% Fe, 0.1% C, 0.4% Si, 0.5% Mn, 0.02% La, 0.015% B, 2% Mo, 0.3% Al	Simulated PFBC Operating Conditions				
	650°C (1200°F) 242 and 500 Hrs	760°C (1400°F) 258, 524.5, and 1016 Hrs	815-840°C (1500-1550°F) 259, 476, and 986 Hrs	840°C (1550°F) — No SO ₂ — 253, 546.5, and 1014 Hrs	840°C (1550°F) — SO ₂ & Na — 225 and 496 Hrs
Physical Integrity	Intact	Intact	Intact	Intact	Intact Densified external surface (496 hrs)
Open Porosity*	Retained after 500 hrs	Retained after 258 hrs Limited after 524.5 and 1016 hrs	Retained after 259 hrs Limited after 986 hrs	Limited after 253 hrs Closure of pores resulted after 546.5 and 1014 hrs	Limited after 225 hours; Closure of pores after 496 hrs
Surface Oxidation*	~2.5-3 µm O-Cr-Ni (Fe) spinel after 500 hrs	External ~3-5.5 µm NiO layer formed; With time, underlying subsurface oxide transitioned from O-Cr-Ni-based layer to a chromia- enriched layer	Initially fibers were encapsulated with an external chromia-enriched layer, and a subsurface chrome-nickel oxide layer After 476 hrs, chromia was identified as the primary oxide phase After 986 hr, an ~1.5-6 µm Fe-Ni oxide spinel or NiO layer formed	With continued exposure time, the thickness of the surface oxide layer tended to increase: ~1.5 µm at 253 hrs; ~5.5 µm at 546.5 hrs; ~2.5-15 µm at 1014 hrs Initially an external chromia and subsurface O-Cr-Ni spinel formed; With time, a Ni- enriched outer surface resulted with an underlying O-Ni-Fe-Cr-enriched subsurface layer	Thickness of the external oxide layer increased with time: ~1-25 µm after 225 hrs ~25-50 µm after 496 hrs Outer surface layer after 225 and 496 hrs was enriched with Ni and O; Subsurface oxides consisted of O-Cr-Ni-Fe phases
Internal Oxidation*	Internal oxidation between adjacent fibers resulted after 500 hrs	Intragranular oxidation resulted within fibers, between sinter bonded fibers, and at the fiber/structural mesh support interface Void formations at the fiber oxide/base metal interface were observed	Intragranular oxidation resulted within fibers; Oxidation resulted between sinter bonded fibers Void formations at the fiber oxide/base metal interface resulted; Void formations became more pronounced with continued exposure time	After 253 hrs, void formations and internal oxidation were evident	Internal oxidation and numerous voids were observed after 225 hrs
Recommendation for PFBC Use	Acceptable for short-term use	Not recommended for long-term use	Not recommended for long- term use	Not recommended for long- term use	No

* Based on SEM/EDX analyses.

USF Haynes 214 75% Ni, 16% Cr, 3% Fe, 0.05% C, 0.2% Si, 0.5% Mn, 0.01% B, 4.5% Al, 0.1% Zr, 0.01% Y	Simulated PFBC Operating Conditions				
	650°C (1200°F) 242 and 500 Hrs	760°C (1400°F) 258, 524.5, and 1016 Hrs	815-840°C (1500-1550°F) 259, 476, and 986 Hrs	840°C (1550°F) — No SO ₂ — 253, 546.5, and 1014 Hrs	840°C (1550°F) — SO ₂ & Na — 225 and 496 Hrs
Physical Integrity	Intact	Intact	Intact	Intact	Removal of the filtration media and outer mesh within 496 hrs
Open Porosity*	Retained after 500 hrs	Retained after 1016 hrs	Retained after 259 hrs; Limited after 476 and 986 hrs	Retained after 253 hrs; Limited after 1014 hrs	Limited with pore closure after 225 hrs; Pore closure after 496 hrs
Surface Oxidation*	~0.5-1.5 µm chromia/ O-Cr-Fe spinel; Minor sulfur sorption after 500 hrs	Multiple layers: ~2-5 µm After 258 hrs: External O-Ni-(Cr) Internal O-Cr-Ni layer formed After 524.5 hrs: External: Chromia-Ni Sublayer/particles: O-Ni-Cr-(Al) Adjacent to substrate: O-Ni-Al-(Cr) After 1016 hrs: External: Chromia-Ni Sublayer/particles: O-Ni-Cr-(Al) Adjacent to substrate: O-Cr-Ni-(Al) Sinter bond between adjacent fibers appeared to be retained	Multiple layers: ~3-10 µm After 259 hrs: External: NiO Subsurface: O-Cr-Ni (Al) Adjacent to substrate: O-Cr-(Ni-Al) After 476 hrs: External: NiO-(Fe) Subsurface: O-Ni-Fe-Cr Adjacent to substrate: Chromia-Ni After 986 hrs: External: Chromia	Multiple layers: ~3.4- >14 µm After 253 hrs: External: Chromia Subsurface: NiO-(Cr) Interface: O-Ni-Cr-(Al) After 546.5 hrs External: NiO-(Fe-Cr) Subsurface: O-Cr-Ni Adjacent to substrate: O-Ni-(Cr) Extensive oxidation after 1014 hrs	Thickness of the external oxide layer increased with time: 4-16 µm after 225 hrs >18 µm after 496 hrs with pore closure After 225 hrs: External: O-Cr-Ni-Fe Subsurface: Chromia-(Ni) Adjacent to substrate: O-Cr-Ni-S-(Al) After 496 hrs: External: NiO-(Fe) Subsurface: Fe-Ni-O Interface: Chromia-(Ni-Fe)
Internal Oxidation*	Pitting observed along the external surface of the fibers; Void formations observed along the sinter bond interface between adjacent fibers	Significant internal oxidation along the periphery of the fibers penetrating to depths of ~4-8 µm	Significant internal oxidation initially along the periphery of the fibers penetrating to depths of ~13-16 µm after 259 hrs; Oxidation observed throughout the cross-sectioned fibers after 986 hrs	Significant internal oxidation along the entire cross-sectioned fiber surface	Significant internal oxidation along the entire cross-sectioned fiber surface
Recommendation for PFBC Use	Acceptable for short-term use	Possible use; Integrity of the fibers questioned	Integrity of the fibers questioned	Integrity of the fibers questioned	No

* Based on SEM/EDX analyses.

USF Haynes 188 Co Bal; 21-23% Cr, 20-24% Ni, 13-15% W, 3% Fe, 0.05-0.15% C, 0.2-0.5% Si, 1.25% Mn, 0.03-0.12% La, 0.015% B	Simulated PFBC Operating Conditions				
	650°C (1200°F) 242 and 500 Hrs	760°C (1400°F) 258, 524.5, and 1016 Hrs	815-840°C (1500-1550°F) 259, 476, and 986 Hrs	840°C (1550°F) — No SO ₂ — 253, 546.5, and 1014 Hrs	840°C (1550°F) — SO ₂ & Na — 225 and 496 Hrs
Physical Integrity	Intact	Intact	Intact	Intact	Expansion and separation of filtration media radiating from the circumferentially welded metal joiner rings within 496 hrs (i.e., elephant foot formation); Longitudinal cracks within filter media
Open Porosity*	Retained after 500 hrs	Retained after 258 and 524.5 hrs; Slight reduction after 1016 hrs	Retained after 259 hrs; Reduced after 476 hrs; Pore closure after 986 hrs	Retained after 253 and 546.5 hrs; Limited after 1014 hrs	Pore closure after 225 hrs
Surface Oxidation*	Multiple layers: ~1-2 µm ~1 µm external O-Cr-Co or O-Cr-Fe layer; ~1 µm chromia subsurface layer after 500 hrs	Multiple oxide layers formed; Crack formations evident with removal of material; Separation of the oxide from the base metal substrate After 258 hrs: ~2.4-8 µm oxide External: O-Fe-Co-Cr Subsurface: O-(Co-Cr)-(Fe-Ni) Adjacent to substrate: Chromia After 524.5 hrs: ~3-6 µm External: CoO-(Ni-Fe) Subsurface: O-Cr-Co Adjacent to substrate: Chromia After 1016 hrs: ~6-10 µm External: O-Co-(Ni-Cr) Subsurface: O-Cr-Co Adjacent to substrate: Chromia	Extensive oxidation; Areas evident where removal of the oxide resulted After 259 hrs: ~4-9.2 µm External: O-Cr-Co-(Fe) Subsurface: O-Cr-(Co) Adjacent to substrate: O-Cr After 476 hrs: ~10-20 µm External: O-Cr-Co-Fe Subsurface: O-Cr-Co After 986 hrs: ~12-15 µm External: O-Cr-Co-Fe Subsurface: O-Cr-(Co)	Extensive oxidation; Crack formations evident with removal of the oxide After 253 hrs: ~0.8-4 µm External: O-Cr Subsurface: Chromia After 546.5 hrs: ~4-11 µm External: O-Cr-Co-(Fe) Precipitates: O-Cr-Co-(Ni) Subsurface: O-Cr-(Co); O-Co-(Ni-Cr) O-Cr-Co Extensive oxidation after 1014 hrs: ~7-14 µm	Extensive oxidation; Crack formations evident within the oxide
Internal Oxidation*	Internal oxidation along the surface of the fibers; Sinter bond impacted	Oxidation observed between adjacent sinter bonded fibers Penetration of oxide intermittently along the periphery of the fibers to depths of ~4-10 µm increases as a function of time	Intergranular oxidation evident; Oxidation at sinter bond between adjacent fibers evident Oxidation along the periphery of the fiber base metal substrate penetrating to depths of ~10 µm after 259 hrs; ~8 µm after 476 hrs; and ~20 µm after 986 hrs. Extensive oxidation entirely throughout the cross-sectioned fiber after 986 hrs	After 253 hrs, intermittent oxidation resulted along the periphery of the fibers extending to depths of ~5-7.5 µm After 546.5 hrs, significant oxidation was evident throughout the cross-sectioned fibers; Also observed were areas with intermittent oxidation extending to depths of ~12 µm After 1014 hrs, extensive oxidation resulted throughout the cross-sectioned fibers	After 225 hrs, intermittent penetration of oxide resulted along the periphery of the fiber to depths of ~4-10 µm After 496 hrs, extensive oxidation resulted throughout the cross-sectioned fibers
Recommendation for PFBC Use	Possible use	Integrity of the fibers questioned	Integrity of the fibers questioned	No	No

* Based on SEM/EDX analyses.

USF Haynes 556 Fe Bal; 22% Cr, 20% Ni, 18% Co, 2.5% W, 0.1% C, 0.02% La, 3% Mo, 0.02% Zr, 0.6% Ta	Simulated PFBC Operating Conditions				
	650°C (1200°F) 242 and 500 Hrs	760°C (1400°F) 258, 524.5, and 1016 Hrs	815-840°C (1500-1550°F) 259, 476, and 986 Hrs	840°C (1550°F) — No SO ₂ — 253, 546.5, and 1014 Hrs	840°C (1550°F) — SO ₂ & Na — 225 and 496 Hrs
Physical Integrity	Intact	Isolated removal of the outer filtration mat occurred within 1016 hrs	Isolated removal of the outer filtration mat occurred within 259 hrs	Isolated removal of the outer filtration mat occurred within 253 hrs	Expansion and separation of filtration media radiating from the circumferentially welded metal joiner rings within 496 hrs (i.e., elephant foot formation); Longitudinal cracks within filter media
Open Porosity*	Retained after 500 hrs	Retained after 258 hrs; Limited after 524.5 hrs; Limited with pore closure after 1016 hrs	Pore closure limiting porosity after 259 hrs; Continued exposure enhanced pore closure	Pore closure resulted after 253 hrs	Pore closure resulted after 225 hrs
Surface Oxidation*	Multiple layers: ~1-10 µm External O-Fe-Cr layer; Underlying chromia-enriched layer; Through crack formations observed within the oxide scale	Multiple layers: 4-12.5 µm After 258 hrs: External: FeO-(Co-Ni) Subsurface: Chromia Adjacent to substrate: Chromia; O-Fe-(Ni-Cr) After 524.5 hrs: External: O-Fe-Ni-Cr Subsurface: Chromia Adjacent to substrate: Chromia; Sulfur sorption	Extensive oxidation resulted; Separation of oxide from underlying substrate Multiple layers: ~6-15 µm After 259 hrs: External: FeO-(Co-Ni) Subsurface: O-Fe-Cr-(Co-Ni) Adjacent to substrate: O-Cr-Co-Fe After 476 hrs: External: O-Fe-(Ni-Co) Subsurface: O-Fe-Cr-(Ni-Co); O-Cr-Co Precipitates: O-Co-(Fe) Adjacent to substrate: O-Cr-(Co) spinel After 986 hrs: Extensive oxidation	Extensive oxidation resulted: ~6-18 µm Separation of internal oxide layer adjacent to substrate from the surrounding external oxide After 253 hrs: External: O-Fe-Co-(Cr-Ni) Subsurface: O-Fe-(Co-Ni-Cr) Precipitates: O-Cr-Ni Adjacent to substrate: O-Cr-(Fe-Co-Ni) or O-Cr-Co After 546.5 hrs: External: O-Cr-(Fe) Subsurface: O-Cr-(Fe) Adjacent to substrate: O-Cr After 1014 hrs: External: O-Fe-(Co) Subsurface: O-Fe-(Ni) Precipitate: O-Cr-(Ni-Fe) Adjacent to substrate: O-Cr-(Co)	Extensive oxidation resulted after 225 hrs. Thickness of the external oxide was ~8-13 µm External: O-Fe-(Co-Ni-Cr) Subsurface: O-Cr After 496 hrs, extensive oxidation resulted. The thickness of the oxide was ~7.5-20 µm. External: O-Co-Cr-Fe Subsurface: O-Cr
Internal Oxidation*		Intermittently observed along the periphery of the fibers; Depth of penetrations: ~5-8 µm; Also intermittently observed entirely throughout the fiber; Possible grain boundary oxidation Oxidation along adjacent sinter bonded fibers Sulfur sorption within cross-sectioned fiber	With time, the extent of internal oxidation within the fiber increased After 259 hrs, intermittent oxidation was observed across the fiber; Intergranular oxidation was evident; Oxidation resulted along adjacent sinter bonded fibers After 476 hrs, significant oxidation resulted along the periphery of the base metal of the fiber After 986 hrs, significant oxidation resulted across the entire surface of the cross-sectioned fibers	After 253 hrs, extensive internal oxidation resulted along the periphery of the fiber, extending to depths of ~2.5 µm; Intermittently, oxidation was observed entirely throughout the cross-sectioned fiber After 546.5 hrs, extensive internal oxidation was observed across the entire cross-sectioned fiber After 1014 hrs, extensive oxidation was observed across the entire cross-sectioned fiber; Internal oxides detected	Oxidation observed along sinter bond formed between adjacent fibers After 225 hrs, extensive oxidation resulted across the entire fiber cross-sectioned surface; Alternately penetration to depths of ~5 µm was observed along the periphery of the fibers After 496 hrs, extensive internal oxidation was observed along the entire cross-sectioned surface of the fibers
Recommendation for PFBC Use	Possible short-term use	No	No	No	No

* Based on SEM/EDX analyses.

USF Hastelloy X 47% Ni, 22% Cr, 19% Fe, 9% Mo, 1.7% Co, 0.6% W, 0.5% Mn, 0.3% Si, 0.08% C	Simulated PFBC Operating Conditions				
	650°C (1200°F) 242 and 500 Hrs	760°C (1400°F) 258, 524.5, and 1016 Hrs	815-840°C (1500-1550°F) 259, 476, and 986 Hrs	840°C (1550°F) — No SO ₂ — 253, 546.5, and 1014 Hrs	840°C (1550°F) — SO ₂ & Na — 225 and 496 Hrs
Physical Integrity	Intact	Intact	Intact	Intact	Expansion and separation of filtration media radiating from the circumferentially welded metal joiner rings within 496 hrs (i.e., elephant foot formation); Longitudinal cracks within filter media; Embrittlement and cracking of the filtration media
Open Porosity*	Retained after 242-500 hr	Retained. Localized areas of pore closure after 258-1016 hrs	Retained. Localized areas of pore closure after 986 hrs	Pore closure resulted after 546.5-1014 hr	Extensive pore closure restricted porosity after 225-496 hrs
Surface Oxidation*	~0.6 µm after 242 hrs; ~4 µm after 500 hrs External: O-Ni-Fe-Cr Crack formations evident within this layer Ni-chromate subsurface layer	~3.75-6.6 µm oxidation layer External O-Fe-Ni-Cr spinel, and underlying chromia-enriched layer	~1.6-4.3 µm oxidation layer After 259 hrs: External: O-Cr-Ni-Fe spinel; Chromia subsurface After 476 hrs: O-Fe-Ni/Cr-rich spinel Chromia subsurface After 986 hrs: O-Ni-Fe-Cr Chromia subsurface	Along discrete fibers, the oxidation layer ranged between ~2.7-4.2 µm External: Spinels (O-Cr-Ni-Fe; O-Fe-Ni-Cr) Chromia subsurface	Along discrete fibers, the oxidation layer ranged between ~1.8-2.5 µm External: O-Ni-Cr-Fe spinel Chromia subsurface Low concentrations of sulfur detected in subsurface chromia layer
Internal Oxidation*	Negligible/limited	Internal oxidation along the periphery of the fiber, and along the sinter bond between adjacent fibers	Internal oxidation along the periphery of the fiber, and along the sinter bond between adjacent fibers	Evident along the periphery of the fiber, and along the sinter bond between adjacent fiber; Extensive after 546.5 hr	Extensive internal oxidation after 225-496 hrs
Recommendation for PFBC Use	Yes	Possible short-term use; Integrity of the fibers questioned with extended operating time	Possible short-term use; Integrity of the fibers questioned due to crack formations resulting throughout the oxide layer formed on the o.d. and i.d. filter surfaces	No Crack formations within the oxide along the i.d. surface after 546.5 hrs	No

* Based on SEM/EDX analyses.

Pall Hastelloy X 47% Ni, 22% Cr, 19% Fe, 9.0% Mo, 1.7% Co, 0.6% W, 0.5% Mn, 0.3% Si, 0.08% C	Simulated PFBC Operating Conditions				
	650°C (1200°F) 242 and 500 Hrs	760°C (1400°F) 258, 524.5, and 1016 Hrs	815-840°C (1500-1550°F) 259, 476, and 986 Hrs	840°C (1550°F) — No SO₂ — 253, 546.5, and 1014 Hrs	840°C (1550°F) — SO₂ & Na — 225 and 496 Hrs
Physical Integrity	Intact	Intact	Intact	Intact	Surface scaling
Open Porosity*	Retained after 242 hrs	Retained	Retained	Retained	Generally retained
Surface Oxidation*	~4 µm oxide intermittently formed along the outer surface of the particles After 242 hrs: External: Ni-Cr-Fe-O contained numerous cracks Subsurface: Continuous nickel chromate phase	Multiple layers After 258 hrs: o.d. surface: ~6 µm External: O-Ni-Fe Subsurface: Chromia Mid-section: Intermittent i.d. surface: ~4.3-5.7 µm External : NiO Subsurface: O-Fe-Ni Interface: Chromia After 524.5 hrs o.d. surface External: O-Ni-Fe Subsurface: Chromia i.d. surface External: O-Cr-Ni Interface: Chromia After 1016 hrs o.d surface: ~2.8-5 µm External: O-Cr-Ni Subsurface: Chromia i.d. surface: ~3.5-8.75 µm External: O-Ni-Fe-Cr Subsurface: Chromia	Multiple layers After 259 hrs: ~2.4 µm on o.d. ~4 µm on i.d. External: O-Ni-Fe Interface: Chromia After 476 hrs: ~8-10 µm on o.d. After 986 hrs: ~9 µm on o.d.; ~5-9 µm on i.d External: O-Fe-Ni Interface: Chromia	Multiple layers After 253 hrs: ~2-4 µm o.d. surface: Chromia Mid-Section: External: O-Ni-Fe Subsurface: Chromia i.d surface: Separation of oxide layers After 546.5 hrs: ~2.6-3.4 µm o.d. surface: Chromia i.d surface: Crack formations throughout the oxide; Removal of oxide After 1014 hrs o.d. surface: ~16-20 µm External : O-Fe-Ni Subsurface: Chromia i.d. surface: ~3 µm Crack formations throughout the oxide; Removal of oxide Pulse cycling reduced the adherence and integrity of the oxide formation to remain attached to the base metal substrate particle	Multiple layers After 225 hrs: o.d. surface: Continuous oxide formation External : ~2.5-8.8 µm O-Fe-Ni Subsurface: ~1.8-5 µm Chromia After 496 hrs: o.d. surface : External : ~6.5-8.5 µm O-Fe-Ni Subsurface: ~1.6-4 µm Chromia Near the center of the cross-sectioned filter wall, and particularly along the i.d., crack formations were readily evident through the oxide; Where present, the thickness of the residual chromia oxide was ~2-5 µm Removal of the external oxide from the chromia-enriched surface was expected to have resulted along the i.d. surface of the filter material after 496 hrs
Internal Oxidation*	Limited/negligible	Internal oxidation, and oxidation along the grain boundaries evident after 524.5 hrs	Internal oxidation along the periphery of the particles evident after 259 hr, and tended to increase with time; Oxidation also observed along the grain boundaries evident after 259 hrs	Internal oxidation along the periphery of the particles, as well as along the sinter bonds observed after 253 hrs; Internal oxidation became more pronounced with extended exposure time	After 225-496 hrs, internal oxidation along the periphery of the particles, and along the sinter bonds between adjacent particles was observed
Recommendation for PFBC Use	Yes	Yes	Yes; Stability of matrix questioned with extended exposure time	Possible short-term use; Integrity of the particles questioned with extended operating time	Yes; Stability of matrix questioned with extended exposure time

* Based on SEM/EDX analyses.

USF Inconel 600 Ni Bal; 15.5% Cr, 8% Fe, 0.3% Mn, 0.2% Si, 0.2% Al, 0.2% Ti	Simulated PFBC Operating Conditions				
	650°C (1200°F) 242 and 500 Hrs	760°C (1400°F) 258, 524.5, and 1016 Hrs	815-840°C (1500-1550°F) 259, 476, and 986 Hrs	840°C (1550°F) — No SO ₂ — 253, 546.5, and 1014 Hrs	840°C (1550°F) — SO ₂ & Na — 225 and 496 Hrs
Physical Integrity	Intact	Intact	Intact	Intact	Intact; Densified external surface
Open Porosity*	Retained after 242 hrs; Limited after 500 hrs of operation	Retained. Localized area of external surface pore closure after 524.5-1016 hrs	Localized areas of external surface pore closure after 259-986 hrs	Localized areas of external surface pore closure after 253-546.5 hrs; Continuous amorphous external surface oxide layer formed after 1014 hrs	Extensive pore closure after 225-496 hrs
Surface Oxidation*	Multiple layers: ~8-11 µm External: ~2 µm thick O-Ni-Fe or O-Ni layer; Sulfur identified to be present within the NiO layer, forming a filament-like structure Subsurface: ~8 µm nickel chromate layer Along the o.d. surface of the filtration media, removal of the oxide from the fibers was observed; Retention of the oxide along the i.d. surface resulted	~1.5 µm after 258 hrs; ~7.5 µm after 1016 hrs External: Chromia/Ni-O or chrome-nickel spinel after 258 hrs Fe/Ni-O formed along the outer surface of the continuous oxide layer; Subsurface chromia; Crack formations intermittently observed After 1016 hrs, NiO formed along the outer surface of the continuous oxide; Subsurface Fe-Ni-O spinel and nickel chromate phases	Multiple layers: ~7.6 µm after 259 hrs; ~4-7.2 µm after 476 hrs; ~8-10 µm after 986 hrs External: NiO Subsurface: O-Ni-Fe, O-Ni, O-Ni-Cr, O-Cr-Ni Crack formations were observed within the external oxide layer after 259 hrs	Multiple layers: ~6.25 µm after 253 hrs; ~7.5 µm after 546.5 hrs; ~8.3-10 µm after 1014 hrs External: Ni/NiO Subsurface: Nickel chromate; Chromia Crack formations within, and separation of the external oxide phase from the underlying subsurface oxides evident after 253 hrs	Extensive oxidation of the fibers resulting in pore closure and restriction of gas flow through the filter media A continuous, amorphous, Ni/NiO-enriched layer formed along the o.d. surface of the filter element Along discrete, sinter bonded fibers, cracks were observed through the ~6.7 µm thick surface oxide External: O-Ni/Fe Subsurface: Ni/NiO, O-Ni-Cr/Fe, O-Ni-Cr Adjacent to substrate: Chromia
Internal Oxidation*	Negligible	Internal oxidation initially along the periphery of the fiber; Extensive oxidation with extended time	Internal oxidation along the periphery of the fiber, as well as within the cross-sectioned fiber	Void formations through the cross-sectioned fiber, increasing with extended operating time	Within discrete fibers, void formations were evident along the cross-sectioned fiber surface
Recommendation for PFBC Use	Possible short-term use; Integrity of the fibers/element questioned with extended operating time	Possible short-term use; Integrity of the fibers/element questioned with extended operating time	Possible short-term use; Integrity of the fibers/element questioned with extended operating time	Possible short-term use; Integrity of the fibers/element questioned with extended operating time	No

* Based on SEM/EDX analyses.

Mott Inconel 600 Ni Bal: 15.5% Cr, 8% Fe, 0.3% Mn, 0.2% Si, 0.2% Al, 0.2% Ti	Simulated PFBC Operating Conditions				
	650°C (1200°F) 242 and 500 Hrs	760°C (1400°F) 258, 524.5, and 1016 Hrs	815-840°C (1500-1550°F) 259, 476, and 986 Hrs	840°C (1550°F) — No SO ₂ — 253, 546.5, and 1014 Hrs	840°C (1550°F) — SO ₂ & Na — 225 and 496 Hrs
Physical Integrity	Intact	Intact	Intact	Intact	Crack detected near circumferential weld to ring with 225 hrs
Open Porosity*	Retained after 500 hrs	Retained	Retained with the exception of localized areas of pore closure	Retained with the exception of localized areas of pore closure after 253-546.5 hrs; Extensive pore closure after 1014 hrs	Pore closure resulted along the o.d. surface; Open porosity was retained throughout the remainder of the filter wall
Surface Oxidation*	<p>After 242 hrs:</p> <p>o.d. surface: External: ~3-3.5 µm Nickel chromate-iron Subsurface: ~4.5 µm NiO, nickel chromate Thickness of the oxide was not continuous along the outer surface of the particle, but appeared to be preferential to the direction of the process gas</p> <p>Mid-section: Limited oxidation</p> <p>i.d surface : ~3.2 µm oxide</p> <p>After 500 hrs: ~1-2 µm along o.d. ~1 µm along mid-section ~2-4 µm along i.d.</p>	<p>Multiple layers External: NiO Subsurface: Nickel chromate Interface: Chromia</p> <p>Crack formations and debonding of oxide observed after 258-1016 hrs</p> <p>Oxide thickness where retained: After 258 hrs: ~3.3 µm After 524.5 hrs: ~6 µm After 1016 hrs: 4-11.7 µm</p>	<p>Multiple layers: External: NiO Subsurface: Nickel chromate, O-Fe-Ni, chromia Interface: NiO</p> <p>More extensive oxidation resulted along the o.d. and i.d. surface of the filter element in comparison to along the mid-section</p> <p>Crack formations were typically observed through the oxide; Separation of the oxide from the base metal evident after 986 hrs along o.d. surface of the filter element</p> <p>Oxide thickness: After 259 hrs: ~8.3-16.6 µm After 476 hrs: ~1.6-7.5 µm After 986 hrs: ~4.6-10 µm</p>	<p>Multiple layers</p> <p>After 253 hrs: External: Ni/Cr-O Subsurface: NiO, Nickel chromate Interface: Chromia</p> <p>After 546.5 hrs: External: Nickel chromate Subsurface: O-Ni-Fe, O-Ni-Cr Interface: Chromia</p> <p>After 1014 hrs: External: NiO Subsurface: O-Ni(Fe), O-Cr-Ni Interface: Chromia</p> <p>Oxide thickness: o.d. surface: ~7.2-17.2 µm i.d surface : ~8.8 µm</p>	<p>Multiple layers</p> <p>After 225 hrs: o.d. surface: ~2.2 µm Cr/Ni-O i.d. surface: ~0.1-0.26 µm</p> <p>After 496 hrs: o.d. surface: ~3 µm Cr-Fe-Ni-O Mid-section: Dendritic chromia-enriched whiskers extended from the surface of the particles; Subsurface: ~6 µm Cr/Ni-O i.d. surface: ~5 µm External: Nickel chromate Subsurface: Chromia</p> <p>Crack formations through the oxide, separation, and removal of the oxide evident after 225 hrs</p>
Internal Oxidation*	Limited penetration into particle after 500 hrs of operation	Internal oxidation within and along the periphery of the particles, as well as along the sinter bonds observed after 258 hr. Tendency to increase with time, particularly on i.d. surface of filter media	Extensive internal oxidation along the periphery of the base metal resulted after 259 hr along the o.d. and i.d. surfaces of the filter element	An internal oxidation zone was typically present in localized areas beneath the external oxide layer Penetration: o.d. surface: ~4.8-7.2 µm i.d. surface: ~12-14 µm Internal oxidation within the particles and along the sinter bonds more frequently observed on i.d. surface in comparison to o.d. or mid-section of the filter element	Along the o.d. surface of the filter element, internal oxidation penetrated to depths of ~2.0 µm after 225 hrs, and ~10 µm after 496 hrs Internal oxidation was limited along the i.d. surface of the filter element
Recommendation for PFBC Use	Yes	Yes	Possible short-term use; Integrity of the particles/element questioned with extended operating time	Possible short-term use; Integrity of the particles/element questioned with extended operating time	Possible short-term use; Integrity of the particles/element questioned with extended operating time

* Based on SEM/EDX analyses.

USF 310S Fe Bal; 25% Cr, 20.5% Ni, 1.6% Mn, 0.75% Mo, 0.5% Si, 0.05% C	Simulated PFBC Operating Conditions				
	650°C (1200°F) 242 and 500 Hrs	760°C (1400°F) 258, 524.5, and 1016 Hrs	815-840°C (1500-1550°F) 259, 476, and 986 Hrs	840°C (1550°F) — No SO ₂ — 253, 546.5, and 1014 Hrs	840°C (1550°F) — SO ₂ & Na — 225 and 496 Hrs
Physical Integrity	Intact	Intact	Intact	Intact	Intact; Densified external surface
Open Porosity*	Limited open porosity due to coalescence of fibers after 500 hrs	Localized pore closure; Loss of open porosity increased with time particularly along the o.d. surface of the filter element	Localized pore closure; Loss of open porosity increased with time particularly along the o.d. surface of the filter element	Pore closure restricting gas flow through the filter element after 253-1014 hrs	Pore closure restricting gas flow through the filter element after 225-496 hrs
Surface Oxidation*	<p>After 242 hrs: External: ~1 µm Iron oxide layer Subsurface: ~1 µm O-Cr-Fe-Ni</p> <p>After 500 hrs: Thicknesses of both external and subsurface oxides increased to ~3-5 µm; Typically cracks extended through the oxide formations</p>	<p>Extensive oxidation after 258-1016 hrs External: FeO, Fe/Ni-Cr-O Subsurface: Chromia</p> <p>Oxide thickness: After 258 hrs: ~3 µm After 524.5 hrs: ~6.6-13.3 µm After 1016 hrs: ~3.5 µm</p> <p>Separation between the oxide layers, as well as at the base metal interface</p> <p>Crack formations observed through external surface oxide layer along i.d. surface of the filter element</p>	<p>Extensive oxidation External: Fe-Ni-O spinel Subsurface: Chromia</p> <p>Separation between the oxide layers, as well as at the base metal interface</p>	<p>Extensive oxidation</p> <p>After 253 hrs External: Fe-Ni/Cr-O spinel Subsurface: Chromia</p> <p>After 1014 hrs External: Fe-Cr-O Subsurface: Fe-Cr-Ni-O, Cr-Fe-O, Ni-Fe-Cr-O</p> <p>Crack formations within the oxide(s) observed</p>	<p>Extensive oxidation Multiple layers External: Fe-Ni-O spinel Subsurface: FeO, Fe-Ni/Cr-O</p> <p>Primarily along the i.d. surface of the filter element, cracks were observed within the oxide formations, as well as between adjacent sinter bonded fibers</p>
Internal Oxidation*	Negligible	Extensive internal oxidation along the periphery, as well as within the fibers, and at the sinter bonds	Extensive internal oxidation	Extensive internal oxidation	Extensive internal oxidation
Recommendation for PFBC Use	Acceptable for short-term use; Integrity of the particle questioned with extended exposure time	No	No	No	No

* Based on SEM/EDX analyses.

Pall 310S Fe Bal; 25% Cr, 20.5% Ni, 1.6% Mn, 0.75% Mo, 0.5% Si, 0.05% C	Simulated PFBC Operating Conditions				
	650°C (1200°F) 242 and 500 Hrs	760°C (1400°F) 258, 524.5, and 1016 Hrs	815-840°C (1500-1550°F) 259, 476, and 986 Hrs	840°C (1550°F) — No SO₂ — 253, 546.5, and 1014 Hrs	840°C (1550°F) — SO₂ & Na — 225 and 496 Hrs
Physical Integrity	Intact	Circumferential failure at metal ring weld within 258 hrs	Circumferential failure at metal ring weld within 259 hrs	Circumferential failure at metal ring weld within 253 hrs	Circumferential failure at metal ring weld within 225 hrs
Open Porosity*	Retained after 242-500 hrs	Retained Expected decrease	Not characterized	Not characterized	Extensive pore closure along both outer and inner external surfaces of the filter element; Open porosity retained along ~1 mm thick mid-section of the filter element
Surface Oxidation*	After 242 hrs: ~1 µm Cr-Fe-O After 500 hrs: ~4-7 µm Chromia; Iron chromate; Crack formations were frequently observed, with spallation, exposing base metal	Multiple oxide layers After 258 hrs, ~6 µm oxide thick oxide formed External: Fe-O, Fe-Cr-O Subsurface: O-Ni-Cr or O-Cr-Ni-Fe Interface: Chromia Frequently the external oxide was separated from subsurface spinel layer	Not characterized	Not characterized	Multiple oxide layers o.d. surface: ~6.6-8.3 µm oxide External : Fe-O Subsurface: O-Ni-Fe Mid-section: Chromia-enriched whiskers Interface: Chromia i.d. surface: External: Fe-O Subsurface: Chromia
Internal Oxidation*	Limited internal oxidation; Limited oxidation along sinter bonds	Along the periphery of the particles; Within the cross-sectioned particles	Not characterized	Not characterized	Extensive internal oxidation observed along both outer and inner surface-contained particles Along the mid-section of the filter wall, void formations along the periphery of the particles, as well as within the particles, and along sinter bonds between particles were observed
Recommendation for PFBC Use	Acceptable for short-term use; Integrity of the particle questioned with extended exposure time	Limited short-term use	No	No	No

* Based on SEM/EDX analyses.