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for

CRADA Number 0602

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January 14, 2008
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Final Report – CRADA 0602 with Pall Corporation

DEVELOPMENT OF IMPROVED IRON-ALUMINIDE FILTER TUBES AND ELEMENTS

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PURPOSE

The purpose of this Cooperative Research and Development Agreement (CRADA) was to explore and develop advanced manufacturing techniques to fabricate sintered iron-aluminide intermetallic porous bodies used for gas filtration so as to reduce production costs while maintaining or improving performance in advanced coal gasification and combustion systems..

INTRODUCTION

The use of a power turbine fired with coal-derived synthesis gas will require some form of gas cleaning in order to protect turbine and downstream components from degradation by erosion, corrosion, and/or deposition. Hot-gas filtration is one form of cleaning that offers the ability to remove particles from the gases produced by gasification processes without having to substantially cool and, possibly, reheat them before their introduction into the turbine. This technology depends critically on materials durability and reliability, which have been the subject of study for a number of years (see, for example, refs. 1-6).

BACKGROUND

Potential of iron-aluminide filters for hot-gas cleanup

Iron aluminides have shown good to excellent high-temperature corrosion resistance in sulfur-bearing environments relevant to coal-derived energy production systems[7-11] and thus have potential for use as the material of construction for metallic filters used to clean fossil-fuel-derived gases prior to their introduction into gas turbine systems. Consequently, background information for consideration of iron-aluminide alloys for filter applications is given in terms of a brief summary of the physical metallurgy and relevant high-temperature corrosion behavior of iron aluminides.

Iron aluminides have been of interest since the 1930s when the excellent corrosion resistance of compositions with more than about 18 at. % Al was first noted.[11] During the 1980s and 1990s, extensive R&D was conducted on alloys based on the Fe_3Al and FeAl compositions. In addition to their superior corrosion resistance, these alloys also offer relatively low material cost, a lower density than stainless steels, and tensile strengths that compare favorably with many ferritic and austenitic steels. The limited ductility at ambient temperatures and reduced strength at temperatures above 600-650°C hindered their introduction into the commercial marketplace. The work of the 1980's and 90's has resulted in substantial progress in understanding the many variables that affect properties in the Fe-Al system. Notable among the examples of this increased understanding is that the environment, rather than any inherent property, is the cause of embrittlement at ambient temperatures.[12] Atomic hydrogen, which forms through a reaction between moisture in the air and aluminum on the surface of the specimen, penetrates the alloy resulting in environment-induced hydrogen embrittlement. Alloy development efforts[13,14] have demonstrated that improved ductility of iron aluminides can be achieved in wrought Fe_3Al- and FeAl-based alloys through control of composition and microstructure. Chromium additions of from two-to-five percent result in considerable improvement in ductility. Controlled additions of Nb, Mo, Zr, and other elements have improved the hot strength through both precipitation and solid-solution mechanisms. All of these advancements have resulted in Fe_3Al- and FeAl-based alloys with ambient temperature tensile ductilities of 10-20% and tensile yield strengths of as high as 500 MPa on both a research scale and a commercial scale. These properties are more than adequate for hot-gas filter service.

In many service environments, particularly those found in integrated coal gasification combined cycle systems, sulfur coexists with oxygen, carbon, hydrogen, and various contaminant gases such as chlorine compounds. In the gasification environment, the sulfur partial pressure is high and oxygen partial pressure is typically low, but still sufficient to form Al_2O_3 on Fe_3Al- and FeAl-based alloys.[7] Thus, the high-temperature corrosion resistance of iron aluminides derives from the thermodynamic stability as well as the relatively slow growth of the alumina surface product that is formed across a wide range of oxygen partial pressures. Accordingly, the corrosion behavior of these alloys is controlled by the integrity and adherence of the protective alumina surface product.[7]

Because of their corrosion resistance in elevated-temperature, sulfur-bearing environments, Fe_3Al-based alloys appeared to offer distinct temperature and reliability advantages (relative to ceramics) as materials for hot-gas filters used in advanced coal plants utilizing gas turbines. These include those based on integrated gasification combined cycle (IGCC) and pressurized fluidized bed combustors (PFBC). Numerous metallurgical and mechanical evaluations of porous Fe_3Al-based alloys exposed under environmental conditions appropriate to IGCCs and PFBCs were performed and have been reported elsewhere.[5,6,16,17] These have confirmed the efficacy of iron aluminide as a filter material in the IGCC and PFBC environments.

Pall Corporation development of commercial iron-aluminide filters

Pall Corporation has reported on the successful development of commercial manufacturing processes for iron-aluminide filters.[18,19] The Pall development effort included production of seamless cylinders from water-atomized iron-aluminide powder, compaction of the cylinders and optimization of the sintering cycle. The step-wise process developed and described by Pall involves a first step of centrifugally casting a thickened suspension of iron aluminide in water to form a uniform deposit on the interior of a ceramic tube. The balance of the liquid is decanted and the ceramic tube-iron - aluminide deposit preform assembly is dried. The second step in the Pall process involves the isostatic compression of the preform assembly via reusable rubber bladders that are added to the inside and outside of the preform assembly. The resultant dried and compressed tubes are then sintered and cut to length. Connectors and end fittings are welded to the porous metal filter elements to yield a filter unit of any desired length.

Figure 1 is a photograph of the Pall Gas/Solid Separations System (GSS) in its triad element design configuration. These filters exhibit excellent performance efficiencies. Typically, more than 99.99% of the particulate matter entrained in the hot gases being filtered is removed on the surface of the filter. Sintered metal powder media are inherently strong and durable for long-term service. The media are formed into elements capable of withstanding the rigors of repeated cycles in the reverse flow direction (blowback). The S Grade PSS elements are up to 100 inches/2.54 m in overall length, consisting of four 2.38 in/60 cm diameter sections, each 19 inches/0.48 m long. The sections are joined by welding solid joiner rings to the sintered metal tubes for maximum physical strength. Each element provides 4 ft^2/0.37 m^2 of filter area. All solid hardware is type 304 stainless steel. The element is closed at one end by a welded end cap. A suitable adapter is welded to the open end for fitting to a tube sheet. Standard PSS medium thickness is 0.09 in/0.24 cm for the optimal combination of strength with low pressure drop characteristics. These standard elements can withstand pressure drops of 100 psid/6.9 bar in both the forward and reverse flow directions.

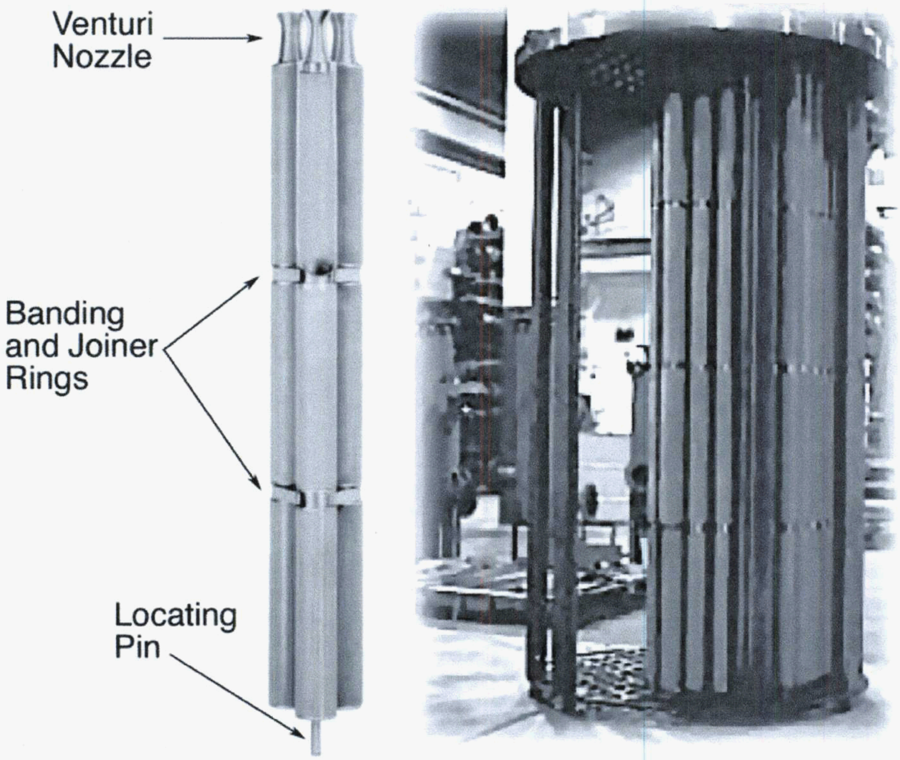

Figure 1. Pall PSS triad element design and filters in a tubesheet assembly.

The Pall PSS elements are then incorporated into a filtration system such as that also shown in Fig. 1. Filter elements have been evaluated in facilities such as the Southern Company Power Systems Development Facility (PSDF) in Wilsonville, Alabama, and other coal gasification and petroleum refining units.[5,6,17] Notable issues that have been identified and addressed include the necessity for preoxidation of the filter elements to form an alumina surface as it is the alumina surface that provides the oxidation and sulfidation resistance for the alloy, as described above. Also, the filters must not be operated under conditions that result in moisture condensing on the filter surfaces (that is, below the dew point). The alloy, Fe_3Al containing about 2% chromium, used for the Pall iron-aluminide filters will corrode under these conditions. Usual atmospheric control measures employed in coal gasification systems are sufficient to prevent this type of degradation. Pall has moved forward with its commercialization efforts and the Pall PSS iron-aluminide filters and filter systems are being used in several plants around the world.[19] Presently, the PSDF has a full complement, 91 filters, in one of its filter vessels. Furthermore, Pall is presently producing a full complement of over 1500 of these filters for an IGCC plant.

RESULTS FOR IRON ALUMINIDES EXPOSED UNDER GASIFICATION CONDITIONS

Metallurgical and mechanical evaluations of porous Fe_3Al-based alloys exposed in test beds that simulate environments associated with IGCCs and PFBCs have been conducted.[5,6,17] Results for as-fabricated porous iron-aluminide filter materials showed good high-temperature corrosion resistance in air, air + SO_2, and H_2S-containing environments. The corrosion resistance was further improved by a preoxidation treatment. The hoop strength of the filters was not significantly affected by the preoxidation treatment or by 100-h exposures in air or air plus SO_2 at 800 and 900°C.

Iron-aluminide filters have also been exposed in an actual gasification plant.[6] Iron-aluminide filter-element or o-ring specimens were characterized after exposure at the Wabash River Plant for times of approximately 400 to 6200 h. Several factors appeared to be important to the length of service of these filters. These included the preoxidation conditions during fabrication of the filter, time and temperature of exposure, and composition of the iron aluminide. The general mode of corrosion failure involved the formation of iron sulfide that grew into and occluded the pores, resulting in blockage of the filter and reduction in mechanical strength. This process appeared to be accelerated at longer exposure times, possibly due to the depletion of aluminum from the filter alloy matrix and the resulting breakdown of the protective alumina layer and its inability to reform. However, it was found that, with appropriate preoxidation treatments (those that produce a thin protective surface alumina), iron-aluminide filters can have extended lifetimes in coal-derived synthesis-gas environments.

PALL ACCUSEP™ FILTERS

Pall Corporation has, with the Oak Ridge National Laboratory's Inorganic Membrane Technology Laboratory, developed a line of elements called Accusep™ filters (see Fig. 2).[19] These are sintered metal seamless tubular filters produced by a proprietary process derived from ORNL's inorganic membrane fabrication methods. The Accusep™ filters are produced commercially from several alloys including 316L stainless steel, 304L stainless steel, 310SC stainless steel, Hastelloy X, and Inconel 600. Standard sizes of the Accusep™ filters include nominal outer diameters of 0.5 in/1.27 cm or 0.75 in/1.90 cm and lengths up to 8 ft/2.4 m. The filter medium is quite thin, i.e., 0.018 in/0.046 cm and 0.025 in/0.063 cm depending on tube diameter, yet quite strong and very uniform. The processing methods yield a structure that is up to three times more permeable than conventional sintered metal tubes.

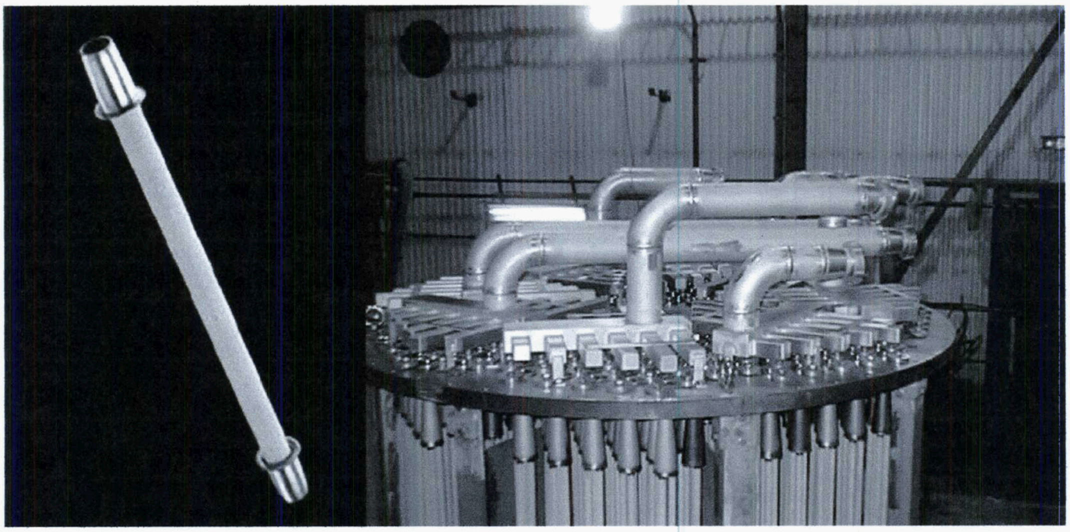

Figure 2. Pall Accusep™ filter element (left) and filter system showing blowback manifold.

Several advantages accrue from these filter elements compared to conventional filters.[19] Among these are:

- seamless construction in small diameters and extended nonsegmented lengths
- greater effective surface area and high packing density, resulting in lower filtration costs and smaller filter systems
- a proprietary inorganic medium that gives higher permeability and highly uniform pore size (longer filter life, smaller filter systems, and consistent operating flux in a cleanable filter)

For hot-gas filtration in an IGCC plant, a multi-tube assembly with single open ended tubular elements connected to a blowback venturi is used.

Given the special benefits of the Pall Accusep™ system and the particular advantages of iron aluminides as hot-gas filtration media for use in sulfur-bearing environments (see above), successful production of iron-aluminide filters in a similar form would be a very attractive advance in state-of-the-art hot-gas filtration for integrated gasification combined cycle systems. Consequently, ORNL and Pall Corporation have collaborated to examine the facility of fabricating iron-aluminide filters by the manufacturing process used for the current Accusep™ products. The balance of this paper describes progress to date in this regard.

ADVANCED MANUFACTURING TECHNIQUES FOR IRON-ALUMINIDE POROUS BODIES

In this CRADA project, advanced manufacturing techniques for sintered iron-aluminide metal porous bodies of the Accusep™ configuration were studied so as to reduce production costs while, hopefully, maintaining or improving the performance of particle filters for coal gasification and combustion systems.* This was done by evaluating the effects of selected processing variables on the microstructural, mechanical, and filtration characteristics of the porous products produced from iron-aluminide powders by ORNL's inorganic membrane fabrication techniques. Specific steps in the process were:

- characterization of the starting iron-aluminide powder (size distribution, particle shape)
- formation of compacts (disks) or tubular bodies
- sintering (various temperatures, times, atmospheres)
- characterization of as-sintered compacts or tubes
  - o permeability
  - o microstructure
  - o mechanical integrity and/or strength

Compacts were initially used in this project to conserve powder and to scope, relatively quickly, the time, temperature, and other processing conditions as they affected pore size, permeance, and mechanical integrity. A study of 79 sintered compacts made from Ametek water-atomized powder (see Table 1) was conducted. Each was characterized for void fraction and permeability. The range of processing conditions that yielded higher density compacts with adequate strength (based on a qualitative evaluation) and permeability was used as the starting point for fabrication of tubular elements of the Accusep type.

From a manufacturing perspective, it was important to find powder and processing conditions for appropriately sized tubes that minimized both the sintering temperature and time while providing acceptable properties (as determined here by pore size and mechanical behavior) so as to assure cost-

*Some effort was also directed toward addressing issues associated with the successful DOE approval for commercial release.

effective production of Accusep™ filters. Thus, the sintering trials and subsequent characterization of the tubes formed the essence of this investigation. Sintering temperatures were varied from 1115 to 1310°C over 30-60 min in argon or hydrogen atmospheres.

Table 2 lists the various types of starting powders used for the experimental runs and, for each, the approximate number of sintered tube elements produced. Figure 3 shows a representative scanning electron image of each powder. As shown in Table 2, gas-atomized powders with several different particle size distributions were examined in this study. Because the amount of appropriately sized metal powder from the atomization process has significant implications with respect to cost-effective manufacturing, the highest possible yields of powder in an acceptable particle size range are desired. Accordingly, in support of this investigation, the Metals Preparation Center at Ames Laboratory produced helium-gas-atomized iron-aluminide powder using a high-shear gas-atomization process developed as an industrial prototype.[20] This approach has the potential for substantially higher yields in the appropriate particle size range (see below). This powder was sieved and classified to obtain different size fractions.

Table 2. Iron-aluminide powder data

Type	Manufacturer	Mean Particle Size, μm	Composition, wt% (balance Fe)	Number of Filter Tubes Produced
Water Atomized	Ametek	82	16.4Al – 2.2Cr – 0.2Zr – 0.5O – 0.02C	~200
Ar Atomized	Ametek	15,35	16.8Al – 2.2Cr – 0.04O – 0. 02C	88
Ar Atomized	Deloro Stellite	15	16.8Al – 2.2Cr – 0.2Zr – 0.01B (O,C not analyzed)	53
He Atomized	Ames Lab	12, 14	16Al – 2Cr (nominal)	29

As noted above, PSS-type filters are fabricated from water-atomized iron aluminide powder. Development work at Pall showed that the irregular nature of these particles (when compared to the gas-atomized case, see Fig. 3) yielded sound sintered iron-aluminide filters. Microstructural characterization and mechanical testing of such showed fully developed sinter bonds that exhibited ductile fracture.[17] Therefore, water-atomized iron-aluminide powder was used in the initial forming and sintering trials based on the inorganic membrane fabrication techniques. However, the resulting formed bodies and sintered products showed great variability in soundness and properties such that it wasn't possible to consistently fabricate acceptable filter tubes. On the other hand, it was found that sound tubes of Accusep™ filter dimensions were successfully made using gas-atomized iron aluminide powder. The reason(s) for this difference with respect to the nature of the starting powder between the standard Pall production technique and that used in the present study is (are) not known. Based on oxygen analyses, it appeared that, as expected, the starting oxide coating on the inert-gas-atomized powder was thinner. This perhaps allowed its breakup during the forming process and improved sintering behavior. Also, the substantially larger particle size of the water-atomized powder (see Table 1) hinders full sintering (see below). Regardless of the underlying reasons, it was clear that the inorganic membrane fabrication approach worked best with the spherical, inert-gas-atomized particles

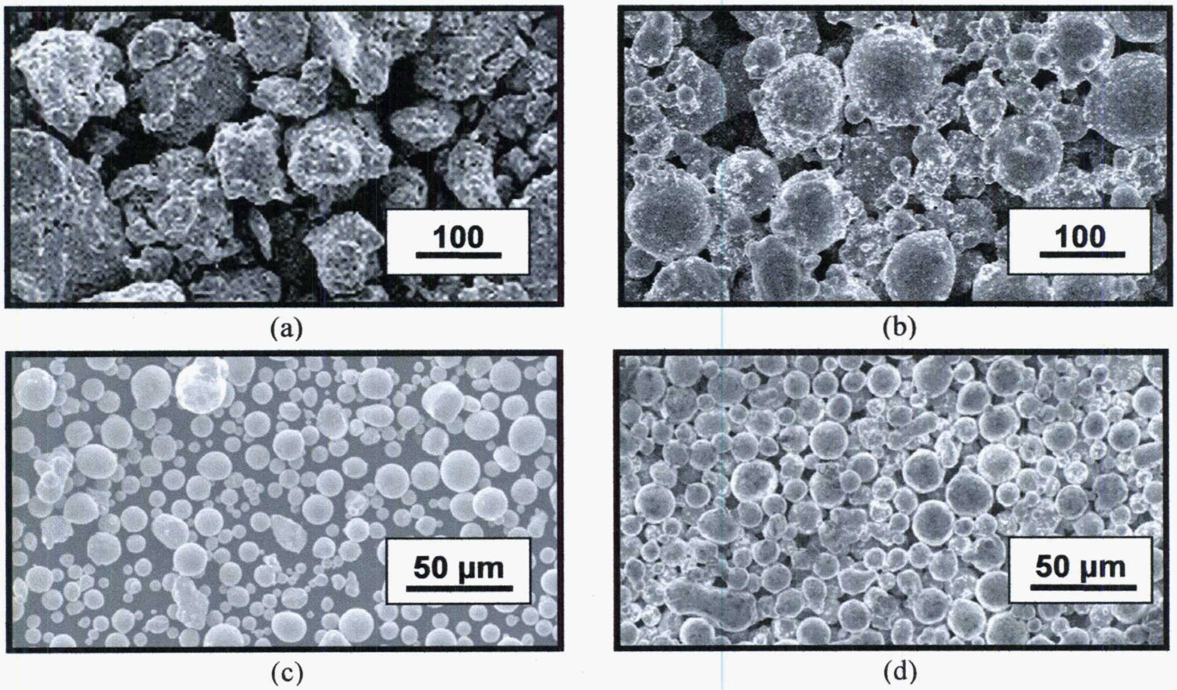

Figure 3. Iron-aluminide powder. (a) water atomized, Ametek. (b) Ar atomized, Ametek. (c) Ar atomized, Deloro Stellite. (d) He atomized, Ames Lab

and subsequent efforts focused on the use of this type of powder and the minimization of sintering times and temperatures.

Typically, sintering times and temperatures to achieve a desired pore size and distribution decrease with decreasing particle size. Consistent with this, iron-aluminide powder with a higher mean particle size required higher sintering temperatures to produce acceptable filter tubes: bodies formed from the powder with a ~35-µm mean particle size required sintering temperatures of 1200°C or above. The use of gas atomized powder with average particle size ≤20 µm was required to produce sound filter tubes with sintering temperatures at or below 1150°C and times amenable to cost effective manufacturing. Some characterization data for filter tubes produced from the finer Ames Lab powder and sintered at 1150°C are shown in Table 3. Despite the overlap in standard deviations, the differences in average pore sizes appeared to be significant as the average pressure drop tracked linearly (inversely) with this variable. The average tensile yield stress of the tubes was considered more than sufficient for the filter application; a nominal yield of ~42 MPa (6000 psi) is typically considered adequate. However, ductility of iron-aluminide elements may be problematical as elongations (ε) in the range $0.3\% \leq \varepsilon < 1\%$ were measured.

Based on data in Table 3 and other results, it was found that the powder that was classified to yield particle sizes in the approximate range of 5-15 µm did not lead to further decrease in the sintering temperature; powder with <20 µm particle sizes (with and without fines) yielded good quality tubes as defined in terms of pore size and yield strength. From a manufacturing viewpoint, this result indicates that the expense of powder classification below 20 µm will not be required. As the yield of <20 µm powder from the Ames Lab process was approximately 30%, this percentage of the initial charge for atomization would need no further classification before processing. (The balance of the powder may, of course, be reintroduced into the process for the manufacture of additional powder.)

Table 3. Characterization data for filter tubes fabricated from <20 μm powder from Ames Lab

Mean Particle Size, μm	Average Pore Size of Filter Tube, μm	Average Pressure Drop, kPa (psi)	Average Tensile Yield Stress MPa (psi)
11.7	4.0 ± 0.5	2.9 (0.42) ± 0.3 (0.05)	
13.1	4.8 ± 0.4	2.1 (0.31) ± 0.4 (0.05)	66.3 (9618) ± 17.3 (2506)
11.7, 13.1	5.5 ± 0.6	1.6 (0.23) ± 0.2 (0.03)	

SUMMARY

Over the past twenty years, alloy research and development efforts have shown that iron aluminides offer excellent corrosion protection at elevated temperatures in the presence of H_2S and have adequate strength and ductility for use as hot-gas filters for coal gasification applications. Pall Corporation had consequently developed a commercially available filter system based on iron aluminide and the corrosion behavior in simulated and actual gasification streams has been characterized. Advanced manufacturing techniques to fabricate filter tubes of an alternative design that offers distinct gas handling advantages were investigated. Unlike the process for the current commercial product, which uses water-atomized powder, the present manufacturing approach requires inert-gas-atomized particles of iron aluminide to achieve the best results. Furthermore, it was found that a gas-atomized powder with average particle size <20 μm could be used to produce sound filter tubes with sintering temperatures (at or below 1150°C) and times amenable to cost effective manufacturing.

ACKNOWLEDGEMENTS

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