Iron Aluminide Hot Gas Filters

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Schedule and Milestones

FY 96 Program Schedule

	M	J	J	A	S	O	N	D	J	F	M	A	M	J	J	A	S
NEPA Info	-----	-----	-----	-----													
Test Plan	-----	-----	-----	-----	-----												
Devel., Quali. Testing					-----	-----	-----	-----	-----	-----	-----						

Introduction

Currently, high temperature filter systems are in the demonstration phase with the first commercial scale hot filter systems being installed on integrated gasification combined cycle (IGCC) and pressurized fluid bed combustion cycle (PBFC) systems (70 MW). They are dependent on the development of durable and economic high temperature filter systems. These filters are mostly ceramic tubes or candles. Ceramic filter durability has not been high. Failure is usually attributed to mechanical or thermal shock: they can also undergo significant changes due to service conditions.

Research sponsored by the U.S. Department of Energy's Morgantown Energy Technology Center, Theodore J. McMahon METC Project Manager, under Contract DE-AC21-95MC31215 with Pall Aeropower, Kenneth B. Zella Program Manager, 6301 49th Street North, Pinellas Park Florida, 34665-5798; fax (813) 528-0516.

Objectives

The overall objective of this project is to commercialize weldable, crack resistant filters which will provide several years service in advanced power processes.

The specific objectives of this project are to develop corrosion resistant alloys and manufacturing processes to make Iron Aluminide filter media, and to use a "short term" exposure apparatus supported by other tests to identify the most promising candidate (alloy plus sintering cycle). The objectives of the next phases are to demonstrate long term corrosion stability for the best candidate followed by the production of fifty filters (optional).

Approach

Seamless cylinders have over the last several years become widely accepted for the manufacture of stainless steel filters. It follows, therefore, that much of the technology needed to produce seamless cylinders in a different alloy such as Iron Aluminide has already been worked out. For example, experimental Iron Aluminide seamless cylinders are made on the same equipment used for the production of seamless cylinders in stainless steel. Some important changes are obviously needed and they are addressed in this project. Basically three steps must be added or revised. To produce a high strength cylinder in Iron Aluminide it is necessary to first, tailor some processing details during the production of the cylinders, second, add a compaction step for the cylinders and, third, develop an optimized sintering cycle. The manufacture of seamless cylinders is a proprietary process.

There is a wealth of information on solid Iron Aluminide determined and/or compiled by Oak Ridge National Laboratory (ORNL). This provides a valuable resource (1).

Project Description

The project is being carried out in five tasks. Task 1 provided the management plan and the necessary NEPA information. Task 2 provided the test plan. The first two tasks have been completed. Task 3 provides the experimental work to implement the test plan: it is now underway. Figure 1 shows the sequence of steps involved in making a seamless filter. It starts with securing a supply of powder and proceeds to welding the components together to make a filter. Task 3 includes any subsequent steps such as preoxidation that would be carried out before placing the filter in service. The upgrading and operation of a test apparatus for "short term" exposure testing of Iron Aluminide candidates is also covered. Experimental work is authorized to the completion of Task 3. At the end of Task 3 the best composition and processing sequence will be chosen for further development.

In Task 4 the most promising candidate will be subjected to further long term exposure. This will be followed by the manufacture of 50 filters (optional).

FIGURE 1
PRODUCTION OF IRON ALUMINIDE SEAMLESS FILTERS

- **POWDER QUALIFICATION**
- **SPINNING OF SEAMLESS CYLINDERS**
- **COMPRESSION OF SEAMLESS CYLINDERS**
- **VACUUM SINTERING**
- **WELDING END FITTINGS**

Candidates

There are four candidates. The first three candidates have different chemical compositions and will be preoxidized (2) before being placed in service:

- Fe_3Al with 2%Cr (Similar to FAS but with 0.19% by weight Zr and without B)
- Fe_3AL with 5%Cr (Similar to FAL with 0.19% by weight Zr but without B)
- FeAl (with 0.19% by weight of Zr plus 0.004 % B).

The fourth candidate is the first composition but without preoxidation.

The preoxidation treatment (800C in air for seven hours) was developed by ORNL (2). The preoxidation treatment favors the formation of a surface enriched in alumina: this is particularly beneficial for resistance to gases containing hydrogen sulfide.

Seamless Cylinders

The preferred product form for Iron Aluminide porous media is as seamless cylinders. The benefits are identified in Figure 2. The uniformity of the pore structure combined with good void volume account for the first four points. The use of seamless cylinders allows the use of advanced alloys such as Iron Aluminide that are tough enough for the intended service but whose ductility may not be sufficient to allow them to be fabricated from flat sheet. The use of a seamless cylinder eliminates the need for a longitudinal seam weld and the resultant heat affected zones (HAZ) on either side of the weld bead. The tensile hoop stresses (during blowback) are twice as high as the stresses at the junction of the endcap with the filter medium. The process produces an intrinsically uniform and inexpensive medium since the key steps are preprogrammed and are under computer control. This removes them from operator variability. Finally, the use of seamless cylinders is economical in that there is almost no powder wastage, the ceramic tubes and the bladders are reusable, etc.

Satisfactory results have been achieved using either water atomized or gas atomized powder. There is a mild preference to use water atomized powder because it promotes high green strength due to its rough surface, as can be seen in Figure 3.

The first step in making a seamless cylinder is to take a ceramic tube and temporarily seal it at one end. Measured quantities of a thickened water based solution and Iron Aluminide powder are mixed together and are poured into the ceramic tube which is again temporarily sealed at the other end. The ceramic tube is then spun in a lathe where the suspended powder deposits uniformly on the interior of the ceramic tube. The remaining liquid is decanted and the assembly is dried. The residual thickener at this point functions as a temporary bonding agent for the powder.

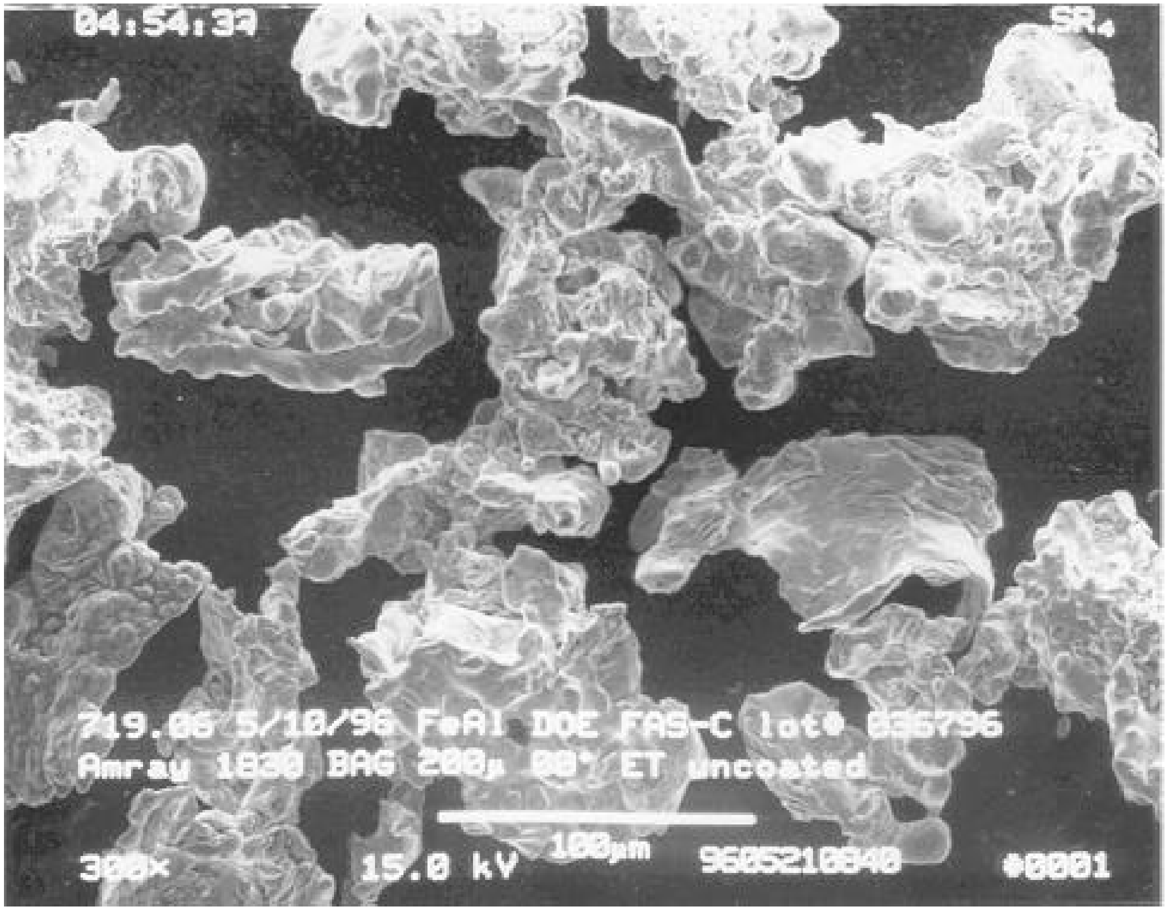
Compression of Preform Seamless Cylinders

With Iron Aluminide the preform assembly is isostatically compressed. Reusable rubber bladders are added to the inside and the outside of the preform assembly and they are sealed to prevent the entry of water during the compression step.

FIGURE 2
BENEFITS OF SEAMLESS TUBE FILTERS

- **HIGH VOID VOLUME AND UNIFORM PORE STRUCTURE**
- **UNIFORM WALL THICKNESS AND TUBE DIAMETER**
- **BLOWBACK IS UNIFORM DUE TO CONSISTENT PORES**
- **PRESSURE DROP IS LOWER THAN CONVENTIONAL CONSTRUCTION**
- **READILY ADAPTS TO HIGH PERFORMANCE ALLOYS SUCH AS Fe_3Al AND FeAl**
- **FREE FROM WEAKNESS OF LONGITUDINAL WELD HAZ**
- **CONSISTENT PROGRAMMED MANUFACTURE**
- **ECONOMIC - LITTLE SCRAP, MOLDS REUSABLE, etc.**

FIGURE 3
IRON ALUMINIDE POWDER AS WATER ATOMIZED - SEM AT 300X



Vacuum Sintering

The tubes are usually sintered in the vertical position. The Iron Aluminide preform is supported by the ceramic tube as the assemblies are heated to the sintering temperature - that is until a point is reached where the Iron Aluminide shrinks markedly as it sinters. There is a straightforward explanation. The Iron Aluminide is held in compression as it is heated since it has a higher coefficient of thermal expansion than the surrounding ceramic. Once the sintering operation is complete the Iron Aluminide seamless cylinders are removed from their protective ceramic tubes. The seamless cylinders are then cut to length.

Welding End Fittings

The end fittings can be 310S (a readily available high temperature stainless steel) or solid Iron Aluminide. Generally speaking, the welding of porous Iron Aluminide to solids is preferably done using a single pass Gas Tungsten Arc (or TIG) weld. The filler can be either 310S or Iron Aluminide. Both fillers have been used successfully but it is distinctly easier to use the 310S. Figure 4 shows that the test specimens are small welded filters.

"Short Term" Exposure Apparatus

As part of this project a "short term" exposure test apparatus has been markedly upgraded. Mixing of the gases that compose the test atmosphere is now done by mass flowmeters. An eight channel programmable controller directs the mass flowmeters.

The test apparatus is composed of two connected tube furnaces. In the first furnace the premixed gases and the injected water are preheated. The preheated atmosphere flows into the second furnace where it contacts the test filters. Figure 5 shows a schematic of this second furnace. The containment tubes for both furnaces are aluminized 310 stainless steel.

Three upgrades have been added in addition to the mass flowmeters. Each was chosen because it allows an important service condition to be studied in greater detail:

* First , the test atmosphere now flows through the wall of the test filter as in actual service,
* Second, an instrumented and programmable blowback capability has been added,
* Third, potential corrosion accelerators such as (HCl + NaCl + KCl) can now be added to the furnace atmosphere.

The upgrades to the test apparatus have been completed and the apparatus is now fully qualified. It is ready for the "short term" testing to begin. At the conclusion of this testing program the results will be analyzed and the best overall composition and processing sequence will be chosen.

Test Results to Date

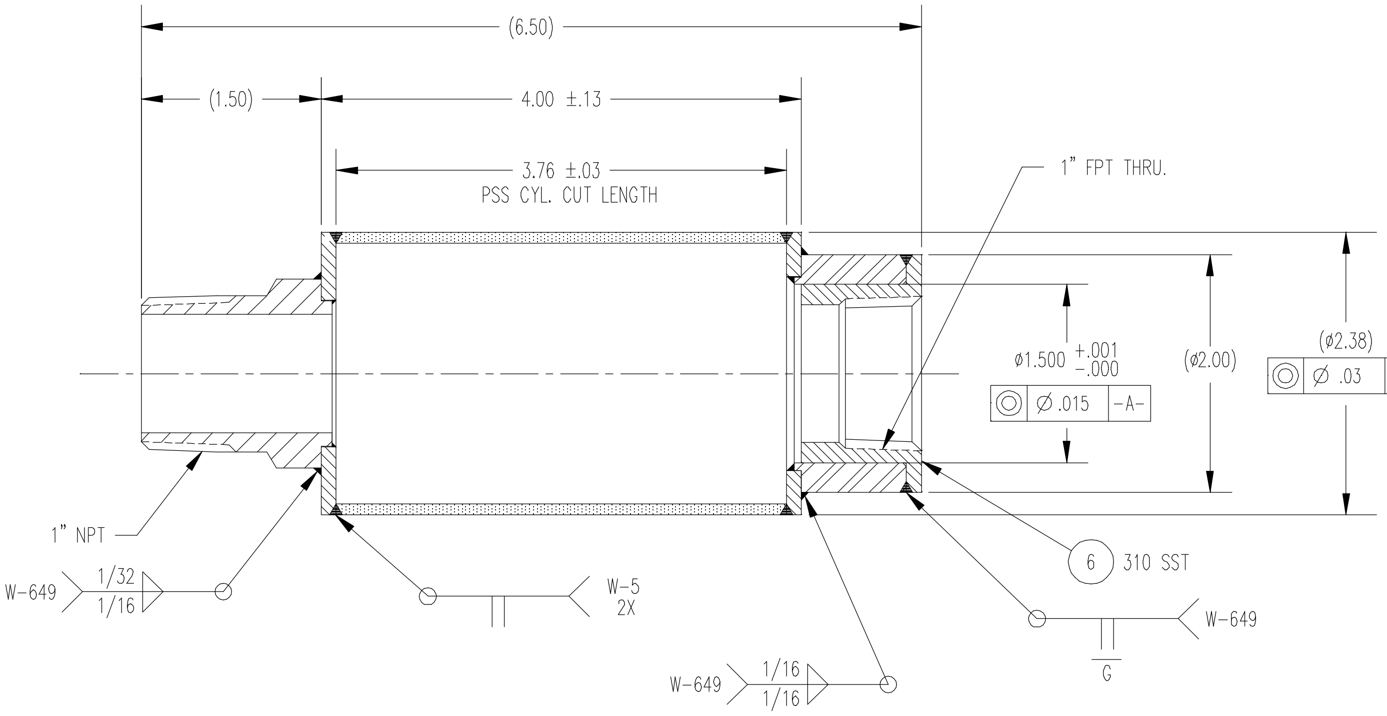
Powder Supply

A supply of powder for each of the three compositions has been purchased. Air induction melts were made followed by water atomization. Two hundred pounds of each composition have been sieved to the -100 +325 range.

FIGURE 4

IRON ALUMINIDE FILTER MEDIUM IS CIRCUMFERENTIALLY WELDED

GAS TUNGSTEN ARC (TIG) PROCESS USED.

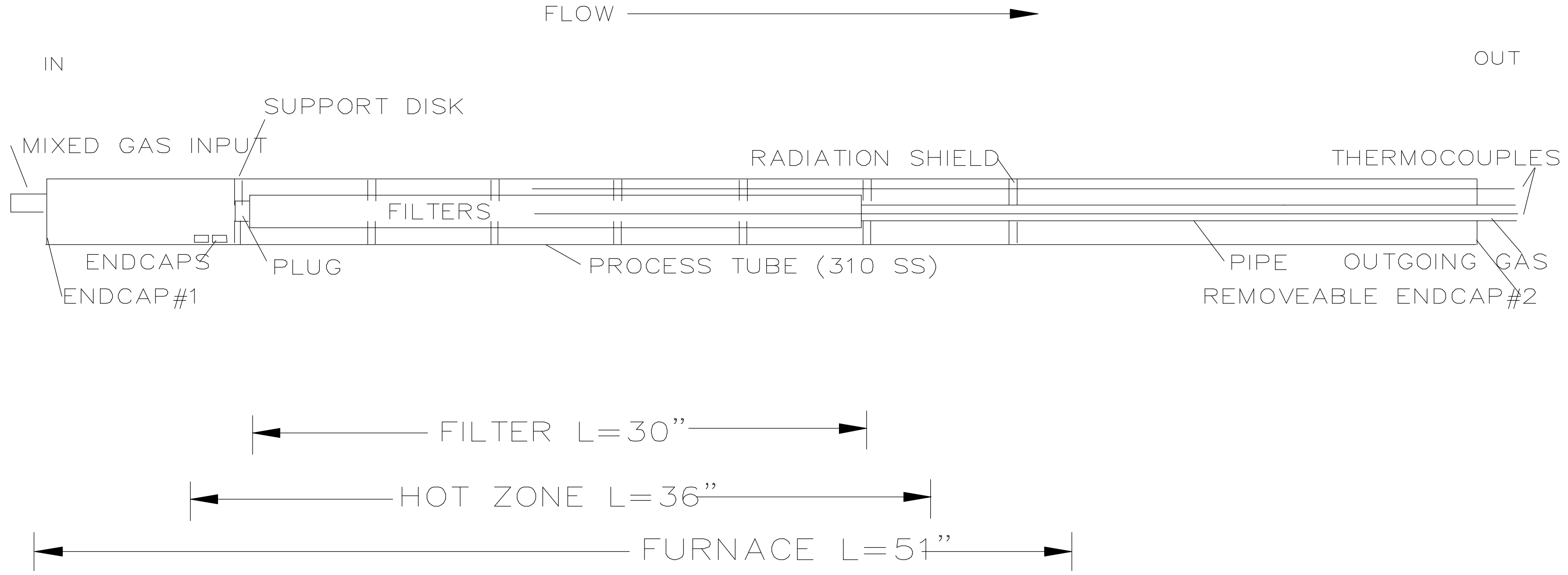


FILTER FOR SHORT TERM TESTING

FIGURE 5

CLOSE-UP SCHEMATIC OF PROCESS TUBE FOR SHORT TERM EXPOSURE

- FLOW THROUGH DESIGN
- PROVISION FOR BLOWING BACK - INTERMITTENT OPERATION
- PROVISION TO ADD HCl+NaCl+KCl TO PREMIXED H_2S CONTAINING ATMOSPHERE



The target and reported analyses for the first three candidates are listed below in weight percent:

		Fe	Al	Cr	O2	C	Zr	B
FAS	T	81.2	16.4	2.2	--	.007	.19	---
	R	BAL	17.1	2.2	.50	.038	.17	---
FAL	T	77.9	16.4	5.5	--	.007	.19	---
	R	BAL	15.8	5.5	.38	.046	.17	---
FeAl	T	76.2	23.6	---	--	.007	.19	.004
	R	BAL	22.8	---	.73	.024	.17	.008

T = Target Comp. R = Reported Comp.

Oxygen contents appear to increase with increasing aluminum content as might be expected. After evaluation it was concluded that powder from all three compositions was satisfactory.

Making Seamless Cylinders and Compressing Them

Procedures for making seamless cylinders in Iron Aluminide are firmly established. Procedures for compressing seamless preforms are well established as well. There is a beneficial major densification in this compression step.

Vacuum Sintering of Seamless Cylinders

Figure 6 shows the Iron Aluminide filter medium after vacuum sintering to 2310F.

The carbon content was higher than expected indicating that some of the carbon from the pyrolyzed (residual) binder had alloyed with the powder. This might add 0.15% carbon over what otherwise would be expected. The carbon, incidentally, is probably improving the high temperature load carrying ability which is a characteristic that is needed (stress rupture, creep strength) with these alloys. In any event, experiments are underway to reduce carbon pickup from this source - by changing processing details, type of binder, etc.

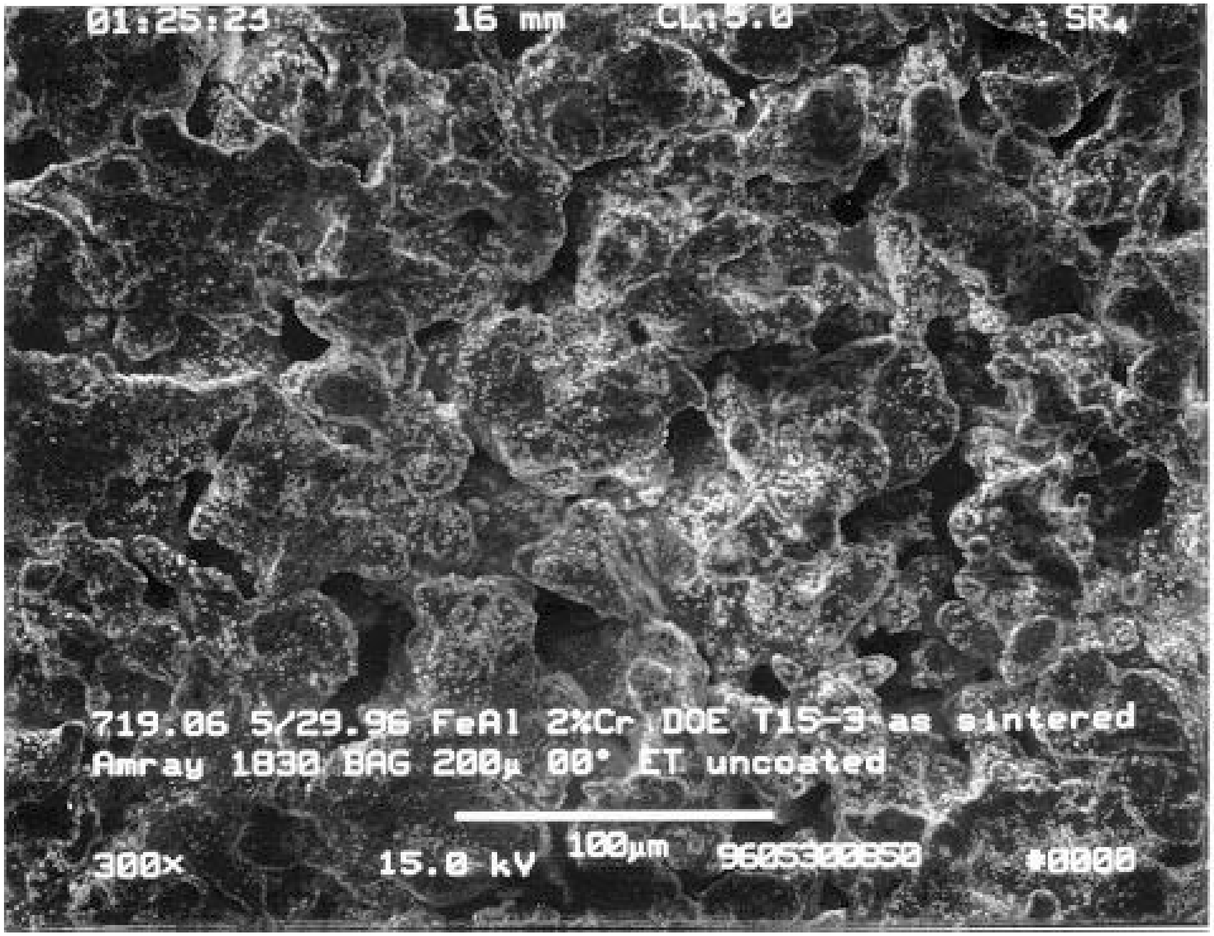
Evaluation of Sintered Seamless Cylinders

Ductility (in an empirical test which measures % contraction in the tube diameter before fracture) was better with all three compositions for the higher sintering temperature. This is seen below:

* Sintering at 2310°F - Gave values of 3.2, 4.9 and 5.9 % ductility before cracking for the FeAl type (0 % Cr), FAL type (5 % Cr) and FAS type (2 % Cr) respectively.

* Sintering at 2345°F - Gave values of 5.3, 5.6, and 7.3 % ductility before cracking for the FeAl type (0 % Cr), FAL type (5 % Cr) and FAS type (2 % Cr) respectively.

FIGURE 6
IRON ALUMINIDE FILTER MEDIUM
AS VACUUM SINTERED - SEM AT 300X



* Sintering at 2385°F - Gave values of 6.6, 5.9, and 6.9% ductility before cracking for the FeAl type (0% Cr), FAL type (5% Cr) and FAS type (2% Cr) respectively.

Experiments are being run to insure that the intrinsic strengths of the tubes are being reported. A new test is being adopted that will be used as a supplement to or perhaps a replacement for "O" ring testing.

Application

Prototype Filters

Three Iron Aluminide candle filters were requested this spring for inclusion in tests that DOE is planning to run at METC. They were needed by the beginning of June. It was decided that the opportunity to develop service experience took precedence over other considerations. A promising alloy and a satisfactory sintering cycle were chosen recognizing that neither had been optimized as yet. Accordingly, it was decided to make the filters from the FAS (2% Cr) type composition, sintered to 2310F, with 310 hardware that was TIG welded. The filters were preoxidized.

Tensile strengths (Av.) for each of three Iron Aluminide cylinders used to make the filters were 9.28, 12.9 and 7.05 ksi.

Preoxidation caused a weight gain of 0.67% most of which appears to be attributable to alumina enrichment of the surface. Preoxidation caused only a 0.7 % average increase in Air /\P (28SCFM) from 27.6 to 27.8 inches of water. First bubble points averaged 21.5 inches of water. The first bubble points were all in the media which is favorable - with none at or immediately adjacent to the welds. The open bubble points of the filters averaged 30.7 inches of water: this corresponds with the "fizz" point for these filters.

Figure 7 shows the Iron Aluminide filter medium after vacuum sintering and preoxidizing in air. There is evidence that the white spots are particles of alumina. Preoxidation of the filters also provided a highly effective stress relief treatment without distortion.

Figure 8 shows the completed filters. Each has a nominal area of one square foot (2 3/8 inch Diameter by 19 1/2 inches long of Fe_3Al). The filter media had Void Volumes of 42.5, 41.9 and 45.8% respectively.

In addition to the prototype filters sent to METC, other prototype filters are being prepared for field evaluations.

FIGURE 7

IRON ALUMINIDE FILTER MEDIUM AS VACUUM SINTERED AND PREOXIDIZED (800°C, 7 HOURS) - SEM AT 300X

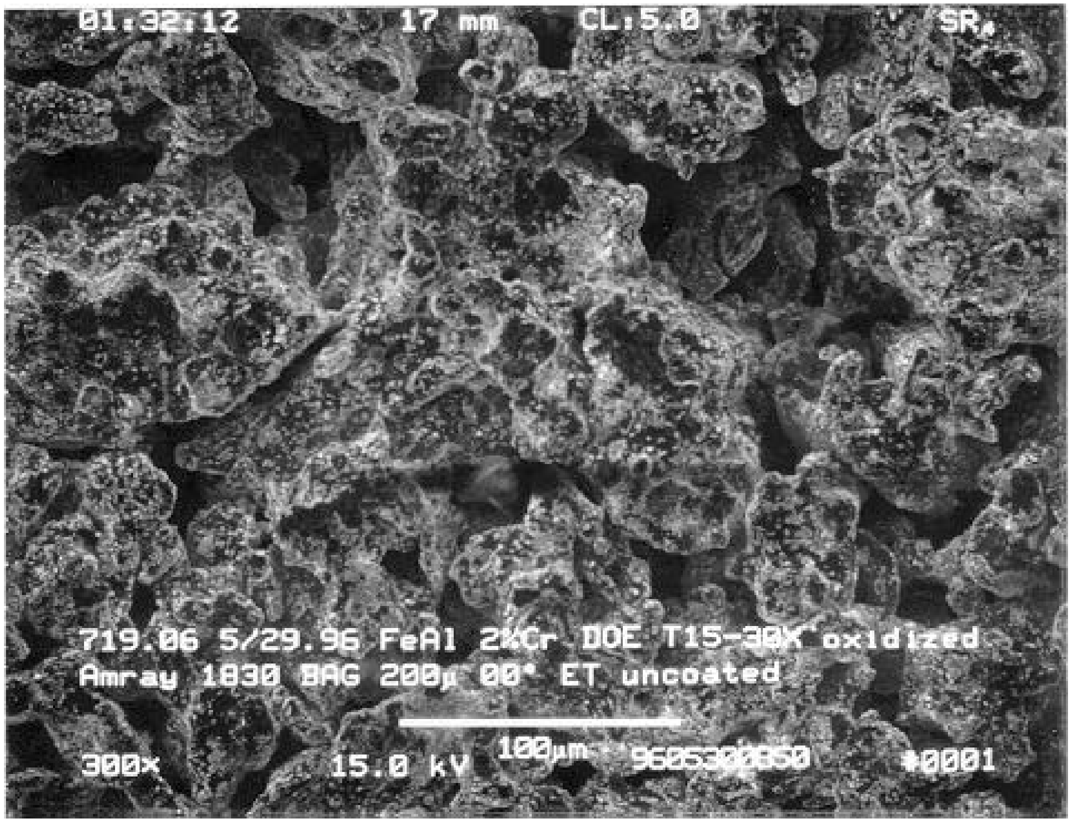


FIGURE 8
THREE IRON ALUMINIDE PROTOTYPE TEST FILTERS

Benefits

To summarize(several observations and points made in this paper), there are four basic advantages offered by this technology:

- First, there is a wealth of information on iron aluminide already available (developed by and/or compiled by ORNL) on the physical properties, mechanical properties and high temperature corrosion resistance - particularly in the presence of hot hydrogen sulfide. While this data was determined for solid materials adjustments can be made to predict performance of porous media.
- Second, an existing manufacturing process is being adopted to produce iron aluminide as seamless cylinders. In fact all the steps for making experimental iron aluminide filters are done on full scale production equipment.
- Third, it is estimated that seventeen thousand square feet of iron aluminide filters could conservatively be produced per year without significant capital expenditures and without interfering with on going production of other grades.
- Fourth, compared to ceramics iron aluminide filters offer crack resistance. Furthermore, fabrication is greatly simplified because iron aluminide can be welded.

Future Activities

In Task 4 the best composition overall will be tested for long term corrosion resistance. This will be followed in Task 5 by the manufacture of 50 filters (optional).

Acknowledgements

Want to acknowledge the advice and encouragement of our METC Project Manager Ted McMahon. Special thanks are due to several individuals at Pall including Steve Geibel (PED) for guidance, to Joe Puzo (MMD) for help in the spinning and vacuum sintering of seamless cylinders and to Keith Rekczis (PED), a team member, for running experiments and for help in upgrading the exposure apparatus. Special thanks are due as well to several staff members at Oak Ridge National Laboratory: this includes Peter Tortorelli and Jack de Van (Ret.) for calculating equilibrium atmospheres.

References

(1). Data Package on Fe3Al-FeAl Based Alloys Developed at ORNL, Compiled by Vinod K. Sikka, January 20, 1993.

(2). J. H. De Van, "Development of Surface Treatments and Alloy Modifications for Corrosion Resistant Oxide Scales", Article In *Proceedings of the Fourth Annual Conference on Fossil Energy Materials*, Compiled by R. R. Judkins and D. N. Braski, August 1990, pgs. 299- 309.