Novel Oxide-Oxide Fiber Reinforced Hot Gas Filter Development

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1.0 INTRODUCTION

This report describes the fabrication and testing of continuous fiber ceramic composites (CFCC) based hot gas filters. The work was divided into two primary tasks. In the first task, a preliminary set of compositions was fabricated in the form of open end tubes and characterized. The results of the first task were then used to identify the most promising compositions for sub-scale fabrication and testing. In addition to laboratory measurements of permeability and strength, exposure testing in a coal combustion environment was performed to assess the thermo-chemical stability of the CFCC materials. The results of this testing were then used to down-select the filter composition for full-scale filter fabrication and testing in the optional Phase II of the program.

2.0 BACKGROUND INFORMATION

Pressurized fluid bed combustion (PFBC) and integrated gasification combined cycle (IGCC) systems are among the advanced coal-based energy cycles being considered for low cost, clean power generation. Hot gas filters are required to remove particulates from the high temperature inlet stream to the turbine in order to protect turbine components from excessive erosive wear and to meet clean air requirements.

The level of mechanical durability exhibited by the currently available filters in field tests indicates that more rugged filters are required to meet the demands of large power generation systems. Furthermore, long term corrosion resistance of currently available filters has yet to be demonstrated in PFBC systems.

The essential requirements of a composite material designed to meet the program objective for a toughened hot gas filter include the following:

o stable continuous fiber
o rigid porous matrix
o engineered fiber-matrix interface
o cost effectiveness

3.0 PROGRAM OBJECTIVES

The objectives of this program are to develop toughened ceramic hot gas filters and evaluate these filters for application in Pressurized Fluidized Bed Combustor (PFBC) and Integrated Gasification Combined Cycle (IGCC) power generation systems.

4.0 APPROACH

Composite preforms were fabricated by a modified filament winding process. The preforms were then bonded and heat treated and machined to produce the test samples. Laboratory testing was used to refine the initial compositions which were then evaluated through additional laboratory and field tests.

Filament winding technology developed under the CFCC program was transferred to the B&W ERI-Lynchburg manufacturing division. Particular attention was given to the development of a consistent macro/micro-structure over the length of the test specimen in terms of the relative amounts and distributions of the chopped and continuous fiber. Controlling this distribution is considered to be essential to achieving the desired properties. Four candidate compositions were included in this task. These compositions, included variations in the type and amount of continuous fiber as well as alternate bond systems and fiber coatings. The compositions were evaluated in terms of permeability, corrosion resistance, and mechanical properties. The characterization results were then used to refine the test compositions used in the full-scale fabrication task.

Based on CFCC results, Mitsui's Almax alumina fiber and 3M's Nextel 610 fibers were used as the reinforcements in the candidate composites. The Almax fiber consists of 1000 filament tows of polycrystalline fibers. The Nextel 610 fibers are comprised of 400 filament tows. The unit weights of the fibers are 2250 denier and 1500 denier for the Almax and Nextel 610 respectively. Saffil chopped fiber was used for all test compositions. Ten samples of each of the compositions given in Table 1 were fabricated in the form of sub-scale filter elements (2.4" diameter by 12" long).

Fiber coatings are generally required to control the fiber/matrix interface and to protect the continuous fibers from degradation during processing and/or service. While the fiber architecture used in this work does not actually have a continuous matrix phase it is important that the fiber to fiber bond does not degrade the continuous fibers. Fugitive carbon coatings have been shown to be effective means of protecting the fibers in many composite systems. The fact that the coating disappears in service is expected to have less of an affect on the long term properties of an oxide composite system than for non-oxide systems. For the oxide fibers utilized in this program, the primary requirement was to protect the fibers during processing with the acid stabilized bonding solutions.

An improved fiber coating process based on the work of Hay[1] was implemented under the CFCC program for the sub-scale fabrication task because the high temperature mechanical properties of samples containing pyrolyzed fiber sizings exhibited brittle failure . This process utilized the surface tension differences in immiscible liquids to minimize the coating bridges that normally occur within the fiber tow.

Sample filter elements were characterized in terms of their microstructure, permeability, corrosion resistance, and mechanical properties. The permeability of test specimens was determined from the face velocity and the associated pressure drop across the specimen. Compressive C-ring tests were performed on a computer controlled mechanical test machine using calibrated load and deflection sensors. All C-ring testing was performed at 1600 °F. Five one inch wide C-rings were tested from each sub-scale filter element. .

The distribution and relative amounts of continuous fiber, chopped fiber, and bond phase was determined by examination of polished sections in an Etech scanning electron microscope (SEM) equipped with energy dispersive spectroscopy (EDS). The samples were vacuum impregnated with low viscosity resin in order to minimize open porosity in the mount.

The corrosion resistance of the candidate filter compositions was determined by an exposure test in a coal combustion environment. The original plan was to perform the test in the Tidd PFBC. Because the Tidd operation ended in March, 1995, an alternate test site was investigated. The 55 MWe circulating fluid bed (CFB) combustion unit located at Ebensburg, PA was selected because it provided a good simulation of the atmosphere and ash characteristics of a PFBC. The disadvantage of the CFB concerned the much higher ash loading in the gas stream which would require careful erosion protection of the samples.

Since the Ebensburg CFB is a commercial utility boiler, it was obviously not possible to shut the unit down to install and retrieve samples. A sample probe was therefore designed to be installed through standard 3" observation ports in the side wall of the CFB while the boiler was on-line. The outer assembly was fabricated from solid and perforated 304 stainless and was intended to protect the sample from excessive erosion by the heavily ash loaded gas stream of the circulating fluid bed combustor. Prior to sample probe insertion, low pressure seal air was injected into the port to prevent the escape of the combustion gas stream. During the 250 hour exposure period, the ash/sorbent was expected to penetrate through the perforated tube and accumulate around the sample. Two samples of each composition received static exposures at temperatures of 1500 °F and 1625 °F.

The exposure test was originally intended to be a static exposure test; however, one additional sample probe of each composition was modified to provide periodic back pulsing with ambient air as part of the CFCC program to add a thermal fatigue component to the test. Plant air at about 90 psi was plumbed to the center of the probe and triggered by a timer controlled pneumatic valve (COAX model VMK20). A single timer/pilot valve was used to control the pulse duration and cycle time of the four coax valves. A pulse duration of about 0.4 seconds was applied to the samples at 15 minute intervals for a total of approximately 1000 cycles.

5.0 RESULTS AND DISCUSSION

The properties of the sub-scale filter elements are summarized in Table 2. Typical high temperature C-ring results for each of the sub-scale compositions in the "as fabricated" condition are given in Figure 1. Composition C1 exhibited relatively low strength but with an extended strain at near maximum load. Composition C2 produced a very brittle fracture behavior in the as-fabricated condition. Composition C3 exhibited high strength and good fracture behavior. The contrast in failure behavior between composition C3 and C2 is not understood since both compositions use the same bond system and carbon coated fiber. The compressive C-ring results for composition C4 are very similar to C1 in terms of maximum strength and failure behavior. As discussed above for composition C1, the distribution of the bond phase in composition C4 appears to account for the lower observed strength.

Figure 2 summarizes the C-ring results for the samples taken from the 1500 °F region of the CFB. Composition C1 increased strength by approximately 16 percent compared to the as-fabricated condition and maintained good failure behavior. Composition C2 retained approximately 80 percent of the as-fabricated strength and exhibited non-brittle failure behavior following the CFB exposure. Composition C3 retained 69 percent of the as-fabricated strength and exhibited non-brittle failure behavior. The compressive C-ring strength of composition C4 increased by about 47 percent and displayed a non-brittle failure behavior.

The high temperature C-ring results for the samples located in the 1625 °F region of the CFB are shown in Figure 3. These results are very similar to 1500 °F CFB samples. Composition C1 increased in strength by about 21 percent with a non-brittle type of failure. Composition C2 retained approximately 74 percent of its as-fabricated strength and also exhibited non-brittle failure behavior. Composition C3 exhibited a strength retention of about 70 percent with a non-brittle failure. Composition C4 produced a 73% increase in strength with a non-brittle failure.

The post-test high temperature C-ring results for CFB thermal fatigue samples are shown in Figure 4. These samples received approximately 1000 back pulses and were located in the 1500 °F region of the CFB. The composition C1 sample showed a 25 percent increase in strength with a relatively non-brittle failure. The composition C2 sample retained 80 percent of the as-fabricated strength and exhibited non-brittle failure. Composition C3 exhibited a retained strength of 72 percent with a non-brittle failure behavior. Composition C4 increased in strength by about 18 percent and also exhibited non-brittle failure.

In general, the SEM examination revealed very little evidence of ash or sorbent penetration into the samples following the thermal fatigue test exposure in the CFB. In composition C1, the distribution of the Almax fiber tows appeared relatively uniform but widely spaced due to the large number of filaments in each tow. The bond phase in this sample was more concentrated near the outside of the sample. This concentration gradient from the OD to the ID most likely caused the low strength and low permeability of these samples.

The microstructure of composition C2 revealed a much more uniform distribution of bond phase compared to composition C1 described above. In composition C3, the increased concentration of Nextel 610 was apparent. The distribution of continuous fibers appears more uniform at the 2:1 Nextel to Saffil ratio. The microstructure is otherwise similar to C2 in terms of there being no evidence of bond phase concentration gradients.

The cross-section of sample C4-4 following the CFB thermal fatigue exposure test also exhibited a bond phase concentration gradient from OD to ID is very similar to that observed in composition C1. There appears to be good potential to improve the properties of this composition if the bond phase can be distributed more uniformly.

Based on the mechanical properties, permeability, and the microstructure results described above, composition C3 was selected for Phase II, the full scale fabrication and simulated PFBC testing and characterization. The overall status of the project is summarized in Table 3 which compares the filter requirements to the current status of the B&W filter manufacturing process.

6.0 BENEFITS

This program has demonstrated a hot gas filter concept and fabrication method that resulted in an oxide-oxide composite based filter with improved strength and toughness compared to monolithic filter materials. In addition, the low density of these filter elements results in substantial weight savings in the filter system (approximately 10 lb. per filter element or 60,000 lb. in a 350 MWe plant).

7.0 FUTURE ACTIVITIES

Four near full size filter elements have been fabricated and will be tested in the Westinghouse High Temperature High Pressure filter test facility during July. These samples along with two baseline samples will be characterized to guide future filter improvements. In addition, an optimization/cost reduction effort is underway to reduce the amount of continuous fiber and/or eliminate the need for a fiber coating. The results of the filter optimization task will be utilized in the production of 50 filter elements for testing in a DOE demonstration facility. In order to guide future filter development activities, a better definition of the mechanical loads imposed by the mounting method as well as in service is required to establish the wall thickness and flange configuration. The method used in this program can accommodate thicker walls but this has a predictable effect on cost.

8.0 ACKNOWLEDGEMENTS

The assistance of T. J. McMahon, METC COR, is gratefully acknowledged. In addition, the U.S. Department of Energy Continuous Ceramic Fiber Composite program has provided valuable material development support for this project. Finally, the cooperation of Ebensburg Power for the 55 MWe Circulating Fluid Bed sample exposure testing is gratefully acknowledged.

9.0 REFERENCE

1. R. S. Hay, "Sol-Gel Coating of Fiber Tow", Ceram. Eng. Sci. Proc. 12[7-8] pp. 1064-1074 (1991).

Table 1. Candidate sub-scale filter compositions.

	C1	C2	C3	C4
continuous fiber	Almax	Nextel 610	Nextel 610	Nextel 610
chopped fiber	Saffil	Saffil	Saffil	Saffil
continuous to chopped ratio	1:1	1:1	2:1	1:1
continuous fiber coating	none	carbon	carbon	none
bond type	B1	B2	B2	B1
continuous fiber architecture	45° helical	45° helical	45° helical	45° helical

Table 2. Properties of sub-scale hot gas filter elements

sample comp.	comments	% fiber	% saffil	% bond	delta P @ 10ft/min (inches H_2O)		C-ring 1600 F (psi)
					pre test	post test	
C1	as fabricated	38.2	41.0	20.8	6.3	na	825
C1	CFB	36.5	40.4	23.1	5.6	10.3	995
C2	as fabricated	31.4	38.0	30.6	6.2	na	1704
C2	CFB	35.5	40.4	24.2	3.8	9.0	1330
C3	as fabricated	52.6	28.1	19.3	11.9	na	1752
C3	CFB	52.3	28.5	19.2	5.9	10.2	1228
C4	as fabricated	37.4	42.8	19.8	5.3	na	856
C4	CFB	34.1	40.4	25.5	9.6	13.5	1249

Table 3. Hot Gas Filter Summary

Property	Req't.	Status	Challenge
size	2.4 x 60"	2.4 x 43"	minor
shape	flanged, closed end tube	flanged, closed end tube	complete
pressure drop @10ft/min	10	10-15	moderate
strength	1 - 4 ksi	0.8 - 1.7 ksi	moderate
toughness	non-brittle failure	non-brittle failure	moderate
thermal shock	ambient air back pulse	72 - 125% retained strength after 1000 back pulses	moderate
corrosion resistance	3 year life	70 - 146% retained strength after 250 hrs in CFB	moderate

Figure 1

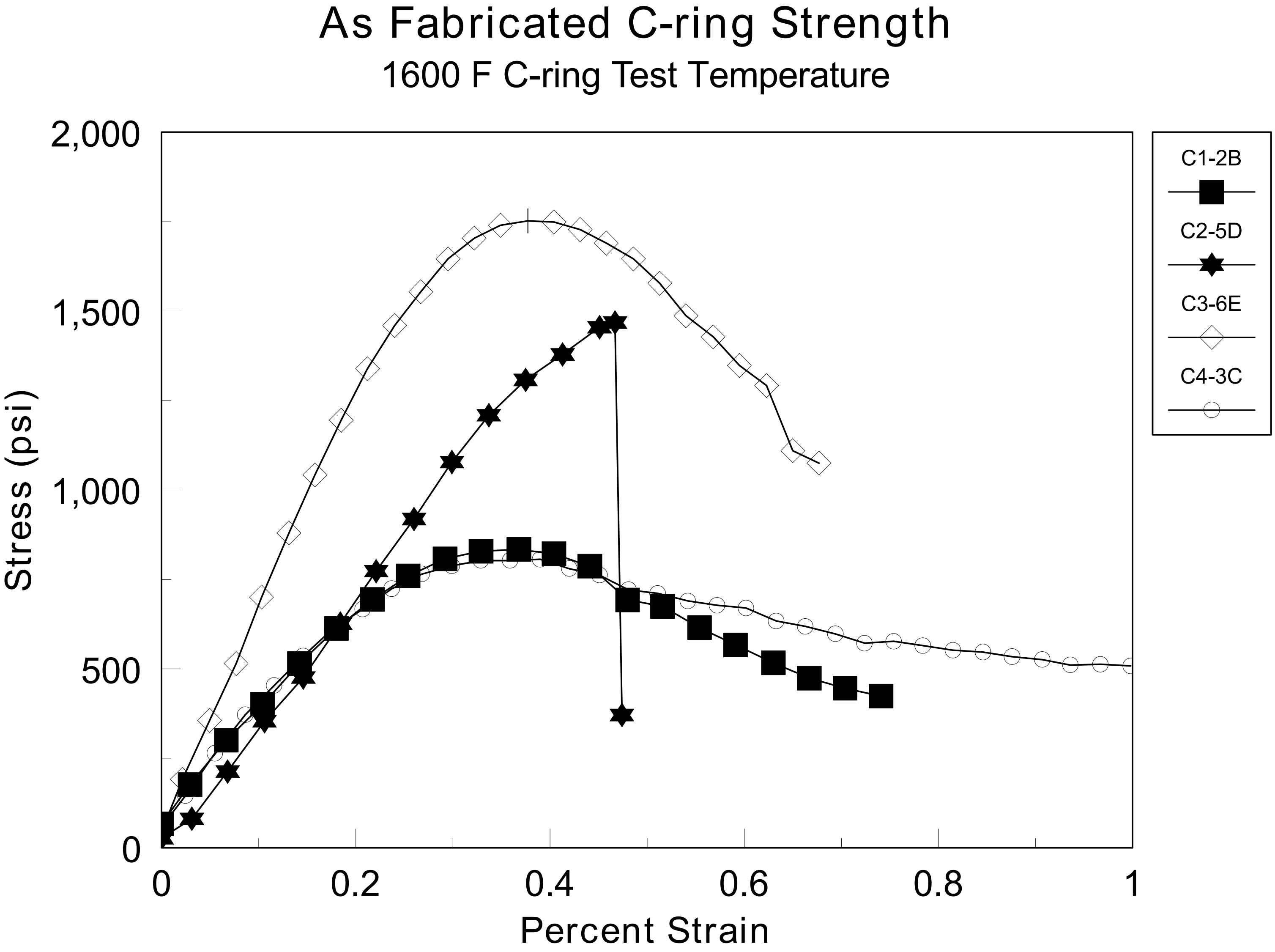

As Fabricated C-ring Strength
1600 F C-ring Test Temperature
Stress (psi)
2,000
1,500
1,000
500
0
0
0.2
0.4
0.6
0.8
1
Percent Strain
C1-2B
C2-5D
C3-6E
C4-3C

Figure 2

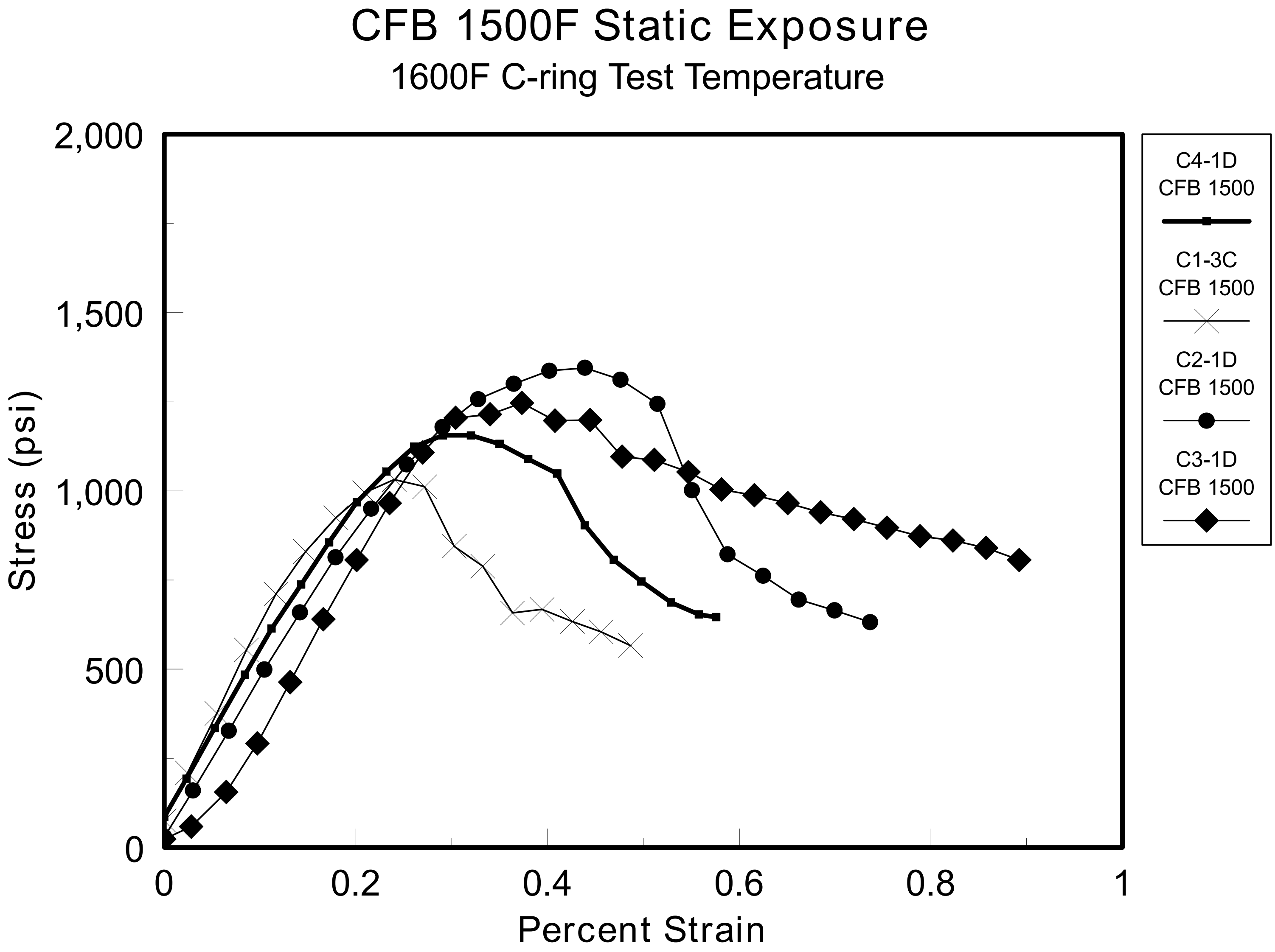

CFB 1500F Static Exposure
1600F C-ring Test Temperature
Stress (psi)
2,000
1,500
1,000
500
0
0
0.2
0.4
0.6
0.8
1
Percent Strain
C4-1D CFB 1500
C1-3C CFB 1500
C2-1D CFB 1500
C3-1D CFB 1500

Figure 3

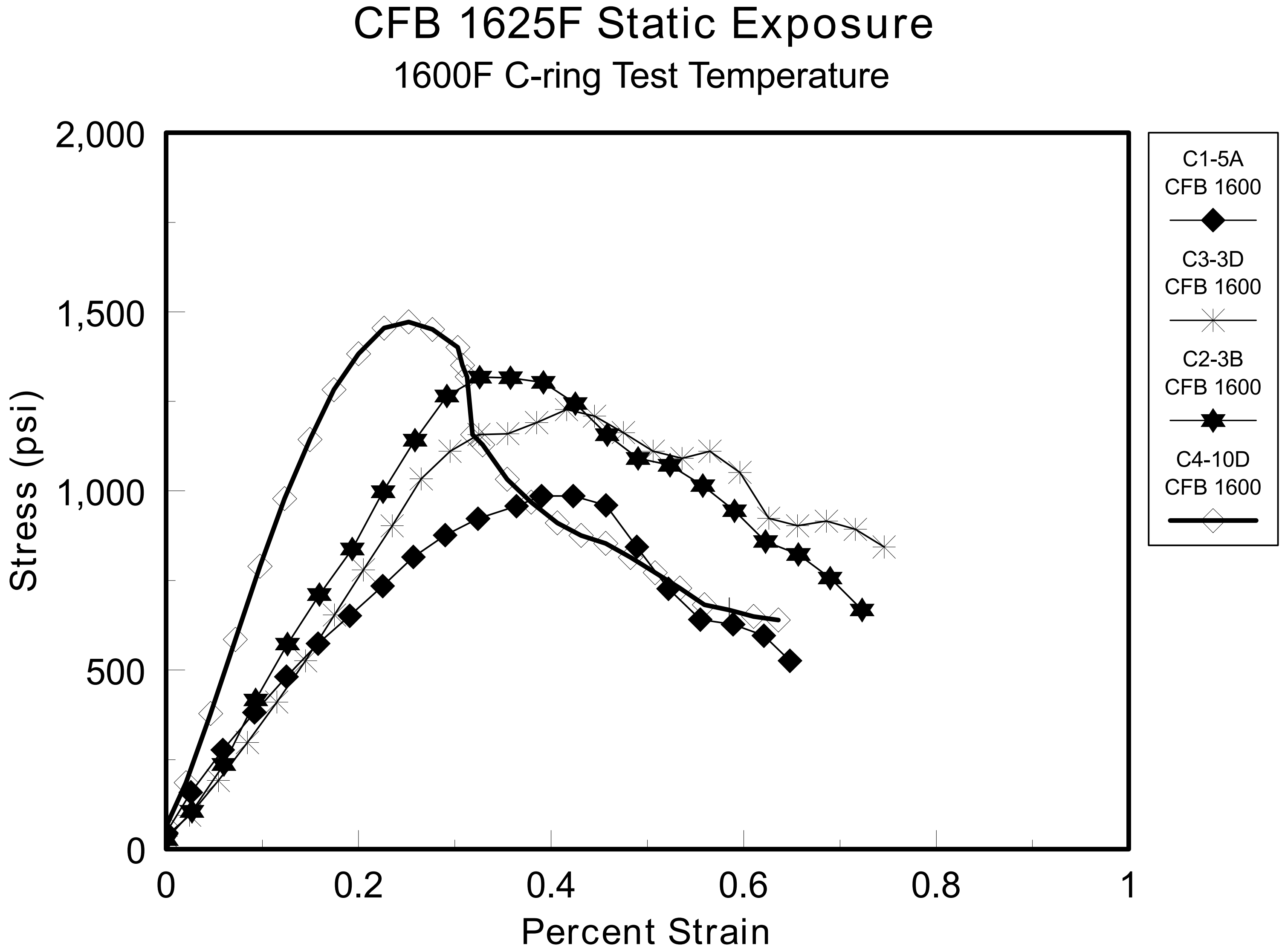
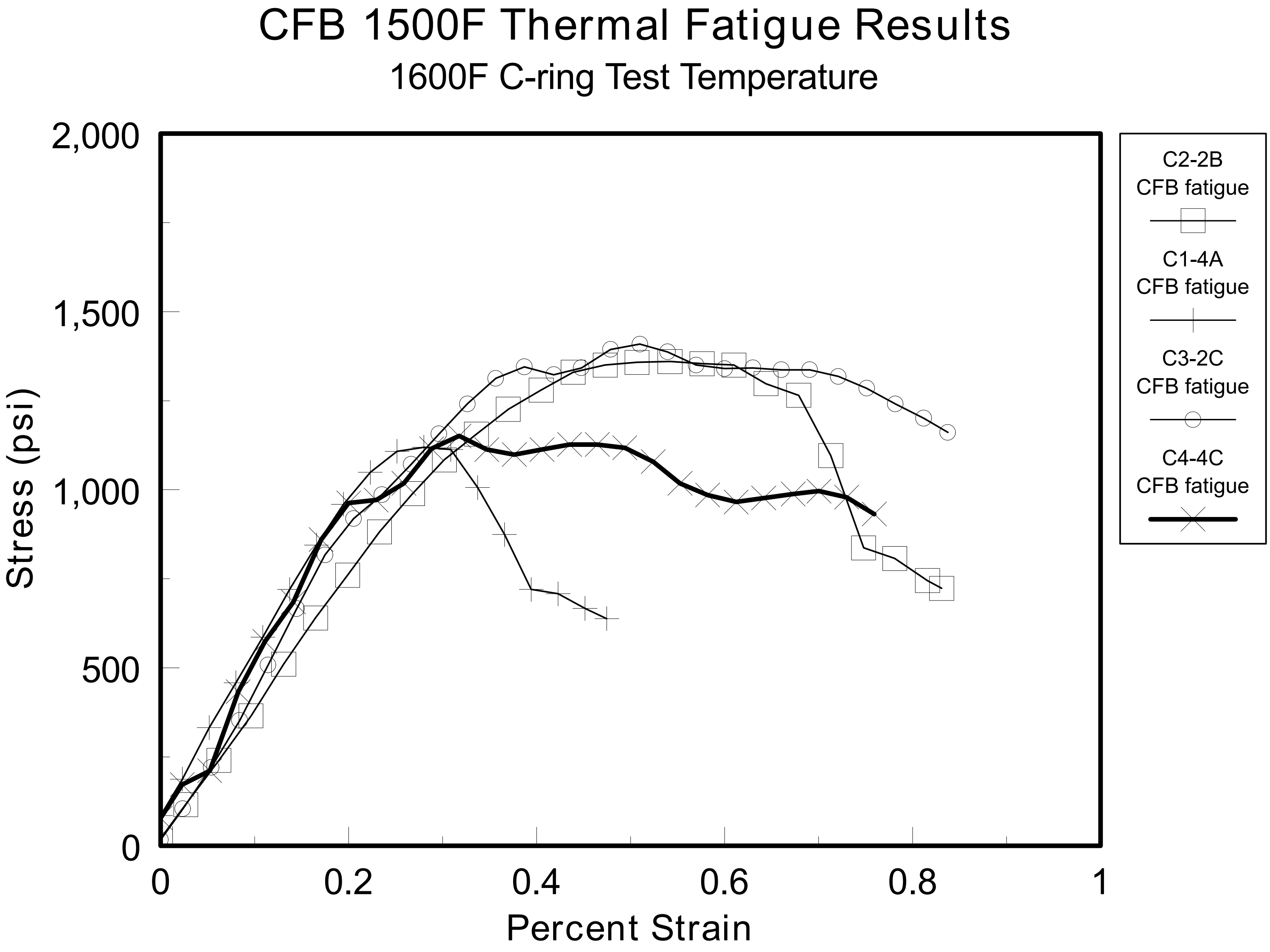

CFB 1625F Static Exposure
1600F C-ring Test Temperature
2,000
1,500
1,000
500
0
Stress (psi)
0
0.2
0.4
0.6
0.8
1
Percent Strain
C1-5A CFB 1600
C3-3D CFB 1600
C2-3B CFB 1600
C4-10D CFB 1600

Figure 4


CFB 1500F Thermal Fatigue Results
1600F C-ring Test Temperature
Stress (psi)
2,000
1,500
1,000
500
0
0
0.2
0.4
0.6
0.8
1
Percent Strain
C2-2B
CFB fatigue
C1-4A
CFB fatigue
C3-2C
CFB fatigue
C4-4C
CFB fatigue