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PFBC HGCU Test Facility
Technical Progress Report

Fourth Quarter, CY 1993

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Prepared for:

The United States Department of Energy
Under DOE Instrument No. DE-FC21 89MC26042

January, 1994

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I. INTRODUCTION

This is the seventeenth Technical Progress Report submitted to the Department of Energy (DOE) in connection with the cooperative agreement between the DOE and Ohio Power Company for the Tidd PFBC Hot Gas Clean Up Test Facility. This report covers the period of work completed during the Fourth Quarter of CY 1993.

During this quarter, the Tidd Hot Gas Clean Up System remained out of service for repairs following the September shutdown. Engineering activity this quarter was concentrated on implementing system enhancements to improve the reliability of the system in subsequent tests. The filter was fitted with all new candle elements and other modifications as discussed below.

On October 20, 1993, we presented the preliminary results of the Advanced Particle Filter (APF) testing during the previous quarter to the DOE at Morgantown.

II. WORK ACCOMPLISHED DURING THE REPORTING PERIOD

2.1 Detailed Design and Engineering

A detailed design was made for the addition of nine purge nozzles to the APF hopper. The nozzles were added to the hopper during the fourth quarter outage. Six nozzles were added at one elevation and three other nozzles were added at a lower elevation. With the three original nozzles at the bottom of the hopper, there are now a total of twelve purge points. Figure 1 shows the design of the new nozzles.

An air reservoir was also added to one of the original purge nozzles in case a large blast of air is required to dislodge accumulated ash.

Based on operating experience with the APF, it was concluded that the filter pressure differential tends to become unstable, i.e., keeps increasing at gas temperatures above approximately 1400°F. In order to allow full load operation of the Tidd Plant, which will produce gas temperatures as high as 1550°F and not impose excessively high pressure differentials across the APF, a means for cooling the gas upstream of the APF was devised. A new 6-inch line was engineered and installed this quarter which will allow 550°F - 580°F process air to mix with the gas in a pipe spool upstream of the filter. This line will be used as necessary to maintain the filter inlet temperature no higher than 1400°F during the next test run.

As previously reported, the primary cyclone upstream of the APF was partially detuned late in the last test run and its effects were inconclusive. During this quarter, the shutoff valve in the spoiling air line was replaced with a ball valve to increase the capacity of the spoiling air. Further engineering studies have produced a plan for totally spoiling the cyclone, should that become necessary for stable filter operation. Basically, the method involves introducing additional air at the ash pickup nozzle at the bottom of the cyclone, thus preventing the removal of the ash from the cyclone dip leg. Eventually, the ash will fill up the dip leg and then be carried out with the main gas stream.

If complete cyclone detuning is attempted, it means that the dust loading to the filter will increase by a factor of 40, from 500 to 20,000 ppm. The ability of the ash removal system to handle up to 3000 lb/hr of ash was evaluated carefully. The screw cooler should be able to pass the required ash loading, provided the cooling water temperature is reduced from 340°F to 100°F. The lockhopper system will handle the higher flow but will be cycling every ten minutes or so. If the lockhopper system malfunctions even temporarily, the APF could fill with ash unless an emergency ash removal system is immediately available. The existing emergency ash removal line does not have sufficient capacity. Therefore, a second emergency ash removal line was engineered. This will consist of a 2-inch double extra strong pipe that will run from the screw cooler outlet pipe to a modified tee bend in the existing primary cyclone ash line. A tungsten carbide nozzle will be installed at the end of the line to control the velocity in the pipe and dissipate the excess pressure drop. Materials were ordered for the new line and it will be

installed during the next quarter.

Following the last test series, the back pulse solenoid valves were inspected and found to be galled on both the piston and cylinder walls. Much engineering activity was devoted to developing a plan for modifying the valves to eliminate this problem. The valve pistons were Stellite coated and the valve body bores were nickel-boron plated to improve resistance to galling. Following these modifications, the valves successfully underwent accelerated cycle testing to verify the design.

One back pulse tube was cut up and examined for degradation. Microcracking was evident on the inside surface of the tube, with cracks as deep as 0.020 inches. Based on this observation, it was decided to replace the Incoloy 800HT material with Haynes 230 alloy, which has better resistance to thermal fatigue. The new back pulse tubes were fabricated and installed during this quarter.

Engineering activity was also expended in following miscellaneous repairs and enhancements to the system during this quarter. These activities included the following:

1. Repair of anchors in the APF head liner
2. Repair of thermocouples in the APF
3. Replacement of ash sampling isolation valves
4. Replacement of several other ball valves with high performance valves
5. Repair of the ash pickup nozzle in the backup cyclone ash vessel

6. Installation of the revised APF hopper vibrator, mounted outside the vessel, as described in the previous quarterly report.

2.3 Westinghouse Engineering and Design

See attached appendix.

7.0 Hazardous Air Pollutants

Engineering time was spent reviewing the design of the sampling hardware that is being supplied by Radian Corporation for hazardous air pollutant sampling. Several enhancements were given to Radian for inclusion in their design.

Figure 1.

	3Q89	4Q89	1Q90	2Q90	3Q90	4Q90	1Q91	2Q91	3Q91	4Q91	1Q92	2Q92	3Q92	4Q92	1Q93	2Q93	3Q93	4Q93	1Q94	2Q94
Original	0.8	3.2	8.1	15.6	23.6	31.8	40.0	48.2	57.3	69.2	77.4	84.7	88.6	90.9	93.1	94.9	96.7	98.6	99.3	100.0
Current	0.6	2.0	3.4	4.8	7.1	14.3	23.3	40.2	53.4	62.2	70.2	77.8	82.5	83.8	87.2	90.5	93.7	96.7	98.4	100.0
Actual	0.6	2.0	3.4	4.8	7.1	14.3	23.3	40.2	53.4	62.2	70.2	77.8	82.5	87.9	91.6	94.5	96.9	98.9		

Figure 2.

	(\$000)																			
	3Q89	4Q89	1Q90	2Q90	3Q90	4Q90	1Q91	2Q91	3Q91	4Q91	1Q92	2Q92	3Q92	4Q92	1Q93	2Q93	3Q93	4Q93	1Q94	2Q94
3/91 Budget	18.7	70.1	106.3	152.3	383.1	1142.4	2108.4	3120.3	5213.0	8220.2	12683.5	14656.2	16524.3	18108.5	19483.3	20039.8	20372.3	20672.6	20681.8	20796.5
9/92 Budget			90.5	146.7	477.6	1531.3	2040.6	3059.1	5387.9	6280.4	9162.2	12522.0	15711.7	16998.3	18284.9	19571.4	20453.8	20567.8		
Actual			90.5	146.7	477.6	1531.3	2040.6	3059.1	5387.9	6280.4	9162.2	12522.0	14397.6	15569.3	16362.4	17753.2	18254.2	18835.3		

Figure 3.

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ADVANCED PARTICLE FILTER

Technical Progress Report No. 14
October through December 1993

Prepared by

Westinghouse Science and Technology Center
Pittsburgh, Pennsylvania

For

American Electric Power Service Corporation
Columbus, Ohio

AEPSC Contract No. C8014

TIDD ADVANCED PARTICLE FILTER

1.0 INTRODUCTION

The APF was shut down on September 23, 1993 and no operation was performed during this quarter. This report summarizes inspection, candle reinstallation, retrofit and accelerometer testing conducted during this three month outage.

2.0 INSPECTION

The APF internals were removed from the vessel on September 30, 1993. Inspection of the candles, including the distribution of failures, the nature of the fractures, the extent of ash bridging and the changes in material properties, has been discussed in quarterly technical report number 13. Additional observations to note include the following:

- (1) Some cast filter holders were slightly bent - generally in a direction radially outward from the cluster. (It is speculated that the same stresses from ash bridging that may have caused candle failure could also have produced creep and deformation in the type 310 stainless steel holders at operating temperature.)
- (2) The APF vessel head liner was deformed inward around the opening for the outlet nozzle. Three of six studs used to hold this liner in place were broken. (It is speculated that this deformation occurred as a result of misalignment of the outlet nozzle liner and the adjoining downstream pipe work during assembly.)

- (3) Cross sectional examination of one Incoloy 800 HT back pulse pipe showed cracking had initiated on the inside pipe surface. (Similar cracking noted for pulse pipes in the APF at Karhula is attributable to thermal fatigue arising from repetitive "cold" pulsing of this normally hot structure.)
- (4) Thermocouples and pressure taps passing through insulation in the APF vessel head were noted to be badly corroded and/or embrittled. It is likely that this condition contributed to some lost data during the last run.

Section 4 discusses the fixes and rework conducted to address these issues.

3.0 CANDLE REINSTALLATION

Concern regarding potential residual candle damage in the unbroken candles suggested that all candles should be replaced with new ones. The only exceptions to this decision were nine thoroughly tested surveillance candles which were reinstalled at position numbered 16 for each of the plenums. This position was selected because it was deemed most visible from the vessel inspection nozzles located at 0° (North), 120° (ESE) and 240° (WSW). A fail-safe/regenerator device was installed in the filter holder directly above each of these nine surveillance candles. (This fail-safe design has been recently operated in the APF at Karhula, Finland.)

Prior to candle installation, ash was removed outside (dirty side) and inside (clean side) of the metal structures by brushing and vacuuming. Filter holders were checked for plumb. Approximately 25 holders in the middle and bottom plenums were found to vary from plumb by more than $\pm 1^\circ$. These were straightened by mechanical bending. Of the filter holders which were straightened, one cracked at the juncture

between its 1-1/2" neck and 3-1/2" body. The crack was circumferential and approximately 1" long. The crack is attributable to reduced ductility of type 310 stainless steel following exposure in the temperature range for formation of sigma phase.

The filter holder was repaired by seal welding. Weldability was first checked by applying a bead on plate weld to a surveillance coupon removed from the vessel head. Visual and penetrant inspection confirmed soundness of the test weld and associated heat affected zone. The filter holder crack was then repair welded, visually inspected and penetrant tested.

All candles were reinstalled using new gaskets and new bolting hardware. New filter holder rings, with a collar on their inside diameter to ensure gasket containment, had previously been installed in all bottom plenum filter holders (ref. quarterly report no. 12, p 1). Such rings were also used for top and middle filter holders during the present installation. Bolting holes in all bottom cast nuts were redrilled to increase the hole diameter from 0.281 inch to 0.343 inch. This enlarged hole has been demonstrated to facilitate bolting during candle installation in the APF at Karhula.

4.0 RETROFIT WORK

Deformation of the vessel head liner was of modest magnitude and, since it did not interfere with APF functions, it was not reformed. All accessible gaps were filled with Fiberfrax. The three support studs were repaired by adding a threaded extension rod to the remaining end of the stud. Support was then reestablished as usual by placing a large washer over the stud, threading a nut over the extension and tack welding the nut to the rod to fix it in place. A small weld crack around the liner outlet was seal welded.

Cracking of Incoloy 800 HT back pulse pipes has been addressed at Karhula by inserting a tube inside of the pipe to act as a thermal barrier. This approach was investigated to resolve the cracking noted in pulse pipes at Tidd, however, sleeving was not utilized because of limited bore diameter of the APF outlet nozzles. Alternatively, the pulse pipes were remanufactured using a more fatigue resistant material - Haynes Alloy 230.

Essential thermocouples and pressure taps in the vessel head were replaced with new material. Where compression fittings were used to connect pressure tubing, the bore of the tube was reamed at its ends to clean out swaged material to the nominal tube inside diameter. All new thermocouples installed were clad of Inconel 600 sheath. This material may provide improved resistance to corrosion and embrittlement and may extend the life of the thermocouple leads.

Unreliable performance of back pulse valves has been attributed to galling. Parallel path approaches were initiated to resolve this issue. These actions are described in the memo in Appendix A. The nearest term approach that has been demonstrated to resist galling involves weld overlay cladding of Stellite 6 on wear surfaces of valve pistons and electroplating of nickel-boron on wear surfaces of valve bodies. At this time all 7 (3 primary, 3 backup and 1 spare) pulse valves at Tidd have been reworked with Stellite and nickel-boron.

Continuing concern regarding ash discharge from the APF vessel has prompted additional actions to enhance ash flow. They include:

- (1) Relocation of the liner vibrator from inside the manway to a mount on the outside of the manway flange. This arrangement is anticipated to improve

the reliability of air supply/exhaust hoses and to enhance the serviceability of the vibratory device.

- (2) Addition of an "air cannon" for nozzle 2. An air accumulator tank has been installed to provide a sudden blast of air in the APF hopper if discharge problems are encountered.
- (3) Addition of nine (9) new purge air nozzles. Nozzles have been added to the APF hopper to provide additional flexibility to dislodge ash by purging at various elevations and locations.

5.0 ACCELEROMETER TESTING

A series of tests have now been completed to evaluate the dynamic behavior of the Westinghouse candle cluster during a pulse cleaning event. A second test has now been performed to measure the acceleration of a bottom plenum of the APF resulting from room temperature pulse cleaning. Three accelerometers were epoxied to the B cluster bottom plenum plate. The x and y accelerometers were mounted on the side of the plate 90 degrees apart and the z accelerometer was mounted on the bottom surface of the plate as close to the center as possible (4 inches in from the edge of the plate). The plenum acceleration resulting from 12 room temperature pulses of cluster B bottom were measured with an HP 3562A Dynamic Signal Analyzer and captured to diskette. Two analyzer setups were used:

High Frequency Setup with a measurement period of 1.5 seconds and frequencies up to 5 kHz.

Low Frequency Setup with a period of 15.5 seconds and frequencies to 500 Hz.

A high frequency and low frequency measurement was taken for each accelerometer at each of the two pulse pressures (350 and 700 psig) for a total of 12 measurements.

The low frequency acceleration appears to be damped. The high frequency measurements show the highest accelerations. The highest acceleration (16 g's) occurred during a 700 psig pulse in the x direction. This plenum acceleration data will be used by Adapco Analysis & Design in a candle failure analysis.

In June, 1993 APF candle acceleration tests were performed during room temperature pulse cleaning at 350 and 700 psig. Consistent with the plenum tests described above, low frequency (0-312 Hz) measurements indicated low candle accelerations (0.7 g's maximum in the radial direction).

6.0 KARHULA PARTICLE FILTER TESTING

A 500 hour test run was completed on the Westinghouse candle filter installation at the Ahlstrom 10 MW_t pressurized fluidized bed combustion pilot plant facility at Karhula Finland. The filter unit met all performance goals while operating on a U.S. powder river coal (Black Thunder). Inspection of the 128 candle filter element unit following the test period showed the system to be in excellent condition. Several of the candle elements have been returned to STC for material characterization follow-up. Operation of the unit is scheduled to resume early February.

Characterization of candle filters exposed prior to the most recent (500 hour) test run is described in Appendix B to this report.

APPENDIX A

ATKOMATIC PULSE SOLENOID VALVE REWORK

ATKOMATIC PULSE SOLENOID VALVE REWORK

G. J. Bruck
Advanced Fossil Energy Systems
December 17, 1993

1.0 INTRODUCTION

Severe adhesive wear (galling) has been noted between pistons and valve bodies for the 2 inch Atkomatic model 35871 pulse solenoid valves at Tidd. Maintenance work has included frequent honing and hand sanding (deburring). Such extensive rework during operation is unacceptable. This brief memo documents the current status of corrective actions undertaken to improve valve performance.

2.0 CORRECTIVE ACTIONS

2.1 Unsuccessful Valve Changes

The galling was noted to be located next to flat areas on the valve pistons (and the corresponding surfaces within the valve bodies). The first action taken to correct galling was to minimize the size of these areas. New pistons were supplied by Atkomatic with flats shortened from 1.000 inch to 0.300 inch (see sketches in Appendix A.) Subsequent operation with the newly designed pistons still produced galling that required frequent maintenance.

The second action to correct galling involved Teflon coating of all wear surfaces within the valve. Atkomatic supplied Teflon coating of both pistons and valve body

bores. Subsequent testing of such coated valves proved unsuccessful. In separate tests Teflon coated cylinder with Teflon coated body and Teflon coated cylinder with uncoated body, both failed in the open position in less than 200 cycles.

A third action to correct galling involved using an Atkomatic-supplied piston made of Naval Brass. Tests with this cylinder in uncoated and Teflon coated bodies also resulted in valve failure in less than 200 cycles.

2.2 Valve Changes Under Current Test

Alternative valve changes being undertaken involve changing valve body and piston materials to improve resistance to galling. The following table summarizes the status of these part preparations and tests. Technical information on the Gall-Tough stainless steel and bronze alloy 24059 may be found in Appendix B. For the Gall-Tough and bronze pistons, two piston geometry options are being offered. The first has a 0.250 inch long flat area. The second has the same 0.250 inch flat with an additional 35° bevel. Material certificates and design details are described in Appendix C.

Piston	Body	Due Date	Test Results	Number of Units
W/Bronze Reinforced Teflon Rings	—	rec'd	TBD	2
Stellite 6 clad	NiB electroplated	rec'd	Early tests inconsistent Recent 1300 pulse test at 1000 psi OK Recent 200 pulse test at 1000 psi OK	7
Gall Tough SS Op. 1	—	12-20-93	TBD	3
Op. 2	—	12-20-93	TBD	3
Bronze Alloy 24059 Op. 1	—	12-20-93	TBD	3
Op. 2	—	12-20-93	TBD	3
—	Stellite 6 clad	1-17-94	TBD	3

APPENDIX A

SKETCHES OF ORIGINAL AND MODIFIED PISTON DESIGN

APPENDIX B

TECHNICAL INFORMATION
ON
GALL-TOUGH STAINLESS STEEL
AND
Mil-B-24059 Aluminum Bronze

2

Gentlemen,

Please find attached technical information on the materials we are proposing to use and comparable information on the materials we have tried previously.

"Gall-Tough" is the trade name of a Carpenter stainless steel specifically formulated to resist galling. It contains high-levels of manganese and silicon, and it has been nitrogen strengthened. The testing indicates it is substantially resistant to galling. I have also included some background information on galling and some of Carpenter's test results.

I have included some information on standard 316L stainless steel for comparison with the above.

Please find attached specifications for aluminum bronze C63200, MIL-B-24059, this material also has a high-manganese content to improve its galling characteristics. It also has a much higher tensile strength than the brass we tried previously. Atkomatic has had success in the past using this material to relieve galling problems.

I have included some information on standard Leaded Naval Brass for comparison with the above.

(PROPOSED
MATERIAL)

3

Gall-Tough® Stainless

U.S. Patent No. 4,814,140

(S20161)

Type Analysis

Carbon 0.15% max.
Manganese 4.00/6.00%
Phosphorus 0.040% max.
Sulfur 0.040% max.
Silicon 3.00/4.00%

Chromium 15.00/18.00%
Nickel 4.00/8.00%
Nitrogen 0.08/0.20%

Description

Gall-Tough® stainless is a high-silicon, high-manganese, nitrogen strengthened, austenitic stainless alloy which possesses superior self-mated galling and metal-to-metal wear resistance.

This alloy displays higher strength and high temperature oxidation resistance than Type 304 stainless with comparable corrosion resistance depending on the environment.

Galling and Metal-to-Metal Wear Resistance

When compared to other standard stainless steels, the self-mated galling resistance and metal-to-metal wear resistance of Gall-Tough

stainless are outstanding. Optimum galling resistance is obtained when both mating components are made of Gall-Tough stainless.

Galling and Wear Test Properties—Various Alloys

Galling test involves rotating a compressively loaded 1/2" (12.7 mm) diameter button against a block counterclockwise 360°, clockwise 360°, and counterclockwise 360° and determining the highest stress at which visible galling damage does not occur. Metal-to-metal wear tests were conducted for 40,000 cycles using crossed metal cylinders per ASTM G83.

Alloy	Galling Test		Wear Test	
	Threshold Galling Stress		Avg. Total Volume Loss, mm ³	
	ksi	MPa	100 rpm	400 rpm
Gall-Tough Stainless	15*	103*	5	4
Type 440C (HRC 56)	2	14	9.5	5.5
Type 304	< 1**	7**	29	25
Type 430	< 1**	7**	230	172

*Testing at higher stress not performed

**Galled at lowest stress evaluated

Applications

Gall-Tough stainless may be considered for applications in which parts are in relative motion under load without lubricants.

Potential applications include:

- Chain link conveyor belts and other components in food and drug processing industries
- Bolts, nuts and other fasteners
- Fittings for pumps and valves
- Luggage carousels in air terminals

(4)

Corrosion Resistance

Gall-Tough stainless is resistant to atmospheric corrosion. Its resistance to some acids and corrosive environments is comparable to that of Type 304 stainless and superior to that of Type 430 stainless.

Intergranular corrosion resistance may be impaired if the material is

heated between 800/1650°F (427/899°C) or cooled slowly through that range.

For increased corrosion resistance from this alloy, surfaces must be free of scale and foreign particles and finished parts should be passivated.

Typical Corrosion Properties—Gall-Tough Stainless Annealed condition

Environment	Time of Test	Average Corrosion Rate (mpy)		
		Gall-Tough Stainless	Type 304	Type 430
5 w/o H ₂ SO ₄ —RT	1-48h period	18	12	1300
50 w/o acetic acid—boiling	"	0	0	8
1 w/o HCl—RT	"	34	7	248
5 w/o formic acid—178°F (80°C)	"	41	0	694
65 w/o HNO ₃ —boiling	5-48h periods	82	59	46
5 w/o NaCl fog—95°F (35°C)	200 h	10-40% rusted	occasional rust spots	5-10% rusted

Scaling

The safe scaling temperature for continuous service is 1800°F (982°C).

Physical Properties

Specific gravity..... 7.65

Density
lb/in³..... 0.276
kg/m³..... 7650

Mean specific heat
Btu/lb • °F (126-216°F)..... 0.123
J/kg • K (52-102°C)..... 514.5

Mean coefficient of thermal expansion

Test Temperature		10 ⁻⁴ /°F	10 ⁻⁴ /K
77°F to	25°C to		
212	100	9.60	17.28
302	150	9.66	17.38
392	200	9.74	17.54
482	250	9.85	17.73
672	300	9.93	17.87
682	350	10.01	18.02
752	400	10.13	18.24
842	450	10.23	18.41
932	500	10.29	18.53
1022	550	10.36	18.65
1112	600	10.46	18.83
1202	650	10.54	18.98
1292	700	10.63	19.14

Thermal conductivity

Test Temperature		Btu-in/(ft ² •h•°F)	W/m•K
°F	°C		
122	50	84.72	12.22
212	100	93.58	13.49
392	200	100.25	15.33
672	300	118.10	17.03
752	400	128.93	19.60

Modulus of elasticity (E)

ksi..... 24.0 × 10³
MPa..... 171.1 × 10³

5

Gall-Tough® Stainless

Heat Treatment

Annealing
Heat to 1900/2000°F (1038/1093°C) and quench in water. Typically, hardness as annealed is Rockwell B95.

Hardening
Gall-Tough stainless cannot be hardened by heat treatment; however,

this material can be hardened through cold work.

Workability

Hot Working

Gall-Tough stainless can be readily forged, hot rolled, hot headed and upset.

For hot working, heat uniformly to 2100/2300°F (1149/1260°C). Preheating to an intermediate temperature is not required. Do not forge below 1700°F (927°C). Forgings can be air cooled without danger of cracking.

For maximum corrosion resistance, annealing after hot working is required.

Cold Working

Gall-Tough stainless is readily cold worked by conventional methods.

Cold working causes martensitic transformation in Gall-Tough stainless, resulting in a significant increase in magnetic permeability.

Because of its metastability, Gall-Tough stainless is characterized by a high tensile strength in both annealed and cold worked conditions.

Machinability

Gall-Tough stainless machines at about 50% of the rates used for Type 304 stainless. Its machinability is similar to that of other nitrogen-strengthened alloys, like 22Cr-13Ni-5Mn stainless. A rigid setup, heavy positive feeds, and ample coolant flow are recommended.

Welding

Gall-Tough stainless can be satisfactorily welded by the shielded fusion and resistance welding processes. Oxyacetylene welding is not recommended since carbon pickup in the weld may occur.

When a filler metal is required, consider AWS E/ER240 welding consumables which should provide welds with strength approaching that of the base metal. If high weld strength is not necessary, then consider E/ER308.

Resistance to intergranular corrosion can be restored by a postweld annealing treatment.

Typical Mechanical Properties

Typical Room Temperature Mechanical Properties—Gall-Tough Stainless

Annealed bar and wire

Section Size	0.2% Yield Strength		Ultimate Tensile Strength		% Elongation in 4D	% Reduction of Area	Rockwell B Hardness
	ksi	MPa	ksi	MPa			
1" (25.4 mm) rd.	53	365	145	1000	59	64	93
250" (6.35 mm) rd.	66	455	156	1076	70	73	93

Typical Elevated Temperature Tensile Properties—Gall-Tough Stainless

Annealed 1" (25.4 mm) diameter bar

Test Temperature		0.2% Yield Strength		Ultimate Tensile Strength		% Elongation in 4D	% Reduction of Area
°F	°C	ksi	MPa	ksi	MPa		
Room Temp		53	365	145	1000	59	64
400	254	30	207	81	559	63	78
800	427	27	188	77	531	57	78
1200	649	25	172	59	407	58	70
1600	871	19	131	20	135	109	90

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content of the stainless steels tends to reduce their corrosion resistance.

Resistance Welding

In the resistance welding methods, the heat required for bonding is provided by the electrical resistance offered by the stainless steel parts to the short-time flow of a high electric current. Force (mechanical pressure) is applied before, during and after the application of current. The thermal and electrical conductivity of the parts, the electrode voltage and current, cleanness of the surfaces to be joined, force, and holding time are all important factors to be considered.

The austenitic stainless steels are easily joined by resistance welding methods. The very high electrical resistance of these grades allows the use of lower currents and shorter times than those necessary for mild steel. Distortion and intergranular carbide precipitation can be minimized by limiting the current flow to the minimum time necessary to produce a sound joint. Ferritic stainless steels can be welded by resistance methods, but welding time should be kept to a minimum to avoid grain coarsening and embrittlement. Martensitic stainless steels can also be resistance welded, but hardening does

occur in the weld zone and the heat-affected zone and this can lead to problems with cracking. Postweld heat treatments are necessary to restore ductility.

Resistance Welding Methods

Spot and Projection Welding: This process is limited to strip and sheet thicknesses using a lap-type joint. Fusion is accomplished by applying a high current to the work under pressure between suitable electrodes. The size and shape of the individual welds are controlled by the size and shape of the electrodes. Projection welds are localized by the predetermined design of one of the mating parts which contains small projections or embossments. Automatic control is usually employed with spot or projection welding. It is very important that the surfaces to be joined be free from dirt, grease or other contamination. The welding pressure should be as uniform as possible, since pressure variations affect both current density and electrical resistance.

Seam Welding: This process is the same as spot welding, except that the parts to be joined are held under pressure between circular electrodes. This results

in a series of overlapping spot welds made progressively along a joint by rotating the electrodes. Warping and carbide precipitation in austenitic stainless steel can be avoided by submerging the weld in water or by directing a continuous flow of water upon the weld immediately after each seam is welded. The ferritic and martensitic grades are not usually so welded.

Butt Welding and Flash Welding: Butt welding the ends of bars or wire to be joined are butted together by clamps which also serve as electrodes. Pressure is applied before the current is turned on and is maintained as melting occurs at the joining faces. Flash welding differs from butt welding in that the surfaces to be joined touch very lightly initially. When the surfaces become molten due to heat produced by arcing between the electrodes, pressure is applied to complete the fusion process. With these methods the surfaces to be joined should be fairly smooth and clean, and proper alignment is absolutely essential.

For further information on welding, consult the American Iron and Steel Institute, 11 16th Street N.W., Washington, DC 20036, request the publication entitled "Welding Stainless Steels and Other Joining Methods."

(SOME BACKGROUND & TEST RESULTS) Galling and Carpenter Stainless Steel

Adhesive wear results from two metals being rubbed together under a load sufficient to break through the oxide film allowing the mating surfaces to come into contact at the high points. When the cohesive force between the two metals exceeds the strength of either metal, adhesion or cold welding occurs. Under low stress, this adhesion usually results in a complex process which wears away one or both of the mating surfaces at a slow rate. At higher stresses, cold welding occurs more rapidly and over a greater area of the mating surfaces. This higher degree of cold welding is referred to as galling, and it may cause equipment to seize or freeze up. If not dealt with at the outset, galling can be a worrisome and recurring problem, particularly in the application of stainless steels.

Prevention

The probability of galling occurring between two metals can be minimized or prevented via 1) control of surface roughness, 2) use of lubricants, 3) decreasing the contact load, and 4) alloy selection. Of these, contact load may be the least subject to manipulation but, nevertheless, must be controlled to the extent that excessive loads are avoided. Control of surface roughness, coupled with the use of a high-quality lubricant such as a moly-disulfide reinforced grease, is all that may be required in many applications to prevent galling. The ideal surface is one that is free of machining burrs, fins and tears and is not overly rough or overly smooth. For most applications, a surface roughness in the range of 15 to 50 microinches (rms) is best.

Alloy Selection

Alloy selection is a useful tool within the family of stainless steels, a relatively recent approach to the control of galling. Most stainless steels are more susceptible to galling than carbon and low alloy steels. However, not all stainless steels are equal in this respect and, in fact, some are quite resistant to galling, that is, they have a high threshold galling stress (TGS).

Threshold galling stress as discussed here is determined via a laboratory test and is the stress required to produce galling when a 1/2" (12.7 mm) diameter specimen is rotated against a flat plate with lubrication. A single revolution of the specimen is normally used. However, when evaluating galling-resistant alloys, it

as Gall-Tough® stainless, a procedure with three alternating revolutions is used to simulate more severe service. The button and plate may be the same or different alloys. Because of the many possible extraneous factors such as lubrication, temperature, roughness and others, TGS is not necessarily the lowest or, conversely, the highest stress which will produce galling in actual practice. Nevertheless, TGS has proved to be a valuable guide in selecting stainless steels with increased resistance to galling.

Threshold Galling Stress for Carpenter Alloys

To provide the user and engineer with data which may be useful when selecting alloys for applications where galling could occur, Carpenter's research laboratory has determined the threshold galling stress via the above procedure for many stainless steels. For some alloys, TGS was determined in two ways. In the first,

both button and base plate were the same alloy while in the second, base plate and button were of two different alloys or conditions. Results from the various test are shown in Tables I, II, and III. Note that Tables III and II present results for single-rotation tests, while Table I presents results for triple-rotation tests.

Large differences in TGS are seen among the various alloys and, of course, as the TGS increases, so does the resistance to galling. Differences less than 2 to 4 ksi (14 to 28 MPa) between alloys, however, are not considered to be significant for the single-rotation test.

Some Pertinent Observations Based on Our Data

Contrary to popular belief, cold working does not increase the resistance of an austenitic stainless steel to galling and, in fact, can be deleterious. On the other hand, increasing hardness via heat treat-

ing is generally beneficial. Martensitic stainless steels such as Type 410 and Type 416 in the annealed or tempered conditions are similar or less resistant to galling than annealed austenitic stainless steels such as Type 304 and 316.

The addition of free-machining additives to both the 300 and 400 series stainless steels and the restriction of nickel in the nitrogen-strengthened alloys increase resistance to galling.

The TGS for a combination of two different alloys frequently lies between the values for the individual alloys in a self-mated condition.

Gall-Tough stainless has the highest TGS of any stainless steel produced by Carpenter and should be considered for those applications now using alloys such as Types 302, 304, 316 and 22Cr-13Ni-5Mn where galling has been a problem, or is likely to be a problem.

Table I
Triple-Rotation
Threshold Galling Stress Results for Various Stainless Steels
Self-Mated, Unlubricated Ground Finish

Alloy	Condition	Rockwell Hardness	Threshold Galling Stress ksi / MPa	Alloy	Condition	Rockwell Hardness	Threshold Galling Stress ksi / MPa
Gall-Tough*	Mill Annealed	B 92	15* 103*	Type 304	Annealed	B 76	<1* <1**
Gall-Tough	Cold Drawn	C 28	15* 103*	18-18 PLUS	Annealed	B 98	1 7
Gall-Tough	Cold Drawn	C 38	15* 103*	18Cr-2Ni-12Mn	Annealed	B 95	2 14
Type 440C	Tempered 400°F	C 58	2 14	Type 430	Annealed	B 77	<1** <1**
Custom 455	H950	C 46	<1* <7**				

* Testing at higher stress not performed

** Galled at lowest stress evaluated

(STANDARD)
316

Project 70® Stainless Type 316L

(UNS S31603)

Type Analysis

Carbon 0.03% max.
Manganese 2.00% max.
Phosphorus 0.045% max.
Sulfur 0.030% max.
Silicon 1.00% max.

Chromium 16.00/18.00%
Nickel 10.00/14.00%
Molybdenum 2.00/3.00%
Iron Balance

Description

Project 70® Stainless Type 316L is an improved machining version of conventional Type 316L. Users report this grade has offered significantly improved machinability characteristics, including faster machining speeds, longer tool life and improved finishes.

In this low-carbon austenitic alloy, control of carbon to a maximum of 0.03% has been shown to minimize

carbide precipitation during welding. Customers report the use of this steel in the as-welded condition in a variety of corrosive applications.

Containing molybdenum with increased percentages of nickel, this alloy displays higher tensile and creep strengths at elevated temperatures and better corrosion resistance than the conventional 18-8 alloys.

Applications

Project 70 Stainless Type 316L has found application in paper pulp handling equipment, process equipment for producing photographic chemicals, ink,

rayon, rubber, textile bleaches and dyestuffs as well as various high temperature equipment applications.

Corrosion Resistance

Project 70 Stainless Type 316L has been used in sulfate pulp mills to resist corrosion by sulfurous acid compounds. Due to its superior corrosion resistance, its use has been extended to handling many of the chemicals used by chemical process industries.

This alloy is more resistant to pitting than conventional 18-8 alloys.

For optimum corrosion resistance, surfaces must be free of scale and foreign particles. Finished parts should be passivated.

Scaling

The safe scaling temperature for continuous service is 1600°F (871°C).

Physical Properties

Specific gravity 7.95
Density
lb/cu in 0.287
kg/cu m 7950
Mean specific heat
Btu/lb·°F (32/212°F) 0.12
J/kg·K (0/100°C) 500

Electrical resistivity (RT)
ohms c/ft 445
microhm-mm 740
Mean coefficient of thermal expansion
10⁻⁶/°F (32/1200°F) 10.3
10⁻⁶/K (0/649°C) 18.5

Heat Treatment

Annealing
Heat to 1850/2050°F (1010/1121°C)
and water quench. Brinell hardness
approximately 150.

Hardening
Cannot be hardened by heat treatment.
Hardens only by cold working.

15/93

14:36

MILLARD METAL

NO. 022

002

9

Aluminum Bronzes (PROPOSED MATERIAL)

ALLOY/ UNS NUMBER	NOMINAL CHEMICAL COMPOSITION					MECHANICAL PROPERTIES									
	Copper	Aluminum	Iron	Nickel	Other (Max.)	Tensile Strength (KSI)		Yield Strength (KSI)		Elonga- tion (%)		Hardness BHN 3000 Kg.		Density Lb/Cu. In.	
						Min.	Typ.	Min.	Typ.	Min.	Typ.	Min.	Typ.		
AMPCO C-3 C86400	86	10.8	4.0	2.8 (Max.)	0.6Mn/ 0.5	75	82	30	34	12	14	143	174	0.269	
AMPCO C-3HT C86400HT	86	10.5	4.0	2.5 (Max.)	0.5Mn/ 0.6	90	96	45	47	6	10	180	192	0.269	
AMPCO D-4 C86500	78.8	10	5	5	0.6	90	96	40	43	10	12	170	187	0.270	
AMPCO D-4HT C86500HT	79.8	10	5	5	0.6	110	116	60	66	10	14	216	228	0.270	
AMPCO M-4+ C86820	77.6	11	4.8	6.1	1.0Mn/ 0.6	125	130	85	106 (Al 0.2% offset)	2	4	265	269	0.289	
AMPCO M-4+ C86820	77.6	11	4.8	6.1	1.0 Mn/ 0.6	135	148	100	116 (Al 0.2% offset)	6	8	282	284	0.289	
AMPCO 48 C86300	80.0	10.0	3.8	5.0	1.0Mn/ 0.6	110	118	66	78	10	18	202	228	0.272	
AMPCO 483 C86800	81	8	4.0	4.5	1.0Mn/ 0.5	85	92	35	39	16	24	159	174	0.276	
AMPCO 483 C863200	81	9	4.0	4.5	1.0Mn/ 0.6	86	105	50	53	18	22		212	0.276	
AMPCO 485 C86700	78	8.0	2.5	2.0	12.5Mn/ 0.5	92	96	42	48	22	28	160	180	0.273	
AMPCO 642 C84200	90.8	7			2.0 Si/ 0.5	88	90	44	47 (Al 0.2% offset)	15	30	130 (1000 Ksi)	166	0.278	

MIL-B-24059

Manganese Bronzes

Manganese Bronzes														
ALLOY/ UNS NUMBER	Copper	Aluminum	Iron	Nickel	Other (Max.)	Tensile Strength (KSI)		Yield Strength (KSI)		Elonga- tion (%)		Hardness BHN 3000 Kg.		Density Lb/Cu. In.
						Min.	Typ.	Min.	Typ.	Min.	Typ.	Min.	Typ.	
AMPCO 62 C86800	58.0	1.0	1.0		0.3 Mn 38.5 Zn	66	72	25	28	25	35	112	131	0.296
AMPCO 64 C86200	64.0	4.0	3.0		3.5 Mn 26.6 Zn	90	95	46	48	20	26	170	192	0.296
AMPCO 64 C86300	62.0	6.0	3.0		3.5 Mn 26.6 Zn	110	116	60	65	12	16	192	223	0.296
A-16														

Aluminum Bronzes (10)

SPECIFICATIONS						FORMS AVAILABLE				
ASTM	ASME	FEDERAL	MILITARY	SAE	AMS	Sheet	Cast	Extruded	Forged	Plate
B-148 C96400 B-271 C96400 B-506 C96400	BB-148 C96400	QQ-C-390 C954		J-482 C96400	4635	.	.	.	.	
B-148 C96400HT B-271 C96400HT B-506 C96400HT	BB-148HT C96400HT	QQ-C-390 C-954HT	C-16346 Alloy 13	J-482 C-96400HT	4636	.	.	.	.	
B-148 C96500 B-271 C96500 B-506 C96500		QQ-C-390 C965	C-16346 Alloy 18 C-22229 Alloy 988	J-482 C-96500		.	.			
B-148 C96600HT B-271 C96600HT B-506 C96600HT		QQ-C-390 C966HT	C-16346 Alloy 14 C-22229 Alloy 988HT	J-482 C-96600HT		.	.			
B-148 C96620 B-271 C96620 B-506 C96620					4661	.	.			
B-150 C63020 B-316 C63020					4590			.	.	
B-150 C63000 B-124 C63000	BB-150 C63000	QQ-C-465 C630	B-16166 C630	J-463 C-63000	4640 46608		.	.	.	
B-148 C96800 B-271 C96800 B-506 C96800		QQ-C-390 C968	C-16346 Alloy 24 B-84480 B-21230 Alloy 1 C-22229 Alloy 988	J-482b C-96800		.	.		.	.
B-150 C63200		QQ-C-465 C63200	B-24066					.	.	
B-148 C95700			B-21230 Alloy 2			.	.	.	.	
B-150 C64200		QQ-C-466			4631C			.		

Manganese Bronzes

ASTM	FEDERAL	MILITARY	SAE	AMS	Sheet	Cast	Extruded	Forged
B-147 C86500 B-271 C86500	B-505 C86500	QQ-C-390 C865	C-15346 Alloy 4 C-22229 Alloy 866	J-462 C-86500	4660 4619	.	.	.
B-147 C86200 B-271 C86200	B-506 C86200 B-584 C86200	QQ-C-390 C862	C-22229 Alloy 862	J-462 C-86200		.	.	
B-22 C86300 B-271 C86300 B-147 C86300	B-505 C86300 B-584 C86300	QQ-C-390 C863	C-15346 Alloy 6 C-22229 Alloy 863	J-462 C-86300 A-17	4662	.	.	

USED
PREVIOUSLY

Copper and Its Alloys — Wrought

C44200 (Brass)	Type and CDA No.	C44300, C44400, C44500* (Admiralty)	C44400, C44500, C44600, C44700* (Naval Brass)	C44500 (Leaded Naval Brass)	C50600 (Phosphor Brass E)	C51000 (Phosphor Brass A)	C52100 (Phosphor Brass C)
87-95, Zn 12, Sn 1-2	NOMINAL COMPOSITION, %	Cu 71, Zn 28, Sn 1	Cu 60, Zn 23.7, Sn 0.75	Cu 60, Zn 37.5, Pb 1.75, Sn 0.70	Cu 88, 7, Sn 1.3 P trace	Cu 94.8, Sn 5 P 0.02	Cu 92, Sn 8
117-0.319 150-1940 89-95	PHYSICAL PROPERTIES						
	Density (lb/in ³)	0.308	0.304	0.305	0.321	0.320	0.318
	Melting Temperature Range (°F)	1850-1720	1830-1850	1830-1850	1900-1970	1750-1920	1820-1880
	Thermal Conductivity, 68°F (Btu-in/hr-ft ² -F)	64	87	87	120	40	36
	Coefficient of Thermal Expansion, 68-872°F (10 ⁻⁶ in/in/F)	11.2	11.8	11.8	9.9	9.9	10.1
10.2 0.09	Specific Heat, 68°F (Btu/lb-F)	0.09	0.09	0.09	0.09	0.09	0.09
28-41	Electrical Conductivity (% IACS)	25	26	28	48	15	13
18	MECHANICAL PROPERTIES*						
	Tension Modulus (10 ⁶ psi)	18	15	15	17	16	16
34-45	Tensile Strength (10 ⁴ psi)	53.55	57	57 75	40, —	47.50	55.80
62-63	Half Hard	—	75	75	55, —	68.85	78, 105
64-76	Hard	—	80	80	65, 79	81, 110	93, 130
74-89	Spring	—	—	—	—	100, 140	112, —
	Extra Spring	—	—	—	—	107, —	120, —
12-18	Yield Strength (10 ⁴ psi)						
43-51	Annealed	22	25	25	14	19, 20	24
63-73	Half Hard	—	53	53	—	55, 80	55
74-87	Hard	—	—	—	50	75	72
48-49	Elongation in 2 in. (%)	65, 80	47	40	48	64, 54	70, 85
12-20	Annealed	—	20	15	18	28, 8	32
4-10	Hard	—	—	—	4	10	10
2-4	Spring	—	—	—	4	4	3
	Extra Spring	—	—	—	—	3	2
F55-70	Hardness (Rockwell)						
860-76	Annealed	F75	B55	B55	—	B28	F75
872-84	Half Hard	—	B82	B82	B64	B78	B84
879-82	Hard	—	—	—	B75	B87	B92
	Spring	—	—	—	B79	B95	B98
31	Extra Spring	—	—	—	—	B97	B100
37	WORKING PROPERTIES						
29	Formability	Excellent	Fair	Poor	Excellent	Excellent	Good
	Formability	Fair	Excellent	Good	Good	Poor	Poor
Excellent	Soldering Temp (°F)	800-1100	800-1100	800-1100	900-1200	900-1250	900-1250
Good 14	Solderability Index*	30	30	70	20	20	20
1468-1858	Soldering	Excellent	Excellent	Excellent	Excellent	Excellent	Excellent
800-1100	Brazing	Excellent	Excellent	Good	Excellent	Excellent	Excellent
20	Oxyacetylene Welding	Good	Good	Not Rec	Fair	Fair	Fair
Excellent	Butt Resistance Welding	Good	Good	Fair	Excellent	Excellent	Excellent
Excellent	Corrosion Resistance	Good resistance to industrial, rural and marine atmospheres; petroleum products, alcohols, dry gases and sea water. Fairly good resistance to weak bases. Resistant to some weak bases and weak organic acids, but generally poor resistance to moist acids and strong bases. Poor resistance to solutions of cyanides and ammonium compounds. Resistant to dezincification.			Generally good resistance to atmospheric, water, salt water, and salt solutions. Some resistance to alkaline solutions and inorganic acids. Poor resistance to organic acids, cyanides, and ferric and ammonium compounds.		
Good	Usable Forms	Plate, wire, tube	Strip, bar, plate, rod, tube shapes	Bar, rod, shapes	Strip, wire	Strip, rod, wire, tube	Strip, rod, wire
Good	Typical Products	Condenser, evaporator and heat exchanger tubes; condenser tube plates; distiller tubes	Fasteners, marine hardware, propeller shafts, valve stems, condenser plates	Marine hardware, screw machine products, valve stems	Electrical contacts, flexible hose, pole line hardware	Bridge bearing plates, chemical hardware, perforated sheets, textile mach., weld rods, boiler bars, cotter pins, diaphragms, fuse clips, fasteners, washers, bushings, springs, switch parts, truck wire	

*Free-cutting brass is 100. This alloy is usually inhibited with either As, Sb or P in the range of 0.02 to 0.10%. Inhibited alloys are identified by C44300, C44400, C44500. When required this alloy may be inhibited with either As, Sb or P in the range of 0.02 to 0.10%. Inhibited alloys are identified respectively by C44300, C44400, C44500.

APPENDIX C

**MATERIAL CERTIFICATES AND DESIGN OPTION DETAILS
FOR GALL-TOUGH AND BRONZE PISTONS**

CERTIFICATE OF TESTS

FORM 7184-33
(2 of 2)

CARPENTER TECHNOLOGY CORPORATION PAGE NUMBER: 1

P.O. BOX 14662
READING, PA. 19612-4662

• THE RECORDING OF FALSE, FICTITIOUS OR FRAUDULENT STATEMENTS OR ENTRIES ON THIS DOCUMENT MAY BE PUNISHED AS A FELONY UNDER FEDERAL STATUTES INCLUDING FEDERAL LAW, TITLE 18, CHAPTER 47.
• THE VALUES AND OTHER TECHNICAL DATA SHOWN REPRESENT THE RESULTS OF ANALYSES AND TESTS MADE ON SAMPLES COLLECTED FROM THE TOTAL LOT. ORIGINAL DATA RECORDS CAN BE TRACED BY REFERENCE TO THE CARPENTER ORDER NUMBER.
• THIS MATERIAL MELTED AND MANUFACTURED IN THE U.S.A.
• MATERIAL IS MANUFACTURED FREE FROM MERCURY, RADIUM AND ALPHA SOURCE CONTAMINATION

DATE 11/20/93

ADDRESS REPLY TO:

ATKOMATIC VALVE CO INC
PO BOX 11258
INDIANAPOLIS, IN 46201

CUSTOMER ORDER NUMBER	CARPENTER ORDER NUMBER	DATE SHIPPED	WEIGHT
9396	RDG7756 L13281		143

HEAT NO.-501174

PRODUCT DESCRIPTION: GALL-TOUGH STAINLESS ANNEALED ROUGH TURNED

SIZE 4.000000 IN. RD BAR

C 0.097	MN 5.47	SI 3.49	P 0.022	S 0.022	CR 16.27
NI 5.26	MO 0.35				

THIS MATERIAL WAS MANUFACTURED IN ACCORDANCE WITH CARPENTER STEEL DIVISION QUALITY PROGRAM MANUAL REVISION 17, DATED 11/91.

HARDNESS AS SHIPPED, HB - 207

I CERTIFY THE ABOVE INFORMATION TO BE A TRUE AND CORRECT RESTATEMENT OF PORTIONS OF THE APPLICABLE TEST AND ORDER DATA.

PART# B335#R
DATE REC'D 11-24-93
P.C. # 9396
R & I # _____
FOR V- _____

ROBERT E. CARNES
SUPERVISOR - METALLURGICAL LAB
CARPENTER TECHNOLOGY CORPORATION

MILLARD UTILITY METAL
Service Center of New Jersey

TO ATKINS & CO. VALVE
ATTN Q.C.
P.O. NO 9377
OUR REF. # 2/2331
ITEM NO 6
QUANTITY 40 LBS.
SIGNATURE Ross Carter
DATE 11/23/93

MATERIAL CERTIFICATION

DATE: NOVEMBER 17, 1993

SOLD TO: MILLARD UTILITY
110 ASIA PLACE
CHRISTADT, NJ 07072

SHIP TO: SAME

CUSTOMER P.O.: N95282N

PACKING SLIP: 3631

MATERIAL: NICKEL ALUMINUM BRONZE ANNEALED MIL-B-24059 AND 38-D

SIZE: 3.500" DIAMETER X 29.4375" LG 80#

HEAT NUMBER: B4434

CHEMICAL PROPERTIES

CU	81.07	NI/CO	4.53
FE	3.80	PB	<0.01
MN	1.36	SI	.01
AL	9.21		

MECHANICAL PROPERTIES

YIELD: 46,847/52,214
TENSILE: 100,076/101,962
ELONG%: 34.0%/30.0%

MATERIAL FREE FROM MERCURY CONTAMINATION

WE CERTIFY THAT THE CHEMICAL ANALYSIS AND
PHYSICAL TEST RESULTS APPLYING ON THE ABOVE
ORDER NUMBER ARE CORRECT AND TRUE.

SIGNATURE: Gardner E. Smith

DATE: 11-18-93

GARDNER E. SMITH, Q.C. MANAGER

PART: B33B%R
DATE REC'D 11-29-93
P.O. # 9397
R & I #
FOR V-

UNLESS OTHERWISE SPECIFIED DIMENSIONS ARE IN INCHES AND TOLERANCES ARE:				MATERIAL: "GALL-TOUGH" STAINLESS STEEL			
				ASTM NUMBER:			
TWO PLACE .XX ±.010		THREE PLACE .XXX ±.005	ANGLES ±1°	FRACTIONS ±.015	☐ SEE FORM PG7171 FOR REQUIRED CERTIFICATIONS ☐ NO CERTIFICATIONS REQUIRED		
REVISIONS					ATKOMATIC VALVE COMPANY, INC. 141 SOUTH SHERMAN DRIVE INDIANAPOLIS, INDIANA, U.S.A.		
LTR.	DESCRIPTION			DATE		APP'D.	
A	CREATED ON AUTOCAD, ECN# 1667			11-15-93		CAP	
A-22					TITLE: 2" PISTON FOR 35871-879		
					DWN BY C.A.P.	DATE 11-18-93	CHNG. NO. 35871-879-1C
					CHNG. BY R.W.L.	DATE 11-17-93	
					SCALE FULL		SHEET 1 OF 1

A-23

SECTION A - A

UNLESS OTHERWISE SPECIFIED DIMENSIONS
ARE IN INCHES AND TOLERANCES ARE:

TWO PLACE DECIMALS
.XX ±.010

THREE PLACE
.XXX ±.005

ANGLES
±1°

FRACTIONS
±.015

MATERIAL: "GALL-TOUGH" STAINLESS STEEL

ASTM NUMBER:

☐ SEE FORM P07171 FOR REQUIRED CERTIFICATIONS

☐ NO CERTIFICATIONS REQUIRED

REVISIONS

LTR.	DESCRIPTION	DATE	APP'D.
A	CREATED ON AUTOCAD, ECHN 1685	12-1-93	CAP

ATKOMATIC VALVE COMPANY, INC.
141 SOUTH SHERMAN DRIVE
INDIANAPOLIS, INDIANA, U.S.A.

TITLE:

2" PISTON FOR
35871-879

DRN. BY	C.A.P.	DATE	12-1-93	DRG. NO.
CHKD. BY	R.W.L.	DATE	12-1-93	35871-879-1F
SCALE	FULL	SHEET	2	OF 2

A-24

A-26

SECTION A - A

UNLESS OTHERWISE SPECIFIED DIMENSIONS ARE IN INCHES AND TOLERANCES ARE:				MATERIAL: MIL-B-24050 BRONZE	
DECIMALS				ASTM NUMBER:	
TWO PLACE .XX ±.010	THREE PLACE .XXX ±.005	ANGLES ±1'	FRACTIONS ±.015	☐ SEE FORM P07171 FOR REQUIRED CERTIFICATIONS ☐ NO CERTIFICATIONS REQUIRED	
REVISIONS					
LTR.	DESCRIPTION	DATE	APP'D.	ATKOMATIC VALVE COMPANY, INC. 141 SOUTH SHERMAN DRIVE INDIANAPOLIS, INDIANA, U.S.A.	
A	CREATED ON AUTOCAD ECN# 1685	12-1-93	CAP		
				TITLE:	
				2" PISTON FOR 35871-879	
				DRN. BY	DATE
				C.A.P.	12-1-93
				CHKD. BY	DATE
				R.W.L.	12-1-93
				SCALE	SHEET
				FULL	2 OF 2

APPENDIX B

Characterization of the Clay Bonded Silicon Carbide Candle Filters after 227 Hours of CFBC Operation at the Karhula/Ahlstrom Facility

CHARACTERIZATION OF THE CLAY BONDED SILICON CARBIDE CANDLE FILTERS AFTER 227 HOURS OF CFBC OPERATION

M. A. Alvin
January 4, 1994

Introduction

Twenty-one clay bonded silicon carbide (seven Schumacher Dia Schumalith F40 and fourteen Pall Vitropore 442T filters), and ninety-one alumina/mullite candles were utilized in the circulating fluidized-bed combustion (CFBC) test facility at Karhula, Finland during testing between Weeks 18-20 and Weeks 21-23. Figure 1 illustrates the candle filter identification numbers and their location within the top, middle, and bottom plenums or cluster arrays in the Westinghouse Advanced Particulate (APF) system. Note that the inner ring of candle filters in the top cluster were removed (i.e., blanked off) during this segment of testing, leaving only the outer candle filters available for use.

During the Week 18-23 test period, the Westinghouse APF system was operated for a period of 227 hours at temperatures of 900°C with fines generated from Kentucky and Newland coal and Wilbur dolomite and Tsukumi limestone. Testing of the twenty-one, as-manufactured, clay bonded silicon carbide candles was initiated in this test segment. The ninety-one alumina/mullite candles had previously been exposed to the CFBC gas environment for a period of 489 hours, and therefore at test termination during Week 23, the alumina/mullite filters had acquired a total of 716 hours of operation at CFBC process conditions.

Although all twenty-one of the clay bonded silicon carbide candle filters remained intact during the 227 hours of CFBC operation, ten of the alumina/mullite candles which were positioned in the bottom plenum of the Westinghouse APF experienced failure. Failure was attributed to the shifting of the bottom shroud contour, and subsequent ash bridging between candles and the shroud in this area of the

Figure 1a - Candle Filter Identification And Location Within The Westinghouse Advance Particulate Filtration System In Karhula, Finland (Weeks 18-20 and Weeks 21-23)

KARHULA MIDDLE PLENUM

(WEEK 18-20,21-23. APRIL 1993)

D.435.MAAMI

Figure 1b - Candle Filter Identification And Location Within The Westinghouse Advance Particulate Filtration System In Karhula, Finland (Weeks 18-20 and Weeks 21-23)

SURVEILLANCE LOCATION

- ⑨ B-1
- ⑩ B-16
- ⑪ B-19
- ⑫ B-35
- ⑬ B-36
- ⑭ B-8
- ⑮ B-12

KARHULA BOTTOM PLENUM

(WEEK 18-20.21-23. APRIL 1993)

D.435.MAAB1

Figure 1c - Candle Filter Identification And Location Within The Westinghouse Advance Particulate Filtration System In Karhula, Finland (Weeks 18-20 and Weeks 21-23)

Westinghouse APF vessel. Since a breach or failure of the ten alumina/mullite filters had occurred, ash fines were permitted to enter the clean side of the APF, and subsequently be redistributed along the remaining ID surfaces of the intact, as well as fractured candle filters during pulse gas cleaning. As a result, dust penetration along the candle filter ID surfaces was evident during both borescope and visual inspection. Figures 2 and 3 indicate that ash fines penetrate to depths of 2-2.5 mm into the ID surfaces of either the 15 mm wall clay bonded silicon carbide Schumacher Dia Schumalith F40 candle, or the 10 mm wall clay bonded silicon carbide Pall Vitropore 442T candle.

This report focuses on the material characterization results for the clay bonded silicon carbide candle filters which were removed from the Westinghouse Advanced Particulate Filtration system after 227 hours of operation in the CFBC gas environment. Candle filters which were destructively characterized in this effort included a Schumacher Dia Schumalith F40 candle designated as S2855/374C which had been positioned in the Westinghouse APF cluster array location T12, and a Pall Vitropore 442T candle designated as R3-0061 which had been positioned in location M19. Characterization of these filters included:

- Room temperature and hot strength (900°C) analysis (i.e., C-ring compression and tension)
- Burst strength analysis and determination of Young's Modulus
- Scanning electron microscopy/energy dispersive x-ray analysis (SEM/EDAX)
- Gravimetric leaching and qualitative x-ray analysis (XRD).

Similar characterization of as-manufactured Schumacher and Pall candle filters (i.e., S4828/315C and R1-0061, respectively) was performed in order to provide baseline information regarding the various material and

Figure 2 - Depth Of Fines Penetration Along The ID Surface Of A
Schumacher Dia Schumalith F40 Candle Filter Designated As
S2855/374C (T12)

Figure 3 - Depth Of Fines Penetration Along The ID Surface Of A Pall
Vitropore 442T Candle Filter Designated As R3-0061 (M19)

compositional properties of both clay bonded silicon carbide matrices. The characterization results for both the as-manufactured and field-tested Schumacher and Pall candle filters are provided in the following sections. Similar efforts were also conducted on the alumina/mullite candle filters which were exposed to the additional 227 hours of service during the CFBC test segment in Weeks 18-20 and Weeks 21-23. The results of the alumina/mullite filter characterization will be provided in a subsequent Quarterly Report.

Material Strength Characterization

Table 1 summarizes the room temperature and 900°C process temperature strength data for both the as-manufactured and the 227 hour CFBC-exposed Schumacher Dia Schumalith F40 and Pall Vitropore 442T candle filters. Both filter materials experienced a loss of strength after 227 hours of operation in the 900°C CFBC gas environment. The strength data of the Pall Vitropore 442T candle filters exceed those of the Schumacher Dia Schumalith F40 matrix with the exception of the Schumacher Dia Schumalith candle ID surface strength at process operating temperatures. What is interesting to note in these data is that the ID surface of the Schumacher Dia Schumalith F40 matrix appears to be significantly stronger than its OD surface. A nearly comparable, but slightly stronger OD surface is typical of the Pall Vitropore 442T filter matrix. Although the Schumacher Dia Schumalith filter has a 15 mm wall thickness, while the Pall Vitropore 442T candle has a 10 mm wall thickness, the differences observed in the OD vs ID candle filter strengths are attributed to the silicon carbide grain size and binder composition, as well as the individual vendor's manufacturing process and firing temperature profile which perhaps influence the bonding characteristics between adjacent silicon carbide grains within the various regions of the candle filter wall.

TABLE 1
SUMMARY OF THE CLAY BONDED SILICON CARBIDE CANDLE FILTER
MATERIAL STRENGTH

Candle ID	Process	Temp. °C	Time, Hrs.	C-Ring Strength, psi			
				RT Comp.	RT Tension	900°C Comp.	900°C Tension
Schumacher Dia Schumalith F40							
S482/3150	---	---	---	1790*112	2308*275	1569*90	3158*271
S2855/3740 (T-12)	CFBC	900	227	824* 76	1268*139	987*87	2392*305
Pall Vitropore 442T							
R1-0081	---	---	---	3152*130	2914*289	2747*351	2936*303
R3-0081 (M-19)	CFBC	900	227	2230*203	1829*247	2203*156	2014*291

Table 2 provides a summary of the percent strength retention for both filter elements after 227 hours of exposure in the 900°C CFBC gas environment. A greater strength reduction occurred along the OD surface of the Schumacher Dia Schumalith filter matrix, while a greater loss of material strength resulted along the ID surface of the Pall Vitropore 442T filter matrix. Thermal fatigue along the candle filter ID surface, as well as thermal/chemical degradation along the candle OD surfaces and through the filter walls are considered to be responsible for the loss of strength of the binder phase in both clay bonded silicon carbide filter materials.

Burst Strength And Young's Modulus Determination

Table 3 summarizes the results of the burst strength tests which were performed on 10 inch sections which were removed at comparable locations within both as-manufactured, as well as 227 hour exposed Schumacher Dia Schumalith and Pall Vitropore 442T candle filters. As with the C-ring compression and tension strength data previously discussed, the ultimate hoop stress of both filter elements decreases after exposure in the CFBC gas environment. Similarly Young's Modulus for both materials decreases during hot gas filtration in the CFBC gas environment.

Scanning Electron Microscopy/Energy Dispersive X-ray Analyses

Schumacher Dia Schumalith F40

Schumacher Dia Schumalith F40 clay bonded silicon carbide candle filters which were manufactured in 1992 were utilized in the hot gas filtration testing in the Karhula CFBC test facility. The morphology of the as-manufactured Schumacher Dia Schumalith filter matrix is shown in Figure 4. The binder phase which encapsulates the silicon carbide grains consists principally of a silica-enriched matrix, with minor contributions of alumina, as well as potassium, sodium, and

TABLE 2
CANDLE FILTER PERCENT STRENGTH RETENTION

Matrix	RT Compression	RT Tension	900°C Compression	900°C Tension
Schumacher Dia Schumalith F40	46.03	54.94	62.91	75.74
Pall Vitropore 442T	70.75	62.76	80.20	68.60

TABLE 3
BURST STRENGTH ANALYSIS AND YOUNG'S MODULUS
OF CFBC-EXPOSED CLAY BONDED SILICON CARBIDE CANDLE FILTERS

Candle ID	Process	Temp. °C	Time, Hrs,	Ultimate Hoop Stress, psi	Young's Modulus psi x 10 ⁶
<hr/>					
Schumacher Dia Schumalith F40					
S153/317B	---	---	---	1905	5.67
S2855/374C (T-12)	CFBC	900	227	1221	4.90
<hr/>					
Pall Vitropore 442T					
R1-0081	---	---	---	1816	8.04
R3-0081 (M-19)	CFBC	900	227	1186	5.98
<hr/>

Figure 4a - Morphology Of The As-Manufactured Schumacher Dia Schumalith
F40 Clay Bonded Silicon Carbide Filter Matrix (S482/315C)

Figure 4b - Higher Magnification Micrograph Of The Binder Phase Which Encapsulates The Silicon Carbide Grains In The Schumacher Dia Schumalith F40 Filter Matrix (S482/315C)

Figure 4c - Morphology Of The Clay Bonded Silicon Carbide Grains At An Alternate Location Along The Schumacher Dia Schumalith F40 Filter Matrix (S482/315C)

Figure 4d - Morphology Of The Clay Bonded Silicon Carbide Grains At An Alternate Location Along The Schumacher Dia Schumalith F40 Filter Matrix (S482/315C)

iron phases. Micron and submicron particulate-like fines are evident along the as-manufactured binder surface. Typically there are areas along the binder surface where crystalline rod-like features are evident. The composition of the crystalline features shown in Area 1 of Photo 7 in Figure 4 includes 49.81% O, 38.81% Si, 6.64% Al, 1.99% K, 1.40% Na, and 1.37% Fe (atomic percent basis). The composition of the fines which appear to adhere to the binder surface (Area 2, Photo 7, Figure 4) includes 58.69% O, 34.58% Si, 3.75% Al, 1.41% K, 1.32% Na, and 0.24% Fe.

In order to begin to explore the morphology of the Schumacher Dia Schumalith F40 filter matrix after 227 hours of exposure in the 900°C CFBC gas environment, a section of the matrix was removed from candle filter S2855/374C. The section was carbon coated so as not to mask the presence of sulfur within the matrix when EDAX analyses were conducted.

Figure 5 illustrates the morphology of the cross-sectioned CFBC-exposed Schumacher Dia Schumalith candle filter matrix near the OD wall. Note the relatively open pore channel structure along the cross-sectioned candle matrix, indicating that pore plugging via fines penetration had not occurred. The integrity of the fibrous aluminosilicate membrane along the OD surface of the filter remained virtually intact, serving as the protective barrier which is supported by the underlying silicon carbide grains.

Photos 3 through 5 in Figures 5b and 5c illustrate the morphology of the fibrous OD membrane of the Schumacher Dia Schumalith F40 candle filter. Area scan analysis of the fibrous membrane area shown in Photo 5, Figure 5c indicates the presence of 63.64% O, 21.96% Si, 13.84% Al, and 0.57% K.

Figure 5a - Morphology Of The Cross-Sectioned Schumacher Dia Schumalith F40 Matrix After 227 Hours Of Exposure To The 900°C CFBC Gas Environment. Penetration Of Fines Is Not Evident Along The OD Fibrous Membrane Or Pore Structure.

Figure 5b - Higher Magnification Micrographs Illustrating The Morphology Of Fibrous OD Membrane Of The Schumacher Dia Schumalith F40 Matrix After 227 Hours Of Exposure In The 900°C CFBC Gas Environment

Figure 5c - Higher Magnification Micrograph Illustrating The Morphology Of The Fibrous OD Membrane Of The Schumacher Dia Schumalith F40 Matrix After 227 Hours Of Exposure In The 900°C CFBC Gas Environment

Figure 6 shows the morphology of the clay bonded silicon carbide grains at approximately two grain layers below the OD fibrous membrane. The morphology of the binder phase (Area 1, Photo 6, Figure 6a) contains not only holes and ripples, but also what appears to be a phase change as indicated by the micron and submicron nodules that result along the smooth, as-manufactured binder surface (Photo 9, Figure 6b). The relatively large nodule in Area 1 contains 70.42% O, 20.85% Si, 6.69% Al, 1.13% Na, and 0.90% Fe, while the submicron mat (Area 2) consists of 61.24% O, 35.36% Si, 1.91% Al, 0.68% Na, 0.42% Fe, 0.26% K, and 0.12% Ti.

Higher magnification of the binder phase which interconnects adjoining silicon carbide grains (Area 2, Photo 6, Figure 6a) is shown in Figure 7. Note the extensive coalescence of the nodules along the binder coated silicon carbide grain surface, as well as separation and/or depletion of the matrix surrounding the raised nodular formations. EDAX characterization of the raised nodules in Area 1, Photo 14, Figure 7c indicate the presence of 77.65% O, 17.19% Si, and 5.15% Al which is comparable to the composition of the background binder phase (77.92% O, 17.30% Si, and 4.78% Al; Area 2, Photo 14, Figure 7c).

An extensive layer of fines is evident along the ID surface of the carbon coated cross-sectioned Schumacher Dia Schumalith F40 filter matrix. Extensive pore channel plugging via entrapped fines is not evident along the cross-sectioned filter ID surface shown in Photo 16, Figure 8a. The composition of the fines as determined by EDAX analysis consists of 65.62% O, 14.68% Si, 12.28% Al, 1.59% Ca, 1.42% Na, 1.25% Fe, 1.17% K, 0.70% Mg, 0.53% Ti, 0.46% Cl, and 0.30% P. The composition of the binder phase along the ID surface silicon carbide grains consists of 66.20% O, 33.06% Si, 0.47% Al, and 0.28% Na. Similarly the composition of the binder ligaments which joins adjacent grains along the ID surface consists of 67.04% O, 27.91% Si, 3.16% Al, 1.13% K, and 0.77% Na. The binder phase which remains along the cross-sectioned

Figure 6a - Morphology Of The Binder Coating Along The Silicon Carbide Grains At Approximately Two Grain Layers Below The Fibrous OD Membrane

Figure 6b - Higher Magnification Micrographs Indicating The Phase Transformations That Result Along The Binder Coating Of The Schumacher Dia Schumalith F40 Silicon Carbide Grains After 227 Hours Of Exposure In The CFBC Gas Environment

Figure 7a - High Magnification Micrographs Illustrating The Phase Transformations That Result Along The Surface Of The Binder Ligaments That Hold Adjoining Silicon Carbide Grains Together In The CFBC-Exposed Schumacher Dia Schumalith F40 Matrix

Figure 7b - High Magnification Micrographs Illustrating The Phase Changes That Result Along The Binder Ligament Surfaces In The CFBC-Exposed Schumacher Dia Schumalith F40 Matrix

Figure 7c - High Magnification Micrograph Illustrating The Coalescence And Phase Transformations Which Occurred Along The Schumacher Dia Schumalith F40 Matrix After 227 Hours Of Exposure In The CFBC Gas Environment

Figure 8a - Morphology Of The Ash Fines Along The ID Surface Of The Schumacher Dia Schumalith F40 Matrix

Figure 8b - Higher Magnification Micrograph Of The Binder Coating Along The Silicon Carbide Grains Near The Cross-Sectioned Filter ID Surface

grain in Area 1, Photo 17, Figure 8b is once again a silica-enriched aluminosilicate phase (62.32% O, 29.06% Si, 6.05% Al, 1.65% K, and 0.93% Na), while the underlying silicon carbide grain contains a thin oxide coating (88.26% Si, 11.74% O). The concentration of carbon is not semi-quantitatively determined by EDAX analysis.

Note that sulfur was not detected along either the binder surface of the CFBC-exposed silicon carbide grains or in the fines that had collected along the filter ID surface.

A second series of SEM/EDAX analyses were performed along the cross-sectioned Schumacher Dia Schumalith F40 matrix in an attempt to demonstrate the homogeneity of the filter matrix after 227 hours of exposure in the CFBC gas environment. As shown in Figure 9, the silicon carbide structure is relatively open, lacking entrapped fines within its pore channels. Frequently several micron-sized holes are evident within the Schumacher Dia Schumalith F40 binder phase (Photos 4 and 5, Figure 9b and 9c). Similar hole formations are evident along the binder coating of the silicon carbide grains shown in Figure 10, as well as the mat-like nodular formations which were previously described.

As shown in Photo 11, Figure 11a, a section of the binder coating which is adjacent to a hole in the binder phase appears to be raised or blistered. EDAX analysis of Area 1, Photo 11, Figure 11a indicates the presence of silicon, oxygen, aluminum, potassium, and sodium. Higher magnification micrographs of the mottled binder surface is shown in Photo 12, Figure 11b. EDAX analysis of the raised 1-2 μm formation in Area 1, Photo 12, Figure 11b indicates the presence of 52.37% O, 42.39% Si, 3.91% Al, 0.94% K, and 0.39% Na. The underlying binder phase which appears to contain rod-like formations consists of 49.05% Si, 39.55% O, 8.60% Al, 2.10% K, and 0.70% Na.

Figure 9a - Morphology Of The Binder Coated Silicon Carbide Grains In The Schumacher Dia Schumalith F40 Filter Matrix After 227 Hours Of Exposure In The CFBC Gas Environment

Figure 9b - Higher Magnification Micrographs Detailing The Micron-Sized Hole Formations In The Binder Coating Of The CFBC-Exposed Schumacher Dia Schumalith F40 Filter Matrix

Figure 9c - Higher Magnification Micrograph Illustrating The Hole Formations In The Binder Phase Of The Schumacher Dia Schumalith F40 CFBC-Exposed Silicon Carbide Grains

Figure 10a - Morphology Of The Clay Bonded Silicon Carbide Grains
At An Alternate Location Within The CFBC-Exposed Schumacher
Dia Schumalith F40 Matrix

Figure 10b - Micrographs Illustrating The Mottled Surface And Hole Formations Along The Binder Coating Of The CFBC-Exposed Schumacher Dia Schumalith F40 Filter Matrix

Figure 11a - Mottled And Blister-Like Appearance Of The Binder Coating Of The Clay Bonded Silicon Carbide Schumacher F40 Matrix After 227 Hours Of Exposure In The 900°C CFBC Gas Environment

Figure 11b - High Magnification Micrograph Illustrating The Nodular Formations That Result From Phase Changes Within The Silica-Enriched Binder Coating Along The Silicon Carbide Grains In The CFBC-Exposed Schumacher Dia Schumalith F40 Filter Matrix

Figure 12 shows the morphology of the binder ligament surface and interface between two adjoining silicon carbide grains. Figure 13 illustrates the rather mottled surface blister-like appearance of the binder phase which coats the silicon carbide grains in the CFBC-exposed Schumacher Dia Schumalith F40 filter matrix.

In order to provide higher resolution of the surface morphology changes that have occurred within the CFBC-exposed filters, an alternate section of the Schumacher Dia Schumalith matrix was removed and coated with gold prior to further SEM/EDAX characterization. Figure 14 shows the morphology of the gold coated fibrous OD membrane and underlying silicon carbide support grains in the CFBC-exposed Schumacher Dia Schumalith F40 matrix. Note the relatively open pore channels in the microstructure, indicating that fines have not penetrated through the outer membrane surface.

Photo 3 in Figure 14b shows the adherence of the fibrous outer membrane to the first silicon carbide grain in the underlying support structure. Fines appear to be attached to the fibers present in the OD membrane (Photo 5, Figure 14c). EDAX analysis of the fines shown in Area 1 (Photo 5, Figure 14c) indicate the presence of 61.92% O, 19.80% Al, 15.01% Si, 1.42% Ca, 1.10% Na, and 0.76% Fe. Round, nodular areas are also evident along the surface of the aluminosilicate fibers. EDAX analysis of these features (Area 2, Photo 5, Figure 14c) indicates the presence of 57.96% O, 27.65% Al, 13.89% Si, and 0.51% Fe.

Figure 15 illustrates the morphology of the binder ligament between two adjacent silicon carbide grains that are directly below the fibrous OD membrane of the CFBC-exposed Schumacher Dia Schumalith F40 filter matrix. Higher magnification of Area 1, Photo 6, Figure 15a is shown in Photo 7, Figure 15a. Note the appearance of mat-like, nodular strands which formed along the binder phase of the clay bonded silicon carbide Schumacher Dia Schumalith F40 filter matrix. EDAX analysis of

Figure 12a - Morphology Of The Ligament And Interconnect Binder Phase
In The CFBC-Exposed Schumacher Dia Schumalith F40 Matrix

Figure 12b - Higher Magnification Micrographs Of The Ligament And Interconnect Binder Phase In The CFBC-Exposed Schumacher Dia Schumalith F40 Matrix

Figure 13 - Blister-Like Appearance Of The Binder Phase Which Coats Silicon Carbide Grains In The CFBC-Exposed Schumacher Dia Schumalith F40 Matrix

Figure 14a - Micrographs Illustrating The Morphology Of The Schumacher Dia Schumalith F40 Matrix After 227 Hours Of Exposure In The 900°C CFBC Gas Environment (Gold Coating Applied During Sample Preparation)

Figure 14b - Higher Magnification Micrographs Detailing The Morphology Of The Fibrous OD Membrane And Its Attachment To The First Underlying Support Silicon Carbide Grain

Figure 14c - High Magnification Micrograph Detailing The Morphology Of
The Fibers In The Schumacher Dia Schumalith F40 OD Membrane
After 227 Hours Of Exposure In The CFBC Gas Environment

Figure 15a - Micrographs Illustrating Phase Transformations Along The Binder Which Coats The Silicon Carbide Grains In The CFBC-Exposed Schumacher Dia Schumalith F40 Filter Matrix

Figure 15b - Higher Magnification Micrograph Illustrating Phase Changes (Crystallization) In The Binder Coating Of The CFBC-Exposed Schumacher Dia Schumalith F40 Filter Matrix

the mat-like formation shown in Area 1, Photo 8, Figure 15b indicates the presence of 51.03% O, 46.42% Si, and 2.55% Al, while the surrounding binder phase in Area 2, Photo 8, Figure 15b consists of 31.49% O, 61.61% Si, 4.38% Al, and 2.52% K.

Figure 16 illustrates the extensive mat-like formation which results during coalescence (crystallization) of the binder phase in the CFBC-exposed clay bonded silicon carbide filters. Note the formation of holes in the binder, as well as rod-like features. EDAX analysis of the 1-2 μm nodules formed along the binder surface indicates the presence of 58.26% O, 32.62% Si, 8.50% Al, 0.40% Fe, and 0.22% K (Area 1, Photo 13, Figure 16b). The rod-like features consist of 46.01% O, 38.94% Si, 12.77% Al, 1.07% Fe, 0.70% K, and 0.51% Ti (Area 2, Photo 13, Figure 16b).

The mat appearance of the binder phase is a surface effect which does not permeate through the ligament thickness of the binder between adjacent silicon carbide grains. This is evident by the smooth texture of the cross-sectioned ligament area shown in Area 1, Photo 16, Figure 17 (53.73% Si, 34.05% O, 7.74% Al, and 4.48% K).

Photo 17, Figure 18 shows the surface of the silicon carbide grain which appears to have undergone debonding of the binder phase. Area 1 of Photo 17, Figure 18 is shown at higher magnification in Photo 18, Figure 18. The thickness of the binder in this area is approximately 10 μm . Note that the mat-like surface features of the binder phase do not extend into the cross-sectioned binder, or directly influence the binder-grain interface after 227 hours of exposure in the 900°C, oxidizing CFBC gas environment.

Figure 16a - Extensive Phase Transformations Along The Binder Surface
Of The CFBC-Exposed Schumacher Dia Schumalith F40 Filter
Matrix

Figure 16b - Higher Magnification Of The Phase Changes That Occurred Along The Surface Of The CFBC-Exposed Schumacher Dia Schumalith F40 Filter Matrix

**Figure 16c - Higher Magnification Of The Phase Changes That Occurred
Along The Surface Of The CFBC-Exposed Schumacher Dia
Schumalith F40 Filter Matrix**

Figure 17 - Morphology Of A Cross-Sectioned Binder Ligament Indicates That Phase Changes Occur Primarily Along The Surface Of The Silica-Enriched Matrix

Figure 18 - Micrographs Illustrate The Thickness Of The Binder Coating, And Surface Phase Changes That Result During Exposure Of The Schumacher Dia Schumalith F40 Matrix To The 900°C CFBC Gas Environment

Frequently areas of the binder contain extensive phase transformations as shown in Figure 19. These areas are often associated with blistered or bubbled areas of the binder which are raised or have cracked, possibly due to outgassing from the underlying substrate.

Figure 20 illustrates the extensive mottling along the surface of the Schumacher Dia Schumalith F40 binder surface. Holes and depressions, as well as cracked and raised blister formations are also evident. The mottled rounded nodular features of the binder appear to be raised above the original binder surface (Figure 20b). The nodule shown in Area 1, Photo 24, Figure 20b consists of 64.29% O, 25.60% Si, 8.26% Al, 0.90% Na, 0.58% Fe, and 0.37% K.

Moving several grains below the OD fibrous outer membrane, similar blisters and mat-like formations are evident in the Schumacher Dia Schumalith F40 matrix (Figure 21). The binder ligament area shown in Area 1, Photo 27, Figure 21b consists of 52.81% O, 37.56% Si, 4.76% Al, 2.24% K, 2.10% Na, and 0.53% Ti.

Peeling and removal of the binder layer is evident in Photo 28, Figure 21b, and at higher magnification in Figure 21c. The smooth binder ligament shown in Area 1, Photo 29, Figure 21c contains 53.17% O, 42.02% Si, 2.32% Al, 1.60% Na, 0.84% K, and 0.34% Ti. The mottled area shown in Area 2, Photo 29, Figure 21c is shown at higher magnification in Area 1, Photo 30, Figure 21c. The composition of the mottled area contains 53.87% Si and 46.13% O (carbon is also present) which implies the formation of a thin oxide layer along the surface of the silicon carbide grain.

Moving approximately 10 mm from the OD fibrous membrane (i.e., 5 mm from the ID surface) we see further blistering and cracking, as well as outgassing of the binder coating along the Schumacher Dia Schumalith F40 silicon carbide grains (Figure 22). Debonding of the

B-55

Figure 19a - Mottled Blister-Like Appearance Of The CRBC-Exposed
Schwacher Die Schwallitz F40 Binder

Figure 19b - High Magnification Micrograph Detailing The Presence Of Cracks In The Blister Formations That Result Along The Schumacher Dia Schumalith F40 Binder Surface After 227 Hours Of Operation In The CFBC Gas Environment

Figure 20a - Micrographs Illustrating The Extensive Mottling Of The Schumacher Dia Schumalith F40 Binder Surface After Exposure To The CFBC Gas Environment

Figure 20b - Higher Magnification Micrograph Illustrating The Morphology Of The Schumacher Dia Schumalith F40 Binder Surface After Exposure To The CFBC Gas Environment

Figure 21a - Blistering And Debonding Of The Binder Surface Along Silicon Carbide Grains In The Schumacher Dia Schumalith F40 Filter That Was Exposed To CFBC Operating Conditions

Figure 21b - Higher Magnification Micrographs Illustrating The Crack Formations And Debindered Areas Of The Clay Bonded Silicon Carbide Schumacher Dia Schumalith F40 Filter Matrix

Figure 21c - Higher Magnification Micrographs Showing Both The Binder Coating And An Area Which Contains A Thin Layer Of A Silica-Containing Phase Along The Silicon Carbide Grain Surface

Figure 22a - Morphology Of The Clay Bonded Silicon Carbide Schumacher
Dia Schumalith F40 Candle Filter Matrix At Approximately
10 mm From The OD Surface Of The CFBC-Exposed Candle Filter

Figure 22b - Micrographs Illustrating Debonding And Cracking Of The Binder Phase In The Schumacher Dia Schumalith F40 CFBC-Exposed Candle Filters

Figure 22c - Presence Of Microcracks Along The Mat-Like Formations Of
The Binder Phase Which Coats The Schumacher Dia Schumalith
F40 Silicon Carbide Grains

binder and crack formations, as well as the formation of the nodular mat-like binder surface features are also evident. The extensive formation of the mat-like binder surface phase is shown in Figure 23. The mat (Area 1, Photo 37, Figure 23) consists of 50.85% O, 45.23% Si, and 3.92% Al. The smooth underlying substrate shown in Area 2, Photo 37, Figure 23 consists of silicon and carbon, indicating the location of the silicon carbide grain. Oxygen was not detected along the silicon carbide grain surface shown in Area 2, Photo 37, Figure 23.

At approximately 1.5 mm from the ID surface of the Schumacher Dia Schumalith F40 clay bonded silicon carbide filter matrix, we see that the pore channels are frequently filled with fines (Figure 24). The fines were forced back into the filter matrix during pulse gas cleaning once failure in the ten alumina/mullite candle filters had occurred. Note the relatively large (20-30 μm) agglomerate of fine particulates which were deposited and retained along the binder surface of the silicon carbide grains.

Figure 25 shows the adherence of fines along the binder coating of the silicon carbide grains at an alternate location which was approximately 1.5 mm from the candle filter ID surface. Frequently microcracks are evident along the binder coating which encapsulates the silicon carbide grains along the filter ID. The top micrograph shown in Photo 44, Figure 25b faintly illustrates the microcrack features. These have been redrawn in the bottom micrograph of Photo 44, Figure 25b to emphasize their existence. Higher magnification micrographs of the fines which adhere to the ID surface silicon carbide grains are shown in Figure 25c. The fines are identified to contain silicon, oxygen, aluminum, calcium, magnesium, sodium, potassium, titanium, and iron.

Figure 23 - Extensive Mat-Like Feature Of The Crystallized Binder Phase Which Initially Coated The Silicon Carbide Grains In The Schumacher Dia Schumalith F40 Filter Matrix

Figure 24a - Morphology Of The Schumacher Dia Schumalith F40 Filter Matrix At Approximately 1.5 mm From The Candle Filter ID Wall. Fines Are Present In The Pore Channels.

Figure 24b - Adherence Of Fines Along The Binder Coated Silicon Carbide Grains

Figure 24c - High Magnification Micrograph Of The Fines Which Were Present Along The Silicon Carbide Grains Near The ID Surface Of The Clay Bonded Schumacher Dia Schumalith F40 Filter That Experience CFBC Operating Conditions

Figure 25a - Micrograph Illustrating The Adherence Of Fines Along
The Binder Coating Of The Silicon Carbide Grains In The
CFBC-Exposed Schumacher Dia Schumalith ID Surface

Figure 25b - Higher Magnification Micrographs Indicating The Presence Of Microcracks In The Binder Coating Of The Clay Bonded Silicon Carbide Candle Filter ID Surface

Figure 25c - High Magnification Micrographs Illustrating The Morphology Of The Ash Fines Retained In The Clay Bonded Silicon Carbide Matrix After Exposure To The CFBC Gas Environment

Figure 26 provides a series of micrographs at an alternate location which is approximately 1.5 mm from the candle filter ID surface. Again the mottled surface features of the binder phase are apparent, as well as the formation of microcracks through the binder coating (Photo 50, Figure 26b).

Fines are shown to fill the pore channels of the silicon carbide microstructure along the Schumacher Dia Schumalith F40 candle ID surface (Figure 27). Again the mat-like micron and submicron formations are evident along the binder surface. A higher magnification micrograph of the mat-like formations is shown in Figure 27c. The composition of the round nodules in the mat consists of 62.41% O, 19.72% Si, 12.42% Al, 1.71% Ca, 1.33% Mg, 0.90% Fe, 0.70% Na, 0.42% Ti, and 0.40% K. Due to the presence of calcium and magnesium, and the higher aluminum content (>7%) but lower silicon content (<35%), the mat-like formation is considered to contain submicron or micron ash particles as opposed to simply a phase transformation that results along the binder surface during operation in the CFBC gas environment at elevated process operating temperatures.

Pall Vitropore 442T Candle Filters

The morphology of the as-manufactured Pall Vitropore 442T clay bonded silicon carbide candle filter matrix is shown in Figure 28. As the Schumacher Dia Schumalith F40 matrix, the Pall filters consist of silicon carbide grains that are bonded together via a high silica-containing binder phase. EDAX characterization of the binder phase shown in Area 1, Photo 4, Figure 28b indicates the presence of 51.22% O, 42.43% Si, 2.98% Na, 2.83% Al, and 0.55% K. Micron and submicron particulate-like features are seen to adhere to the binder phase of the as-manufactured Pall Vitropore 442T filters. These are artifacts of the manufacturing process, and are identified by EDAX to consist of 54.48%

Figure 26a - Mottled Surface Features Of The Binder Phase Along The ID Surface Of The CFBC-Exposed Schumacher Dia Schumalith F40 Candle Filter Matrix

Figure 26b - Higher Magnification Micrographs Illustrating The Mat-Like Features And Microcrack Formations In The Binder Phase Of The CFBC-Exposed Schumacher Dia Schumalith F40 Candle Filter Matrix

Figure 26c - Mat-Like Surface Features Of The CFBC-Exposed Schumacher
Dia Schumalith F40 Binder

Figure 27a - Adherence Of Fines Along The Binder Coated Silicon Carbide Grains In The CFBC-Exposed Schumacher Dia Schumalith F40 Filter Matrix

Figure 27b - Higher Magnification Micrograph Of The Fine Particulates Attached To The Binder Coated Silicon Carbide Grains

Figure 27c - High Magnification Micrograph Illustrating The Morphology Of The Binder Coated Silicon Carbide Grains In The CFBC-Exposed Schumacher Dia Schumalith F40 Matrix

Figure 28a - Micrographs Illustrating The Morphology Of The As-Manufactured Pall Vitropore 442T Filter Matrix

Figure 28b - High Magnification Micrographs Illustrating The Smooth Binder Phase Which Is Present In The As-Manufactured Pall Vitropore 442T Candle Filters

Figure 28c - Morphology Of The Pall Vitropore 442T Candle Filter Matrix At An Alternate Location

0, 36.95% Si, 4.86% Al, 3.11% Na, and 0.59% K (Area 2, Photo 4, Figure 28b). The fine particulate-like features are infrequently detected along the smooth binder phase in the clay bonded silicon carbide Pall Vitropore 442T candle filter matrix.

Based on EDAX analysis of a carbon coated sample of the Pall filter which was exposed to the CFBC gas environment, the ash fines which adhere to the candle filter OD surface were identified to contain 65.21% O, 27.64% Si, 4.48% Al, 1.40% Na, 0.37% Fe, 0.36% Ca, 0.35% K, 0.23% S, and 0.13% Ti. As a result of the low concentration of sulfur in the fines, an additional sample of the CFBC-exposed Pall filter matrix was removed and gold coated in order to generate more distinct, high resolution micrographs. Figure 29 illustrates the morphology of the OD membrane and supporting silicon carbide grains along the cross-sectioned Pall Vitropore 442T filter which was exposed for 227 hours in the CFBC gas environment.

The thickness of the fine grained OD membrane along the Pall Vitropore 442T filter as shown in Photo 1, Figure 29a is approximately 100-300 μm , depending on the contour of the underlying coarse silicon carbide grain support layer. Photos 2 through 5, Figure 29 provide a series of high magnification micrographs which illustrate the morphology of the OD membrane-first grain support layer in the Pall CFBC-exposed matrix. Note the rather mottled surface features of the binder coated silicon carbide grains.

Figure 30 illustrates the morphology of the membrane-first grain layer at an alternate location along the Pall Vitropore 442T matrix. Note the relatively large amorphous area of the material in the micrographs shown in Figure 30. EDAX analysis of Area 1, Photo 7, Figure 30 indicates the presence of 55.23% Si, 36.55% O, 5.29% Al, and 2.94% K (high carbon content also indicated). Based on the EDAX analyses, the amorphous areas are considered to be a combination of both the silicon carbide grain and binder phase.

Figure 29a - Morphology Of The CFBC-Exposed Pall Vitropore 442T
Candle Filter OD Membrane And First Grain Layers

Figure 29b - Morphology Of The Silicon Carbide Grains Near The OD Membrane Surface Of The CFBC-Exposed Pall Vitropore 442T Candle Filter. Mottling Of The Binder Phase Is Apparent, As Well As The Adherence Of What Appears To Be Particulate Ash Fines.

Figure 29c - Higher Magnification Micrograph Illustrating The Mottled Surface Features Of The Binder Phase Which Coats The Silicon Carbide Grains In The CFBC-Exposed Pall Vitropore 442T Candle Filters

Figure 30 - Micrographs Illustrating The Morphology Of The Pall
Vitropore 442T Candle Filter At An Alternate Location Near
The OD Membrane

Figure 31 begins to explore the morphology and phase changes that result along the binder coating of the first grain layer beneath the OD membrane of the CFBC-exposed Pall Vitropore 442T matrix. The outer surface of the binder contains numerous 1-2 μm raised and rounded features. Cracks are also evident in the binder surface. A higher magnification micrograph of the area shown in Area 1, Photo 3, Figure 31b is shown in Photo 4, Figure 31b. The "white" spherical micron feature consists of 59.01% O, 33.13% Si, 6.10% Al, 1.38% Fe, and 0.38% K. Due to the somewhat higher oxygen content, yet lower silicon content, Area 1 may be an ash particle. The grayish raised, approximately 1 μm feature in Area 2 of Photo 4, Figure 31b consists of 50.40% O, 40.46% Si, 6.48% Al, 1.51% Fe, and 1.15% K. The underlying binder substrate (Area 3) consists of 47.56% Si, 39.76% O, 7.75% Al, 2.56% Fe, 1.46% K, and 0.92% of a rare earth additive that is used during the manufacture of the Pall Vitropore 442T filters to provide oxidative stability and/or corrosion resistance to the clay bonded silicon carbide filter matrix.

Figure 32 illustrates the mottled surface features of the binder phase along an alternate grain near the OD membrane. At high magnification (Photo 7, Figure 32b) it is evident that the binder phase has separated from the underlying silicon carbide grain. EDAX characterization of Area 1, Photo 7, Figure 32b indicates that the binder contains 66.47% O, 28.80% Si, 3.70% Al, 0.60% K, 0.25% rare earth, and 0.20% Fe. Area 2 (Photo 7, Figure 32b) along the binder/grain interface appears to contain cracks, and as identified by EDAX consists of 55.12% O, 42.02% Si, 1.95% Al, 0.55% K, 0.25% Fe, and 0.11% rare earth which again implies the presence of the binder layer. The silicon carbide grain is located in Area 3, Photo 7, Figure 32b.

Figure 33 provides an additional series of micrographs detailing the morphology of the binder coated silicon carbide grains at an alternate location near the OD membrane surface of the CFBC-exposed Pall Vitropore 442T filter. Again note the extensive mottling of the

Figure 31a - Micrographs Illustrating The Morphology Of The Binder Phase Along The First Grain Layer In The CFBC-Exposed Pall Vitropore 442T Filter Matrix

Figure 31b - Higher Magnification Micrographs Illustrating The Mottled Surface Features Of The Binder Coating

Figure 32a - Micrographs Illustrating The Morphology Of The CFBC-Exposed Pall Vitropore 442T Matrix At An Alternate Grain Near The OD Membrane Of The Cross-Sectioned Filter

Figure 32b - Higher Magnification Micrograph Illustrating The Raised And Separated Features Of The Binder Coating Along A Silicon Carbide Grain

Figure 33a - Micrographs Illustrating The Extensive Mottling Of The Binder Surface Near The OD Membrane Of The CFBC-Exposed Pall Vitropore 442T Filter

Figure 33b - Additional Micrographs Illustrating The Mottled Surface Features Of The Binder Phase Which Coats The Silicon Carbide Grains Near The OD Membrane Of The CFBC-Exposed Pall Vitropore 442T Filter

Figure 33c - Higher Magnification Micrograph Illustrating The Mottled Features Of The Binder Coating

binder surface features, as well as the adherence of numerous 5-10 μm particles. EDAX analysis of Area 1, Photo 10, Figure 33a indicates the presence of 59.77% O, 29.11% Si, 7.72% Al, 1.55% Na, 0.74% K, 0.57% Mg, and 0.55% Fe. The typical mottled surface features of the binder phase shown in Area 2, Photo 10, Figure 33a consists of 54.03% O, 38.76% Si, 5.65% Al, 1.02% K, 0.52% Fe, and 0.23% Na. The raised agglomerate features shown in Area 3, Photo 10, Figure 33a consists of 57.21% O, 30.42% Si, 6.26% Mg, 3.47% Al, 1.72% Fe, 0.79% Ca, and 0.34% K. Note the close similarity between the composition of the CFBC ash particulates and the binder phase. Area scan analysis of Area 1, Photo 12, Figure 33b indicates the presence of 58.74% O, 26.57% Si, 10.90% Mg, 2.87% Al, 0.81% Ca, 0.66% Fe, 0.42% K, and 0.23% rare earth. The presence of the rare earth in this area of the matrix implies the binder phase of the CFBC-exposed Pall Vitropore 442T filter matrix, while the presence of magnesium and calcium indicates the inclusion of ash fines. The surface features of the binder coated silicon carbide grain shown in Photo 12, Figure 33b are expected to result from phase transformations along the binder surface in the CFBC-exposed Pall Vitropore 442T filters. Higher magnification of the binder surface morphology is shown in Photo 13, Figure 33c. The mottled surface consists of 53.63% O, 37.63% Si, 6.63% Al, 1.22% K, 0.77% Fe, and 0.13% rare earth.

Figure 34 provided a series of micrographs again near the OD surface of the CFBC-exposed Pall Vitropore 442T filter. Note the presence of what appears to be fine particulates adhering to the binder phase shown in Photo 14 and 15, Figure 34b, as well as the convoluted binder layer that formed near the surface of the silicon carbide grain. Area scan analysis of the smooth feature shown in Area 1, Photo 16, Figure 34c indicates the presence of 62.10% Si, 30.45% O, 4.12% Al, 3.09% K, and 0.24% rare earth. The somewhat mottled features shown in Area 2, Photo 16, Figure 34c contain 59.23% O, 40.58% Si, and 0.19% Al.

Figure 34a - Micrograph Illustrating The Morphology Of The Clay Bonded Silicon Carbide Filter Matrix After Exposure To The CFBC Gas Environment

Figure 34b - Micrographs Illustrating The Binder And Adhering Fines
Near The OD Membrane Surface Of The CFBC-Exposed Pall
Vitropore 442T Candle Filter

Figure 34c - Micrographs Illustrating The Morphology Of The Binder Coating Surface Along Silicon Carbide Grains Near The OD Membrane Surface Of The CFBC-Exposed Pall Vitropore 442T Candle Filter

Similar features are evident along the binder phase which coats the silicon carbide grains that are located 3-4 grain layers below the OD membrane surface. EDAX analysis of Area 1, Photo 20, Figure 35 indicates the presence of 47.72% O, 43.18% Si, and 9.10% Al in the surface of the binder phase. EDAX characterization of Area 2, Photo 20, Figure 35 indicates the presence of silicon and carbon, indicating the location of the silicon carbide grain.

Figure 36 shows the morphology of the fractured binder ligament between two adjacent silicon carbide grains which are 3-4 grain layers below the OD membrane surface. The composition of the binder ligament consists of 51.26% O, 36.29% Si, 6.41% Al, 3.30% Na, 1.18% Fe, 1.11% K, and 0.45% rare earth (Area 1, Photo 22, Figure 36).

Similar mottled features along the binder surface are also detected at approximately 5 mm from the OD surface, as well as 5 mm from ID surface of the CFBC-exposed Pall Vitropore 442T candle filter matrix (Figure 37). Cracks and separations are readily evident in the binder coating. High magnification micrographs illustrating the morphology of the mottled binder surface features (Area 1, Photo 26, Figure 37b) are shown in Photo 27, Figure 37c.

A mottled binder coating surface appearance results along silicon carbide grains that are located at approximately 2 mm from the ID surface of the Pall Vitropore 442T filter (Figure 38). Areas are evident where the binder ligaments have been fractured during sampling. The binder ligament in these areas appears to be not only discontinuous, but also contains holes (Area 1, Photo 29, Figure 38a). Area 2, Photo 29, Figure 38a is shown at higher magnification in Photo 30, Figure 38b. The particle-like features (Area 1, Photo 30, Figure 38b) consists of 56.97% O, 25.21% Si, 14.48% Al, 1.91% Na, 0.74% Fe, and 0.70% K. The amorphous features of Area 2, Photo 30, Figure 38b consist of 56.55% O, 32.98% Si, 6.89% Al, 2.50% Na, 0.61% K, 0.25% Ca, and 0.23% rare earth, indicating the presence of the binder phase. The presence of calcium in

Figure 35 - Micrographs Illustrating The Morphology Of The Binder Coated Silicon Carbide Grains Located At 3-4 Grain Layers Below The OD Membrane Surface Of The CFBC-Exposed Pall Vitropore 442T Candle Filter

Figure 36 - Morphology Of A Binder Ligament Between Adjacent Grains That Are Located 3-4 Grain Layers Below The OD Membrane Of The CFBC-Exposed Pall Vitropore 442T Candle Filter

Figure 37a - Micrographs Illustrating The Morphology Of The CFBC-Exposed Pall Vitropore 442T Matrix At Approximately 5 mm Below The OD Membrane Of The Candle Filter

Figure 37b - Higher Magnification Micrographs Illustrating The Extensive Mottling And Crack Formations Which Result In The Binder Coating Along Grains That Were Located 5 mm Below The OD Membrane Of The CFBC-Exposed Pall Vitropore 442T Candle Filter

Figure 37c - Higher Magnification Micrograph Illustrating The Morphology Of The Mottled Binder Phase

Figure 38a - Morphology Of The CFBC-Exposed Pall Vitropore 442T Filter
At Approximately 2 mm From The Filter ID Surface

Figure 38b - Adherence Of Fines Along The Binder Ligament

Figure 38c - High Magnification Micrograph Illustrating The Mottled Surface Features Of The Binder Coating Along Silicon Carbide Grains That Are Located At Approximately 2 mm From The Filter ID Surface

this area is considered to be an artifact of the adjacent adhering fine particle. The mottled binder surface features shown in Area 3, Photo 29, Figure 38a are shown at higher magnification in Photo 31, Figure 38c. The composition of the mottled binder surface includes 60.22% O, 31.76% Si, 5.33% Al, 1.41% Na, 0.75% Fe, 0.38% K, and 0.16% rare earth

Fines are seen to have penetrated the ID surface of the Pall Vitropore 442T filter after failure of the ten alumina/mullite candle filters in the Westinghouse Advanced Particulate Filtration system. The ash fines are shown to adhere to the binder coated silicon carbide grains in Figure 39. The composition of the fines (Photo 33, Figure 39) includes 58.66% O, 22.32% Si, 15.02% Al, 1.74% Fe, 1.41% Ca, 0.54% Ti, and 0.30% K.

Figure 40 provides another series of micrographs along the ID surface of the CFBC-exposed Pall Vitropore 442T filter. Heavy accumulation of fines is evident in the pore channels in this area of the filter. Area scan analyses of the fines shown in Area 1, Photo 35, Figure 40a indicates the presence of 63.26% O, 22.26% Si, 10.57% Al, 1.74% Fe, 1.43% Ca, and 0.73% Ti. Photo 36, Figure 40b illustrates the binder/grain interface along the ID surface silicon carbide grains. The mottled features of the binder phase are evident amidst the adhering particulate fines.

Gravimetric Leaching And Qualitative X-Ray Diffraction Analysis

Samples from both the as-manufactured and field-tested Schumacher Dia Schumalith F40 and Pall Vitropore 442T candle filters were ultrasonically leached with water and then ground in a tungsten carbide ball mill. The samples were weighed into platinum dishes and HF was added. The samples were then taken to dryness, and re-weighed. The resulting weight loss is considered to reflect the concentration of SiO_2 present in each material.

Figure 39 - Micrographs Illustrating The Adherence Of Fines Along The ID Surface Of The CFBC-Exposed Pall Vitropore 442T Candle Filter

Figure 40a - Micrographs Illustrating The Adherence Of Fines Along An Alternate Location Of The ID Surface Of The CFBC-Exposed Pall Vitropore 442T Candle Filter

Figure 40b - High Magnification Micrograph Illustrating The Mottled Surface Features Of The Binder Phase Which Coats The ID Surface Silicon Carbide Filter Grains

As shown in Table 4, the SiO_2 concentration for both the Schumacher Dia Schumalith F40 and Pall Vitropore 442T matrices is approximately 6-8%. XRD analyses are also provided in Table 4 for the coarse and fine fractions of the CFBC-exposed Schumacher Dia Schumalith F40 and Pall Vitropore 442T matrices, as well as the as-manufactured Pall Vitropore 442T matrix. The fine fraction is dominated by the oxide phase since it is easier to crush than the coarser silicon carbide grains. Mullite, if present in the oxide phase, is not expected to dissolve completely in the HF solution, and therefore a lower resulting SiO_2 concentration will be estimated via the HF dissolution technique. The presence of mullite as a result of crystallization of the Schumacher Dia Schumalith F40 binder at CFBC temperatures may be responsible for the lowered SiO_2 content in comparison to the as-manufactured Schumacher Dia Schumalith F40 matrix.

In the as-manufactured Schumacher Dia Schumalith F40 matrix, 4H and 6H polytypes of silicon carbide are present, as well as cristobalite and mullite. Similar x-ray diffraction patterns are evident in the Schumacher Dia Schumalith F40 matrix after 227 hours of exposure to the CFBC gas environment. Note the presence of a trace concentration of tridymite in the crushed oxide fines in the CFBC-exposed Schumacher Dia Schumalith F40 matrix. At this time tridymite is considered to primarily result from the reaction of the as-manufactured binder sodium concentration with silica, as opposed to sorption and reaction of gas phase sodium with the binder matrix. If the concentration of tridymite increases with increased exposure time, we would then conclude that the binder is sorbing gas phase sodium that is released into the effluent CFBC gases.

The Pall Vitropore 442T matrix similarly consists of polytypes of α -silicon carbide, with mullite and a trace of tridymite present in the coarse fraction of the material. Cristobalite is evident in this fraction after the Pall Vitropore 442T matrix is exposed for 227 hours

TABLE 4
GRAVIMETRIC LEACHING AND QUALITATIVE XRD ANALYSIS OF
AS-MANUFACTURED AND CFBC-EXPOSED SILICON CARBIDE CANDLE FILTERS

Matrix	%SiO ₂ *	XRD Analysis
<hr/> Schumacher Dia Schumalith F40		
As-Manufactured	8.06	4H and 6H polytypes of SiC. Evidence of cristobalite and mullite.
S2855/374C ** (T12)	6.16	Hex polytypes of α -SiC, cristobalite, and mullite. Fines: More cristobalite than mullite. Trace of tridymite.
<hr/> Pall Vitropore 442T		
As-Manufactured	6.79	Hex α -SiC, mullite, trace of tridymite. Fines: Glass plus trace of cristobalite. More oxide than the CFBC-exposed Schumacher Dia Schumalith F40 matrix.
R3-0061 ** (M19)	7.57	Hex α -SiC, mullite and cristobalite, trace of tridymite. Fines: Mullite, cristobalite and tridymite. More oxide than the as-manufactured Pall or CFBC-exposed Schumacher matrix.

* Gravimetric Leaching.

** CFBC-Exposed Materials.

to the CFBC gas environment. Notably in the fine fraction of the Pall Vitropore 442T matrix, glass plus a trace of cristobalite are present in the as-manufactured matrix, while mullite, cristobalite and tridymite are identified after 227 hours of CFBC operation. The quantity of oxide present is increased in the CFBC-exposed Pall Vitropore 442T matrix over its original background content, implying that oxidation of the matrix may have occurred.

Conclusions

- Both the Schumacher Dia Schumalith F40 and Pall Vitropore 442T clay bonded silicon carbide candle filters lose room temperature and process temperature (900°C) strength after 227 hours of operation in the CFBC gas environment.
- Although the Pall Vitropore 442T filter is initially stronger than the Schumacher Dia Schumalith F40 filter, the resulting CFBC-exposed hot strength along the Schumacher ID surface is 2392 psi, while the Pall ID surface is 2014 psi. In contrast the CFBC-exposed Schumacher OD surface hot strength is only 987 psi, while the Pall OD hot strength is 2203 psi.
- A greater percentage of strength is lost along the ID surface of the Pall Vitropore 442T candle filters after 227 hours of operation in the CFBC gas environment, implying that the matrix has experienced thermal fatigue during pulse gas cleaning.
- A greater percentage of strength is lost along the OD surface of the Schumacher Dia Schumalith F40 candle filters after 227 hours of operation in the CFBC gas environment, implying that the matrix has experienced thermal (and/or) chemical degradation during hot gas filtration. Note that the Schumacher Dia Schumalith F40 matrix appears to be somewhat more resistant to thermal fatigue along its ID surface in comparison to the Pall Vitropore 442T matrix.

- The OD membrane of the Schumacher Dia Schumalith F40 matrix remains intact after 227 hours of operation in the CFBC gas environment. Fines are not observed to have penetrated through the fibrous aluminosilicate membrane. In contrast, fines are evident within the support grains of the Pall Vitropore 442T matrix. The adhering fines are attached to the binder coated grains along the OD surface and mid-section of the Pall Vitropore 442T filter. The fines do not fill and/or plug the pore channels.

- Although all twenty-one of the clay bonded silicon carbide candle filters remained intact during the 227 hours of operation in the CFBC gas environment, fines were detected to penetrate the ID surfaces of both the Schumacher and Pall filters. These fines were introduced via pulse cleaning after failure of ten alumina/mullite candle filters had occurred. The depth of fines penetration along the ID surface is nearly equivalent (2-2.5 mm) in both filter materials, irrespective of the wall thickness and pore channel dimensions.

- The binder surface in both the Schumacher Dia Schumalith F40 and Pall Vitropore 442T filters undergoes phase transformations during exposure to the CFBC gas environment. These appear to be surface effects which do not propagate through the binder coating or ligament thickness. The expected phase transformation is the formation of cristobalite. The resulting phase transformations within the clay binder of both the Schumacher and Pall filters may be responsible for the loss of material strength at process operating conditions.

- Changes within Schumacher Dia Schumalith F40 matrix include:
 - Surface phase transformations. Coalescence of the silica-rich areas (lower alumina content), leading to a separation and/or depletion of the surrounding binder matrix.

- Formation of holes and microcracks in the binder coating.
 - Formation of raised or blister areas in the binder coating.
 - Evidence of outgassing of subsurface layers.
 - Debonding of the binder phase, revealing what may be a thin cristobalite layer along the silicon carbide grain surface.
- Changes within the Pall Vitropore 442T matrix include:
 - Binder surface phase transformations.
 - Microcracks and separation within the binder surface which encapsulates the silicon carbide grains.
 - Separation of the binder coating from the underlying silicon carbide grain.
 - The rare earth additive which is included in the Pall Vitropore 442T matrix appears to diffuse to the binder coating surface during hot gas filtration in the CFBC gas environment.

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