DOE/METC-82-46
(DE82016257)

**METC CERAMIC CORROSION/EROSION STUDIES—
TURBINE MATERIAL SCREENING TESTS IN
HIGH-TEMPERATURE, LOW-BTU, COAL-DERIVED
GAS COMBUSTION PRODUCTS**

By
Curtis V. Nakaishi
Debbie M. Waltermire
Larry W. Hawkins
Terry L. Jarrett

May 1982

Morgantown Energy Technology Center
Morgantown, West Virginia

and

EG&G WASC, Inc.
Morgantown, West Virginia

TECHNICAL INFORMATION CENTER
UNITED STATES DEPARTMENT OF ENERGY

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency Thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

DISCLAIMER

"This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise, does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof."

This report has been reproduced directly from the best available copy.

Available from the National Technical Information Service, U. S. Department of Commerce, Springfield, Virginia 22161.

Price: Printed Copy A05
Microfiche A01

Codes are used for pricing all publications. The code is determined by the number of pages in the publication. Information pertaining to the pricing codes can be found in the current issues of the following publications, which are generally available in most libraries: *Energy Research Abstracts*, (ERA); *Government Reports Announcements and Index* (GRA and I); *Scientific and Technical Abstract Reports* (STAR); and publication, NTIS-PR-360 available from (NTIS) at the above address.

METC CERAMIC CORROSION/EROSION STUDIES --
TURBINE MATERIAL SCREENING TESTS IN HIGH-TEMPERATURE
LOW-BTU, COAL-DERIVED GAS COMBUSTION PRODUCTS

By

Curtis V. Nakaishi and Debbie M. Waltermire

Morgantown Energy Technology Center
Morgantown, West Virginia 26505

and

Larry W. Hawkins and Terry L. Jarrett

EG&G WASC
Morgantown, West Virginia 26505

May 1982

UNITED STATES DEPARTMENT OF ENERGY
Morgantown Energy Technology Center
Morgantown, West Virginia 26505

TABLE OF CONTENTS

	<u>Page</u>
List of Figures	iii
List of Tables	iv
List of Notation	v
Acknowledgments	ix
Abstract	1
 I. Introduction	
Objectives	2
Participants	2
Approach	3
Summary	5
 II. Material Description	8
 III. Results Discussion	11
Exposure Environment	11
Description of the Reactants	11
Products of Combustion	12
Aerosol Data	26
Material Test History	29
Static Materials	29
Dynamic Materials	47
 IV. Facilities Description	51
Static Facility	51
Static Test Furnace	51
Static Test Specimen Racks	54

TABLE OF CONTENTS
(Continued)

	<u>Page</u>
Static Air/Fuel System	54
Static Exhaust Section	55
Dynamic Facility	55
Dynamic Test Section	58
Dynamic Test Specimen Holder	58
Dynamic Exhaust Section	60
Dynamic Air/Fuel System	60
V. Loading Procedures	61
References	74

LIST OF FIGURES

<u>Figure</u>	<u>Description</u>	<u>Page</u>
1	Corrosion/Erosion Test Facilities	4
2	Test Specimens Configuration	10
3	METC Gasifier/Gas Cleanup System	13
4	Static Facility Specimen Arrangement, Rack 1	30
5	Static Facility Specimen Arrangement, Rack 2	31
6	Static Facility Specimen Arrangement, Rack 3	32
7	Static Facility Specimen Arrangement, Rack 4	33
8	Static Facility Specimen Arrangement, Rack 5	34
9	Static Facility Specimen Arrangement, Rack 6	35
10	Static Facility Specimen Arrangement, Rack 7	36
11	Static Facility Specimen Arrangement, Rack 8	37
12	Static Facility Specimen Arrangement, Rack 9	38
13	Static Accumulated Exposure Time vs. Temperature	39
14	Dynamic Facility Specimen Arrangement	46
15	Dynamic Accumulated Exposure Time vs. Temperature	48
16	Simplified Process Flow Schematic, Static Facility	52
17	Static Facility	53
18	Simplified Process Flow Schematic, Dynamic Facility	56
19	Dynamic Facility	57
20	Dynamic Facility Specimen Holders	59
21	Photo Static Rack 1	64
22	Photo Static Rack 2	65
23	Photo Static Rack 3	66
24	Photo Static Rack 4	67
25	Photo Static Rack 5	68
26	Photo Static Rack 6	69
27	Photo Static Rack 7	70
28	Photo Static Rack 8	71
29	Photo Static Rack 9	72
30	Photo Dynamic Specimens Upstream View	73

LIST OF TABLES

<u>Table</u>	<u>Description</u>	<u>Page</u>
1	Summary Table of the Description of the Products of Combustion	6
2	Test Specimen Material Types and Suppliers	9
3	Bituminous Coal Analysis	14
4	The Gasifiers Operating Parameters	15
5	Average Composition of Fuel Gas	16
6	Multi-Elements Sampled in Fuel Gas	17
7	Average Heating Values for Fuel Gas	18
8	Assumed Composition of Combustion Air	18
9	Static Test Cell 1 POC Composition	20
10	Static Test Cell 2 POC Composition	21
11	Static Test Cell 3 POC Composition	22
12	Dynamic Unit POC Composition	23
13	Comparison of POC Levels at Different Pressures	26
14	Aerosol Mass Loading	28
15	Static Facility Accumulated Exposure	40
16	Dynamic Specimen Exposure Time	47

LIST OF NOTATION

A/F	Air/fuel
AFML	Air Force Material Lab, Wright-Patterson Air Force Base
Ag	Silver
Al	Aluminum
Al ₂ O ₃	Alumina
A/N	Aluminum Nitride
~	Approximation
Ar	Argon
As	Arsenic
ASME	American Society of Mechanical Engineers
atm	Atmosphere
B	Boron
Ba	Barium
Btu/hr	British thermal units/hour
Btu/lbm	British thermal units/pound mass
Btu/scf	British thermal units/standard cubic feet
C	Carbon
Ca	Calcium
CaSO ₄	Sulfate (anhydrite)
Cd	Cadmium
C/E	Corrosion/erosion
cfm	Cubic feet per minute
CH ₄	Methane
C ₂ H ₆	Ethane
Cl	Chlorine
Cm	Curium
cm ³	Cubic centimeters
CNTD	Controlled Nucleation Thermochemical Deposition
Co	Cobalt
CO	Carbon monoxide
CO ₂	Carbon dioxide
COS	Carbonyl sulfide
Cr	Chromium

LIST OF NOTATION

(Continued)

CS ₂	Carbon disulfide
Cu	Copper
CVD	Chemical vapor deposited
DM	Polyvinyl chloride
DOD	Department of Defense
DOE	Department of Energy
EAA	Electrical aerosol analyzer
EDX	Energy dispersive x-ray
°F	Degree Fahrenheit
Fe	Iron
FeSO ₄	Ferrous sulfate
FFU	Fossil Fuel Utilization Division
fps	Feet per second
GC	Gas chromatography
GF	Glass filter
H ₂	Hydrogen
HCN	Hydrocyanic acid
Hg	Mercury
H ₂ O	Water
HP	Hot Pressed
H ₂ S	Hydrogen Sulfide
HSDU	Holmes-Stretford desulfurization unit
IITRI	Illinois Institute of Technology Research Institute
in	Inch
IR	Infrared
K	Kelvin (°K)
kg/s	Kilogram/second
kJ/kg	Kilo Joule/kilogram
kPa	Kilo Pascal
Kw	Kilowatt
Li	Lithium
<	Less than

LIST OF NOTATION

(Continued)

m	Meter
m ³	Cubic meters
METC	Morgantown Energy Technology Center
Mg	Magnesium
MJ/m ³	Mega Joule/cubic meter
mm	Millimeter
Mn	Manganese
Mo	Molybdenum
m/s	Meter per second
m ³ /s	Cubic meters/second
μ	Micro
μm	Micrometers
μg/m ³	Micrograms per cubic meter
N ₂	Nitrogen
Na	Sodium
NASA	National Aeronautics and Space Administration
ND	Not detected
NH ₃	Ammonia
Ni	Nickel
NM	Not Measured
NO	Nitric oxide
NO _x	Nitric oxides
O ₂	Oxygen
Pb	Lead
POC	Product of combustion
ppm	Volume parts per million
psig	Pounds per square inch, gage
QCM	Quartz crystal microbalance
R&D	Research and Development
RMS	Root mean square
RS	Reaction sinistered
S	Sulfur
scfh	Standard cubic feet per hour

LIST OF NOTATION

(Continued)

scfm	Standard cubic feet per minute
Se	Selenium
SEM	Scanning electronic microscope
Si	Silica
SiC	Silicon carbide
Si ₃ N ₄	Silicon nitride
SO ₂	Sulfur dioxide
SO ₃	Sulfur trioxide
SO _x	Oxides of sulfur
Sr	Strontium
Std	Standard
V	Volume
Vol	Volume
wt/wt	Weight/weight
Y ₂ O ₃	Yttrium oxide
Zn	Zinc
ZrO ₂	Zirconia

ACKNOWLEDGEMENTS

The scope and general direction of the program have been developed during Program Coordination Meetings which included the following organizations external to METC.

DOE Fossil Fuel Utilization Division, Program Sponsor:

Gary Voelker
John Fairbanks
George Manning

MITRE, Technical Advisors:

Roy Peterson

Material Characterization Studies:

David Larsen, IITRI
Jane Adams, IITRI
Robert Ruh, AFML

The design, construction, and operation of the facilities at METC has been the responsibility of Technology Development and Engineering Division, John E. Notestein, Director. The METC project members include:

Process Technology and Engineering Branch, Project Office:

L. C. Headley
C. V. Nakaishi
D. M. Waltermire

METC Engineering Test Branch, Operation:

G. R. Fizer

EG&G, Support Engineering Contractors:

J. Bird
L. Hawkins
T. Jarrett
D. Bonnett
A. Kubala

Dynamic Flow, Inc., Consultant:

John Loth

A note of appreciation to METC Word Processing, Pam Stasia, and Carrie McCulloch for typing this report.

ABSTRACT

The Morgantown Energy Technology Center, through its Ceramics Corrosion/Erosion Studies, has participated in the United States Department of Energy's High-Temperature Turbine Technology Program, Ceramic Technology Readiness. The program's overall objective is to advance the turbine firing temperature to a range of 2600°-3000°F (1700-1922K) with a reasonable service life using coal or coal-derived fuel. The Ceramics Corrosion/Erosion Studies' major objective was to conduct a screening test for several ceramic materials to assess their probability of survival in turbine applications. The materials were exposed to combustion products from low heating value coal-derived gas and air at several high temperatures and velocities. The combustion product composition and temperatures simulated actual environment that may be found in stationary power generating gas turbines except for the pressure levels. The results of approximately 1000 hours of accumulative exposure time of material at the specific test conditions are presented in this report.

I. INTRODUCTION

Objectives

The objective of the Ceramics Corrosion/Erosion Studies (C/E) Project was to initiate a technology baseline for the applications of ceramic materials in electric power generating gas turbines fueled with coal-derived gases. The material baseline will be derived from studying the corrosion/erosion effects as a function of exposure duration at various combustion product temperatures and velocities. The purposes of this report are: (1) identify the controlled test conditions; (2) describe the chemical composition and aerosols of the exposure environment; and (3) delineate the individual specimens handling and exposure histories.

Participants

The Department of Energy, Division of Fossil Fuel Utilization (DOE/FFU), had overall responsibility of the project (Reference 1). A variety of different ceramic test specimen materials were provided through separate DOE/FFU contracts with Westinghouse (Reference 2) and Garrett (Reference 3).

IIT Research Institute (IITRI) and the Air Force Material Lab, Wright-Patterson Air Force Base (AFML), had the responsibility of characterization of the materials (Reference 4).

Morgantown Energy Technology Center (METC) had the responsibility of designing and constructing the test facilities, exposing the test specimens to the combusted coal-derived gas, implementing and determining the methods of specimens exposure, describing the specimen exposure history, characterizing the specimen's test environment, and providing the coal-derived gas (Reference 5).

Approach

This study involved the use of an actual coal-derived fuel gas with its inherent variability of composition and heating value. The source of the fuel gas was the METC fixed-bed gasification system. The gasifier was a modified Wellman-Galusha design equipped with a movable stirrer (Reference 6). The testing revolved around determining the degradation of the chosen ceramic material by the coal-derived fuel gas combustion products. The degradation effects were compared at specific test conditions of temperature and velocity for specified time periods. The fuel gas provided to the Corrosion/Erosion Facilities during these tests was from the end of the gasification full stream cleanup system. This gas was relatively clean.

Two separate test units were used. One provided near static, low-velocity combustion products and the other provided dynamic, high-velocity combustion products. Both units were operated simultaneously. The static unit operating conditions in its three different test cells were: (1) separate temperatures of 1850°, 2200°, and 2600°F (1283, 1478, and 1700K); (2) exposure durations of 100, 300, and 1000 hours; and (3) combustion product velocities of less than 10 feet per second (fps) (3.05 m/s). The test conditions for the dynamic unit were: (1) a single temperature of 2600°F (1700K), (2) a duration of 1000 hours, and (3) combustion product velocity of 1000 fps (305 m/s). The test units are shown in Figure 1.

The "dynamic" test should give an indication of the combined corrosive/erosive effects while the "static" test should indicate, predominately, corrosive effects. A comparison of both tests should give a measure of each effect on the ceramic specimens. At a minimum, the tests should help eliminate materials that are not suited for use in a high-temperature, coal-derived combustion gas environment. Although further testing may be needed, the current studies will identify some of the degradation to ceramics due to their exposure in a coal-derived combustion gas environment. The precise effect to the materials will be presented in a report by IITRI after the material characterization is completed.

FIGURE 1

CORROSION/EROSION STUDIES

MORGANTOWN ENERGY TECHNOLOGY CENTER

Summary

To achieve an accumulated exposure duration of 1000 hours at test conditions, the operation of the two test units and the fixed-bed gasifier spanned approximately a year of time. The unloading/loading sequence of the static test specimens was not possible while the unit was at operating temperatures as previously planned. The three required shutdowns for this purpose resulted in natural time exposures of 100, 300, and 600 hours. These durations were called Phases I, II, and III, respectively. Phase I was from November 15 through November 21, 1980. Phase II was from May 29, 1981, through June 16, 1981. Phase III was from July 16, 1981, through August 19, 1981. To maintain comparable conditions, the dynamic unit followed a similar schedule. The length of the non-operating period between each phase depended on repairs to the gasifier, METC scheduling of other projects, and manpower availability.

Bituminous coals were used in the gasification process for producing the fuel gas. Arkwright was used during the first two phases and Lower Kittanning was used in the final phase. The change to a lower sulfur (Lower Kittanning) coal was made because the Holmes-Stretford desulfurization unit (HSDU) was not part of the cleanup system during the last phase. The most probable combustion product exposure environments for all three phases at each of the three test temperatures in both static and dynamic units are shown in Table 1. One of the static test cells operated at the same nominal temperature as the dynamic unit. These test conditions should have shown the effect of the combustion product velocities on the test specimens. The exposure environment gas compositions were slightly different for each unit because they operated with different air/fuel (A/F) ratios to maintain the 2600°F (1700K) temperature.

TABLE 1

Description of the Products of Combustion

Test Unit	Phase	Nominal Temperature	Nominal Velocity	A/F	Chemical Composition					
					CO ₂	H ₂ O (Percentage)	N ₂	O ₂	SO ₂ (ppm)	NO _x
Static 1	I	1850°F (1283K)	>10fps (3 m/s)	3.14	10.70	8.00	73.84	6.63	117	9
	II			3.93	7.26	6.79	75.55	10.35	70	3
	III			3.66	7.61	7.26	75.07	9.98	490	34
Static 2	I	2200°F (1477K)	>10fps (3 m/s)	2.16	8.69	6.51	74.66	9.27	94	4
	II			2.70	9.80	8.48	74.48	7.15	90	7
	III			2.52	10.21	9.08	73.87	6.71	670	37
Static 3	I	2600°F (1700K)	>10fps (3 m/s)	1.52	10.70	8.00	73.84	6.53	117	9
	II			1.82	13.05	10.62	73.12	3.05	120	19
	III			1.71	13.54	11.40	72.32	2.53	900	48
Dynamic	I	2600°F (1700K)	1000 fps (305 m/s)	2.12	10.67	7.98	73.85	6.57	117	14
	II			2.29	11.09	9.31	73.91	5.50	110	9
	III			2.20	12.56	9.85	71.53	5.84	750	32

The dynamic unit combustion product aerosol data indicated average mass loadings of $10,514 \mu\text{g}/\text{m}^3$ in phase I, $3,157 \mu\text{g}/\text{m}^3$ in Phase II, and $178,731 \mu\text{g}/\text{m}^3$ in Phase III. The particle mean diameters varied as follows: $< 0.5 \mu\text{m}$ in Phase I, $< 0.1 \mu\text{m}$ in Phase II, and $\sim 1 \mu\text{m}$ in Phase III. During Phase I the major constituents of the aerosols were Si, Cr, Fe, Cu, and Zn. There were no significant concentrations of organics. Phase II aerosols consisted of high-relative concentrations of aluminum with trace or minor amounts of Si, S, and Ca. Chemical analyses of the aerosol in Phase III indicated the majority consisted of sulfur or sulfur compounds; Cl; and traces of Si, Ca, and Cu.

The static unit consisted of three separate test cells. These test cells operated at the following nominal temperatures: cell 1 at 1850°F (1283K); cell 2 at 2200°F (1478K); and cell 3 at 2600°F (1700K). The products of combustion (POC) velocities in each of the three test cells were calculated to be less than 10 fps (3 m/s). Each test cell had the capacity of exposing two racks simultaneously. Each rack had a duplication of four individual specimens of each of the fourteen different material types (504 specimens) except for the 100-hour, 2600°F (1700K) rack, which had an additional four proprietary carbon-carbon specimens. At the start, each cell was loaded with the racks scheduled for 1000 and 100 hours of exposure. After the first phase, those 100-hour racks were replaced by racks scheduled for 300 hours of exposure. Finally after Phase II (400 hours), the 300-hour racks were removed leaving only the three racks scheduled for the full 1000 hours of exposure. This totaled nine test racks. The durations of exposure times and the specimen arrangement on the nine racks are shown in the Material Test History Section.

It appeared that 498 of the original 508 static test specimens survived intact. Six of the ten specimen degradations were probably a result of the exposure environment. Two of those were the carbon-carbon materials, while the other four were the "C" material type (CNTD AlN coating on HP-AlN). Another material type that showed a visible amount of distortion was "D" (HP- Al_2O_3 with ZrO_2 particulates).

The dynamic unit consisted of a single test section operating at a nominal temperature of 2600°F (1700K). The POC velocity at the specimens was designed to be 1000 fps (305 m/s). At this temperature, this equals ~ Mach 0.4. This unit could only test twelve individual specimens at one time. Only 11 of the total 14 material types were tested in this unit. These dynamic test specimens were scheduled to be exposed for the entire 1000 hours. The specimens were replaced when they were lost out of their holders, broken off, or degraded a significant amount, unless their distortion prevented removal.

Three material types "B" (CVD Si_3N_4 coating on Norton RS- Si_3N_4), "C" (CNTD AlN coating on HP-AlN), and "E" (50/50 mole percent Spinel) were eliminated without achieving significant exposure times in the dynamic unit. There was only a single dynamic specimen provided of the type "B" material. This specimen was lost along with four other specimens after 28 hours of exposure during a test operation interruption. The "C" material type surfaces became cracked, swollen, and finally could not be supported at their test positions. The "E" material also degraded rapidly in the dynamic unit, mainly by the breaking off of the tips of the exposed ends. Also the "D" (HP- Al_2O_3 with ZrO_2 particulates) and "M" (sintered Si_3N_4) deformed by bending over in the dynamic unit. Twenty-seven specimens were used in this test unit during the 1000 hours of exposure. The position, order of replacement, and the exposure times accumulated by each specimen are given in the Material Test History Section. Of the twenty-seven specimens, only four of the original twelve specimens survived to the conclusion of the testing. Most of the specimens when removed at the end of the test period showed signs of material removal from their upstream surfaces. An accurate measurement of material loss was left for IITRI to determine.

II. MATERIAL DESCRIPTION

Fourteen different types of ceramic materials were supplied to METC for test exposure in the C/E facilities. Six of the materials were supplied through Garrett and the other eight through Westinghouse Electric Corporation. All the materials are listed in Table 2. Since the materials

will be discussed at length in this report, they will be referred to only by their assigned letter designations from that table.

TABLE 2
Test Specimen Material Types and Suppliers

<u>Type</u>	<u>Vendor</u>	<u>Description</u>
<u>Garrett Supplied Materials</u>		
A	Carborundum	Sintered α -SiC
B	Norton/DCI Coating	CVD Si_3N_4 Coating on RS- Si_2N_4 (NC-350)
C	Ceradyne/Chemetal Coating	CNTD AlN Coating on HP-AlN
D	AiResearch Casting	HP- Al_2O_3 with ZrO_2 Particulates
E	Montana Energy R&D Institute	50/50 Mole Percent Spinel
F	Greenleaf Corporation	HP-Sialon
<u>Westinghouse Supplied Materials</u>		
G	Norton, Experimental	HP- Si_3N_4 + 4 Percent Y_2O_3
H	Norton	HP-SiC (NC-203)
I	Norton/Chemetal Coating	CNTD SiC Coating on HP-SiC (NC-203)
J	Ceradyne/Westinghouse, Experimental	Al_2O_3 + 60 Percent SiC Composite
K	Carborundum/Chemetal Coating	CNTD SiC Coating on Sintered α -SiC
L	Norton	RS- Si_3N_4 (NC-350)
M	GTE Sylvania	Sintered Si_3N_4
N	Westinghouse, Experimental	HP- Si_3N_4

Each of these material types was provided in two configurations -- one shape for each of the test facility units.* The two configurations used are shown in Figure 2.

The static test specimens had a square cross section of $0.250 \pm .002$ inches ($6.350 \pm .050$ mm). Their length was $3.00 \pm .01$ inches ($76.20 \pm .25$ mm). Corners were beveled, and the edges were chamfered lengthwise by removing 0.005 inches (0.127 mm) of each edge at a

---

*That is, with the exception of materials "F" "J" and "L" which were not prepared for the dynamic test unit. All 14 materials, however, were supplied for the static test unit.

TEST SPECIMENS CONFIGURATION

STATIC TEST SPECIMEN

3 IN (7.6 cm) LENGTH

1/4 x 1/4 IN. (.635 x .635 cm) CROSS SECTION

DYNAMIC TEST SPECIMEN

5 IN. (12.7 cm) LENGTH

1/4 IN. (.635 cm) DIAMETER

FIGURE 2

45 degree angle. The surfaces were finished by being ground in the longitudinal direction to a maximum of 10 Root Mean Square (RMS) parallel to the grinding direction. The dynamic test specimens had a circular cross section of $0.250 \pm .002$ inches ($6.350 \pm .050$ mm) diameter. Their length is $5.0 \pm .1$ inches (127.0 ± 2.5 mm). The surfaces were finished by being ground longitudinally to a maximum of 20 RMS perpendicular to the grinding direction.

III. RESULTS DISCUSSION

The materials to be tested were shipped from IITRI to METC in September 1980. During the following 12 months, the materials were subjected to products of combustion environments in the METC C/E facilities. The materials were returned to IITRI in September 1981 for a qualitative analysis of their reactions to these tests.

Exposure Environment

The exposure environment was defined by its composition, particulate size, particulate, gas velocity, and temperature of the gases.

Description of the Reactants

Coal, air, and steam were combined in the 42-Inch Fixed-Bed Gasifier to produce the low-heating value gas used by the C/E facilities. The coal used in Phases I and II was Arkwright bituminous and in Phase III, it was Lower Kittanning bituminous (References 7, 8, and 9). Table 3 shows the Bituminous Coal Analysis. Table 4 lists the gasifier operating parameters during the three phases of testing.

Before the gas was combusted in the C/E facilities, it was passed through the 42-Inch Fixed-Bed Gasifier full-stream cleanup system. The cleanup system includes several components still in the development stage. After the gasifier, the gas passed through cyclones which removed the heavy ash/dust particles. The flow was then cycled through a humidifier and tar separator which condensed and removed heavy tars from the gas stream. A venturi scrubber was used to remove medium- to low-molecular weight

tars. The gas was then passed through a direct cooler to remove excess water, oil, and low-molecular weight tars before entering the HSDU, where hydrogen sulfide was absorbed by a vanadium salt solution. From the HSDU, the fuel gas was sent through water scrubbers to remove alkali compounds and entrained Stretford liquor. The fuel gas was then piped to the C/E facilities. The gasifier and cleanup system are shown in Figure 3.

Table 5 lists the averaged composition of the fuel gas in all three phases of testing. Note, the increased value of the H_2S in the third phase. Table 6 lists the multi-elements that were analyzed for in the fuel gas. The averaged high and low heating values for the gas are shown in Table 7. Table 8 indicates the assumed composition of the air in each phase.

Products of Combustion

The exposure environment gas composition characterization was achieved using the following four methods: (1) data measured by on-line C/E gas analyzers, (2) data measured by analytical gas chromatography (GC) on grab samples taken from the C/E facilities, (3) composition predicted by a computer program designed to calculate equilibrium compositions of the products of combustion (POC) based on reactants' compositions, and (4) POC compositions predicted by a computer program which assumed complete combustion of reactants.

The first method used the following C/E on-line instruments: Beckman Infrared (IR) analyzers to measure SO_2 (Model 865), CO_2 (Model 864-11), and CO (Model 864-33); a Parametric Model 700 dew point hygrometer to measure H_2O ; and Beckman (Model 955) chemiluminescence analyzer to measure $NO-NO_x$. Nitrogen concentrations were determined by balance. The measurements were time shared between the dynamic and three static test cells.

The second method analyzed grab samples taken at two different locations. In phase II, grab samples were taken directly from the test units and then passes through a condenser into gas collection bottles. This technique was abandoned because the SO_x gases were being absorbed by the

FIGURE 3

METC GASIFIER/GAS CLEANUP SYSTEM

TABLE 3
Bituminous Coal Analysis

Phase	Long Proximate (Weight Percentage)					Ultimate (Weight Percentage)					Heating Value	
	Moisture	Ash	Sulfur	Vol. Matter	Fix Carbon	H ₂	C	N ₂	S	O ₂	Ash	Btu/lbm (kJ/kg)
I	0.98	8.03	2.28	35.72	56.22	4.87	78.02	1.52	2.28	5.28	8.03	13,020 (30,285)
II	0.64	8.64	2.20	38.67	52.05	5.62	77.66	1.54	2.20	4.34	8.04	14,156 (32,927)
III	0.44	9.09	1.02	36.46	54.01	5.16	75.40	1.48	1.02	7.85	9.09	13,633 (31,710)

TABLE 4

The Gasifier Operating Parameters

Phase	Coal		Air				Steam			
	Feed Rates		Pressure		Flowrates		Pressure		Flowrates	
	tons/day	(kg/s)	psig	(kPa)	scfh	std m ³ /s	psig	(kPa)	tons/day	kg/s
I	12.96	(3.27)	247	(1703)	40,600	(0.319)	254	(1751)	6.65	1.68
II	14.40	(3.63)	190	(1310)	40,000	(0.315)	254	(1751)	7.30	1.84
III	19.04	(4.80)	189	(1303)	50,510	(0.397)	200	(1379)	9.75	2.46

TABLE 5

Average Composition of Fuel Gas

Phase	H ₂	CH ₄	CO	O ₂	N ₂	C ₂ H ₆	H ₂ S	COS	CS ₂
I	15.95	3.54	22.07	0.68	50.17	0.56	0.0012	0.0302	0.0024
II (Part 1)	16.54	3.12	22.63	.74	48.96	0.28	.0006	0.027	.0020
II (Part 2)	16.54	3.12	22.63	.74	48.96	0.28	.0539	0.027	.0020
III (Part 1)	18.25	2.63	22.34	0.69	47.07	0.61	0.21	0.017	0.0004
III (Part 2)	18.25	2.63	22.34	0.69	47.07	0.61	0.19	0.015	0.0003

Concentrations of the gas made on grab samples analyzed by a Perkin Elmer Sigma 1B gas chromatograph.

TABLE 6

Multi-Elements Sampled in Fuel Gas

Volume (in liters)	Concentration in Parts Per Billion (wt/wt)			Detection Limit
	510	504	519	
Phase	I	II	III	
Elements				
Al	64	80	30	1
As*	4	9.8	5	5
B	ND	48	ND	5
Ba	ND	ND	ND	1
Ca	ND	30	114	<1
Cd	ND	ND	ND	5
Co	ND	ND	ND	2
Cr	ND	ND	ND	2
Cu	ND	5	10	1
Fe	ND	3	36	7
Hg*	ND	<0.5	ND	5
K*	ND	ND	<10	5
Li	ND	ND	ND	5
Mg	ND	ND	56	<1
Mn	ND	3	ND	1
Mo	ND	ND	ND	5
Na**	16	45	<20	1
Ni	ND	3	ND	5
Pb	ND	ND	ND	5
Se*	3	ND	ND	2
Sr	ND	3	ND	5
V	ND	ND	ND	2
Zn	ND	128	34	2

ND -- Not Detected.

*Analyses done by Atomic Absorption Spectrophotometer.

**Analyses done by Ames On-Line Spectrometer in Phase III.

All others done by Plasma Emissions Spectrometer.

TABLE 7

Average Heating Values for Fuel Gas***

Phase	Higher Heating Value Btu/scf (MJ/m ³)	Lower Heating Value Btu/scf (MJ/m ³)
I	168.8 (6.30)	156.1 (5.82)
II	163.6 (6.10)	151.9 (5.67)
III	170.0 (6.34)	157.0 (5.86)

***Measured by high-speed calorimeter, Fluid Data Model FLO-CAL.

TABLE 8

Assumed Composition of Combustion Air

Phase	N ₂	O ₂	Volume Percent H ₂ O	AR
I	78.03	20.99	--	0.98
II	78.00	19.50	2.50	--
III	78.55	19.50	1.95	--

condensed water. Grab samples taken during the latter part of phase II and all of phase III were obtained after the gas passed through a permeable membrane dryer where water vapor was removed. This was the same sampling point used by the C/E on-line analyzers. The grab samples were analyzed for CO_2 , SO_2 , and O_2 by a Perkin Elmer Sigma 1B gas chromatograph.

For the third method, combustion products (exposure environment) were predicted from the reactants, i.e., the coal-derived fuel gas and the combustion air. This was accomplished by the NASA Complex Chemical Equilibrium Composition program (NASA CCEC) (Reference 13). The chemical equilibrium equations were solved simultaneously by minimization of Gibbs Free Energy. A/F ratios, combustion products temperature, and the reactant composition, temperatures, and pressures were specified. The A/F ratios were calculated from measured flow rates into the separate test units and are shown for each run on Tables 5 and 8. The trace elements in Table 6 were not used. The combustion product temperatures were the test conditions, i.e., 1850°, 2200°, and 2600°F (1283, 1477, and 1700K).

The fourth method was a computer program that predicted the composition of POC by assuming complete combustion of all non-inert gases in the coal-derived gas. Input to this program was identical to input for NASA CCEC. Average reactant compositions for each of the three phases are shown in Tables 5 and 8.

The ceramic specimens' exposure environment varied during the 1000-hour test run, primarily because of intermittent performance by the Holmes-Stretford desulfurization unit. The POC is presented in five separate sections which reflect the variation in concentrations of H_2S in the fuel gas.

Tables 9 through 12 show the composition of the products of combustion for the three static test cells and for the dynamic unit.

POC concentrations predicted by the NASA CCEC were believed to be most representative concentrations except for the NO levels during the first

TABLE 9

Description of the Static Test Cell 1
 Products of Combustion Composition
 Temperature: 1850° ± 50°F (1283K)
 Velocity: <10 fps (3 m/s)

Phase/Part	A/F Ratio	Method	No.	Major Mole Percent				Minor ppm	
				CO ₂	H ₂ O	O ₂	N ₂	SO ₂	NO _x
Phase I Exposure Time = 96 Hours	3.14	On-Line (Wet)	1	13.75	7.73	9.33	69.19	537	9
		NASA (Wet)	3	10.70	8.00	6.53	73.84	117	1679
Phase II/1 Exposure Time = 184 Hours	3.93	On-Line (Wet)	1	9.60	6.40	11.50	72.43	240	3
		NASA (Wet)	3	7.26	6.79	10.35	75.55	70	290
		On-Line (Dry)	1	10.26	--	12.29	77.44	256	3
		Grab (Dry)	2	8.49	--	12.95	78.53	<20	--
		NASA (Dry)	3	7.79	--	11.11	81.07	75	311
Phase II/2 Exposure Time = 122 Hours	3.93	On-Line (Wet)	1	9.60	6.40	11.50	72.43	210	3
		NASA (Wet)	3	7.26	6.79	10.35	75.55	180	290
		On-Line (Dry)	1	10.26	--	12.29	77.44	224	3
		Grab (Dry)	2	8.49	--	12.95	78.53	107	--
		NASA (Dry)	3	7.79	--	11.11	81.07	193	311
Phase III/1 Exposure Time = 361 Hours	3.66	On-Line (Wet)	1	6.98	7.27	11.53	74.12	740	34
		NASA (Wet)	3	7.61	7.26	9.98	75.07	490	290
		Complete (Wet)	4	7.70	7.33	9.87	75.04	510	--
		On-Line (Dry)	1	7.43	--	12.43	79.93	808	36
		Grab (Dry)	2	8.06	--	13.26	78.75	513	--
		NASA (Dry)	3	8.21	--	10.76	80.76	528	715
		Complete (Dry)	4	8.31	--	10.65	80.98	551	--
Phase III/2 Exposure Time = 240 Hours	3.66	On-Line (Wet)	1	NM	NM	NM	NM	NM	NM
		NASA (Wet)	3	7.61	7.26	9.98	75.07	490	290
		Complete (Wet)	4	7.70	7.33	9.87	75.04	510	--
		On-Line (Dry)	1	NM	NM	NM	NM	NM	NM
		Grab (Dry)	2	8.06	--	13.26	78.75	513	--
		NASA (Dry)	3	8.21	--	10.76	80.76	528	715
		Complete (Dry)	4	8.31	--	10.65	80.98	551	--

:ph:514b31

TABLE 10

Description of the Static Test Cell 2
 Products of Combustion Composition
 Temperature: $2200^{\circ} \pm 50^{\circ}\text{F}$ (1477K)
 Velocity: >10 fps (3 m/s)

Phase/Part	A/F Ratio	Method	No.	Major Mole Percent				Minor ppm	
				CO ₂	H ₂ O	O ₂	N ₂	SO ₂	NO _x
Phase I Exposure Time = 96 Hours	2.16	On-Line (Wet)	1	10.50	6.15	10.56	72.79	365	4
		NASA (Wet)	3	8.69	6.51	9.27	74.66	94	765
Phase II/1 Exposure Time = 185 Hours	2.70	On-Line (Wet)	1	11.60	6.90	8.20	73.26	324	7
		NASA (Wet)	3	9.80	8.48	7.15	74.48	90	670
		On-Line (Dry)	1	12.46	--	8.81	78.69	348	--
		Grab (Dry)	2	11.42	--	9.69	78.87	139	--
		NASA (Dry)	3	10.71	--	7.81	81.38	98	733
Phase II/2 Exposure Time = 114 Hours	2.70	On-Line (Wet)	1	11.60	6.90	8.20	73.26	345	7
		NASA (Wet)	3	9.80	8.48	7.15	74.48	250	670
		On-Line (Dry)	1	12.46	--	8.81	78.69	371	--
		Grab (Dry)	2	11.42	--	9.69	78.87	139	--
		NASA (Dry)	3	10.71	--	7.81	81.38	273	733
Phase IIL/1 Exposure Time = 335 Hours	2.52	On-Line (Wet)	1	8.89	8.72	8.07	74.24	766	37
		NASA (Wet)	3	10.21	9.08	6.71	73.87	670	650
		Complete (Wet)	4	10.33	9.17	6.58	73.84	685	--
		On-Line (Dry)	1	8.89	--	8.84	81.32	839	40
		Grab (Dry)	2	10.96	--	10.35	79.06	685	--
		NASA (Dry)	3	11.23	--	7.38	81.34	738	715
		Complete (Dry)	4	11.37	--	7.25	81.30	754	--
Phase II/2 Exposure Time = 269 Hours	2.52	On-Line (Wet)	1	NM	NM	NM	NM	NM	NM
		NASA (Wet)	3	10.21	9.08	6.71	73.87	670	650
		Complete (Wet)	4	10.33	9.17	6.58	73.84	602	--
		On-Line (Dry)	1	NM	NM	NM	NM	NM	NM
		Grab (Dry)	2	10.96	--	10.35	79.06	685	--
		NASA (Dry)	3	11.23	--	7.38	81.34	737	715
		Complete (Dry)	4	11.37	--	7.25	81.30	663	--

1:ph:514b32

TABLE 11
Description of the Static Test Cell 3
Products of Combustion Composition
Temperature: 2600° ± 50°F (1700K)
Velocity: <10 fps (3 m/s)

Phase/Part	A/F Ratio	Method	No.	Major Mole Percent				Minor ppm	
				CO ₂	H ₂ O	O ₂	N ₂	SO ₂	NO _x
Phase I Exposure Time = 53 Hours	1.52	On-Line (Wet)	1	13.75	7.73	9.33	69.19	537	9
		NASA (Wet)	3	10.70	8.00	6.53	73.84	117	1679
Phase II/1 Exposure Time = 150 Hours	1.82	On-Line (Wet)	1	14.80	7.60	3.90	73.66	343	19
		NASA (Wet)	3	13.05	10.62	3.05	73.12	420	1140
		On-Line (Dry)	1	16.02	--	4.22	79.72	371	20
		Grab (Dry)	2	15.14	--	5.80	79.88	216	--
		NASA (Dry)	3	14.61	--	3.41	81.84	134	1276
Phase II/2 Exposure Time = 118 Hours	1.82	On-Line (Wet)	1	14.80	7.60	3.90	73.66	370	19
		NASA (Wet)	3	13.05	10.62	3.05	73.12	330	1140
		On-Line (Dry)	1	16.02	--	4.22	79.72	400	20
		Grab (Dry)	2	15.14	--	5.80	79.88	216	--
		NASA (Dry)	3	14.61	--	3.41	81.84	369	1276
Phase III/1 Exposure Time =	1.71	On-Line (Wet)	1	11.20	8.90	3.95	75.87	779	48
		NASA (Wet)	3	13.54	11.40	2.53	72.32	900	1030
		Complete (Wet)	4	13.69	11.52	2.39	72.31	907	--
		On-Line (Dry)	1	12.29	--	4.34	83.28	855	53
		Grab (Dry)	2	14.58	--	6.23	79.16	777	--
		NASA (Dry)	3	15.28	--	2.86	81.63	1016	1163
		Complete (Dry)	4	15.47	--	2.70	81.73	1025	--
Phase II/2 Exposure Time =	1.71	On-Line (Wet)	1	NM	NM	NM	NM	NM	NM
		NASA (Wet)	3	13.54	11.40	2.53	72.32	900	1030
		Complete (Wet)	4	13.69	11.52	2.39	72.31	797	--
		On-Line (Dry)	1	NM	NM	NM	NM	NM	NM
		Grab (Dry)	2	14.58	--	6.23	79.16	668	--
		NASA (Dry)	3	15.28	--	2.86	81.63	1016	1163
		Complete (Dry)	4	15.47	--	2.70	81.73	901	--

ph:514b33

TABLE 12

Description of the Dynamic Unit
Products of Combustion Composition
Temperature: 2600° ± 50°F (1700K)
Velocity: 1000 ± 50 fps (305 m/s)

Phase/Part	A/F Ratio	Method	No.	Major Mole Percent				Minor ppm	
				CO ₂	H ₂ O	O ₂	N ₂	SO ₂	NO _x
Phase I Exposure Time = 79 Hours	2.12	On-Line (Wet)	1	17.00	7.60	13.50	61.85	450	14
		NASA (Wet)	3	10.67	7.98	6.57	73.85	117	1684
Phase II/1 Exposure Time = 168 Hours	2.29	On-Line (Wet)	1	11.60	7.10	7.43	73.83	370	9
		NASA (Wet)	3	11.09	9.31	5.50	73.91	110	1540
		On-Line (Dry)	1	12.49	--	8.00	79.47	398	10
		Grab (Dry)	2	12.33	--	8.84	78.85	193	--
		NASA (Dry)	3	11.74	--	5.82	78.23	116	1630
Phase II/2 Exposure Time =	2.29	On-Line (Wet)	1	11.60	7.10	7.43	73.83	370	9
		NASA (Wet)	3	11.09	9.31	5.50	73.91	280	1540
		On-Line (Dry)	1	12.49	--	8.00	79.47	398	10
		Grab (Dry)	2	12.33	--	8.84	78.85	193	--
		NASA (Dry)	3	11.74	--	5.82	78.23	309	1630
Phase III/1 Exposure Time =	2.20	On-Line (Wet)	1	10.36	7.90	7.60	74.14	774	32
		NASA (Wet)	3	12.56	9.85	5.84	71.53	750	1500
		Complete (Wet)	4	11.46	9.96	5.18	73.33	759	--
		On-Line (Dry)	1	11.25	--	8.25	80.50	840	--
		Grab (Dry)	2	11.50	--	9.91	78.89	776	--
		NASA (Dry)	3	12.56	--	5.84	79.34	832	1664
		Complete (Dry)	4	12.72	--	5.75	81.44	843	--
Phase III/2 Exposure Time =	2.20	On-Line (Wet)	1	NM	NM	NM	NM	NM	NM
		NASA (Wet)	3	12.56	9.85	5.84	71.53	750	1500
		Complete (Wet)	4	11.46	9.96	5.18	73.33	667	--
		On-Line (Dry)	1	NM	NM	NM	NM	NM	NM
		Grab (Dry)	2	11.50	--	9.91	78.89	776	--
		NASA (Dry)	3	12.56	--	5.84	79.34	832	1664
		Complete (Dry)	4	12.72	--	5.75	81.44	741	--

ph:514b34

phase of testing. NASA CCEC predictions were felt to be most representative because the on-line SO_2 levels were considerably higher than SO_2 levels expected for combustion of gas containing less than 330 ppm total sulfur as H_2S , COS , and CS_2 .

Methods one through three were used to determine exposure environment concentrations for the 100 to 400 hour C/E test period (phase II). During phase II the H_2S concentration in the fuel gas increased from <10 ppm to 536 ppm. Times of exposure at each of these conditions are included in these tables. Note that the gas concentrations are listed on a wet and dry basis. NASA CCEC and the C/E on-line instruments were able to determine the water content in the gas but the analytical gas chromatograph was not. In order to compare the grab sample measurements with the NASA CCEC and C/E on-line concentrations gas concentrations determined by the two latter methods are calculated on a dry basis. The grab sample analyses compared favorably with the NASA CCEC predictions for CO_2 and N_2 concentrations while the on-line N_2 and O_2 levels are comparable to the NASA CCEC predictions. As in phase I, the NASA CCEC predictions are considered to be representative of POC levels during phase II.

All four methods were used to characterize the POC during the last 600 hours of C/E testing (phase III). The 600 hours is divided into two parts because of a large variation of the H_2S levels in the fuel gas. The average H_2S concentration for the first 10 days of phase III was 2096 ppm and 1843 ppm for the remainder. NASA CCEC predictions for all gases except NO correlated well with the predictions of the complete combustion program. Grab samples indicated concentrations of CO_2 , N_2 , and SO_2 (except in the 2600°F static cell) that were within 20 percent of the values predicted by NASA CCEC. On-line measurements of N_2 , CO_2 , and SO_2 (except in 1850°F cell) were within 20 percent of NASA CCEC predictions. O_2 levels predicted by NASA CCEC correlated poorly with both on-line and grab measurements. Higher values of O_2 and N_2 and lower values of CO_2 may have been caused by air leaks in the sampling system. Because of the reasonable agreement between NASA CCEC, complete combustion program and the highly accurate analytical gas chromatograph, the NASA CCEC values for all gases except NO are expected to be realistic concentrations for the POC.

There was a large difference in NO levels as predicted by NASA CCEC and those measured by the C/E on-line analyzers. This can partly be explained by examining the probable way NO forms and its reaction kinetics. There were two sources of nitrogen during the combustion process which contribute to the formation of NO. Elemental nitrogen from the air forms "thermal NO", and nitrogen chemically bound in the fuel gas forms "fuel NO". During phases I and II, the amounts of nitrogen bound in the fuel (HCN and NH_3) were negligible. These gases were probably removed in the HSDU. For these test phases, the NASA CCEC was run with no fuel bound nitrogen. The program computes NO levels with combustion process continuing to equilibrium, with no attempt to determine the effect of rate of formation.

During the last phase, the HSDU was not used and there were measurable quantities of HCN and NH_3 in the fuel gas. This explains in part the higher values of NO during this phase.

When comparing gas concentrations with respect to cell temperature, the effect of dilution by excess air was evident. The CO_2 , SO_2 , and H_2O concentrations increased and O_2 levels decreased with rising temperatures because of less excess air. There is very little change in the equivalence ratios between the test conditions in the three static cells and the dynamic unit. This indicates that the concentrations of CO_2 , SO_2 , H_2O , and O_2 were greatly dependent on the amount of excess air present. The increase in POC water vapor with time is due to the increase in humidity of the combustion air and the lower A/F ratios used during phases II and III. SO_2 levels increased with time because of poor performance by the HSDU.

All C/E tests were conducted at near atmospheric pressure. In order to help compare actual turbine operating conditions, the NASA CCEC was also run at a pressure of 20 atmospheres (2026 kPa). This was used to compare the differences in products of combustion of the same gas combusted at two different pressure levels at and the same temperature. Their respective concentrations are shown in Table 13.

TABLE 13
Comparison of POC Levels at Different Pressures

Pressure atm (kPa)		Gas Concentration						
		Majors				Minors		
		Mole Percent				ppm		
		CO ₂	H ₂ O	N ₂	O ₂	SO ₂	SO ₃	NO
1	(101)	7.61	7.27	75.07	9.98	490	20	290
20	(2027)	7.61	7.27	75.07	9.97	430	80	290

The only significant difference between the POC at the two conditions is the partitioning of the sulfur-containing gases. At higher pressures, the SO₃ level increased by four-fold while the SO₂ level dropped to approximately 88 percent of the level at the lower pressure. Effect of SO₃ upon metal surfaces is thought to be highly corrosive, but its effect on ceramic surfaces is uncertain.

Aerosol Data

Aerosol data was gathered and analyzed by METC Analytical Sciences Branch (References 10, 11, and 12). The references describe the aerosol samples taken from the coal-derived fuel gas, dynamic unit's combustion air and POC, and a bench-scale burner which was operated in parallel with the dynamic and static units. There were no aerosol samples taken from the POC from static test cells. Only the dynamic unit POC aerosols are described here. Sampling was done downstream of the test specimens.

During phase I, aerosol data was solely derived from spectro-grade glass fiber filters (Gelman No. 64878-47 mm). Scanning electron microscopic (SEM) analysis indicated large quantities of very small particles. The crustal appearance implied that the agglomeration occurred during collection which leads to the speculation that the aerosols were very small (<0.5 μ diameter). The energy dispersive X-ray (EDX) analysis revealed that the major constituents were Si, Cr, Fe, Cu, and Zn. EDX analysis does not detect elements having atomic numbers greater than Na. Gas/liquid chromatographic analyses of the extracts from the filters showed no significant concentrations of organics.

In phases II and III, two types of aerosol filters (spectro-grade glass fiber filters, Gelman No. 64878-47 mm, 0.45 and 5.0 μm pre-size silver membrane filters, Selas No. FM47) were used for mass loading and subsequent chemical analyses. The SEM analysis indicated also in phases II and III large quantities of small particles. The bulk of aerosols in phase II was attributed to the degradation of the alumina refractory liner of the dynamic unit with traces indicated by X-ray diffraction of Si, S, and Ca. In phase III, the aerosol loading dramatically increased. The chemical analyses indicated that the aerosols consisted mostly of sulfur or sulfur compounds with traces of chlorine present.

The mass loadings in all three phases are shown in Table 14.

Aerosol size distributions were done in phases II and III using an electrical aerosol analyzer (EAA), TSI Model 3030, and a quartz crystal microbalance (QCM) impactor, Berkeley Controls Model C-1000A. In phase II, the aerosol concentration was $\sim 2 \times 10^7$ particles/cm³ with mean diameter of 0.05 μm . In phase III the QCM impactor indicated a large portion of the aerosols $< 1 \mu\text{m}$ in diameter. Extreme fluctuation in concentration prevented EAA data from being obtained.

At the end of the 1000-hour test period, several types of deposits were noted in various locations of the C/E test facility. A reddish-brown solid was removed from the inside of the water-cooled front dynamic viewing port. This was analyzed by x-ray fluorescence spectroscopy and was found to contain amounts of CaSO_4 and $\text{FeSO}_4 \cdot \text{H}_2\text{O}$. A minor amount of Se and trace quantities of Na and K were also found. A possible source of these compounds was caused by cooling water that leaked through the viewport and then reacted with SO_2 in the POC.

TABLE 14
Aerosol Mass Loading

Phase	Date	Time		Flow Rate		Mass Loading	
		On	Off	scfh	m ³ /s	µg/m ³	Filter
I	11/16/80	1003	1008	40	3.16×10^{-4}	13 500	GF-BB
	11/18/80	1830	1930	40	3.16×10^{-4}	7 700	GF6
	11/19/80	0912	0929	37	2.92×10^{-4}	13 600	GF3
	11/19/80	1207	1257	28	2.19×10^{-4}	8 100	GF5
	11/20/80	1411	1501	28	2.19×10^{-4}	8 100	GF9
	11/21/80	0903	0953	28	2.19×10^{-4}	11 300	GF12
	11/21/80	1130	1220	28	2.19×10^{-4}	11 300	GF13
II	06/01/81	1015	1120	14.1	1.11×10^{-4}	2 840	AG2
	06/02/81	0933	1249	20.5	1.61×10^{-4}	4 450	AG3
	06/03/81	1310	1510	18.7	1.47×10^{-4}	2 180	AG4
III*	07/22/81	0925	0935	14.1	1.11×10^{-4}	261 163	AG6, AG7
	07/22/81	0949	0959	12.0	9.42×10^{-5}	293 039	DM8, DM9
	07/27/81	1025	1320	11.4	8.95×10^{-5}	78 067	AG23, AG24
	07/29/81	0914	1334	10.2	8.01×10^{-5}	82 656	AG20, AG25

GF = Glass fiber.

DM = Polyvinyl chloride.

AG = Silver.

*Phase III samples gathered using two filters in series.

Material Test History

The ceramic test materials, as described in Section II, were sent by their respective material contractors (Garrett and Westinghouse) to IITRI prior to being received at METC. At IITRI they were given a pre-exposure screening, identification markings, and visual as well as x-ray inspections. Additionally, they were photographed and records of weight, dimensions, density, and porosity were made. Each of the material specimens were packaged individually in a paper envelope. No special precautions against humidity, temperature, or oxidation were made. METC received the material samples in September 1980. They were stored in their individual envelopes until they were loaded into the METC test facilities. Cotton photography gloves were worn by all personnel who handled the specimens.

Static Materials

A total of 288 Westinghouse and 216 Garrett material specimens were received for testing in the static test unit. This number represented 36 individual specimens of each of the 14 ceramic material types. The specimens were divided into nine equal groups and placed on nine test racks, i.e., four specimens of each of the 14 materials, a total of 56 specimens, for each of nine racks.¹ Specimens were placed on each rack in a random order which was determined using the specimen identification number and the Table of Random Units, Column 14, from Reference 14. Figures 4 through 12 show the arrangements of the test specimens on the nine static unit racks. Table 15 describes the test conditions prescribed for each of the nine racks and indicates the actual exposure time achieved at those conditions. Figure 13 is the summary of the static specimens exposure times for specific temperature ranges.

---

¹Four proprietary Department of Defense materials were placed on rack No. 1 in addition to the other 56 samples. These were carbon-carbon materials and were to receive a limited exposure time at 2600°F (1700°K).

FIGURE 4

STATIC FACILITY SPECIMEN ARRANGEMENT

TEMPERATURE 2600

DURATION: 100

HOURS

RACK (TEST CONDITION): 1

FIGURE 5

STATIC FACILITY SPECIMEN ARRANGEMENT

TEMPERATURE 2200

DURATION: 100

HOURS

RACK (TEST CONDITION): 2

FIGURE 6

STATIC FACILITY SPECIMEN ARRANGEMENT

TEMPERATURE 1850

DURATION: 100

HOURS

RACK (TEST CONDITION): 3

FIGURE 7

STATIC FACILITY SPECIMEN ARRANGEMENT

TEMPERATURE 2600

DURATION:300

HOURS

RACK (TEST CONDITION): 4

FIGURE 8

STATIC FACILITY SPECIMEN ARRANGEMENT

TEMPERATURE 2200

DURATION: 300

HOURS

RACK (TEST CONDITION): 5

FIGURE 9

STATIC FACILITY SPECIMEN ARRANGEMENT

TEMPERATURE

1850

DURATION: 300

HOURS

RACK (TEST CONDITION): 6

FIGURE 10

STATIC FACILITY SPECIMEN ARRANGEMENT

TEMPERATURE 2600

DURATION: 1000

HOURS

RACK (TEST CONDITION): 7

FIGURE 11

STATIC FACILITY SPECIMEN ARRANGEMENT

TEMPERATURE

2200

DURATION:

1000

HOURS

RACK (TEST CONDITION): 8

FIGURE 12

STATIC FACILITY SPECIMEN ARRANGEMENT

TEMPERATURE 1850

DURATION: 1000

HOURS

RACK (TEST CONDITION): 9

FIGURE 13 Static Accumulated Exposure Time vs. Temperature

TABLE 15
Static Facility Accumulated Exposure

Rack Number	Cell Number**	Total Exposure Time			Total
		Phase I	Phase II	Phase III	
1	3	62	*	*	62
2	2	98	*	*	98
3	1	99	*	*	99
4	3	*	268	*	268
5	2	*	299	*	299
6	1	*	306	*	306
7	3	62	268	482	812
8	2	98	299	604	1001
9	1	99	306	601	1006

NOTE: All time units in hours.

* No exposure time during this phase.

** Cell 1 -- 1850°F (1283K)

Cell 2 -- 2200°F (1477K)

Cell 3 -- 2600°F (1700K)

The static test unit has a loading capacity of two racks in each of its three temperature chambers. The three 1000-hour duration racks (Nos. 7, 8, and 9) were paired with the three 100-hour duration racks (Nos. 1, 2, and 3) and placed into the three temperature cells to begin the test operation. When the 100-hour duration was met, racks 1, 2, and 3 were removed and replaced by the 300-hour duration racks (Nos. 4, 5, and 6). Once their 300-hour duration was achieved, they too were removed leaving racks 7, 8, and 9 alone in the facility. After 600 hours further duration, racks 7, 8, and 9 had reached their 1000-hour duration, thus completing the test of the static unit materials. Unfortunately, the test sequence and rack rotation did not occur in a smooth progression. A record of the events leading to the conclusion of the test is as follows.

Phase I -- At 1:00 p.m. on November 15, 1980, the burners in the static test cells were ignited, thus initiating the materials exposure test. After 14 hours, cells 1 and 2 reached their respective operating test temperatures. After 34 hours, cell 3 reached its operating test temperature. After 50 hours, fuel gas was lost for a short period, and all three cells dropped below operating temperatures to approximately 1600°F (1144K). After 58 hours, cells 1 and 2 again reached operating temperatures. However, before cell 3 could reach operating range, fuel gas was again lost (after 64 hours of operation), and the three cells dropped as low as 1200°F (922K) before gas was available again. After 78 hours, cells 1 and 2 again reached operating temperatures. Eight (8) hours later, cell 3 also reached operating temperatures. Then after 102 hours of operation, fuel gas was lost again, and cell temperatures dropped to about 600°F (589K). When fuel gas was regained, cells 1 and 2 reached operating temperatures after 120 hours of accumulative operation. After 124 hours, cell 3 also reached its operating temperature. When 154 hours of operation were reached, cells 1 and 2 had accumulated about 100 hours of test exposure. Cell 3 had accumulated only 62 hours of test exposure. The cells were allowed to return to ambient temperature at this point in order to make the first rotation of the racks. As described above, these first six racks had undergone a total of four thermal cycles into and out of their respective steady-state conditions.

At this juncture in the testing, the specimens were inventoried on these six racks. All specimens were present from racks 3 and 9 in cell 1 and there was no deformation of the test racks. In cell 2, one "B" material specimen (B22) was lost from rack 2 (100-hour) and none were missing from rack 8 (1000-hour). Both racks were in good condition with only slight deformation of the support members. In cell 3, five samples were missing including one "C" material sample (C9) and two of the DOD carbon-carbon samples (A102 and A103) from rack 1 (100-hour). One each of "B" and "C" samples (B37 and C11) were gone from rack 7 (1000-hour). Each of the racks from cell 3 was extremely deformed. Rack 7 specimens were transferred to a replacement rack and placed back into cell 3 to continue testing. Figures 21 through 23 are photographs of the static specimens after the completion of phase I.

Excluding the DOD samples, which were not being characterized, only "B" and "C" materials were lost. The "B" materials which remained on the racks showed no characteristic changes. However, the remaining "C" materials did show a characteristic condition. These specimens were swollen with signs of separation at all edges. This condition was much more pronounced in cell 3 where actual separation into parts occurred. The separation typically was at a 45 degree angle to each plane, leaving six, four-sided pyramid-shaped pieces.

Phase II -- Racks 1, 2, and 3 were replaced with racks 4, 5, and 6 respectively just prior to continuation of the test operation. On December 4, 1980, testing was started. However, mechanical problems developed in the gas producer shortly after start-up forcing an immediate shut down. Testing was postponed for five months as repairs were made. Several observations were made following the abbreviated test period. All the racks were removed for inspection and inventory. Racks 4, 5, 6, and 9 had all their specimens present and no visible changes. But rack 7 (cell 3) had several support members deformed and while removing it from the test cell, seven specimens (A18, E18, F16, I13, M18, M19, and M21) fell off into the cell. Six of these were recovered with no apparent damage, but sample E18 was shattered. The six recovered specimens were placed back in their original rack positions.

As rack 8 was removed from cell 2, sample B32 fell off the rack and was broken. Thus, one specimen of each of the "B" and "E" materials was lost to further testing. It was again required to transfer the specimens of rack 7 to a new rack. Note that although the test cells did not quite reach operating temperatures, racks 7, 8, and 9 were subjected to a fifth major temperature cycle. Racks 4, 5, and 6 suffered their first thermal cycle.

On May 29, 1981, fuel gas was available once again and the testing was continued. On June 16, 1981 (435 hours later) the static facility was shut off and allowed to cool to ambient as racks 4, 5, and 6 had reached their 300-hour exposure times. The facility had again experienced fuel supply and maintenance problems which caused some further thermal cycling. All six racks suffered six major temperature cycles (i.e., a temperature change greater than 1000°F [811K]) and eight minor temperature cycles. In a related incident, one of the major temperature cycles occurred after 100 hours of the operation when an unknown amount of Stretford solution* contaminated the cells by way of the fuel lines.

The mishap occurred when a droplet separator in the gas cleanup system overfilled, allowing the solution to pass through the fuel gas lines and into the burners. The problem lasted only a few hours before it was discovered and corrected but repair of fouled instruments extended the non-operating period to about 12 hours.

When the static unit was cooled following this operation, the racks were removed for inspection. Unloading began with cell 1 where it was observed that racks 6 and 9 (Figures 26 and 29) were covered with a brown-purple discoloration or residue. Other than the change in color, the two racks' surfaces still appeared smooth. The specimens themselves in many cases were bubbled with the appearance of residue on their surfaces. Each rack had multiple fractures to their support members.

---

*Stretford solution is an aqueous solution containing Vanadium and sodium compounds.

Rack 6 had one support member broken off which allowed four specimens (H29, H30, I35, and J41) to drop off. Specimen H30 and the broken support member were caught below on rack 9 when they fell, specimen H30 landed atop sample E37 of rack 9 and left a blue "foot print" on that specimen. The broken rack piece caught on rack 9 specimens F33, G24, F34, and K24, but left no apparent imprint. The other three specimens were found unbroken in the bottom of the test cell where it is estimated they spent the last 30 hours of the test exposure. Cell 2 was unloaded next. Figure 25 is a photograph of rack 5 specimens. They were removed from their original test rack and placed on a new replacement rack for this photograph. Figure 28 pictures the original rack 8 with its specimens. Discoloration and residue were much less apparent than in cell 1 but still visible on this rack. Specimen surfaces are also less rough in appearance than those of cell 1. When cell 3 was unloaded, it was noted that the racks were extremely deformed. Rack 4 specimens are pictured in Figure 18. They were placed on a new replacement rack for this photograph. The original rack had several broken and deformed parts which allowed five specimens (A33, E29, J16, L28, and N28) to fall to the cell floor. Two of these specimens (L28 and N28) fell and fused together. The E29 specimen was damaged (chipped) on one end. Figure 19 shows rack 7 specimens. They were placed on original rack 5 used only to set up these specimens for photographing. Both racks removed from cell 3 showed little visible signs of contamination on either of the racks or the specimens. Thus, all static unit specimens survived this 300-hour operation even though some fell off the racks and had to be recovered from the bottom of the cell. Racks 7*, 8, and 9 were placed back into the test cells, alone, to receive their final 600 hours of exposure time. At this point, the specimens had been subjected to a total of 19 thermal cycles. Figures 24 through 26 are photographs of static specimens after Phase II.

Phase III -- On July 16, 1981, fuel gas was available once again and the 600-hour phase of testing began. Five (5) weeks later on August 19,

---

*Rack 7 was modified somewhat when an additional centerspan support member was added to the rack to improve its strength.

the test was terminated when racks 7, 8, and 9 achieved their goal of 1000 hours duration of exposure. The racks went through three more major temperature cycles and 10 more minor temperature cycles. This brought the total number of thermal cycles to 33 for racks 7, 8, and 9. The number was significant, not only to the test racks, but also to the test facility as well. Including checkout phases, the static unit had undergone more than 50 thermal cycles. During the final 100 hours of this final test operation, a portion of the interior lining of cell 3 failed due to these severe conditions. Three pieces of dense refractory, weighing from 5 to 10 pounds, fell a distance of about 12 inches onto rack 7. Of the 53 test specimens on rack 7, twenty-five remained on the rack and twenty-eight fell to the floor of the cell. Only five samples which fell to the floor were broken -- three "C" and two "B" materials. Every specimen was recovered, but the rack was totally destroyed. The racks removed from cells 1 and 2 were not damaged. Only rack 8 was missing any specimens, C12 and C13, both probably due to exposure conditions. Figures 27 through 29 are photographs of the static specimens after phase III.

Racks 8 and 9 which had been contaminated and discolored during phase II testing showed no signs of increase or decrease of those conditions at the conclusion of phase III testing.

Other than the failure of the refractory lining of cell 3, no significant occurrences were observed in this concluding test phase. When racks 7, 8, and 9 were removed from the facility, a final inventory was made. Of the 504 samples exposed, 496 survived the testing, intact. Although there were eight specimens damaged, only four were probably due to the exposure environment. All four belonged to material group "C" (CNTD AlN coating on HP-AlN). The only material to exhibit what appeared to be sag or creep was material group "D" (HP-Al₂O₃ with ZrO₂ particulates). This observation was made at 400 hours of exposure, but there seemed to be no further progression following another 600 hours of exposure.

FIGURE 14

DYNAMIC FACILITY SPECIMEN ARRANGEMENT

1000 HOURS DURATION

* INDICATES THE SPECIMEN WAS REPLACED

VIEW IS THE UPSTREAM SIDE OF SPECIMEN

Dynamic Materials

The dynamic test was conducted, for the most part, concurrently with the static test. A total of 51 test samples were received for testing in the dynamic unit. That number represents only 11 of the material types which were tested in the static unit.¹ Included are five each of "A"

TABLE 16

Dynamic Specimen Exposure Time/Specimen Holder Position

Specimen Number	1	2	3	4	5
Material Type	Exposure Time/Position				
A	849/1*	820/10	***	***	***
B	---	---	28/10	---	---
C	305/5	614/5	78/12	30/5*	42/5
D	809/3	***	***	6/3	32/3
E	0/1	32/1	6/1	38/1	1/7
M	32/8	817/8	***	***	***
Specimen Number	51	52	53	54	55
G	849/2	780/7	***	***	***
H	849/2	***	***	***	***
I	78/9	780/9	***	***	***
K	***	***	441/12*	638/6	405/12
N	849/4	***	***	***	***

* Phase II position, see Figure 8 for Phase I position.

--- No samples received.

*** Not exposed.

Exposure time in hours.

¹Materials "F," "J," and "L" were not available in the configuration required for the dynamic test unit.

FIGURE 15 Dynamic Accumulated Exposure Time vs. Temperature

"C" "D" "E" "G" "H" "I" "K" "M" and "N" materials and one "B" material specimen. The first 12 of these material specimens were positioned in the dynamic test section randomly using their identification numbers and the Table of Random Units, Column 14, Reference 14. Numbers 1 through 11 were selected, noting that 1 is material "A," 2 is material "B," and so on, skipping past materials "F," "J," and "L." Figure 19 shows the initial specimen arrangement and subsequent replacements for the entire test period. Table 16 lists the exposure time accumulated by each of the specimens. The loading procedures for the dynamic test are discussed later in this report.

Phase I -- The dynamic test unit was brought to test conditions shortly before midnight on November 15, 1980. By midnight the specimens had preheated and were placed into exposure position in the POC gas stream. During this placement, a single sample, E1 in holder 1, was dislodged from its holder. It was immediately replaced by sample E2. After 39 hours of operation, fuel gas was lost, and the dynamic unit was shut down along with the static unit. Prior to this shutdown, excess cooling water downstream of the test section had been accumulating in a preheat air recuperator cooling system. When the gas flow stopped, water in the form of steam entered the test section. This event resulted in the loss from their respective holders or damage of five of the 12 specimens. Specimens D5 and M1 were completely lost from holders 3 and 8, respectively. Specimens E2 and B3 in holders 1 and 10 had parts of their exposed ends broken off. And specimen C3 in holder 12 had multiple, longitudinal cracks and swelling along its exposed end. Specimens E2, D5, M1, and B3 were replaced with specimens E3, D4, M2, and A2, respectively. All specimens were withdrawn from the gas stream until the facility could reach test conditions again. After the facility lost fuel gas once again and was brought up to test conditions a third time, two more specimens were broken off. Those specimens, E3 and D4, were replaced by E4 and D1, respectively. At the 100-hour exposure plateau, the dynamic facility was shut down along with the static unit. The specimens were inspected and two (C3 and E4) were found to be eroded and broken. They were replaced by C4 and E5. As in the static test, the

dynamic test specimens had experienced four temperature cycles. Nineteen (19) specimens representing 11 materials had been exposed. Six of these specimens from five materials survived the exposure from the start. The "B" material was eliminated when its lone specimen was destroyed in the steam incident. The last "E" material was placed in its holder awaiting continuation of the test.

Phase II -- When the test was continued on December 4, 1980, specimen I51 was blown out of its holder by a sudden burst of cooling air. Cooling air was used to maintain the stainless steel holder temperature to <1000°F (811K). It was replaced by I52. Seven (7) hours after start-up, specimen E5, the final "E" material, disappeared. This specimen was replaced by G52. A few hours later, the fixed-bed gasifier failed, and the test was temporarily discontinued. On May 29, 1981, fuel gas was available again and the testing operation was continued. After 318 hours of operation, the dynamic unit was shut down along with the static unit again. During the operation, the "C" material specimens were replaced on three occasions. Specimen C4 had broken off 2 inches from the exposed end after only 12 hours of exposure. Its replacement, C5, survived 22 hours before it broke off 1 inch down from its exposed end. The replacement, C1, survived about 100 hours more of exposure. When the Stretford solution entered the test section, no specimens were lost. Specimen C1 was removed and sent to IITRI for analysis of the contaminant. The specimen had been selected because it was not expected to survive further exposure. C1 was replaced by C2. No other samples required replacement for the duration of this test phase, but specimens D1 and M2 began bending to about 45 degree angles in the direction of the downstream flow. All the other samples (A1, G51, N51, K54, G52, I52, A2, H51, and K53) showed no visible signs of fatigue or deformity. The specimens were left in their positions at the end of this test period to await the start of the final test period. At this point, the facility had provided approximately 400 hours of exposure time at test conditions.

Phase III -- Then on July 16, 1981, the final test period begun as material testing continued. The dynamic unit was operated for 762 hours and achieved about 500 hours more exposure time before it was shut down along

with the static unit. Thus, the dynamic unit fell short 100 hours of reaching the required 1000-hour accumulative exposure plateau. Only three specimen changes were made in this final operation. Specimen K54 was replaced by specimen C1 at the very start of this operation. C1, had been sent to IITRI for analysis and was returned in time to begin this operation. Specimen K54 was recovered from the facility before test conditions were reached. When K53 was blown from its holder early in the run it was replaced by K55. As the test progressed, C1 degraded and completely eroded away at the exposed end. It was replaced by K54 which had earlier been lost and recovered. Specimen C2 had also been eroded away but was left in place until the conclusion of the run. Specimen K53 was later recovered from the facility at the conclusion of testing. Specimens D1 and M2, which had bent to 45 degree angles during the first 400 hours of test exposure, deformed very little beyond that initial shape. A total of 27 specimens had been placed in the dynamic unit and exposed. The total hours of exposure accumulated by each specimen is given in Table 16. Figure 30 shows the dynamic specimens after phase III.

IV. FACILITIES DESCRIPTION

Static Facility

The major components of this facility were the test cells, test specimens racks, exhaust section, and air/fuel system. The static facility process flow sheet is shown in Figure 21 and the facility itself is shown in Figure 22.

Static Test Furnace

The furnace consisted of four separate equal volume test cells enclosed in a single-steel housing. The cells were constructed with inner walls of high-density (85 percent) alumina. In addition, there was insulating brick that separated each cell and protected the steel housing. The exterior metal temperature was designed to be less than 300°F (422K) during operation. The ceiling of each cell was cast in one piece. Each

FIGURE 16   **STATIC FACILITY**
SIMPLIFIED PROCESS FLOW SCHEMATIC

FIGURE 17 Static Facility

cell had a top-fired, single nozzle-mix burner. Three used coal-derived gas, while the fourth used natural gas.

In addition, the fourth cell housed the static unit's preheated combustion air heat exchangers and helped maintain the temperature of the facility. The burners were placed about four feet (1.22 meters) above the higher of the two specimen racks to prevent flame impingement on the specimens. This also provided uniform velocity and temperature profiles at the test specimens. A viewport was built into the roof of each cell to allow a view of the upstream side of the specimens.

Static Test Specimen Racks

There were two levels of supports for the test specimen racks in each of the test cells. The specimens were positioned on the upper and lower racks to allow unobstructed POC flow and visual inspection from the viewports.

The racks were constructed of high-alumina tubes and each held up to 57 specimens. The tubular construction assured that there was only line contact between the rack and the specimen.

Static Air/Fuel System

Preheated air and coal-derived gas were mixed for combustion in three test cell burners while natural gas and a separate air supply were combined for combustion in the fourth.

The combustion air for the three test cells was maintained at a near constant mass flow rate. Temperature was controlled by automatically adjusting the fuel gas flow rate. The air was supplied at a constant rate by a 500 scfm (0.236 std.m³/s) compressor and divided equally between the three cells by a manifold system. Once the air was divided, it entered cell 4 where it was preheated to 500°F (533K) prior to entering the burners.

The gas flow rate was measured by an orifice meter. But a closed loop control system based on temperature feedback was used to control the pressure (flow rate) through each fuel gas regulator.

The nozzle-mix burners were made by Pyronics with modified air tubes to permit the use of low heating value fuel gas. The three (3) different maximum heating capacities of the burners were 775,000, 650,000, and 525,000 Btu/hr (227, 190, and 154 kw) respectively. The pilot flames, as well as the fourth burner, operated on natural gas and a separate air supply.

Exhaust Section

The four cells shared a common exhaust flue located in the center of the furnace. Air was blown in through a duct in the center of the furnace floor to control the draft and to cool the exhaust POC. The exhaust was released to the atmosphere through an exhaust stack.

Dynamic Facility

The major components of this facility were the combustion chamber, test section, specimen holders, exhaust section, and the air/fuel system. The dynamic facility is shown in Figure 23 and the simplified process flow sheet is shown in Figure 24.

The chamber consisted of the burner section, the combustion section, and the transition section. The chamber created a combustion area with a diameter of 42 inches (1.07 meters) and a length of 51 inches (1.3 meters). The metal housings of each section were bolted together. The refractory insulating material inner layers were overlapped to reduce heat paths to the metal housing.

The frustrum-shaped burner section had six equally spaced nozzle-mix burners offset by 30 degrees which produced a swirl or vortex-type pattern of flow. A viewport was located in the center of the flat end of the section and allowed observation of the upstream side of the specimens. The viewport was water cooled.

FIGURE 18 DYNAMIC FACILITY
SIMPLIFIED PROCESS FLOW SCHEMATIC

FIGURE 19 Dynamic Facility

The range cylindrical-shaped combustion section was of sufficient length so that near complete combustion could take place and direct flame impingement on the specimens was prevented. Pressure wall taps and redundant temperature probes measured the internal gas temperature. Thermocouples also measured the refractory temperature.

The POC was accelerated to the design velocity in a standard ASME nozzle. The test specimens were positioned inside the constant area nozzle throat. The pressure wall tap in the nozzle throat and wall tap in the combustion section provided the signal for velocity control.

Dynamic Test Section

The cylindrical-shaped test section (inside diameter of 5.5 inches [0.14 meters]) had 12 equally spaced radial ports through which the test specimen holders positioned each specimen. Refractory material was removed which tapered upstream and downstream of the specimen. This created an area approximately equal to the cross-sectional area of the 12 specimens. This provided a constant flow velocity by maintaining the same cross-sectional area for the POC.

Dynamic Test Specimen Holder

The test specimen holders were air-cooled, stainless steel devices (Figure 25) which were individually inserted or removed from the test section at all stages of operation. The test specimens were held in place by the four clamping jaws of the specimen holder much like a mechanical drafting pencil holds its lead. Four spring-loaded jaws provided a uniform clamping force around its circumference along a 0.5 inch (.013 meter) length of the fixed end of the specimen. A layer of alumina paper between each jaw and around the end of the specimen reduced any chemical interaction between the specimen and the metal of the holder. A fully inserted position exposed approximately 2.25 inches (0.057 meter) of the specimen to the POC flow. The holders also permitted safe storage of the specimen while out of the test section.

FIGURE 20 Dynamic Facility Specimen Holder

Dynamic Exhaust Section

This section consisted of the catcher section, elbow section, extension section, and the recuperator. In the catcher section, an expansion nozzle transformed the 5.5 inch (0.14 meter) diameter to a 12 inch (0.305 meter) diameter. This section also had a trap to recover broken specimens and two viewing ports of 2 inch (0.05 meter) diameter for downstream observation of the specimens. The elbow allowed the POC to flow from the catcher section to the extension section. The extension section contained a spray ring to quench the POC gas temperature to approximately 1800°F (1255K) before entering the recuperator. All sections up to the recuperator were refractory lined. The recuperator heated the combustion air to approximately 500°F (533K). Another spray ring after the recuperator further cooled the POC gas down to 500°F (533K) before exiting to the atmosphere through the stack. A metal bellows allowed for expansion of the piping after the recuperator.

Dynamic Air/Fuel System

Preheated air and coal-derived gas were mixed in the six burners. The flowrates as well as the A/F ratio were controlled to maintain the specified test and velocity temperature.

The combustion air was taken from the test area and passed through a turbo-compressor which was rated at 1450 cfm (0.683 m³/s) at 100 inches water column (24.8 kPa). The turbo-compressor passed the outlet air through the exhaust gas-to-air recuperator which heated the air to approximately 500°F (533K). The preheated air then moved through an automatic damper system which regulated the pressure of the air to 2.5 psig (17.2 kPa). The excess air was bled into the exhaust system. The combustion air temperature and pressure was controlled as well as its flowrate. The hot air flow was divided into three separate pipelines. In each line there was a control valve which maintained the required air flowrate. The butterfly valve was slaved by a mechanical linkage to a mating butterfly valve in the fuel gas pipeline. After the butterfly valve, each of the three lines were divided again to supply air to each burner pair.

For control purposes, as well as analysis, the flowrate, pressure, and temperature of the coal-derived gas were all measured. The gas pressure was then regulated to set the air/fuel ratio and controlled by the temperature measurement from the test section. The flow was divided into three separate pipelines. Each line had a manually tripped safety valve and a control valve which controlled flowrate. The control signal for the valve came from the differential pressure reading from the dynamic test unit. Each pipeline was further separated so that each supplied one pair of burners. A solenoid valve was used to shut off each pair of burners.

The burners were 4 inch (0.1 meter) Maxon series "WR" Wide-Range burners specially drilled for low-Btu gas. These burners were a nozzle mixing type with multiple-stage, burner block tunnels. Each burner was rated at 1,000,000 Btu/hr (293 kilowatts). The pilot assemblies operated on natural gas and separate air supply. Each burner was equipped with an Ultra Violet (UV) flame detector which controlled the manually reset safety valves. These detectors shut off the fuel gas in the event flame was lost. Additionally, if the pilot flame was lost, a solenoid closed the natural gas line.

LOADING PROCEDURES

To establish the best possible material exposure data base, it was vital that the maximum number of exposed ceramic specimens be returned to IIT Research Institute for analysis. Thus, careful handling of the materials before, during, and after test exposure was imperative. More than 500 material test specimens were placed in the static and dynamic test facilities at METC and tested. Better than 95 percent of those were successfully returned for analysis at IITRI. Less than 5 percent of the tested specimens were damaged or destroyed due to test exposure conditions and handling. This section describes the handling procedures employed in loading and unloading the test specimens for each of those facilities.

Static Unit -- The configuration of the specimens tested in this facility is described in Section II of this report. These specimens were removed from their shipping packages and four of each material type were placed randomly on each of nine support racks. This procedure is described in Section III under Static Materials. Each specimen placed on the first six test racks was cemented in place using Ultra Bond 552 Ceramic Adhesive*. The adhesive was used to prevent the specimens from falling off the racks during transport or testing. However, when the first six racks of specimens were removed from the facility following 100 hours of exposure, it was found that the cement had failed in bonding the specimens and the rack parts. Some specimens had moved out of their original positions and onto their sides as though they had "popped" free. In sections of the racks where the cement held, the rack parts were broken. The ceramic adhesive was not used for the remainder of the test for either the racks or the specimens. When testing in this facility was concluded, 504 specimens had been exposed and 496 survived. Eight specimens were destroyed, four due to exposure failure and four due to droppage from the racks. Each surviving specimen was carefully placed back into its shipping package and returned to IITRI.

Dynamic Unit -- The configuration of specimens tested in this facility is described in Section II of this report. These specimens remained in their shipping envelopes until they were individually removed and placed in 1 of the 12 specimen holders. The procedure of specimen positioning is outlined in Section III under Dynamic Materials. Unlike the static unit racks which held 57 specimens each, the dynamic unit specimen holders held only 1 specimen each. However, their design permitted immediate placement or removal of individual specimens during test operations without disturbance to the other specimens. The holder is a

---

*This cement was also used in the construction of the support racks which were made from 98.9 percent Al_2O_3 ceramic tubes. The support rack is detailed on METC drawing number D790099.

spring loaded, clamping device, similar in operation to a mechanical pencil, and loaded as follows. One-half ($\frac{1}{2}$) inch of the numbered end of a ceramic specimen was first wrapped with two layers of 0.5 mm thick ceramic paper. It was then placed into the depressed specimen holder clamp. The clamp was then released exerting a holding force of about 10 psi. The ceramic paper was used to prevent bonding of the specimen to the metal clamp. The specimen was then aligned and a metal seal installed at its base in the clamp. Once the specimen was thus secured in the holder, the holder was mounted into 1 of 12 ball valve assemblies located on the circular test section of the facility. The final step in loading required the dynamic unit to be at test conditions. When those conditions were reached the ball valves in a closed position up to this point, were then opened. One at a time the end of each holder, preceded by its specimen, was passed through the ball valve to a point where the tip of the specimen was within the refractory wall but out of the POC gas stream. The specimen was left in this position for several minutes until it was preheated to at least 1000°F. Then the specimen was gradually inserted into the POC gas stream until approximately $2\frac{1}{4}$ inches (0.057 m) of its end was exposed. Unloading was accomplished by reversing the above procedure. Twenty-seven (27) specimens were placed into the test section in the course of this 1000-hour test. Seventeen (17) of those specimens were either damaged or destroyed due to handling or exposure conditions. Those 17 losses occurred as follows. Eight were degraded or damaged due to the exposure conditions, five were damaged by the accidental introduction of steam into the test section, and four were lost due to the failure of the specimen holders to sufficiently clasp the base of the specimen.

At the conclusion of testing, all surviving specimens and recovered specimen parts were placed in their original shipping packages and returned to IITRI.

Figure 21 Static Rack 1 after
Phase I (2600°F/62 Hours)

Figure 22 Static Rack 2 after
Phase I (2200°F/98 Hours)

Figure 23 Static Rack 3 after
phase I (1850°F/99 Hours)

Figure 24 Static Rack 4 after
phase I (2600°F/ 268Hours)

Figure 29 Static Rack 9 after
phase III (1850°F/1006 Hours)

Figure 30 Dynamic Specimens
after phase III

REFERENCES

1. "Final Report, Concept Feasibility Study of Ceramics for Use in Electric Utility Gas Turbines Fired with Coal-Derived Fuels," by Committee on the Use of Ceramics in Industrial Gas Turbines, University of Pittsburgh, Pittsburgh, Pennsylvania. Report SETEC DO 76-024F, dated October 1976.
2. Rieke, K. L. (Westinghouse Electric Corporation), November 1979, "Ceramics Technology Readiness Development Program, Phase 1: Conceptual Designs and Material Screening," Final Report, U.S. Department of Energy Report No. FE-2786-38, dated November 1979.
3. Elkins, R. T., B. B. Heath, and T. E. Yonushonis, "Innovative Design of Ceramic Utility Gas Turbines," ASME Journal of Engineering for Power, October 1979, Volume 101.
4. Larsen, D. C. and J. W. Adams (IIT Research Institute), "IITRI-AFWL Contributions to DOE-CTR Program, METC Corrosion/Erosion Tests: Evaluation of the Corrosion/Erosion Behavior of Various Ceramic Materials," dated May 1, 1980.
5. Nakaishi, C. V. and L. K. Carpenter, "Ceramic Corrosion/Erosion Project Description," Report No. DOE/METC/SP-111, U.S. Department of Energy, dated February 1981.
6. "Topic Report: Fixed-Bed Gasification," by Morgantown Energy Technology Center, Report No. DOE/METC/SP-184, dated January 1981.
7. Panson, A. G., and J. J. Kovach, "Analytical Data -- Producer/Full-Flow Cleanup System -- Run No. 96," METC IR No. 977, dated February 25, 1981.
8. Panson, A. G. and J. J. Kovach, "Analytical Data -- Producer/Full-Flow Cleanup, Run No. 97," METC IR No. 1080, dated July 24, 1981.

9. Panson, A. G. and J. J. Kovach, "Analytical Data -- Producer Full-Flow Cleanup System, Run No. 98," METC IR No. 1108, dated September 21, 1981.
10. Anderson, R. J., U. Grimm, and J. O. Mapstone, "A Study of Aerosols Contained in Low-Btu Gas and Products of Combustion of the Gas During Run No. 96 of the METC Gas Producer," dated February 1981.
11. Anderson, R. J., U. Grimm, and J. O. Mapstone, "A Study of Aerosols Contained in Low-Btu Gas and Products of Combustion of the Gas During Run No. 97 of the METC Gas Producer," METC IR No. 1091, dated August 21, 1981.
12. Anderson, R. J. and J. O. Mapstone, "The Measurement of Aerosols Generated During the Gasification of Coal and the Subsequent Combustion of the Low-Btu Gas During Run No. 98 of the METC Gas Producer," METC IR No. 1159, dated December 9, 1981.
13. Gordan, S., and B. J. McBride, "Computer Program for Calculation of Complex Chemical Equilibrium Composition, Rocket Performance, Incident and Reflected Shocks, and Chapman-Jouguet Detonations," Report No. NASA SP-273, dated March 1976.
14. CRC Standard Mathematical Tables, 15th Edition.