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BARTLESVILLE, OKLAHOMA

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PROGRESS REPORT NO. 1

FOR

NAVY BUWEPS CONTRACT NOW 64-0443-d

EFFECT OF JP-5 SULFUR CONTENT ON HOT CORROSION OF SUPERALLOYS
IN MARINE ENVIRONMENT

By

H. T. Quigg

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S U M M A R Y

An exploratory program was undertaken to indicate the effect of test variables on the hot corrosion of turbine blade and turbine nozzle guide vane materials. This information will be used to establish the test procedures for a subsequent statistical study to evaluate the effect of JP-5 sulfur content.

This program included an evaluation of the effect of low (0.0002) and high (0.40 weight per cent) fuel sulfur content at six gas temperatures (1200, 1400, 1600, 1800, 2000 and 2200 F) on metal loss of six superalloys (Udimet 700, Stellite 31, Hastelloy R-235, Haynes Alloy 25, Sierra Metal 200 and Udimet 500) in the presence of 10 ppm "sea salt" in the combustor air. A cascade test specimen holder was designed and fabricated which permitted simultaneous evaluation of the effect of sulfur on six superalloys.

The results obtained showed that (1) fuel sulfur inhibited metal loss at gas temperatures of 1800 F and above and (2) fuel sulfur increased metal loss for some superalloys at 1600 F and below. This reversal may be associated with the presence or absence of solid sodium sulfate (freezing point of 1623 F) and a future program should include one temperature below this point.

Aug. 4, 1964

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I. INTRODUCTION

The effect of fuel sulfur on "hot section" durability of aircraft gas turbine engines has been the subject of a number of limited investigations which have been conducted by Phillips Petroleum Company under U.S. Navy Bureau of Naval Weapons Contracts (Ref. 1, 2, 3, 4, 5 and 6). These investigations have been summarized and the problem of hot corrosion in marine environment of superalloys in aircraft engines of advanced design was reviewed in Progress Report No. 3 for Contract NOw 63-0406-d (Ref. 7). This report (Ref. 7) included results of a statistically designed program used to evaluate the effect of three sulfur concentration levels in the fuel (0.0002, 0.040 and 0.40 weight per cent) at three sea salt concentration levels in the air (zero, 1.50 and 15.0 parts per million), and also any sulfur X "sea salt" interactions for two nickel-base alloys (Inconel 713 C and Sierra Metal 200) exposed to vitiated air from the Phillips 2-inch combustor. Test conditions used were

- (1) 56 air-fuel ratio.
- (2) 2000 F gas temperature.
- (3) 15 atmospheres pressure.
- (4) 500 feet per second gas velocity.
- (5) Five hours cyclic operation (55 minutes fuel on and five minutes fuel off).

From this investigation it was concluded that decreasing sulfur concentration in fuel, from the current JP-5 specification maximum of 0.40 to 0.040 weight per cent, did not reduce "sea salt" corrosion significantly. However with the complex interaction found with ingested "sea water", additional data were needed before a recommendation as to the maximum sulfur limit in JP-5 could be made. A test program was proposed to obtain the additional information needed using more superalloys evaluated over a range of gas temperatures.

During the first quarterly period (April through June 1964) under Navy BuWeps Contract NOw 64-0443-d a cascade test specimen holder has been designed and fabricated which permits the evaluation of the effect of sulfur and "sea salt" under various operating conditions on six superalloys simultaneously. An exploratory program has been conducted using single test specimens of six superalloys at each of six gas temperatures and at two levels of fuel sulfur with one level of "sea salt". These data will aid in selection of the three temperature levels which will be used in a statistically designed program during this contract.

II. TEST EQUIPMENT

A. PHILLIPS 2-INCH COMBUSTOR

A schematic diagram of the Phillips 2-inch combustor installation used in this study is shown in Figure 1. Design details of this combustor have been described previously (Ref. 8). Basically, it embodies the principal features of a modern aircraft gas turbine combustor. It was a straight-through, can-type, combustor with fuel atomized by a single, simplex-type nozzle. The flame tube was fabricated from 2-inch, Schedule 40, Inconel pipe, with added internal deflector skirts for film cooling surfaces exposed to the flame.

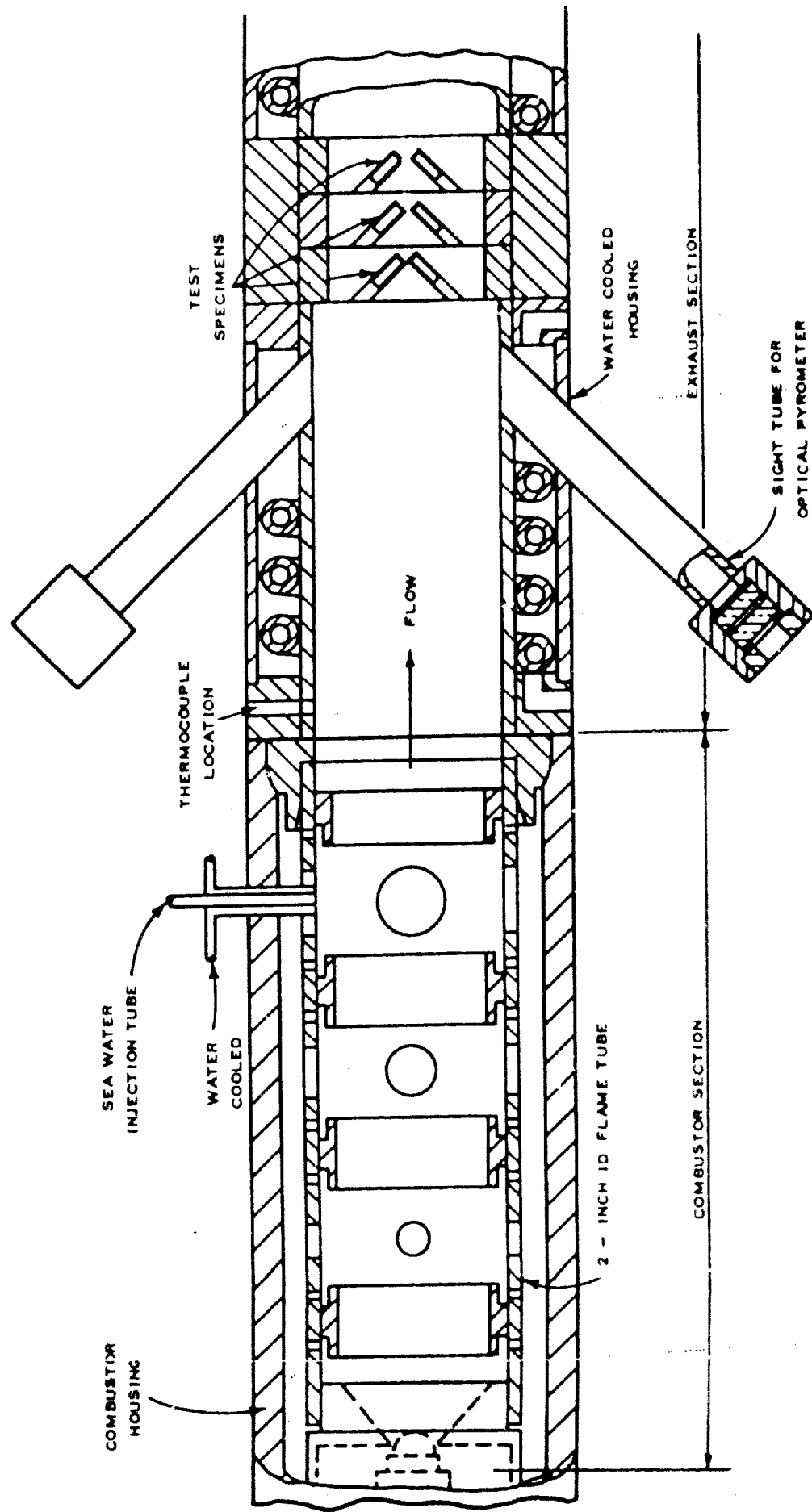


FIGURE 1
PHILLIPS 2 - INCH COMBUSTOR INSTALLATION FOR HOT CORROSION STUDIES

The supporting test facility has been described previously in detail (Ref. 9). Briefly, air was supplied by rotary Fuller compressors, filtered by a Selas Vape-Sorber, and preheated by a Thermal Research heat exchanger. Fuel was supplied by nitrogen pressurization of its supply tank. Also, "sea water" was supplied by nitrogen pressurization of its supply tank.

The design of the combustor installation provides for easy access to the fuel nozzle, flame tube, and test specimens. The combustor installation was disassembled, inspected, and reconditioned after every test.

The location of the "sea water" injection point in the quench zone of the combustor and the water jacketed exhaust section, shown in Figure 1, were selected on the basis of a previous investigation (Ref. 7).

B. SPECIMEN HOLDER

The test specimen holder employed in earlier work (Ref. 7) was modified to permit three holders to be combined in a cascade with each successive holder rotated 120 degrees to prevent channeling of the hot gas flow. The general location with respect to the 2-inch combustor is shown in Figure 1. It is separated from the 2-inch combustor by a six-inch long water cooled spool and is followed by another water cooled spool one foot in length. The holder for a pair of specimens is shown in Figure 2 and the cascade is shown in Figure 3.

The holders maintain the test specimens at an angle of 45 degrees to the axis of the pipe in which they are located. This provides for acceleration of the gas flow over the surface of the test specimens, much as over the turbine blading in an actual engine.

C. SPECIMEN ELECTROCLEANING

Specimen scale or bulk oxide after exposure to hot corrosion was removed by an electrocleaning technique described previously (Ref. 7). Briefly

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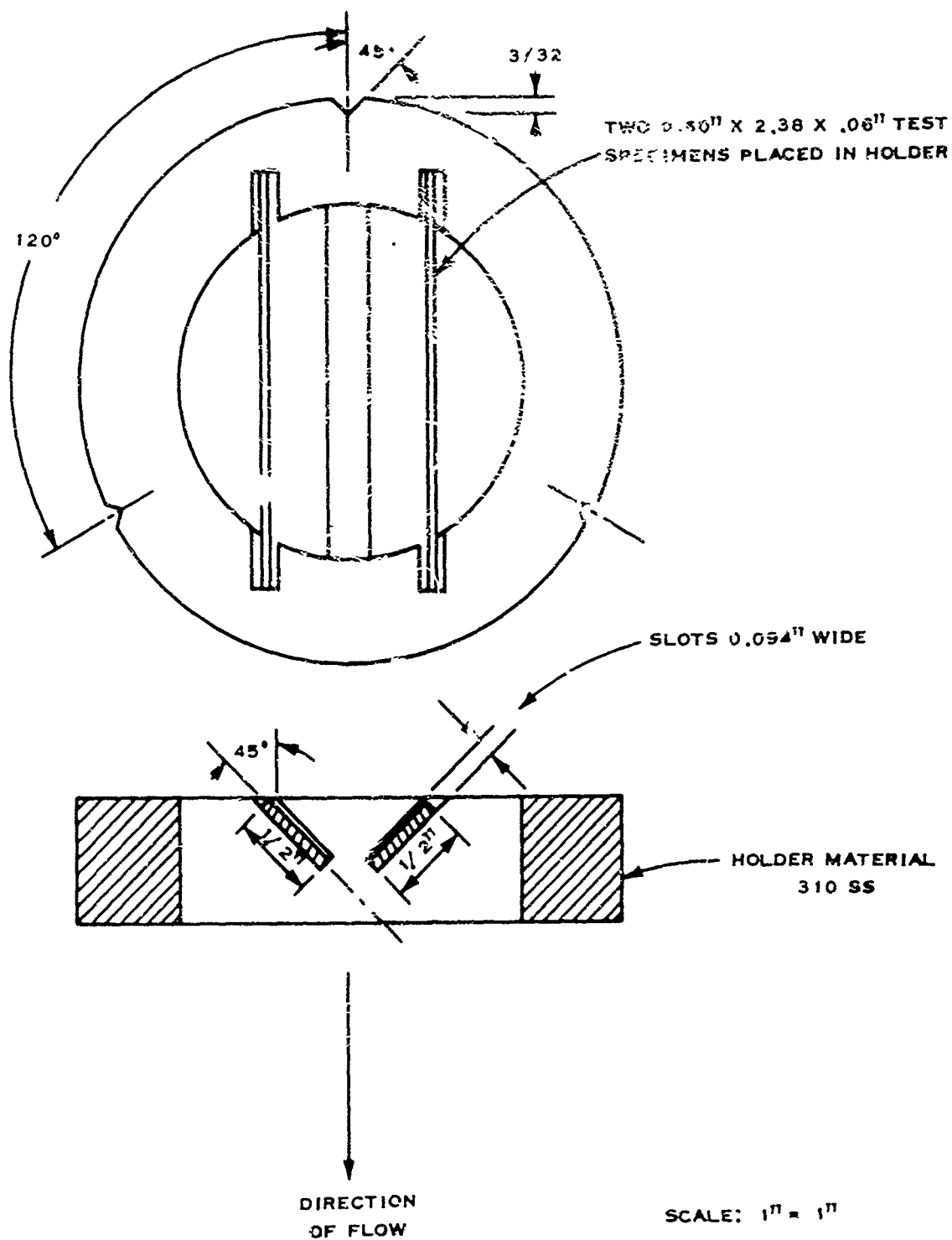
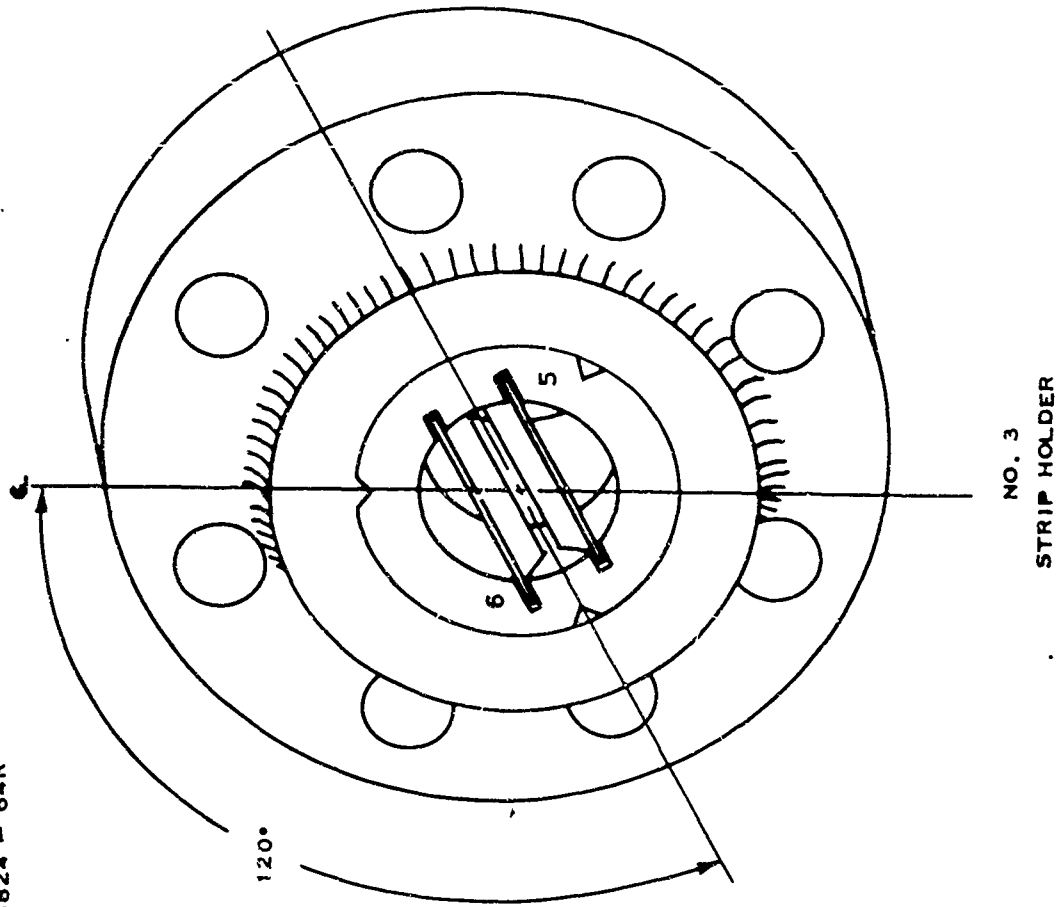


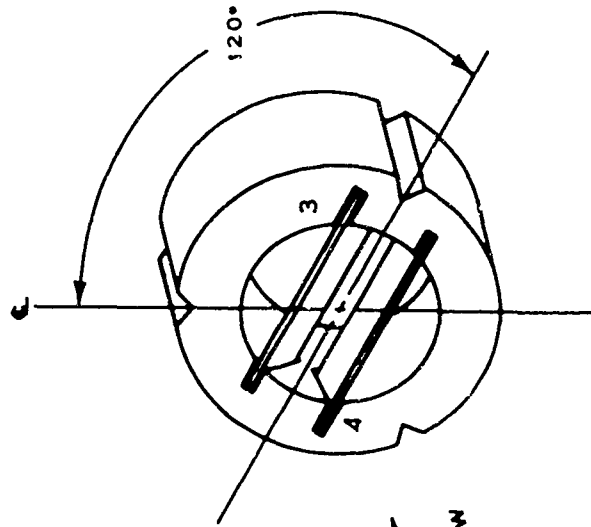
FIGURE 2
TEST SPECIMEN HOLDER FOR PHILLIPS 2- INCH COMBUSTOR

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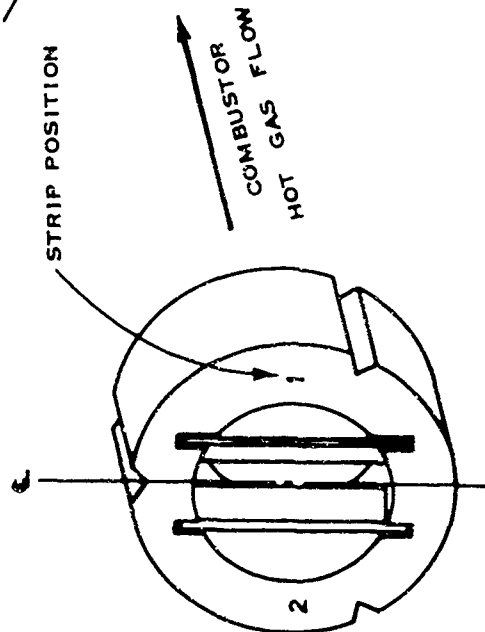
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NO. 3
STRIP HOLDER



NO. 2
STRIP HOLDER



NO. 1
STRIP HOLDER

FIGURE 3
CASCADE STRIP HOLDER ASSEMBLY
FOR CORROSION STUDIES

this consists of immersing the specimens in molten sodium hydroxide (750-790 F) and passing about 1/3 ampere/cm² through the specimens for a period of 10 minutes. This is followed by a water quench.

III. TEST MATERIALS

A. TEST FUELS

The base fuel used in this study was a segregated sample of production ASTM Type A aviation turbine fuel. Physical and chemical properties of this fuel are shown in Table I.

The base fuel was essentially sulfur-free, containing only 0.0002 weight per cent sulfur. The higher sulfur content test fuel was produced by blending to 0.40 weight per cent sulfur using ditertiary butyl disulfide.

B. "SEA WATER"(a)

A synthetic "sea water" was used in this study. Its formulation was taken from the Standard Method of Test for Rust-Preventing Characteristics of Steam-Turbine Oil in the Presence of Water, ASTM Designation D-665-60. The components and their concentrations are shown in Table II.

C. SUPERALLOYS

Six different superalloys were used as test specimens during this study. Four of these superalloys were nickel-base (Udimet 700, Hastelloy R-235, Sierra Metal 200 and Udimet 500) while the other two superalloys were cobalt-base (Stellite 31 and Haynes Alloy 25). Three of the superalloys were prepared as investment castings (Udimet 700, Stellite 31 and Sierra Metal 200) while the other three were cut from sheets. These six metals were selected to represent a wide range of chromium contents. The chemical analyses for the superalloys used are shown in Table III.

(a) "Sea water" and "sea salt" (i.e. in quotes) are used throughout this report to indicate the synthetic composition shown in Table II.

TABLE I

PHYSICAL AND CHEMICAL PROPERTIES OF TEST FUEL

<u>Distillation Temperature, F</u>	<u>Test Fuel Base (a)</u>
Initial Boiling Point	329
5 Volume per cent evaporated	344
10	350
20	359
30	368
40	377
50	388
60	400
70	417
80	435
90	460
95	478
End Point	498
Gravity, degrees API	46.2
Gum, milligrams per 100 milliliters	0.2
Smoke Point, millimeters	26.2
Composition, weight per cent	
Sulfur	0.0002 (b)
Metals (c)	
Iron	< 0.0001
Vanadium	< 0.0001
Nickel	< 0.0001
Copper	< 0.0001
Hydrocarbon Types	
Normal Paraffins	27 (d)
Isoparaffins	23 (d)
Cycloparaffins	36 (d)
Olefins	0 (d)
Aromatics	14

(a) Segregated sample (BJ63-8-G49) of production ASTM Type A Aviation turbine fuel, processed from West Texas crude and finished by hydrotreating.

(b) Higher sulfur content test fuel obtained by blending to desired level using ditertiary butyl disulfide.

(c) X-ray fluorescence analysis.

(d) Typical value for this product.

TABLE II

COMPOSITION OF ASTM D665 SYNTHETIC "SEA WATER"

<u>Salt (a)</u>	<u>Formula</u>	<u>grams per liter (b)</u>
Sodium Chloride	NaCl	24.54
Magnesium Chloride	MgCl ₂ ·6H ₂ O	11.10
Sodium Sulfate	Na ₂ SO ₄	4.09
Calcium Chloride	CaCl ₂	1.16
Potassium Chloride	KCl	0.69
Sodium Bicarbonate	NaHCO ₃	0.20
Potassium Bromide	KBr	0.10
Boric Acid	H ₃ BO ₃	0.03
Strontium Chloride	SrCl ₂ ·6H ₂ O	0.04
Sodium Fluoride	NaF	0.003
	TOTAL	41.953

(a) Use cp chemicals.

(b) Use distilled water.

TABLE III
COMPOSITION OF SUPERALLOY TEST SPECIMENS

<u>Alloying Elements</u>	Chemical Analysis, Per Cent					
	* Udimet 700 (a)	* Stellite 31 (a)	Hastelloy R-235 (b)	Haynes Alloy 25 (b)(c)	Sierra Metal 200 (a)	Udimet 500 (b)
Nickel	Balance (53.05)	10.41	Balance (63.91)	10.0	Balance (60.34)	Balance (51.04)
Cobalt	18.54	Balance (53.62)	0.38	Balance (49.4)	9.80	18.7
Chromium	14.89	25.05	15.29	20.0	9.14	19.0
Molybdenum	5.10	--	5.48	--	--	4.35
Tungsten	---	7.68	--	15.0	12.12	---
Aluminum	4.32	---	2.05	--	4.78	3.10
Titanium	3.02	--	2.48	--	2.00	2.99
Manganese	Nil	0.73	0.03	1.5	0.04	< 0.10
Iron	0.75	1.37	9.96	3.0	0.53	0.36
Zirconium	0.042	--	--	--	0.068	< 0.01
Columbium	--	--	--	--	0.99	--
Silicon	0.18	0.62	0.26	1.0	0.01	0.15
Boron	0.031	--	--	--	0.015	0.003
Sulfur	0.006	0.012	0.009	--	--	0.005
Carbon	0.06	0.49	0.15	0.1	0.17	0.09
Phosphorus	0.001	0.015	0.001	--	--	--
Copper	0.01	--	--	--	--	< 0.10

(a) Investment casting.

(b) Cut from sheet material.

(c) Typical analysis.

IV. TEST PROGRAM

A. PROCEDURE

The operating conditions selected for this program are shown in Table IV. It is seen that operation was at 15 atmospheres with a mass air flow of 5400 pounds per hour. Combustor inlet air temperature and fuel flow were adjusted to give the desired gas temperatures. Synthetic "sea water" was injected at the rate of 10.0 ppm "sea salt" based on inlet air and fuel sulfur contents were 0.0002 and 0.40 weight per cent.

The procedure consisted of a five-hour cyclic test with 55 minutes of exposure of the test specimens to hot gases followed by 5 minutes with fuel turned off. On completion of the test, samples of deposits were removed by scraping and saved for future analysis. The test specimens were then electrochemically cleaned using a procedure previously described (Ref. 7) and loss of weight determined.

This program was an exploratory program in which a single test specimen of each superalloy was exposed to each set of test conditions. Six superalloys were included in each test with the specimens mounted in pairs in specimen holders arranged in a cascade as shown in Figure 3. The position of a specific superalloy within the cascade was held constant throughout this program. It can be observed that only the specimens in the first holder are exposed to hot gas only, while those in the second and third holders may also be exposed to corrosion products from the preceding superalloys.

TABLE IV

OPERATING CONDITIONS OF PHILLIPS 2-INCH COMBUSTOR

Test Variables	Test Conditions (a)					
	1200	1400	1600	1800	2000	2200
Temperature, F						
Exhaust Gas	1195	1375	1590	1780	1990	2090(b)
Profile (c)	20	210	135	135	150	200
Test Specimens (d)	(e)	1610(f)	1650(f)	1685	1865	1955
Combustor Inlet Air	800	800	800	1000	1000	1000
Pressure, atmospheres						
Combustor Inlet Air	15.0	15.0	15.0	15.0	15.0	15.0
Test Specimen Drop	1.4	1.5	1.6	1.7	1.7	1.5
Mass Flow Rate, pounds per hour						
Air	5400	5400	5400	5400	5400	5400
Fuel	36	54	72	72	90	108
Air Fuel Ratio	150	100	75	75	60	50
Flow Velocity, feet per second						
Combustor Reference (g)	170	170	170	200	200	200
Exhaust Gas (h)	180	195	220	240	260	275
at Test Specimens (i)	330	370	410	450	490	510
Test Duration, hours (j)	5	5	5	5	5	5

(a) Average values.

(b) Temperature at exhaust gas core averaged 2175 F but the overall lower average resulted from heat losses to water cooled wall.

(c) Maximum variation between four thermocouples on equal area centers.

(d) First pair of specimens.

(e) Below operating range of optical pyrometer.

(f) Test specimens probably reflecting flame radiation to give fictitiously high readings with optical pyrometer.

(g) Cold flow, based on 2.66 in.² exit area in flame tube.

(h) Based on 3.36 in.² area at outlet from combustor.

(i) Based on 1.80 in.² unblocked area in first test specimen holder.

(j) Operating cycle of 55 minutes at test conditions, followed by 5 minutes fuel off.

V. DISCUSSION OF RESULTS

b) The metal loss data obtained in this exploratory program are shown in Table V expressed as weight loss per unit area and in Table VI expressed as per cent weight loss of the test specimen. Metal loss as a function of temperature for two fuel-sulfur concentrations are shown in Figures 4 through 9. An examination of these data show that with each of the superalloys, metal loss was inhibited by the fuel sulfur content at temperatures of 1800 F and above. At 1600 F and below the data are not consistent for all of the superalloys; however, with Hastelloy R-235 and to a lesser degree with Sierra Metal 200, Udimet 700 and Udimet 500 sulfur in the fuel increases metal loss. This reversal of sulfur effect may be associated with the presence of solid sodium sulfate on the test specimens. The freezing point of sodium sulfate is 1623 F. Although air-fuel ratio varied from 150 at 1200 F to 50 at 2200 which resulted in an increase in sulfur to "sea salt" ratio, comparisons of low (0.0002 weight per cent) and high (0.40 weight per cent) sulfur fuels at a given temperature would not be affected.

gh While a difference in the nature of attack of the specimens at 1600 F and below from attack at 1800 F and above with changes in fuel sulfur content are shown, data are not available at this time to establish the mechanisms of attack. Analysis of specimen scale deposits and photomicrographs of test specimens will be obtained which may aid in determining the type of attack at the various conditions.

Previous exploratory studies (Ref. 7) have indicated that the characteristic sulfidation attack at temperatures below the melting point of sodium sulfate gives way to gross oxidation at higher temperatures when the metal is no longer covered by a heavy deposit of sodium sulfate.

TABLE V
WEIGHT LOSS (MG/CM²) OF SUPERALLOY TEST SPECIMENS

Gas Temp., F	Fuel Sulfur Wt. %	Weight Loss, Mg per cm ² (a)					
		Udimet 700	Stellite 31	Hastelloy R-235	Haynes Alloy 25	Sierra Metal 200	Udimet 500
1200	0.0002	8.7	3.0	11.5	7.4	4.0	10.2
1200	0.40	42.4	3.8	116.7	3.5	11.3	11.2
1400	0.0002	36.4	5.2	72.8	11.2	16.3	10.8
1400	0.40	6.8	3.4	174.4	5.8	60.3	52.2
1600	0.0002	58.5	11.4	169.2	10.0	91.7	33.6
1600	0.40	1.7	5.8	343.6 (b)	11.5	63.9	7.6
1800	0.0002	84.3	16.3	112.0	10.8	201.1	8.7
1800	0.40	8.4	2.7	9.1	8.7	74.6	3.6
2000	0.0002	100.1	30.4	72.9	34.8	427.0 (c)	15.6
2000	0.40	5.6	11.6	9.5	11.9	7.0	7.5
2200	0.0002	107.2	82.1	147.9	234.6	480.0	70.3
2200	0.40	24.1 (d)	24.5	66.8	31.0	90.9	---(e)

- (a) Area of specimen = 17.67 cm².
- (b) Part of specimen burned out.
- (c) Center missing.
- (d) Specimen broken.
- (e) Specimen missing at end of test.

TABLE VI
PER CENT METAL WEIGHT LOSS OF SUPERALLOY TEST SPECIMENS

Gas Temp., F	Fuel Sulfur, Wt. %	Per Cent Metal Weight Loss							
		Udimet 700	Stellite 31	Hastelloy R-235	Haynes Alloy 25	Sierra Metal 200	Udimet 500		
1200	0.0002	1.7	0.5	1.9	1.2	0.7	2.0		
1200	0.40	7.7	0.6	19.6	0.6	2.0	2.2		
1400	0.0002	7.0	0.9	12.3	1.8	3.0	2.1		
1400	0.40	1.2	0.6	29.4	1.0	11.0	10.2		
1600	0.0002	10.8	1.8	28.6	1.6	16.4	6.6		
1600	0.40	0.3	0.9	57.5(1)	1.9	11.4	1.5		
1800	0.0002	16.2	2.8	19.0	1.8	36.3	1.7		
1800	0.40	1.6	0.4	1.5	1.5	13.3	0.7		
2000	0.0002	18.8	5.2	12.2	5.8	79.8(2)	2.9		
2000	0.40	1.0	2.0	1.6	2.0	1.3	1.4		
2200	0.0002	19.5	14.0	25.0	40.5	88.2	13.1		
2200	0.40	4.4(3)	4.0	11.4	5.2	16.3	—(4)		

- (1) Part of specimen burned out.
- (2) Center missing.
- (3) Specimen broken.
- (4) Specimen missing at end of test.

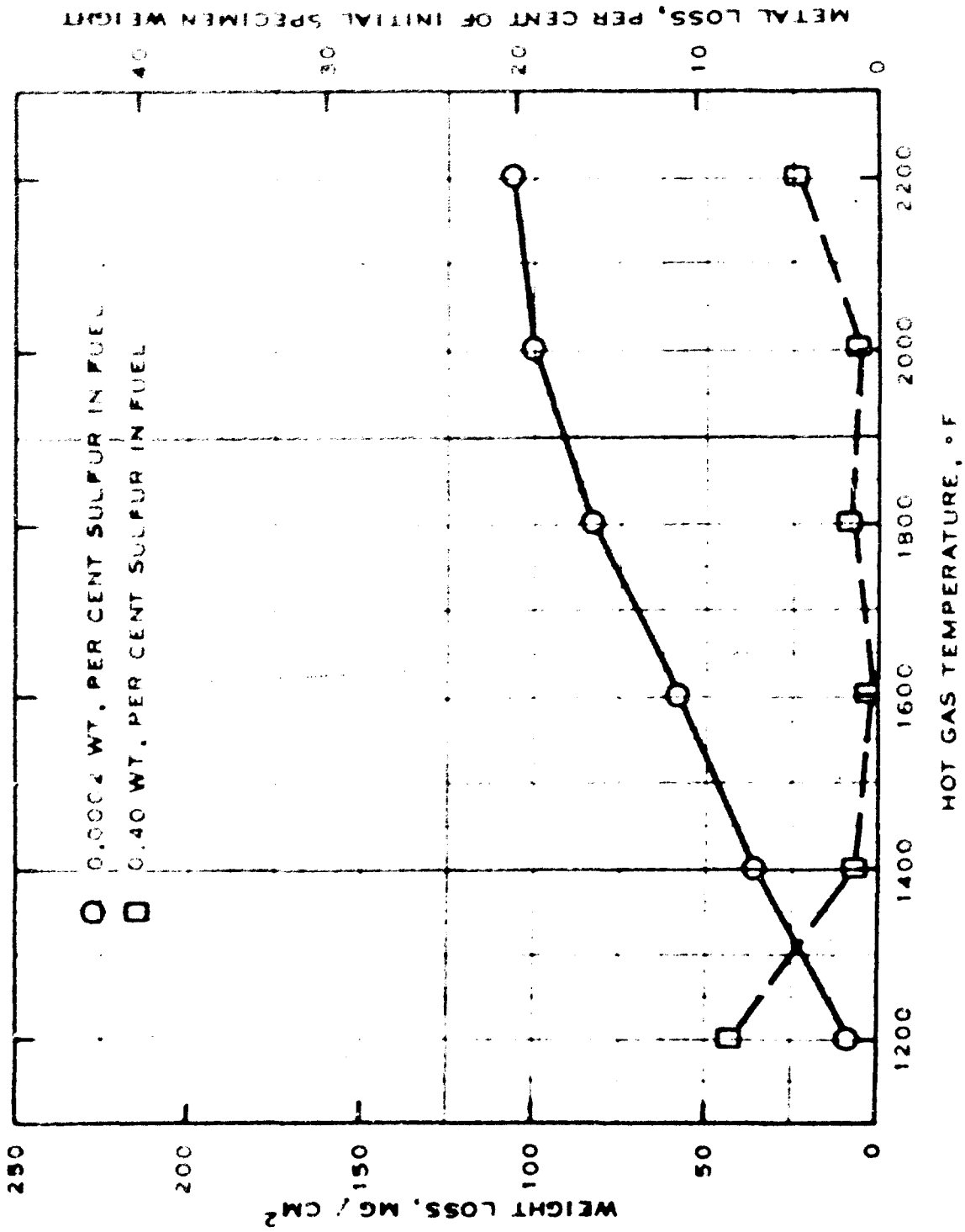
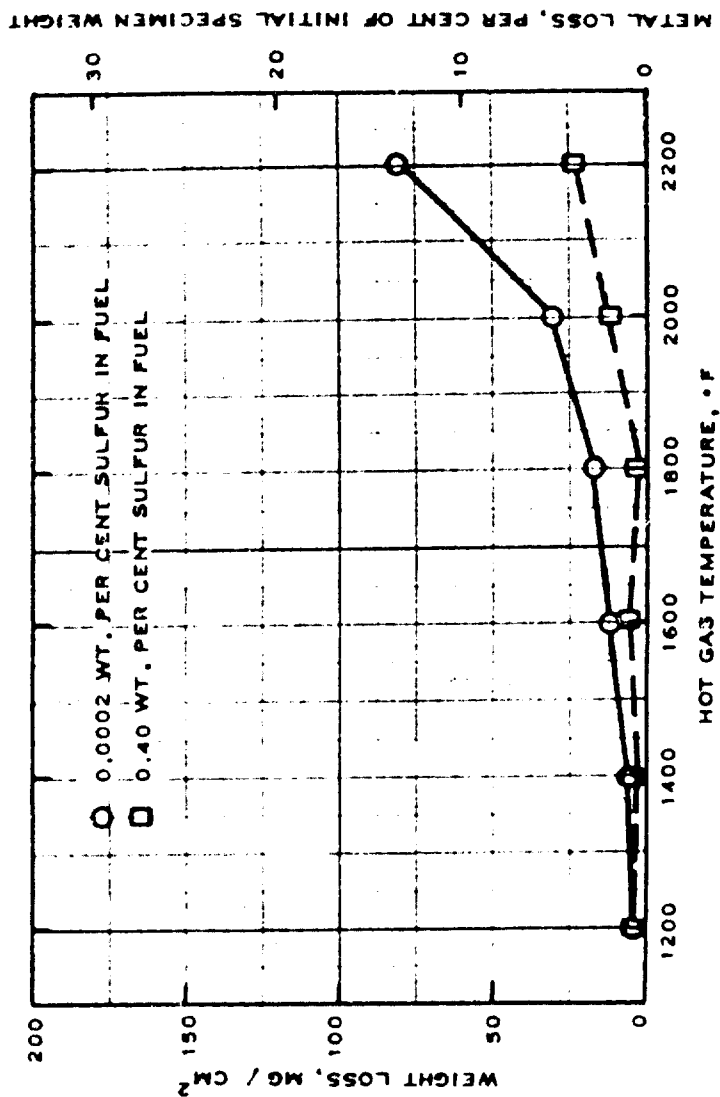


FIGURE 4
EFFECT OF JP - 5 SULFUR CONTENT ON HOT GAS
CORROSION OF UDIMET 700 IN MARINE ENVIRONMENT



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FIGURE 5
EFFECT OF JP - 5 SULFUR CONTENT ON HOT GAS
CORROSION OF STELLITE 31 IN MARINE ENVIRONMENT

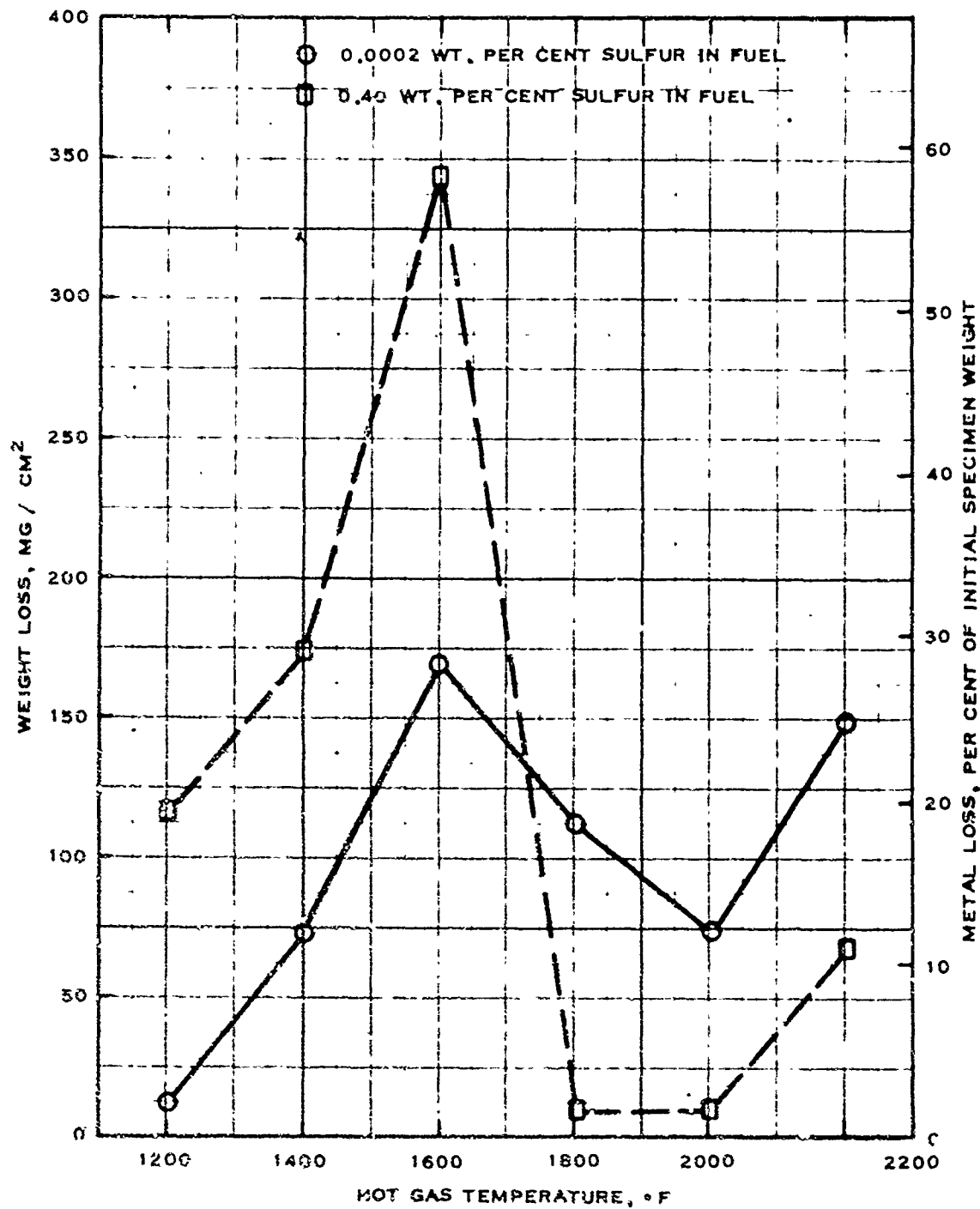


FIGURE 6
 EFFECT OF JP - 5 SULFUR CONTENT ON HOT GAS CORROSION
 OF HASTELLOY R - 235 IN MARINE ENVIRONMENT

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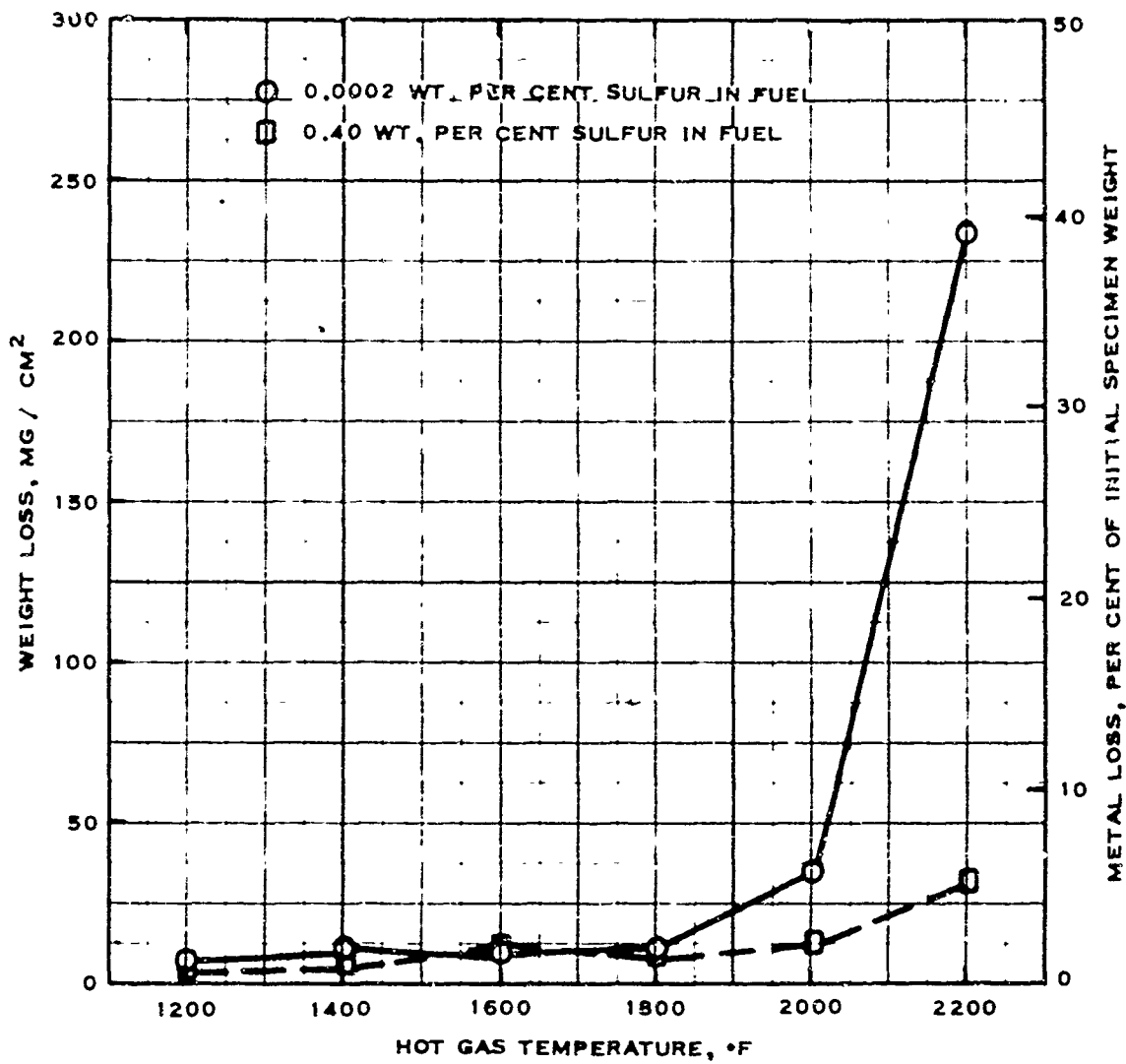


FIGURE 7
EFFECT OF JP - 5 SULFUR CONTENT ON HOT GAS
CORROSION OF HAYNES ALLOY 25 IN MARINE ENVIRONMENT

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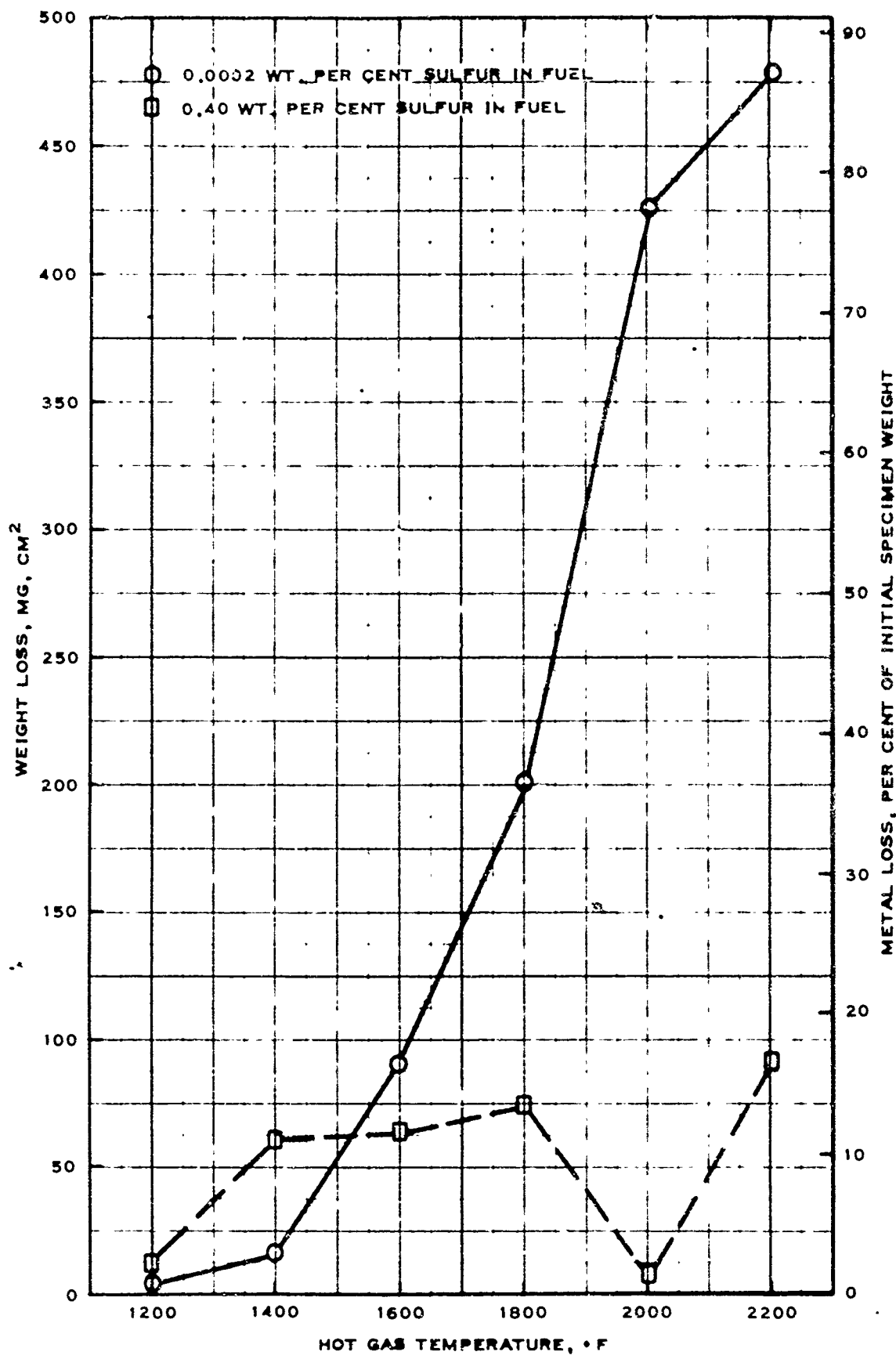


FIGURE 8
EFFECT OF JP - 5 SULFUR CONTENT ON HOT GAS
CORROSION OF SIERRA METAL 200 IN MARINE ENVIRONMENT

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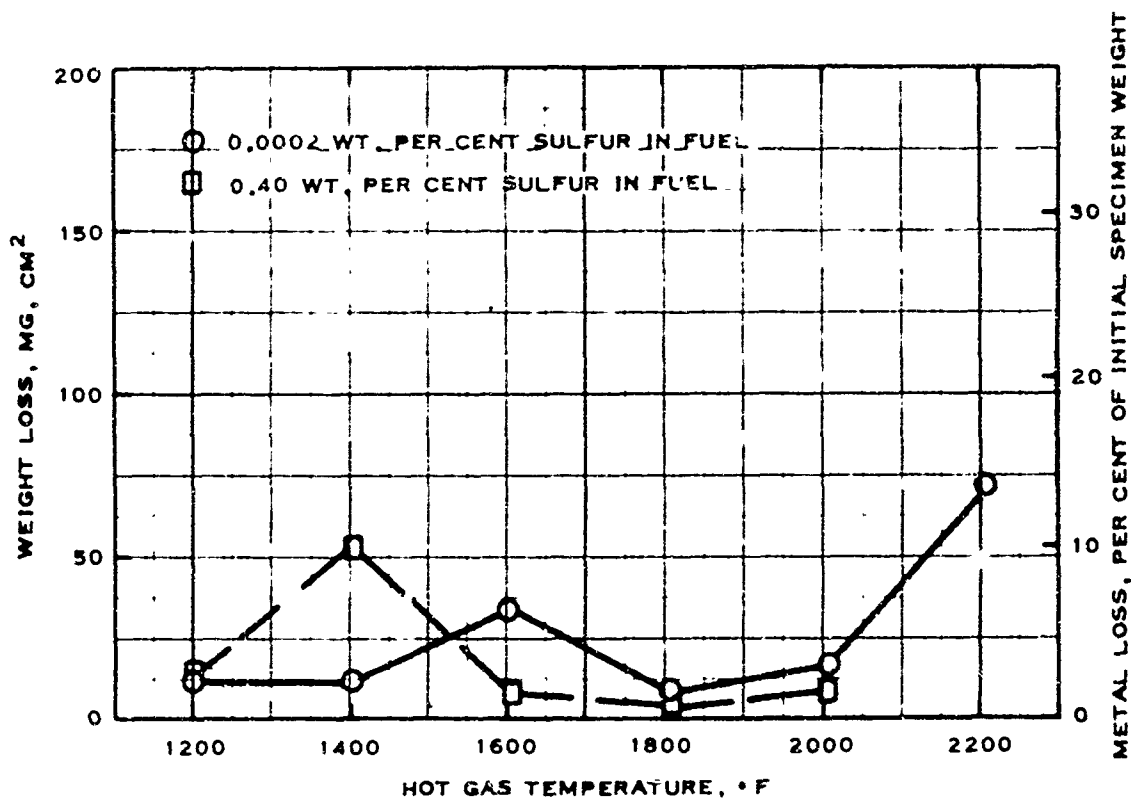


FIGURE 9
EFFECT OF JP - 5 SULFUR CONTENT ON HOT GAS
CORROSION OF UDIMET 500 IN MARINE ENVIRONMENT

VI. CONCLUSIONS

An exploratory program on hot corrosion of superalloys at high pressure (15 atmospheres) and high velocity (330 to 510 ft/sec at the test specimen) with 10 ppm "sea salt" in combustor air over a range of temperatures from 1200 to 2200 F show that:

1. Fuel sulfur inhibited metal loss at gas temperatures of 1800 F and above.
2. Fuel sulfur increased metal loss for some superalloys at 1600 F and below.

VII. RECOMMENDATIONS

This exploratory study was made to determine the effect of fuel sulfur on hot corrosion over a range of temperatures in a marine environment. Because two types of corrosive attack were observed, it is recommended that when the statistical program previously proposed (Ref. 7) is conducted, one of the temperatures should be below the freezing point of sodium sulfate (1623 F). Since the number of temperatures that can be investigated is limited to three by the overall size of the program, it is proposed that 1500, 1850 and 2200 F be used rather than the 1800, 2000 and 2200 F previously proposed.

VIII. FUTURE WORK

During the second quarterly period under Contract NOw 64-0443-d it is planned to continue exploratory testing of the effect of sulfur, under a variety of conditions on hot corrosion in a marine environment. The following operating and test variables are being investigated.

1. Hot gas temperature at test specimens.
2. Hot gas pressure at test specimens.
3. Hot gas velocity at test specimens.

4. "Sea salt" concentration in hot gas.
5. Air-fuel mixture ratio to combustor.
6. Location of test specimens in cascade holder.
7. Test duration.

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