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EFFECT OF JP-5 SULFUR CONTENT AND SEA WATER INGESTION ON HOT GAS CORROSION OF SUPER ALLOYS

PROGRESS REPORT NO. 3

NAVY BUWEPS CONTRACT NOw 63-0406-d

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PHILLIPS PETROLEUM COMPANY

RESEARCH DIVISION

BARTLESVILLE, OKLAHOMA

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S U M M A R Y

An experimental investigation was conducted to determine whether the maximum sulfur content of 0.4 weight per cent, currently allowed in grade JP-5 aviation turbine fuel, is a safe level for the protection of turbine blade alloys used in high-performance engines.

Specimens of two nickel-base alloys (Inconel 713C and Sierra Metal 200) were exposed to vitiated air from the Phillips 2-inch combustor (56 air-fuel ratio) at high temperature (2,000 degrees Fahrenheit), high pressure (15 atmospheres), and high velocity (500 feet per second) during a five-hour cyclic test (55 minutes fuel-on and 5 minutes fuel-off). A statistically designed test program was 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 interaction. The significance of test specimen metal losses and changes in tensile properties was established by analyses of variance, made at a confidence level of 95 per cent.

Both super alloys showed good resistance to oxidation and erosion, in the absence of sulfur and sea salt. Little or no evidence was found of sulfur corrosion in the absence of sea salt. Catastrophic sea salt corrosion was encountered with both super alloys in some instances. A significant sulfur x sea salt interaction was shown by both super alloys; but, while hot gas corrosion of Inconel 713C was accelerated, hot gas corrosion of Sierra Metal 200 was inhibited.

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, the complex interaction found with ingested sea water does not allow for a recommendation as to the maximum sulfur limit in JP-5, without additional data. It is recommended that this study be extended to include additional super alloys, evaluated over a range in exhaust gas temperature. A suggested test program is outlined to obtain the recommended additional information.

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JUN 29 1964

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PHILLIPS PETROLEUM COMPANY

BARTLESVILLE, OKLAHOMA

Progress Report No. 3

Navy BuWeps Contract NOw 63-0406-d

EFFECT OF JP-5 SULFUR CONTENT AND SEA WATER INGESTION ON HOT GAS CORROSION OF SUPER ALLOYS

I. INTRODUCTION

A. Gas Turbine Engine Operation in Marine Environment

In the past, the 0.4 weight per cent of sulfur allowed in grade JP-5 aviation turbine fuel has not accelerated the corrosion of engine hot section parts significantly. The concentration of sulphurous gases in the combustor exhaust stream has not been high enough, under the oxidizing conditions and at the temperatures prevailing, to result in sulfidation of turbine blades. The chromium content of the nickel-base alloys involved, which aids the super alloy in resisting attack by oxygen and sulfur, has been near 20 per cent and operating temperatures have not exceeded 1,700 F.

Continued development of the aircraft gas turbine engine, to decrease specific fuel consumption and increase specific power, has required the development of new alloys to permit higher cycle temperatures. Engines of advanced design are operating now with turbine inlet gas temperatures of 2,100 F. These new alloys are characterized by a reduction of chromium content to near 10 per cent in order to increase the concentration of high temperature strengthening elements. While their resistance to oxidation appears satisfactory, reports of accelerated corrosion by sulfidation must be evaluated carefully by the Navy.

Catastrophic corrosion of nickel-base alloys having low chromium content has been encountered where operation has been over or near the sea (Ref. 1 and 2). Traces of sodium sulfate, a major constituent of sea salt, has been detected on corroded turbine blades. It has been possible to reproduce the essential features of the attack on the new type super alloys in laboratory tests by exposure to sea salt.

Required aircraft operational patterns exclude control over sea water and sea salt ingestion by the engine. However, other approaches to limiting sulfidation of hot section components are being investigated by the Navy. The use of transpiration air cooled turbine blades has been suggested as a design approach to lower metal operating temperatures (Ref. 3). Another approach might be the use of fuel of low sulfur content to reduce the concentration of sulphurous gases (Ref. 4).

Changes in metallurgy to improve corrosion resistance do not appear promising, since they would lose the physical properties which made a desirable structural material in the first place. However, the application of suitable protective coatings to resist attack by corrosive materials is being vigorously pursued, with much promise (Ref. 5). Perhaps suitable coatings of a self-healing nature might be made by the use of proper fuel additives, as has been the practice with industrial gas turbine engines operating on residual fuels. Such a feature would be of great value, for even under the best circumstances coatings are susceptible to random defect failures and reliability remains as the major problem confronting the user. The perfection of a self-healing coating system might allow the use of refractory-metal-base alloys operable at temperatures near 3,000 F.

B. Hot Gas Corrosion of Super Alloys

Super alloys (nickel-chromium-cobalt base with smaller amounts of aluminum, titanium, columbium, molybdenum, etc.) have been developed for service in oxidizing atmospheres at temperatures near 2,000 F. Their superior physical properties are required by aircraft gas turbine engines for hot section components, such as rotating turbine blades, where mechanical and thermal stresses are at a maximum. Their resistance to oxidation damage results from the growth of stable adherent metal oxide scales; therefore, these parts do suffer from oxidation damage, but it is usually not significant until after hundreds, or even thousands, of hours of operation.

Under the oxidizing conditions existing in the hot section of an aircraft gas turbine engine, sulfur attack is normally no more severe than oxygen attack, due to the formation of a surface oxide film on the alloy (Ref. 6). However, the deposition of ash from the air or fuel opens the way for catastrophic rates of sulfidation, associated with the fluxing action of molten phases formed on the surface of the metal structure (Ref. 7, 8, 9, and 10). A common source of such deposits is sea salt, which is rich in sodium sulfate. The deposits may serve to collect and concentrate sulfur, which is normally present in harmless concentrations in the exhaust gas and convey it to the metal surface beneath the deposit.

There is no generally satisfactory solution to this problem short of removal of the molten phase from contact with the metal. With operation in a marine environment, it is not usually feasible to prevent the ingestion of sea water and sea salt. The alternate has been to keep the temperature below the fusion temperature of the predominately sodium sulfate deposit, or to use an alkaline earth additive to raise its fusion temperature above the temperature of operation.

C. Review of Previous Work by Phillips Petroleum Company

Limited investigations of the effect of fuel sulfur on "hot section" durability of aircraft gas turbine engines have been conducted by Phillips Petroleum Company working under U. S. Navy Bureau of Naval Weapons Contracts NOas 58-310-d, NOas 60-6009-c, NOw 61-0590-d, and N600 (19)-58219 (Ref. 11, 12, 13, and 14). Much of this work has been summarized in a paper presented to the Institute of Petroleum (Ref. 15). In addition, a

small amount of exploratory work was conducted during the first quarterly period of the present Contract NOw 63-0406-d (Ref. 16). These investigations have shown that:

1. The form in which sulfur exists in the fuel (i.e., organic sulfur compound type) is unimportant to hot gas corrosion as compared to the gross sulfur content of the fuel. (Ref. 11 and 12)

2. The extent of sulfidation is a linear function with time, indicating that its mechanism is not diffusion controlled by a coherent barrier layer of scale. (Ref. 11, 12, 13, 14, 15, and 16)

3. The aromatic content of the fuel (0 to 25 per cent) had no measurable effect on hot gas corrosion rates, indicating that variations in exhaust gas soot content and flame radiant heating at high pressure were not significant. (Ref. 16)

4. The relative rates of hot gas corrosion for a group of super alloys at atmospheric pressure did not correlate with rates obtained at high pressure, indicating that sulfidation reactions are pressure dependent and vary with alloy composition. (Ref. 14)

5. Sulfur had little, or no, effect on hot gas corrosion--in the absence of sea salt. (Ref. 15)

6. Sea salt accelerated hot gas corrosion at high temperatures 1,600 to 2,000 F, but had no effect at 1,350 F. (Ref. 13 and 16)

D. <u>Contract NOw 63-0406-d</u>

An experimental investigation of corrosion by hot gases of modern super alloys used in aircraft turbine engines of advanced design was conducted by Phillips Petroleum Company during the third quarterly period, October through December, 1963, of U. S. Navy Bureau of Naval Weapons Contract NOw 63-0406-d. Coupons of aluminum-titanium-hardened nickel-chromium-base alloys were exposed to high velocity gases, at high temperature and high pressure, to evaluate the effect of the latter's sulfur and sea salt content.

This study was made to determine whether the maximum sulfur limit of 0.4 weight per cent, currently allowed in grade JP-5 aviation turbine fuel, is a safe level for the protection from hot gas corrosion of turbine blade alloys used in advanced engines when operating in a marine environment. If not, information was sought to show whether a reduction in the sulfur limit for JP-5 would alleviate hot gas corrosion significantly.

The use of oversimplified test methods, such as furnace exposure to high temperature or torch exposure to high temperature and high velocity, does not provide exposure to the full range of variables encountered in actual service. While a limited amount of such data are available from the literature (Ref. 4, 6, 7, 8, 9, and 10), they cannot be accepted with confidence.

A more restrictive limitation on the amount of sulfur allowed in JP-5 carries with it the certainty of decreased availability, and the potential of increased cost. The former can be very important in the event of a national emergency, while even a small increment in the latter can amount to a substantial sum because of the large volumes involved.

Therefore, this investigation was carefully designed, using a high-pressure burner rig to obtain exposure of super alloy test specimens to conditions closely simulating those prevailing in service. For maximum severity of test conditions, a high compression ratio aviation gas turbine operating at sea-level take-off conditions was simulated. The super alloy test specimens were exposed to the exhaust gas from a combustor operated at the following conditions:

1. Combustor inlet temperature of 1,000 F.

2. Combustor pressure of 15 atmospheres.

3. Combustor exhaust gas temperature of 2,000 F.

4. Combustor reference velocity of 200 ft/sec.

5. Cyclic operation each hour, with 55 minutes at temperature.

A statistically-designed test program was conducted to show whether the concentration of sulfur in the fuel accelerated sea salt corrosion significantly. The extent of hot gas corrosion was based on evaluation of:

1. Test specimen weight loss.

2. Deterioration in test specimen tensile properties.

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. 17). Basically, it embodied the principal features of a modern aircraft gas turbine combustor. It was a straight-through, can-type, combustor with fuel atomization by a single, simplex-type, nozzle. The flame tube was fabricated from 2-inch, Schedule 40, Inconel

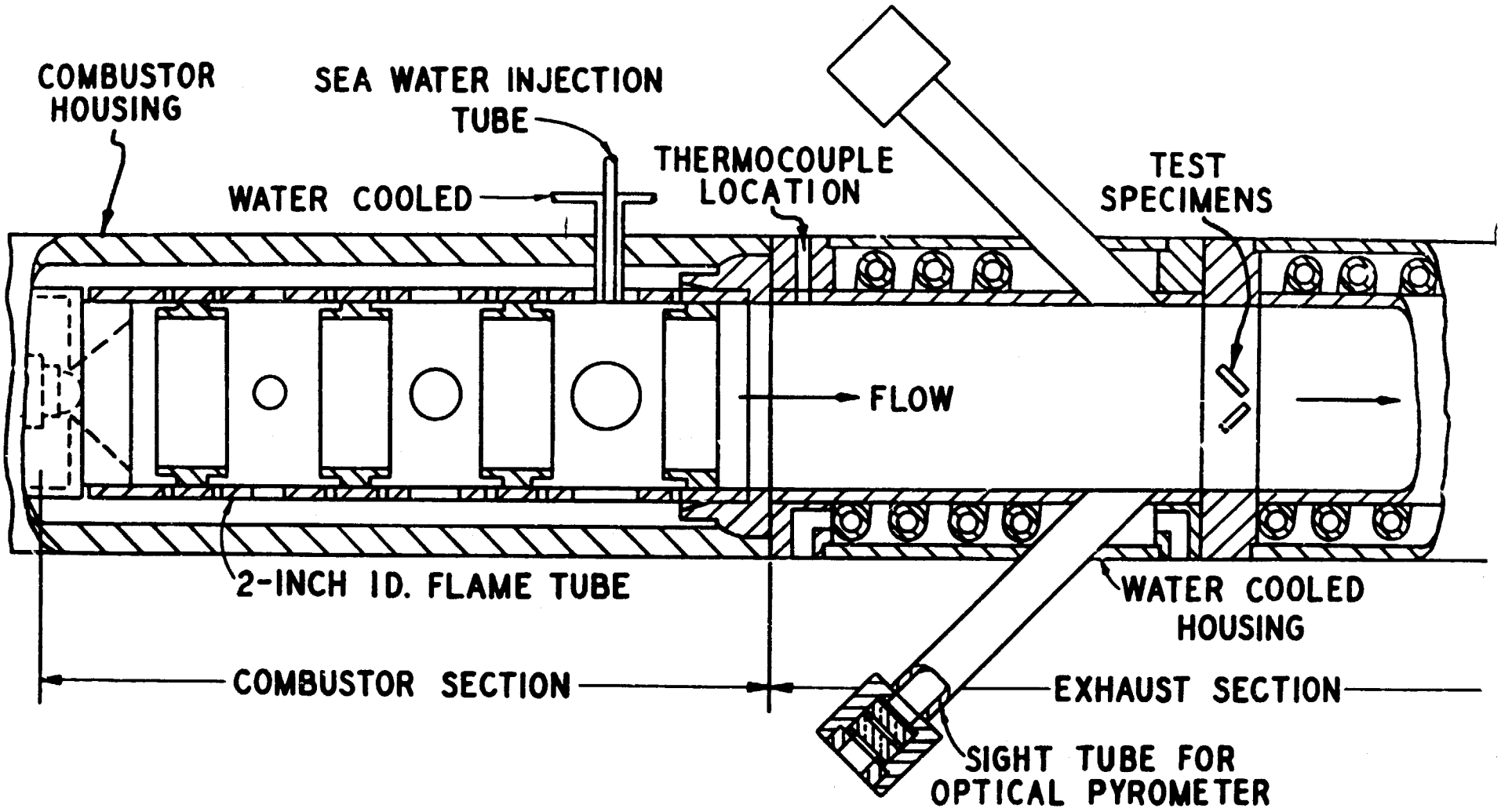

FIGURE 1
PHILLIPS 2-INCH COMBUSTOR INSTALLATION FOR HOT GAS CORROSION STUDIES

pipe, with added internal deflector skirts for film cooling surfaces exposed to the flame.

The supporting test facility has been described previously in detail (Ref. 18). 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.

During preliminary testing, sea water was injected into the primary zone of the combustor, near the fuel nozzle. The injection probe was water cooled to prevent vaporization of sea water, and plugging of the orifice with sea salt. It became evident immediately that flame tube life would be limited to a single 5-hour test with sea water because of severe corrosion of the internal deflector skirts. Therefore, the sea water injection point was moved downstream to the quench zone of the combustor, as shown in Figure 1. This markedly improved flame tube life.

During preliminary testing, an air cooled, 310 stainless steel exhaust section was used. It soon became evident that this would not stand extended periods of operation with 2,000 F exhaust gas temperature. Therefore, it was cooled by water jacketing, with excellent results.

It is of interest to compare data from the preliminary tests with that obtained following relocation of the sea water injection probe and addition of water cooling to the exhaust section. It will be noted from Table I that these modifications in test equipment had a negligible effect on hot gas corrosion of Inconel 713C. Therefore, it was concluded that important changes in the sea salt did not occur as a result of its exposure to either flame temperature or longer residence time in the combustor prior to contacting the test specimens. In addition, it was concluded that an important reduction in test specimen temperature did not result from the lower exhaust section wall temperature.

Good test repeatability was indicated by the data presented in Table I. An excellent spread in weight loss is also evident between tests with the sulfur free (two parts per million) base fuel and the base fuel with added sulfur plus ingested sea water. In view of these data, it was decided to inject the sea water into the quench zone of the combustor and to water jacket the exhaust section. During the test program that followed, no failures of either the flame tube or the exhaust section were noted.

TABLE I

EFFECT OF TEST EQUIPMENT MODIFICATIONS ON LEVEL OF HOT GAS CORROSION

Test Equipment Modification		Average Weight Loss for Two Inconel 713C Test Specimens at 2,000 F Test Condition, Milligrams	
Sea Water Ingestion	Exhaust Section	Base Fuel	Base Fuel with 0.40 Per Cent Sulfur, 15 ppm Sea Salt in Air
Combustor Inlet (Flame Zone)	Air Cooled	52	2098 2166 (Check Test)
Combustor Outlet (Quench Zone)	Water Cooled	20 26 (Check Test)	2167 2246 (Check Test)

B. Specimen Holder

The test specimen holder was of the same design employed in earlier work (Ref. 16). Its general location with respect to the 2-inch combustor has already been indicated 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. It is mounted in a suitable cavity in a flange located between these two water cooled spools.

The cross sectional area of the 2-inch pipe in which the test specimen holder is located is 3.36 in.^{2}; however, the unblocked area in the test specimen holder is only 1.80 in.^{2}. The holder maintains the test specimens at an angle of 45 degrees to the direction of flow of the exhaust gas, as shown in Figure 2. 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.

While the 310 stainless steel test specimen holder is subjected to considerable attack by the hot exhaust gases, this design provides for easy removal of the test specimens--and replacement of the holder when necessary.

It is apparent from the location of the test specimens relative to the flow direction that the specimens were being subjected to an appreciable gas pressure loading while a test was in progress. The pressure drop across the specimens is a measure of the loading and for most of the test results discussed in this report, amounted to 5 lb/in.^{2}.

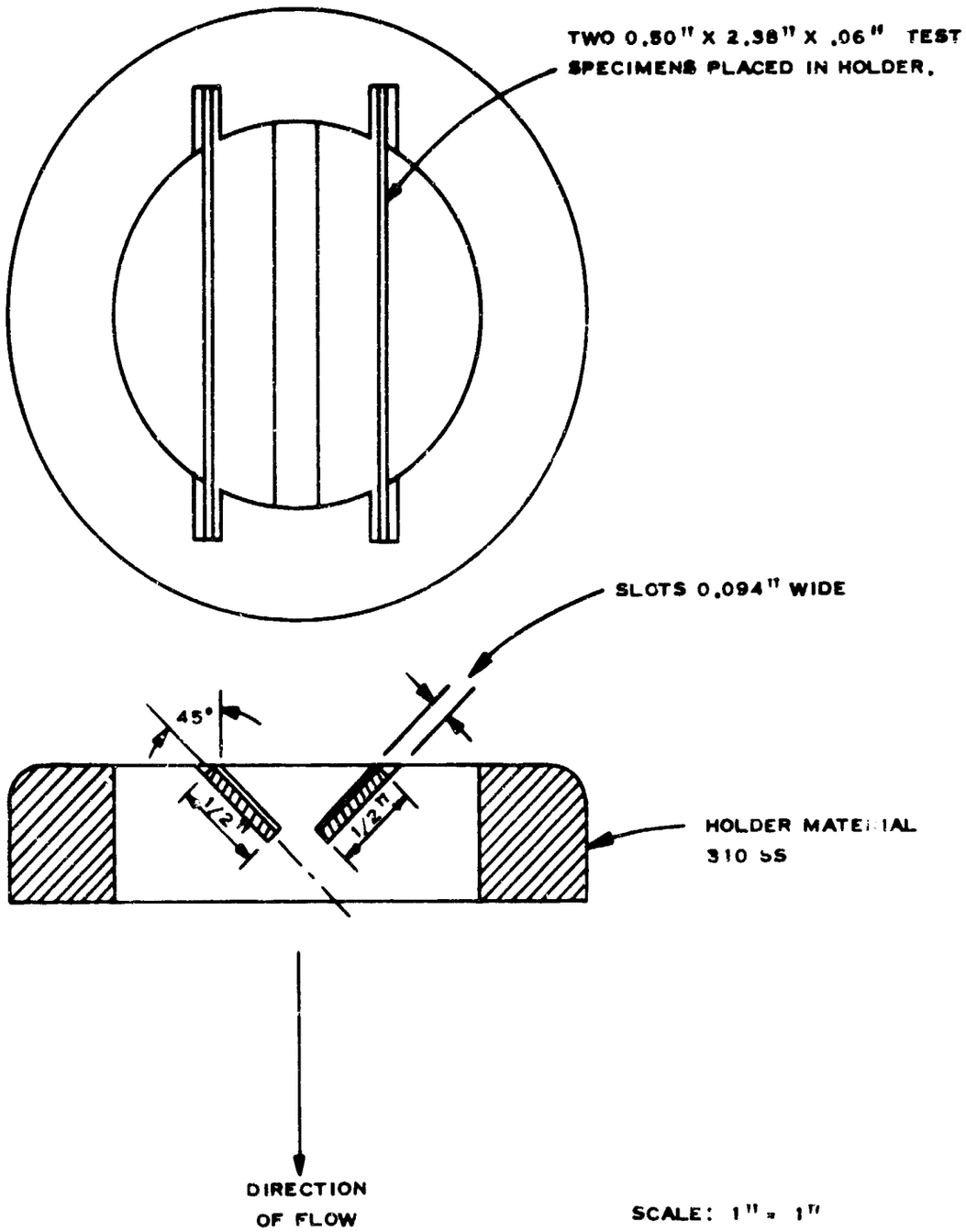

FIGURE 2
TEST SPECIMEN HOLDER FOR PHILLIPS 2-INCH COMBUSTOR

C. Specimen Electrocleaning

A very satisfactory technique for the removal of specimen scale or bulk oxide after exposure to hot gas corrosion has been described by Shirley (Ref. 8). This technique was used in this study. A schematic diagram of the cathodic descaling apparatus is shown in Figure 3. Test specimens have a No. 30 drill hole in one end for hanging in the caustic bath. Briefly, the technique involves immersing the specimens in molten sodium hydroxide (750-790 F) and passing about 1/3 ampere/cm^2 through the specimens for a period of 10 minutes. Thus for two test specimens with a total surface area of 35.28 cm^2, a direct current of 12 amperes was used. This is followed by a water quench. With new, unexposed specimens, only a negligible amount of metal is lost with this technique. The effects of cathodic descaling are discussed in greater detail in the Results section of the report.

D. Specimen Modification for Tensile

As shown in Figure 2, a test specimen consisted of a coupon of super alloy 0.50 in. wide, 2.38 in. long, and 0.06 in. thick. After exposure to hot gas corrosion in the combustor, preweighed specimens were cleaned as described in Section II-C and then reweighed to determine the weight loss.

Prior to the measurement of the tensile properties, both before and after exposure to hot gas corrosion, the new or cleaned specimen was filleted as shown in Figure 4. The width of the tensile specimen, 0.250 ± 0.002 in., was easily measured with a micrometer. However, when the specimen had been exposed to hot gas corrosion, the irregular surfaces of the top and bottom sides made the thickness more difficult to measure. Therefore, there was a minor uncertainty in the cross-sectional area to be used in the calculation of the ultimate tensile strength of specimens after exposure to hot gas corrosion.

The tensile specimens were pulled in an Instron Model TTC1 tensile machine operated at a crosshead travel rate of 0.1 in./min. The gauge length of the tensile specimen was 0.875 in.

III. TEST MATERIALS

A. Fuel

1. Suflur in Petroleum

Natural crude oil is composed of hydrocarbons, primarily. However, normally there are small amounts of organic compounds of sulfur, oxygen, and nitrogen present, in addition to very small amounts of metallo-organic compounds of vanadium, nickel, iron, and copper. These non-hydrocarbon constituents are usually concentrated in the higher-boiling-temperature portion of the crude oil, along with polynuclear aromatics and multi-ring

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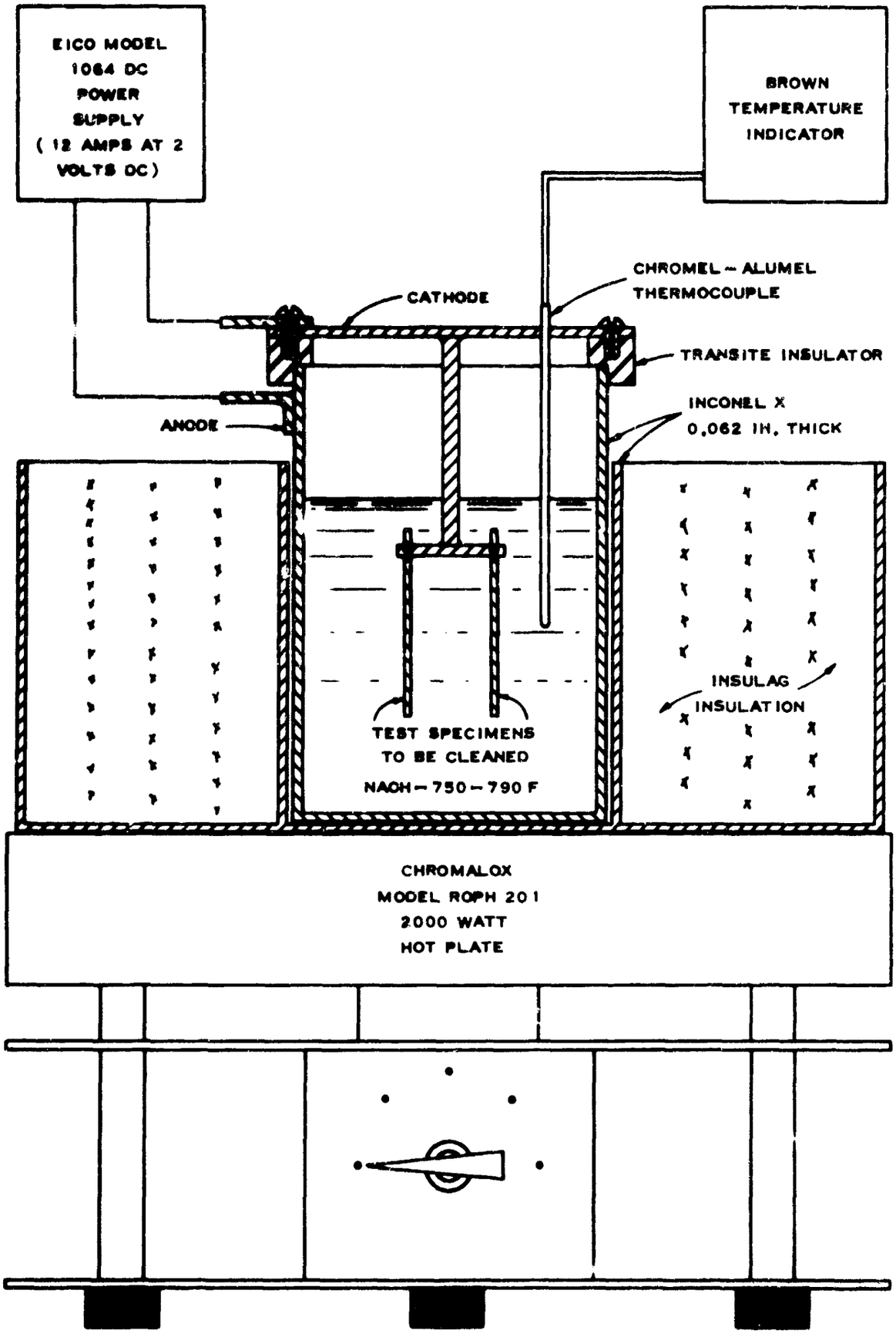

FIGURE 3
CATHODIC DESCALING APPARATUS
FOR CLEANING TEST SPECIMENS
FOLLOWING EXPOSURE TO HOT GAS CORROSION

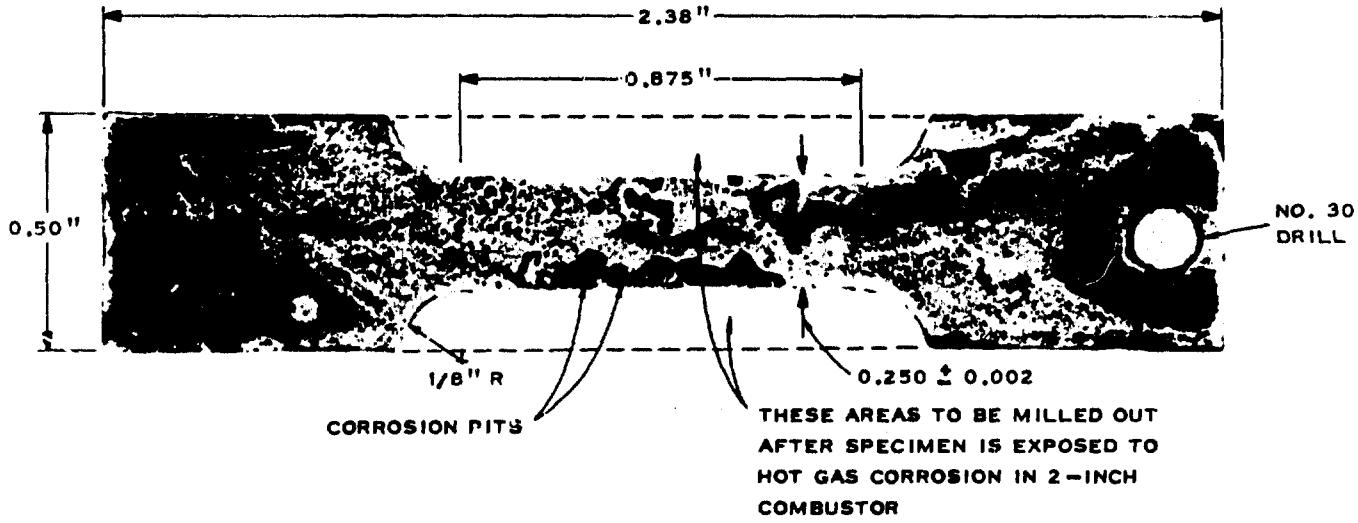

FIGURE 4
MODIFICATION OF TEST SPECIMEN FOR TENSILE EVALUATION
FOLLOWING EXPOSURE TO HOT GAS CORROSION

cycloparaffins. Generally, they have been avoided in aviation turbine fuels by specification of fractions boiling below 550 F.

Sulfur is the exception. Decomposition of high molecular weight sulfur compounds occurs during refining operations, to add lower-boiling sulfur compounds which were not constituents of the original crude oil. Thus, a kerosine type fraction, such as JP-5, usually contains sulfides (RS), disulfides (RS_2), etc.

The amount of sulfur in crude oil varies over a range of several orders of magnitude; i.e., from about 0.05 to 5 weight per cent. South American, Near and Middle East crude oils contain, on the average, more sulfur. However, crude oils produced from a given geographical region can vary greatly.

Conventional refinery distillation of crude oil normally concentrates about 95 per cent of the sulfur in the heavy distillate fraction and residual portion. This leaves the middle distillate fraction, used for aviation turbine fuel, relatively free of sulfur. Further removal of sulfur has long constituted an important part of refinery practice to stabilize products with respect to odor, color, and gum formation.

For more detailed discussions of the above information, see Ref. 19.

2. Sulfur in JP-5

The sulfur content of 54 samples of grade JP-5 aviation turbine fuel, representative of production in the United States from 1957 through 1963, averaged 0.102 weight per cent, but the median value was only 0.060 weight per cent sulfur (Ref. 20). These data are tabulated in Table II. It is pertinent to point out that the precision of the ASTM Lamp Method (D-1226) used by the manufacturers to obtain these data is 0.01 weight per cent sulfur, since 11 per cent of the samples were at, or below, this level. The majority of the samples, 54 per cent, were between 0.02 and 0.10 weight per cent in sulfur content. However, this left 35 per cent which approached the JP-5 specification maximum of 0.40 weight per cent sulfur.

It is of interest to note that 288 samples of the more volatile grade JP-4 aviation turbine fuel, averaged over the same period of time, showed a sulfur content of 0.044 weight per cent (Ref. 20). This is 43 per cent of the average sulfur content reported for JP-5.

On the basis of this and other similar information, it was decided to conduct this study using test fuels having three levels of sulfur concentration. One was chosen at the maximum sulfur content of 0.40 weight per cent allowed by the specification for grade JP-5 aviation turbine fuel, since a considerable quantity of production fuel approaches this level. Another was selected at an order of magnitude less in sulfur content, 0.040 weight per cent, to represent the level characteristic of the major portion of aviation turbine fuel produced. A decrease in sulfur content of another

order of magnitude, to 0.0040 weight per cent, seemed reasonable for the third fuel; however, it was felt that an even lower level would be desirable to allow testing with an essentially sulfur-free base fuel.

TABLE II

SULFUR CONTENT OF REPRESENTATIVE U. S. PRODUCTION SAMPLES OF JP-5

U. S. Bureau of Mines Petroleum Product Survey (Ref. 20)

	Total Sulfur Content, weight per cent							
Year	1957	1958	1959	1960	1961	1962	1963	
	0.02	0.024	0.01	0.004	0.005	0.005	0.01	
	0.063	0.053	0.022	0.038	0.023	0.01	0.027	
	0.16	0.072	0.027	0.078	0.032	0.024	0.033	
	0.18	0.160	0.031	0.182	0.035	0.027	0.039	
	0.22	0.16	0.036	0.23	0.046	0.028	0.050	
		0.29	0.04		0.056	0.067	0.087	
			0.23		0.22	0.08	0.09	
			0.35		0.23	0.097	0.15	
					0.25	0.18	0.19	
						0.18	0.32	
						0.32		
								Total
Average	0.129	0.110	0.093	0.106	0.100	0.093	0.100	0.102
Median	0.16	0.116	0.034	0.078	0.046	0.067	0.069	0.060

3. Base Fuel

The base fuel selected for use during this study was a segregated sample of production ASTM Type A aviation turbine fuel. Its physical and chemical properties of interest to this investigation are presented in Table III. For comparison purposes, the average values of pertinent properties from the Bureau of Mines Petroleum Product Survey over the period from 1957 through 1963 are shown for grades JP-5 and JP-4 aviation turbine fuel (Ref. 20). It should be noted that the physical and chemical properties of the base fuel closely approximate the averages for JP-5, with the exception of its very low sulfur content. The base fuel also was analyzed for metal content, to be certain that its iron, vanadium, nickel, and copper content were negligible; since, if present, they would concentrate as ash and might significantly alter the scale composition on the test specimen being exposed to the combustor exhaust gas.

The base fuel was essentially free of sulfur, containing only 2 parts sulfur per million parts of fuel by weight; i.e., 0.0002 weight per cent sulfur. The higher sulfur content test fuels were produced by blending to 0.040 and 0.40 weight per cent sulfur using ditertiary butyl disulfide. This dithiaalkane has been widely used in past research to obtain high sulfur content test fuels, since it is relatively inexpensive and available at adequate purity. Also, earlier work has shown organic sulfur compound type to be unimportant in hot gas corrosion studies (Ref. 12).

TABLE III

PHYSICAL AND CHEMICAL PROPERTIES OF TEST FUEL

	Test Fuel Base (a)	Average JP-5 (b)	Average JP-4 (b)
Distillation Temperature, F			
Initial Boiling Point	329		
5 volume per cent evaporated	344		
10 volume per cent evaporated	350	382	210
20 volume per cent evaporated	359		
30 volume per cent evaporated	368		
40 volume per cent evaporated	377		
50 volume per cent evaporated	388	413	307
60 volume per cent evaporated	400		
70 volume per cent evaporated	417		
80 volume per cent evaporated	435		
90 volume per cent evaporated	460	455	412
95 volume per cent evaporated	478		
End Point	498		
Gravity, degrees API	46.2	42.1	53.2
Gum, milligrams per 100 milliliters	0.2	1.0	1.0
Smoke Point, millimeters	26.2	23.1	28.1
Composition, weight per cent			
Sulfur	0.0002(c)	0.10	0.04
Metals (d)			
Iron — less than	0.0001		
Vanadium — less than	0.0001		
Nickel — less than	0.0001		
Copper — less than	0.0001		
Hydrocarbon Types			
Normal Paraffins	27 (e)		
Isoparaffins	23 (e)		
Cycloparaffins	36 (e)		
Olefins	0 (e)		
Aromatics	14.0	14.3	11.2

Notes:

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

(b) U. S. Bureau of Mines Petroleum Product Survey. (Ref. 20)

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

(d) X-ray fluorescence analysis.

(e) Typical value for this product.

B. Sea Water

1. Composition

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 IV.

Table V compares the composition of this synthetic sea water with the average composition of sea water, as reported by Goldberg (Ref. 21). Only the elements present in sea water at concentrations of 1 ppm, or greater, have been tabulated. Smaller concentrations of elements present in sea water are not included in the synthetic formula. It will be noted that the abundance of the various elements in the synthetic formula compares very favorably with the average sea water composition. The one exception is silicon, and its exclusion from the synthetic sea water seems justified in the light of its reported variation in abundance from one water-mass to another by a factor of 1000, or more.

It is pertinent to point out that sea water will leave a residue of approximately 4.2 per cent by weight of sea salt upon evaporation of the water. Thus, for our purposes, a concentration of 24 parts of sea water per million parts of air is equivalent to a concentration of sea salt in air of 1 ppm. This sea salt contains approximately 2 per cent by weight sulfur, combined with about 20 per cent of the available sodium as sodium sulfate, Na_2SO_4. The remaining sodium is available to combine with sulfur from the fuel to produce additional sodium sulfate, as noted by Simons, Browning and Liebhafsky (Ref. 7). If the sulfur contributed by the fuel were completely scavenged from the hot gas stream, it would require a fuel sulfur content of only 0.0005 weight per cent at an air-fuel ratio of 60 to convert the excess sodium to sodium sulfate with a sea salt ingestion rate of 1.0 ppm in air.

TABLE IV

COMPOSITION OF ASTM D665 SYNTHETIC SEA WATER

Salt (a)	Formula	grams per liter (b)
Sodium Chloride	$NaCl$	24.54
Magnesium Chloride	$MgCl_2.6H_2O$	11.10
Sodium Sulfate	Na_2SO_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_3BO_3	0.03
Strontium Chloride	$SrCl_2.6H_2O$	0.04
Sodium Fluoride	NaF	0.003
	Total	41.953

Notes:
(a) Use cp chemicals.
(b) Use distilled water.

TABLE V

COMPOSITION OF SEA WATER

Elements (a)	Principal Species	Abundance, grams per liter: Average Composition (Ref. 21)	Abundance, grams per liter: Synthetic Sea Water (ASTM D665)
Oxygen	H_2O; $O_2(g)$; SO_4^{2-}	857	857
Hydrogen	H_2O	108	108
Chlorine	Cl^-	19.0	19.8
Sodium	Na^+	10.5	11.0
Magnesium	Mg^{2+}; $MgSO_4$	1.35	1.33
Sulfur	SO_4^{2-}	0.89	0.92
Calcium	Ca^{2+}; $CaSO_4$	0.40	0.42
Potassium	K^+	0.38	0.39
Bromine	Br^-	0.065	0.068
Carbon	HCO_3^-; H_2CO_3; CO_3^{2-}; Organic	0.028	0.028
Strontium	Sr^{2+}; $SrSO_4$	0.008	0.013
Boron	$B(OH)_3$; $B(OH)_2O^-$	0.005	0.005
Fluorine	F^-	0.001	0.001
Silicon	$Si(OH)_4$; $Si(OH)_3O^-$	0.003 (b)	-----

Notes:
(a) Elements present at an abundance greater than 1 part per million.
(b) Silicon varies in abundance from one water-mass to another by a factor of 1000, or more.

Thus, the amount of sodium sulfate in the ash is likely to be limited only by the total sodium content of the hot gas stream.

2. Ingestion Rate

Establishing a realistic level for the concentration of sea salt in the air ingested by a gas turbine engine operating in a marine environment is difficult from the available literature. Woodstock and Gifford (Ref. 22) report a concentration at 50 feet over the calm ocean near Bermuda of approximately 0.003 parts by weight of sea salt per million parts of air (ppm). Cadle (Ref. 23) comments that salt particles over the ocean may at times be as concentrated as 100 particles per cubic centimeter, although one per cubic centimeter is more common. From this information, we can estimate a sea salt concentration over a rough sea of about 0.3 ppm. Of course, this level may be augmented by the vehicle. In fair agreement, unpublished data has indicated a sea salt concentration at the compressor intake of one marine application to be approximately 0.01 ppm under normal conditions, rising to 0.5 ppm in rough weather. Graves and Carleton (Ref. 24), U. S. Navy Bureau of Ships, point out that marinized gas turbine engines should be capable of satisfactory operation with a sea salt ingestion rate of 1 ppm. Other unpublished data report a sea salt ingestion rate of 1.5 ppm for a helicopter hovering at 20 feet above the ocean, with the rotor tip vortex action creating a considerable spray.

On the basis of this information, it was decided to conduct this study using three levels of sea salt ingestion. First, a corrosion base line was obtained with no sea salt added to the combustion system. Second, a realistic level of 1.5 ppm sea salt in air was obtained by the injection of synthetic sea water into the quench zone of the combustor, as indicated in Figure 1. Third, an accelerated corrosive effect was obtained by the ingestion of sea water at the level of 15.0 ppm sea salt in air. While the latter imposes artificially severe conditions, it is a common metallurgical practice to rely on accelerated testing for guidance.

C. Super Alloys

Two different nickel-base alloys were used as test specimens during this study. The selection of Inconel 713C and Sierra Metal 200 was made to obtain cast alloys representative of materials being used for turbine blades in engines of advanced design. The chemical analysis for the heats from which the investment castings were made are shown in Table VI. The inspection standards for these castings are shown in Table VII.

Inconel 713C has been widely used by aircraft gas turbine engine manufacturers for both turbine blades and turbine nozzle guide vanes. It possesses excellent strength properties up to 1800 F, and exhibits remarkable resistance to oxidation at that temperature. It is of interest to note that its introduction in 1956 led a series of cast alloys which permitted an increase in operating temperatures of about 100 F above previously available wrought materials.

Sierra Metal 200 is one of the newer cast alloys, introduced to obtain another 100 F increase in operating temperature; i.e., to 1900 F. This has required a reduction in chromium content to obtain high temperature strength properties, which has resulted in some lowering of oxidation resistance. It is of significance to note the unusually high tungsten content of this super alloy, for subsequent data show large amounts of sodium tungstate in the scale of test specimens suffering catastrophic rates of corrosion.

The mean initial area of the test specimens was 17.67 cm^2 The mean initial weight of the Inconel 713C test specimens was 9386 milligrams, and of the Sierra Metal 200 test specimens was 9754 milligrams.

TABLE VI

COMPOSITION OF SUPER ALLOY TEST SPECIMENS

Alloying Elements	Chemical Analysis, per cent Inconel 713C	Sierra Metal 200
Nickel	Balance (70.63)	Balance (60.34)
Cobalt	0.38	9.80
Chromium	12.93	9.14
Molybdenum	4.64	---
Tungsten	---	12.12
Aluminum	6.48	4.78
Titanium	0.82	2.00
Manganese	0.01	0.04
Iron	1.26	0.53
Zirconium	0.14	0.068
Columbium	2.25	0.99
Silicon	0.31	0.01
Boron	0.012	0.015
Sulfur	0.007	---
Carbon	0.13	0.17

TABLE VII

INSPECTION STANDARDS FOR SUPER ALLOY TEST SPECIMENS

1. Dimensional

   1.1 Investment castings were finished to 0.06 in. by 0.5 in. by 2.38 in., with a tolerance of ± 0.01 in.

   1.2 Positive roughness was removed, or reduced, by finishing to obtain a "smooth" section surface.

2. Visual

   2.1 Negative defects which did not exceed 1/16 in. diameter by 1/64 in. deep, and separated by a distance equal to the diameter of the larger defect, were acceptable.

   2.2 Evidence of mold crack, or partline, to 1/64 in. high, or deep, was acceptable.

   2.3 Positive roughness to 1/64 in. high was acceptable.

3. Fluorescent Penetrant (Zyglo)

   3.1 Cracks and through porosity were not acceptable.

   3.2 Large faintly fluorescent areas (1/4 in. diameter as a guide) in which definite glowing areas do not exceed 1/16 in. were not cause for rejection.

4. X-ray

   4.1 Cracks were not acceptable.

   4.2 Gas and inclusions up to 3/32 in. in their greatest dimension were acceptable.

IV. TEST PROGRAM

A. Procedure

The operating conditions selected as representative of modern, high performance aircraft are shown in Table VIII under the column heading 2000 F. It is seen that operation was at a pressure of 15 atmospheres, a reference inlet air velocity of 200 ft/sec, an inlet air temperature of 1000 F and an exhaust gas temperature closely approaching 2000 F. The resultant test specimen temperature was 1830 F and the flow velocity over the specimens was 500 ft/sec. Under these conditions, synthetic sea water was injected at the rates 0.0, 1.5 and 15 ppm sea salt with fuel sulfur contents of 0.0002, 0.040 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 the fuel turned off. On completion of a test, the specimens were cathodically cleaned, as described in Section II-C, for determination of weight loss. The tensile properties (ultimate tensile strength, ultimate load and per cent elongation) were measured after preparing the specimens for this purpose as described in Section II-D.

Photomicrographs of the specimens were made for examination to determine the depth and the type of the corrosion attack. X-ray diffraction analyses of the scale from some of the specimens were also made to provide information concerning the chemical composition of corrosion products.

In addition, a brief investigation was made of the effect of temperature on the hot gas corrosion of super alloy Inconel 713C. Tests, similar to those described above, were run at exhaust gas temperatures of 1500 and 1750 F under the conditions shown in Table VIII. In these tests, the sulfur content of the fuel was 0.40 weight per cent and the sea salt concentration in the combustor air was 15 ppm. A single test was made at each temperature and one specimen was used for weight loss measurement and the other for X-ray diffraction analysis of the scale and for photomicrographs.

Under the high temperature conditions (2000 F), supplementary tests were made with essentially sulfur free fuel substituting other sources of sodium compounds (sodium chloride and sodium hydroxide) for sea salt. Weight loss was measured and the scale was analysed by X-ray diffraction.

TABLE VIII

OPERATING CONDITIONS OF PHILLIPS 2-INCH COMBUSTOR

Test Variables	Test Conditions (a) 2000 F	1750 F	1500 F
Temperature, degrees Fahrenheit			
Exhaust Gas	1993 ± 15	1746 ± 11	1498 ± 7
Profile (b)	210	190	110
Test Specimens	1830	1720 (c)	1590 (c)
Combustor Inlet Air . . .	1000 ± 10	900 ± 10	800 ± 10
Pressure, atmospheres			
Combustor Inlet Air . . .	15.0 ± 0.1	15.0 ± 0.1	15.0 ± 0.1
Combustor Drop	0.6	0.8	1.0
Test Specimen Drop	0.3	0.4	0.5
Mass Flow Rate, pounds per hour			
Air	5480 ± 40	6120 ± 40	6840 ± 40
Fuel	98	90	81
Air-Fuel Ratio	56	68	84
Flow Velocity, feet per second			
Combustor Reference (d). .	200	210	220
Exhaust Gas (e)	270	270	270
at Test Specimens (f)	500	500	500
Combustion Efficiency, per cent (g)	100	100	100
Test Duration, hours (h) . . .	5.00 ± 0.01	5.00 ± 0.01	5.00 ± 0.01

Notes:

(a) Average values, with standard deviation shown for control points.

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

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

(d) Cold flow, based on 2.66 in.^{2} exit area in flame tube.

(e) Based on 3.36 in.^{2} area at outlet from combustor.

(f) Based on 1.80 in.^{2} unblocked area in test specimen holder.

(g) Calculated using mean specific heats and temperature at exhaust gas core to minimize error from heat loss to water cooled wall.

(h) Operating cycle of 55 minutes at test condition, followed by 5 minutes with fuel off.

B. Statistical Design

To evaluate the effect of three levels of fuel sulfur content and three levels of sea salt ingestion with air on hot gas corrosion of super alloys, an experiment was set up for each super alloy consisting of duplicate tests at each of the nine possible combinations of sulfur and sea salt. These 18 tests on each alloy were run in a random order. In each test two specimens of the super alloy were exposed to hot gases from the burner. This type of experiment is known as a split-plot design. The effects of sulfu sea salt and possible sulfur x sea salt interaction are evaluated in the main plots and position-to-position interactions are evaluated in sub-plots. Evaluations are based on analysis of (1) weight loss from exposure (2) ultimate load after exposure (3) ultimate tensile strength after exposure and (4) per cent elongation after exposure.

V. RESULTS

A. Basis for Analysis of Data

Four parameters (weight loss, ultimate tensile strength, per cent elongation and ultimate load) were studied for Inconel 713C and Sierra 200. Statistical methods were used in analyzing these data. Notations and tests used are more fully described in texts such as Snedecor (Ref. 25). An analysis of variance was made on each variable. Since this experiment was set up on a split-plot design, error (a) is the mean square to use in setting up variance ratios for "F" tests for significance of sulfur, sea salt or sulfur x sea salt effects while error (b) to test for position or position interactio effects. Throughout this analysis a confidence level of 95 per cent was used in testing for significance of effects, determining confidence limits and determining least significant difference (LSD) between means. In each analysi of variance table, "*" is used to indicate a significant effect.

In an analysis of variance when an interaction is found to be significant test for significance of main effects cannot be considered. Comparisons in this case can only be made of one factor with the level of the other specified. If the interaction is not significant comparisons can be made of the overall means of the main effects.

In the analysis of weight loss data, logarithms of the weight losses were used since the standard deviation varies approximately directly with the means. The antilogarithms of the average of the logarithms for a given test condition is the geometric mean of the data.

The method used in comparing weight losses was to determine the rati of means at two test conditions and establish confidence limits on the ratio. If the confidence includes "one" it can be concluded that there is no signifi cant difference in the weight losses. Ratios and confidence intervals were obtained as follows.

(1) Ratio. Obtained by taking difference between logarithms of the test conditions being compared. The antilogarithm of the difference is the ratio desired.

(2) Confidence intervals were obtained by adding and subtracting the least significant difference (LSD) to the differences in logarithms and then taking antilogarithms to obtain the upper and lower confidence intervals.

In comparing properties of samples after exposure with properties of new metal the LSD for unpaired observations and unequal variance was calculated from error (a) mean square and the error mean square from the variance analysis of the new metal. /Steel and Torrie (Ref. 26)/ This is a conservative approach and will tend to under rather than overstate significance.

B. Inconel 713C at 2000 F Test Conditions

1. Weight Loss

The weight loss obtained for each Inconel 713C test specimen at each test condition is shown in Table IX.

TABLE IX

WEIGHT LOSS (MG) OF INCONEL 713C

Sulfur in Fuel, Wt. %	Sea Salt in Air, ppm		
	0.0	1.50	15.0
0.0002	23.4	23.1	1008.7
	28.7	21.6	1335.3
	22.0	28.2	327.8
	18.0	8.7	598.6
0.040	18.5	106.9	906.0
	18.2	206.4	1008.4
	16.6	63.7	671.1
	13.0	69.6	919.8
0.40	19.3	294.8	1516.4
	9.5	209.2	2817.9
	7.4	74.3	1817.3
	17.8	69.4	2674.1

An analysis of variance of logarithms of weight loss is shown in Table X. The sulfur x sea salt interaction is significant and comparisons of one factor can be made only with the level of the other factor specified. The effect of position and the position interactions .. not significant.

TABLE X

ANALYSIS OF VARIANCE OF LOGARITHMS OF WEIGHT LOSS (MG) FOR INCONEL 713C

Source of Variation	Degrees of Freedom	Sum of Squares	Mean Square	F
Total	35	24.1394		
Sulfur	2	0.7748	0.3874	5.40 (1)
Sea Salt	2	20.7292	10.3646	144.35 (1)
Sulfur x Sea Salt	4	1.5694	0.3924	5.46*
Error (a)	9	0.6462	0.0718	
Position	1	0.0091	0.0091	0.38
Sulfur x Position	2	0.0151	0.0075	0.32
Sea Salt x Position	2	0.0880	0.0440	1.84
Sulfur x Sea Salt x Position	4	0.0925	0.0231	0.97
Error (b)	9	0.2152	0.0239	

(1) With a significant interaction, the test of main effects is vitiated.

The geometric means and the confidence limits are shown in Table XI.

TABLE XI

SUMMARY OF METAL WEIGHT LOSS OF INCONEL 713C

Sulfur in Fuel, Wt. %	Geometric Means, Mg. Sea Salt in Air, ppm		
	0.0	1.50	15.0
0.0002	10.1 ≤ 22.7 ≤ 45.6	9.3 ≤ 18.7 ≤ 37.6	357 ≤ 717 ≤ 1440
0.040	8.2 ≤ 16.4 ≤ 33.0	49.5 ≤ 99.4 ≤ 200	431 ≤ 866 ≤ 1740
0.40	6.2 ≤ 12.5 ≤ 25.0	66.5 ≤ 134 ≤ 268	863 ≤ 2130 ≤ 4290

Lower confidence limit ≤ geometric mean ≤ upper confidence limit

All of the possible comparisons of ratios of weight losses for sulfur concentrations with fixed sea salt concentrations and ratios of weight losses for sea salt concentrations with fixed sulfur concentrations are shown in Table XII.

TABLE XII

COMPARISONS OF RATIOS OF WEIGHT LOSS FOR CHANGE IN ONE VARIABLE WITH OTHER FIXED FOR INCONEL 713C

Sea Salt in Air, ppm	Comparison of Sulfur Concentrations		
	0.040/0.002 (1)	0.40/0.040 (1)	0.40/0.0002 (1)
0.0	0.22 ≤ 0.72 ≤ 1.94	0.28 ≤ 0.76 ≤ 2.03	0.20 ≤ 0.55 ≤ 1.47
1.50	1.98 ≤ 5.32 ≤ 14.3*	0.50 ≤ 1.34 ≤ 3.60	2.66 ≤ 7.16 ≤ 19.2*
15.0	0.45 ≤ 1.21 ≤ 3.24	0.92 ≤ 2.46 ≤ 6.61	1.11 ≤ 2.97 ≤ 7.99*
Sulfur in Fuel, Wt, %	**Comparison of Sea Salt Concentrations**		
	1.50/0.0 (1)	15.0/1.50 (1)	15.0/0.0 (1)
0.0002	0.31 ≤ 0.82 ≤ 2.21	14.3 ≤ 38.3 ≤ 103*	11.8 ≤ 31.6 ≤ 84.7*
0.040	2.26 ≤ 6.06 ≤ 15.9*	3.25 ≤ 8.71 ≤ 23.4*	19.6 ≤ 52.8 ≤ 141*
0.40	3.99 ≤ 10.7 ≤ 28.7*	5.96 ≤ 15.9 ≤ 42.9*	63.8 ≤ 170 ≤ 459*

(1) Ratios of geometric mean weight losses at concentrations indicated. Lower confidence limit ≤ ratio of means ≤ upper confidence limit.

The following conclusions can be drawn from these data.

(1) At 0.0 ppm sea salt, fuel sulfur concentration does not affect metal weight loss.

(2) At 1.50 ppm sea salt, fuel sulfur concentration of 0.0002 per cent causes less metal weight loss than 0.040 or 0.40 per cent sulfur.

(3) At 15.0 ppm sea salt, fuel sulfur concentration of 0.0002 per cent causes less metal weight loss than 0.40 per cent sulfur.

(4) Increased sea salt concentrations increased metal weight loss, with the exception of an increase in sea salt concentration from 0.0 to 1.50 ppm at the low (0.0002 per cent) concentration of fuel sulfur.

(5) Changes in sea salt concentration have a much greater effect on metal weight loss than sulfur concentration. The maximum increase for a change in fuel sulfur was 7.16 times for an increase from 0.0002 to 0.40 per cent fuel sulfur at 1.50 ppm sea salt while the maximum increase for a change in sea salt was 170 times for an increase from 0.0 to 15.0 ppm sea salt at 0.40 per cent fuel sulfur.

2. Ultimate Tensile Strength

The ultimate tensile strength for each test specimen at each test condition is shown in Table XIII. The mean tensile strength for each sulfur concentration and for each salt concentration is also shown.

TABLE XIII

ULTIMATE TENSILE STRENGTH OF INCONEL 713C

Sulfur in Fuel, Wt. %	Ultimate Tensile Strength, lb/in.^{2} X 10^{-3} Sea Salt in Air, ppm 0.0	1.50	15.0	Sulfur Mean
0.0002	117	112	122	
	120	121	102	
	128	126	120	
	119	131	123	120.1
0.040	123	121	110	
	126	120	99	
	123	123	117	
	113	125	117	118.1
0.40	131	123	114	
	138	119	129	
	129	115	108	
	129	114	107	121.3
Sea Salt Mean	124.7	120.8	114.0	

An analysis of variance of these data is shown in Table XIV.

TABLE XIV

ANALYSIS OF VARIANCE OF ULTIMATE TENSILE STRENGTH OF INCONEL 713C

Source of Variation	Degrees of Freedom	Sum of Squares	Mean Square	F
Total	35	2437.000		
Sulfur	2	64.500	32.250	0.39
Sea Salt	2	700.667	350.333	4.27*
Sulfur x Sea Salt	4	367.333	91.833	1.12
Error (a)	9	738.500	82.056	
Position	1	2.778	2.778	0.08
Sulfur x Position	2	49.389	24.694	0.68
Sea Salt x Position	2	69.889	34.944	0.97
Sulfur x Sea Salt x Position	4	118.444	29.611	0.82
Error (b)	9	325.500	36.167	

The preceding analysis shows that the effect of sea salt is significant while all other effects and interactions are not significant. From error (a) mean square a LSD for means of 12 values was calculated to be 8.4 (8400 lb/in.^{2}). An examination of the sea salt means in Table XIII shows that the only difference which exceeds 8.4 is for an increase in sea salt concentration from 0 to 15.0 ppm. This is a significant decrease in ultimate tensile strength of 10,700 lb/in.^{2}. A summary of ultimate tensile strength for each test condition is shown in Table XV.

TABLE XV

SUMMARY OF ULTIMATE TENSILE STRENGTH OF INCONEL 713C

Sulfur in Fuel, Wt. %	Ultimate Tensile Strength, lb/in.^{2} X 10^{-3}		
	Sea Salt in Air, ppm		
	0.0	1.50	15.0
0.0002	121.0	122.5	116.8
0.040	121.2	122.2	110.8
0.40	131.8	117.8	114.5

The effect of cathodic cleaning on tensile strength of Inconel 713C is shown in Table XVI. These data were obtained at the same time as data on the test specimens and were included to confirm the fact that this cleaning procedure did not affect the properties of the metal specimens and to obtain a base line on new metal for comparison with samples after test.

TABLE XVI

EFFECT OF CLEANING ON ULTIMATE TENSILE STRENGTH OF INCONEL 713C

	Ultimate Tensile Strength, lb/in.^{2} X 10^{-3}	
	No Cleaning	Cleaned
	112	133
	123	122
	105	124
	124	130
Mean	116.0	127.2
Overall Mean	121.[illegible]	

Analysis of Variance

Source of Variation	Degrees of Freedom	Sum of Squares	Mean Square	F
Total	7	581.875		
Cleaning	1	253.125	253.125	4.62
Error	6	328.750	54.79[illegible]	

These data show that cathodic cleaning had no significant effect on ultimate tensile strength of Inconel 713C. The ultimate tensile strength of new metal was 121,600 lb/in.^{2}. The LSD for comparing the mean of 8 new test specimens with the mean of 12 test specimens for a given sea salt or sulfur concentration is 8.7(8,700 lb/in.^{2}). With this LSD a mean for a sea salt or sulfur concentration of over 130.3 (130,300 lb/in.^{2}) or less than 112.9 (112,900 lb/in.^{2}) would be significantly different than the new metal. None of the means in Table XIII is significant.

3. Per Cent Elongation

The per cent elongation for each test specimen at each test condition is shown in Table XVII. The mean elongation for each sulfur concentration and for each sea salt concentration is also shown.

TABLE XVII

PER CENT ELONGATION OF INCONEL 713C

Sulfur in Fuel, Wt. %	Per Cent Elongation — Sea Salt in Air, ppm: 0.0	1.50	15.0	Sulfur Mean
0.0002	7.07	6.85	6.39	
	8.20	8.44	4.78	
	7.86	13.00	5.24	
	5.70	11.40	5.92	7.57
0.040	6.72	7.64	5.59	
	6.95	6.61	6.84	
	7.06	7.30	3.88	
	5.70	8.55	3.88	6.39
0.40	8.77	5.13	5.92	
	10.24	6.61	4.22	
	9.35	5.48	5.70	
	8.90	5.47	3.65	6.62
Sea Salt Mean	7.71	7.71	5.17	

The analysis of variance of these data is shown in Table XVIII.

TABLE XVIII

ANALYSIS OF VARIANCE OF PER CENT ELONGATION OF INCONEL 713C

Source of Variation	Degrees of Freedom	Sum of Squares	Mean Square	F
Total	35	149.385		
Sulfur	2	9.368	4.684	1.49
Sea Salt	2	51.647	25.823	8.23*
Sulfur x Sea Salt	4	44.226	11.056	3.52
Error (a)	9	28.235	3.137	
Position	1	0.232	0.232	0.20
Sulfur x Position	2	0.233	0.117	0.10
Sea Salt x Position	2	1.092	0.546	0.47
Sulfur x Sea Salt x Position	4	3.962	0.990	0.86
Error	9	10.391	1.154	

The only significant effect shown for the above data is for sea salt. Using error (a) mean square the LSD for means of 12 values was calculated to be 1.64 per cent elongation. An examination of sea salt means in Table XVII shows no difference in elongation for 0.0 and 1.50 ppm sea salt; however, 15.0 ppm sea salt gave significantly less elongation (2.54 per cent) than either 0.0 or 1.50 ppm sea salt. A summary of per cent elongation for each test condition is shown in Table XIX.

TABLE XIX

SUMMARY OF PER CENT ELONGATION FOR INCONEL 713C

Sulfur in Fuel, Wt. %	Per Cent Elongation — Sea Salt in Air, ppm		
	0.0	1.50	15.0
0.0002	7.21	9.92	5.58
0.040	6.61	7.52	5.05
0.40	9.32	5.67	4.87

The effect of cathodic cleaning on per cent elongation of Inconel 713C is shown in Table XX.

TABLE XX

EFFECT OF CLEANING ON PER CENT ELONGATION OF INCONEL 713 C

	Per Cent Elongation	
	No Cleaning	Cleaned
	6.84	7.64
	6.43	7.52
	4.56	12.00
	9.91	7.98
Mean	6.94	8.78
Overall Mean	7.86	

Analysis of Variance

Source of Variation	Degrees of Freedom	Sum of Squares	Mean Square	F
Total	7	35.496		
Cleaning	1	6.845	6.845	1.4
Error	6	28.651	4.775	

No significant effect was found for cathodic cleaning on per cent elongation of Inconel 713C. The per cent elongation of the new test specimens was found to be 7.86 per cent. The LSD for comparing the mean of 8 new test specimens with the mean of 12 test specimens for a given sea salt or sulfur concentration is 2.39 per cent elongation. With this LSD a mean for a sea salt or sulfur concentration of less than 5.47 would be significant. At 15.0 ppm sea salt an elongation of 5.17 per cent was significantly lower than the new metal.

4. Ultimate Load

The ultimate load for each test specimen at each test condition is shown in Table XXI. The mean ultimate load for each sulfur concentration and for each sea salt concentration is also shown.

TABLE XXI

ULTIMATE LOAD OF INCONEL 713 C

Sulfur in Fuel, Wt. %	Ultimate Load, lb × 10^{-3} Sea Salt in Air, ppm 0.0	1.50	15.0	Sulfur Mean
0.0002	1.780	1.820	1.730	
	2.020	1.900	1.520	
	2.060	1.970	1.630	
	1.920	2.210	1.760	1.860
0.040	1.890	1.820	1.780	
	1.940	1.830	1.830	
	1.810	2.070	1.420	
	1.770	1.900	1.310	1.781
0.40	2.000	1.840	1.840	
	2.230	1.810	1.320	
	1.930	1.740	1.470	
	2.090	1.770	1.130	1.764
Sea Salt Mean	1.953	1.890	1.562	

An analysis of variance of the ultimate load data is shown in Table XXII.

TABLE XXII

ANALYSIS OF VARIANCE OF ULTIMATE LOAD OF INCONEL 713 C

Source of Variation	Degrees of Freedom	Sum of Squares	Mean Square	F
Total	35	2.0725		
Sulfur	2	0.0629	0.0314	0.716
Sea Salt	2	1.0609	0.5304	12.085*
Sulfur x Sea Salt	4	0.1956	0.0489	1.114
Error (a)	9	0.3950	0.0439	
Position	1	0.0032	0.0032	0.294
Sulfur x Position	2	0.0285	0.0142	1.307
Sea Salt x Position	2	0.1031	0.0515	4.728*
Sulfur x Sea Salt x Position	4	0.1251	0.0313	2.870
Error (b)	9	0.0982	0.0109	

The only significant main effect shown for the above data is for sea salt. The sea salt x position sub-plot interaction is also significant. The significance of the sea salt x position interaction in the sub-plot analysis was disregarded since there was no logical basis on which to explain its occurrence. Also, based on the analysis of the other variables and the feeling of the researchers involved, it was disregarded. Using error (a) mean square the LSD for means of 12 values was calculated to be 0.193 (193 lb). Comparing the mean ultimate loads for sea salt in Table XXI it can be seen that the mean ultimate load for

15.0 ppm sea salt of 1.562 (1562 lb) is significantly less than for 0.0 or 1.50 ppm sea salt; however, the difference between 0.0 and 1.50 ppm sea salt is not significant. Table XXIII is a summary of the ultimate load for each test condition.

TABLE XXIII

SUMMARY OF ULTIMATE LOAD FOR INCONEL 713 C

Sulfur in Fuel, Wt. %	Ultimate Load, lb X 10^{-3} Sea Salt in Air, ppm 0.0	1.50	15.0
0.0002	1.945	1.975	1.660
0.040	1.852	1.905	1.585
0.40	2.062	1.790	1.440

The effect of cleaning on ultimate load of Inconel 713 C is shown in Table XXIV.

TABLE XXIV

EFFECT OF CLEANING ON ULTIMATE LOAD OF INCONEL 713 C

	Ultimate Load, lb X 10^{-3} No Cleaning		Cleaned
	1.755		2.030
	2.010		1.820
	1.570		1.910
	1.920		2.020
Mean	1.814		1.945
Overall Mean		1.879	

Analysis of Variance

Source of Variation	Degrees of Freedom	Sum of Squares	Mean Square	F
Total	7	0.1768		
Cleaning	1	0.0344	0.0344	1.452
Error	6	0.1424	0.0237	

No significant effect was found for cathodic cleaning on ultimate load of Inconel 713 C. The ultimate load for the new test specimens was 1.879 (1879 lb).

The LSD for comparing the mean of 8 new test specimens with the mean of 12 test specimens for a given sea salt or sulfur concentration is 0.191 (191 lb). With this LSD a mean for a sea salt or sulfur concentration of over 2.070 (2070 lb) or less than 1.688 (1688 lb) would be significantly different than the new metal. In Table XXI the mean for 15.0 ppm sea salt of 1.562 (1562 lb) is lower than the mean for the new metal.

C. Sierra Metal 200 at 2000 F Test Conditions.

1. Weight Loss

The weight loss obtained for each Sierra 200 test specimen at each test condition is shown in Table XXV.

TABLE XXV

WEIGHT LOSS (MG) OF SIERRA 200

Sulfur in Fuel, Wt. %	Sea Salt in Air, ppm 0.0	1.50	15.0
0.002	106.1	1710.1	4045.4
	245.4	2571.2	3722.6
	95.5	1551.8	4084.7
	82.5	1527.9	4827.1
0.040	1081.6	465.6	2092.3
	483.7	436.1	3038.7
	836.2	517.9	1347.7
	331.3	527.8	2704.7
0.40	164.7	1105.3	1173.5
	89.0	194.9	965.2
	118.3	283.0	154.2
	60.4	70.9	235.0

An analysis of variance of logarithms of weight loss is shown in Table XXVI.

TABLE XXVI

ANALYSIS OF VARIANCE OF LOGARITHMS OF WEIGHT LOSS (MG) FOR SIERRA 200

Source of Variation	Degrees of Freedom	Sum of Squares	Mean Square	F
Total	35	11.8215		
Sulfur	2	2.9522	1.4761	13.58 (1)
Sea Salt	2	5.0154	2.5077	23.07 (1)
Sulfur x Sea Salt	4	2.0199	0.5050	4.64*
Error (a)	9	0.9784	0.1087	
Position	1	0.0732	0.0732	7.60 (1)
Sulfur x Position	2	0.2333	0.1166	12.11 (1)
Sea Salt x Position	2	0.1641	0.0820	8.52 (1)
Sulfur x Sea Salt x Position	4	0.2984	0.0746	7.75*
Error (b)	9	0.0867	0.0096	

(1) With a significant interaction, the test of main effects is vitiated.

The sulfur x sea salt interaction is significant and comparisons of sulfur or sea salt can only be made with the level of the other factor specified. The significance of the sulfur x sea salt x position interaction in the sub-plot analysis was disregarded since there was no logical basis on which to explain its occurrence. The mean square for this interaction is less than the main plot error (a) mean square.

The geometric mean and confidence limits of weight loss for each test condition are shown in Table XXVII.

TABLE XXVII

SUMMARY OF METAL WEIGHT LOSS OF SIERRA 200

Sulfur in Fuel, Wt. %	Geometric Means, mg. Sea Salt in Air, ppm 0.0	1.50	15.0
0.0002	50.7 ≤ 120 ≤ 282	762 ≤ 1790 ≤ 4240	1760 ≤ 4150 ≤ 9790
0.040	262 ≤ 617 ≤ 1470	207 ≤ 485 ≤ 1150	930 ≤ 2190 ≤ 5280
0.40	42.8 ≤ 101 ≤ 238	108 ≤ 256 ≤ 603	191 ≤ 450 ≤ 1060

lower confidence limit ≤ geometric mean ≤ upper confidence limit

All possible comparisons of ratios of weight losses for sulfur concentrations with fixed sea salt concentrations and ratios of weight losses for sea salt concentrations with fixed sulfur concentrations are shown in Table XXVIII.

TABLE XXVIII

COMPARISON OF RATIOS OF WEIGHT LOSS FOR CHANGE IN ONE VARIABLE WITH OTHER FIXED FOR SIERRA 200

Sea Salt in Air, ppm	Comparison of Sulfur Concentrations 0.0002/0.040 (1)	0.040/0.40 (1)	0.0002/0.40 (1)
0.0	0.06 ≤ 0.19 ≤ 0.65 (2)	1.81 ≤ 6.11 ≤ 20.6*	0.35 ≤ 1.19 ≤ 3.99
1.50	1.09 ≤ 3.69 ≤ 12.5*	0.56 ≤ 1.89 ≤ 6.39	2.09 ≤ 6.99 ≤ 23.7*
15.0	0.56 ≤ 1.89 ≤ 6.37	1.46 ≤ 4.87 ≤ 16.4*	2.74 ≤ 9.22 ≤ 31.0*
Sulfur in Fuel, Wt. %	**Comparison of Sea Salt Concentrations 1.50/0.0 (1)**	**15.0/1.50 (1)**	**15.0/0.0 (1)**
0.0002	4.46 ≤ 14.9 ≤ 50.6*	0.69 ≤ 2.32 ≤ 7.78	10.3 ≤ 34.6 ≤ 117*
0.040	0.23 ≤ 0.79 ≤ 2.65	1.34 ≤ 4.52 ≤ 15.2*	1.06 ≤ 3.55 ≤ 12.0*
0.40	0.75 ≤ 2.53 ≤ 8.52	0.52 ≤ 1.76 ≤ 5.93	1.32 ≤ 4.46 ≤ 15.0*

(1) Ratio of geometric mean weight losses at concentrations indicated.
(2) Inverse ratio significant.
lower confidence limit ≤ ratio of mean ≤ upper confidence limit

The following conclusions can be drawn from these data.

(1) At 0.0 ppm sea salt, increasing fuel sulfur from 0.0002 to 0.04 per cent increases metal weight loss while increasing fuel sulfur from 0.040 to 0.40 per cent decreases metal weight loss.

(2) At 1.50 ppm sea salt, fuel sulfur concentrations of 0.040 or 0.40 per cent cause less metal weight loss than 0.0002 per cent sulfur.

(3) At 15.0 ppm sea salt, fuel sulfur concentration of 0.40 per cent causes less metal weight loss than 0.0002 or 0.040 per cent sulfur.

(4) Increasing sulfur concentration showed a significant decrease in metal weight loss or was directionally lower in weight loss in the case of non-significance except for a significant increase in weight loss with an increase in sulfur from 0.0002 to 0.040 per cent at 0.0 ppm sea salt.

(5) At 0.0002 per cent sulfur, increasing sea salt concentration from 0.0 to either 1.50 or 15.0 ppm increased metal weight loss.

(6) At 0.040 per cent sulfur, increasing sea salt concentration from 0.0 to 15.0 or 1.50 to 15.0 ppm increased metal weight loss.

(7) At 0.40 per cent sulfur, increasing sea salt concentration from 0.0 to 15.0 ppm increased metal weight loss.

(8) Increasing sea salt concentration showed a significant increase in metal weight loss or was directionally higher in the cases of non-significance except for a non-significant decrease for a change from 0.0 to 1.50 ppm sea salt at 0.040 per cent sulfur.

2. Ultimate Tensile Strength

The ultimate tensile strength for each test specimen at each test condition is shown in Table XXIX.

TABLE XXIX

ULTIMATE TENSILE STRENGTH OF SIERRA 200

Sulfur in Fuel, Wt. %	Ultimate Tensile Strength, lb/in.^{2} X 10^{-3} Sea Salt in Air, ppm 0.0	1.50	15.0
0.0002	123	106	55 (c)
	124	103 (c)	91
	123	122	95
	126	111 (c)	71
0.040	115	111	78 (c)
	118	114	14 (c)
	96	102	76
	115	118	69 (c)
0.40	113	104	84 (c)
	103	108	111
	118	120	118
	102	102	111

(c) Examination of the break indicated a crack in the test specimen.

The number of test specimens that an examination of the break indicated to have been cracked may affect the analysis of the ultimate tensile strength, per cent elongation and ultimate load data. As mentioned earlier these test specimens were free of cracks prior to test. It may be observed that of the seven specimens showing a crack five were from tests with 15.0 ppm sea salt, two with 1.50 ppm sea salt and none with 0.0 ppm sea salt.

An analysis of variance of the ultimate tensile strength data is shown in Table XXX.

TABLE XXX

ANALYSIS OF VARIANCE OF ULTIMATE TENSILE STRENGTH OF SIERRA 200

Source of Variation	Degrees of Freedom	Sum of Squares	Mean Square	F
Total	35	18459.889		
Sulfur	2	1264.889	632.444	4.09
Sea Salt	2	7959.389	3979.694	25.72 (1)
Sulfur x Sea Salt	4	3710.111	927.528	6.28*
Error (a)	9	1392.500	154.722	
Position	1	64.000	64.000	0.25
Sulfur x Position	2	44.667	22.333	0.09
Sea Salt x Position	2	69.500	34.750	0.14
Sulfur x Sea Salt x Position	4	1700.333	425.083	1.70
Error (b)	9	2254.500	250.500	

(1) With a significant interaction, the test of main effects is vitiated.

The above analysis shows a significant sulfur x sea salt interaction and comparisons of main effects must be made with the level of one variable specified. The effect of position and the position interactions are not significant. A summary of ultimate tensile strength for each test condition is shown in Table XXXI.

TABLE XXXI

SUMMARY OF ULTIMATE TENSILE STRENGTH OF SIERRA 200

Sulfur in Fuel, Wt. %	Mean Ultimate Tensile Strength, lb/in.^{2} 10^{-3} — Sea Salt in Air, ppm		
	0.0	1.50	15.0
0.0002	124.0	110.5	78.0
0.040	111.0	111.2	59.2
0.40	109.0	108.5	106.0

From error (a) mean square the LSD for means of 4 values was calculated to be 19.90 (19,900 lb/in.^{2}) With this LSD the following conclusions can be drawn on ultimate tensile strength of Sierra 200.

(1) At 0.0 and 1.50 ppm sea salt, an increase in sulfur concentration made no significant change.

(2) At 15.0 ppm sea salt, fuel concentrations of 0.0002 and 0.040 per cent sulfur have lower ultimate tensile strengths than for 0.40 per cent sulfur.

(3) At 0.0002 and 0.040 per cent sulfur, salt concentration of 15.0 ppm sea salt has a significantly lower tensile strength than for 0.0 or 1.50 ppm sea salt.

(4) At 0.40 per cent sulfur, changes in sea salt concentration have no significant effect on tensile strength.

The effect of cathodic cleaning on ultimate tensile strength of Sierra 200 is shown in Table XXXII.

TABLE XXXII

EFFECT OF CLEANING ON ULTIMATE TENSILE STRENGTH OF SIERRA 200

	Ultimate Tensile Strength, lb/in.^{2} X 10^{-3}		
	No Cleaning		Cleaned
	137		140
	138		140
	143		123
	130		137
Mean	137.0		135.0
Overall Mean		136.0	

Analysis of Variance

Source of Variation	Degrees of Freedom	Sum of Squares	Mean Square	F
Total	7	292.00		
Cleaning	1	8.00	8.00	0.16
Error	6	284.00	47.33	

These data show that cathodic cleaning had no significant effect on ultimate tensile strength of Sierra 200. The ultimate tensile strength of the new metal was 136,000 lb/in.^{2} The LSD for comparing the mean of 8 new test specimens with the mean of 4 test specimens at a given test condition is 15.3 (15,300 lb/in.^{2}). With this LSD any mean in Table XXXI less than 120.7 (120,700 lb/in.^{2}) is significantly lower than the new metal. With 0.0 sea salt at 0.0002 per cent sulfur the tensile strength is unchanged from the new metal while all other operating conditions resulted in a loss in tensile strength.

3. Per Cent Elongation

The per cent elongation for each test specimen at each test condition is shown in Table XXXIII. The mean elongation for each sulfur concentration and for each sea salt concentration is also shown.

TABLE XXXIII

PER CENT ELONGATION OF SIERRA 200

Sulfur in Fuel, Wt. %	Per Cent Elongation — Sea Salt in Air, ppm: 0.0	1.50	15.0	Sulfur Mean
0.0002	15.0	9.0	8.0 (c)	
	16.0	10.0 (c)	13.0	
	16.0	17.0	7.0	
	20.0	13.0 (c)	8.0	12.7
0.040	13.0	11.0	9.0 (c)	
	15.0	18.0	4.0 (c)	
	15.0	16.0	9.0	
	17.0	14.0	7.0 (c)	12.3
0.40	12.0	12.0	10.0 (c)	
	14.0	14.0	10.0	
	19.0	16.0	13.0	
	14.0	17.0	14.0	13.8
Sea Salt Mean	15.5	13.9	9.3	

(c) Examination of break indicated a crack in the test specimen.

An analysis of variance of the elongation data is shown in Table XXXIV.

TABLE XXXIV

ANALYSIS OF VARIANCE OF PER CENT ELONGATION OF SIERRA 200

Source of Variation	Degrees of Freedom	Sum of Squares	Mean Square	F
Total	35	494.750		
Sulfur	2	13.167	6.583	0.668
Sea Salt	2	246.167	123.083	12.482*
Sulfur x Sea Salt	4	54.167	13.542	1.373
Error (a)	9	88.750	9.861	
Position	1	3.361	3.361	0.222
Sulfur x Position	2	2.389	1.194	0.079
Sea Salt x Position	2	1.722	0.861	0.057
Sulfur x Sea Salt x Position	4	37.278	9.319	0.614
Error (b)	9	136.500	15.167	

The only significant effect indicated by the above analysis is for sea salt. Using error (a) mean square the LSD for means of 12 values was calculated to be 2.90 per cent elongation. Directionally an increase in sea salt concentration decreased per cent elongation and 15.0 ppm sea salt had significantly less per cent elongation than 0.0 or 1.50 ppm sea salt. In Table XXXV a summary is shown of per cent elongation for each test condition.

TABLE XXXV

SUMMARY OF PER CENT ELONGATION FOR SIERRA 200

Sulfur in Fuel, Wt. %	Per Cent Elongation — Sea Salt in Air, ppm: 0.0	1.50	15.0
0.0002	16.75	12.25	9.00
0.040	15.00	14.75	7.25
0.40	14.75	14.75	11.75

The effect of cathodic cleaning on per cent elongation of Sierra 200 is shown in Table XXXVI.

TABLE XXXVI

EFFECT OF CLEANING ON PER CENT ELONGATION OF SIERRA 200

	Per Cent Elongation: No Cleaning	Cleaned
	17.0	17.0
	16.0	18.0
	18.0	14.0
	18.0	19.0
Mean	17.2	17.0
Overall Mean	17.1	

Analysis of Variance

Source of Variation	Degrees of Freedom	Sum of Squares	Mean Square	F
Total	7	16.875		
Cleaning	1	0.125	0.125	0.045
Error	6	16.750	2.792	

No significant effect was found for cathodic cleaning on per cent elongation of Sierra 200. The per cent elongation of the new metal was found to be 17.1 per cent. The LSD for comparing the mean of 8 new test specimens with the mean of 4 test specimens at a given test condition is 3.83 per cent. With this LSD any mean in Table XXXV less than 13.27 per cent is significantly lower than the new metal. The per cent elongation for 1.50 ppm sea salt at 0.0002 per cent sulfur is significantly lower than the new metal and at 15.00 ppm sea salt at all three sulfur concentrations elongation is much lower than for new metal.

4. Ultimate Load

The ultimate load for each test specimen at each test condition is shown in Table XXXVII.

TABLE XXXVII

ULTIMATE LOAD OF SIERRA 200

Sulfur in Fuel, Wt. %	Ultimate Load, lb x 10^{-3} Sea Salt in Air, ppm 0.0	1.50	15.0
0.0002	1.820	1.320	0.284 (c)
	1.820	0.980 (c)	0.750
	1.750	1.615	0.500
	1.890	1.355 (c)	0.400
0.040	1.460	1.570	0.780 (c)
	1.660	1.570	0.116 (c)
	1.320	1.505	0.900
	1.730	1.700	0.625 (c)
0.40	1.645	1.165	1.180 (c)
	1.550	1.590	1.500
	1.750	1.785	1.725
	1.[illegible]	1.505	1.645

(c) Examination of the break indicated a crack in the test specimen.

An analysis of variance of the ultimate load data is shown in Table XXXVIII.

TABLE XXXVIII

ANALYSIS OF VARIANCE OF ULTIMATE LOAD OF SIERRA 200

Source of Variation	Degrees of Freedom	Sum of Squares	Mean Square	F
Total	35	8.1948		
Sulfur	2	0.8427	0.4214	9.23 (1)
Sea Salt	2	4.1319	2.0660	45.25 (1)
Sulfur x Sea Salt	4	2.0034	0.5008	10.97*
Error (a)	9	0.4110	0.0457	
Position	1	0.0006	0.0006	0.016
Sulfur x Position	2	0.0023	0.0011	0.032
Sea Salt x Position	2	0.0312	0.0156	0.449
Sulfur x Sea Salt x Position	4	0.4595	0.1149	3.311
Error (b)	9	0.3122	0.0347	

(1) With a significant interaction, the test of main effects is vitiated.

The sulfur x sea salt interaction is significant and comparison of sulfur or sea salt must be made with the other variable fixed. A summary of ultimate load data is shown in Table XXXIX.

TABLE XXXIX

SUMMARY OF ULTIMATE LOAD OF SIERRA 200

Sulfur in Fuel, Wt. %	Mean Ultimate Load lb X 10^{-3} Sea Salt in Air, ppm 0.0	1.50	15.0
0.0002	1.820	1.318	0.484
0.040	1.542	1.586	0.605
0.40	1.622	1.511	1.512

From error (a) of Table XXXVIII the LSD for means of 4 determinations at a given set of conditions was calculated to be 0.342 (342 lb). With this LSD the following conclusions can be drawn from Table XXXIX.

(1) At 0.0 and 1.50 ppm sea salt, increasing sulfur content caused no significant change in ultimate load.

(2) At 15.0 ppm sea salt, ultimate load was less at 0.0002 or 0.040 per cent sulfur than at 0.40 per cent sulfur.

(3) At 0.0002 per cent sulfur increasing sea salt from 0.0 to 1.50 ppm decreased ultimate load and increasing sea salt from 1.50 to 15.0 ppm decreased ultimate load.

(4) At 0.040 per cent sulfur, ultimate load with 15.0 ppm sea salt was lower than with 0.0 or 1.50 ppm sea salt.

(5) At 0.40 per cent sulfur, increasing sea salt concentration had no effect on ultimate load.

The effect of cleaning on ultimate load of Sierra 200 is shown in Table XL.

TABLE XL

EFFECT OF CLEANING ON ULTIMATE LOAD OF SIERRA 200

	Ultimate Load, lb X 10^{-3}		
	No Cleaning		Cleaned
	2.100		2.140
	2.120		2.060
	2.170		1.800
	1.960		2.100
Mean	2.088		2.025
Overall Mean		2.056	

Analysis of Variance

Source of Variation	Degrees of Freedom	Sum of Squares	Mean Square	F
Total	7	0.1028		
Cleaning	1	0.0078	0.0078	0.493
Error	6	0.0950	0.0158	

No significant effect was found for cleaning Sierra 200. The ultimate load for the new test specimens was 2.056 (2056 lb). The LSD for comparing the mean of 8 new test specimens with the mean of 4 test specimens at a given test condition is 0.265 (265 lb). With this LSD a mean of less than 1.791 (1791 lb) for any test condition in Table XXXIX indicates a loss in ultimate load. The only test condition which did not cause a loss in ultimate load was 0.0 ppm sea salt at 0.0002 per cent sulfur.

VIII. DISCUSSION

A. Oxidation and Erosion

In the design of the test program it was desired to evaluate the effects of (a) sulfur, (b) sea salt and (c) the possible interaction of sulfur and sea salt on hot gas corrosion. Data from tests with no sea salt and negligible fuel sulfur permit an evaluation of the effect of oxidation and corrosion on a specific alloy. Inconel 713C is essentially free of oxidation and erosion attack. Metal weight loss was 1.3 mg/cm^2. This amounts to 0.2 per cent per five hour test or 0.04 per cent per hour. Sierra Metal 200 was poorer than Inconel 713C with respect to resistance to oxidation and erosion although the attack was not considered excessive. Metal weight loss for Sierra Metal 200 was 6.8 mg/cm^2. This represents a 1.2 per cent loss of weight from the new specimen or 0.24 per cent loss per hour.

A visual inspection of the test specimens showed considerable bowing (one-eighth inch) of the Inconel 713C in the direction of gas flow and relatively none with the Sierra Metal 200. This relative difference in ability to withstand the force of the high velocity gas stream is in agreement with differences in tensile strength. This observation also indicates that the test specimens were under considerable stress during exposure to hot gas corrosion.

B. Sulfur Corrosion

The effect of sulfur, per se, was noted in the Results with zero sea salt in the air. Basically, in the absence of sea salt, increasing the fuel sulfur concentration from 0.0002 to 0.04 and 0.40 weight per cent respectively, did not significantly affect weight loss, ultimate tensile strength, elongation and ultimate load of Inconel 713C. Visual inspection of these test specimens showed little evidence of corrosion. An attempt has been made to show this by the photographs of test specimens in Figure 5. The insignificant changes in weight loss and tensile properties are shown in Figures 6, 7 and 8.

With Sierra Metal 200, in the absence of sea salt, increasing the fuel sulfur content from 0.0002 to 0.040 increased the metal weight loss slightly while increasing the fuel sulfur from 0.040 to 0.40 weight per cent decreased metal weight loss. Photographs of the Sierra Metal 200 test specimens under these conditions are shown in Figure 9. The variation in metal weight loss is shown graphically in Figures 10 and 11. An increase in fuel sulfur from 0.0002 to 0.040 and 0.40 weight per cent had no significant effect on the tensile properties of Sierra Metal 200, in the absence of sea salt. This is shown in Figure 12.

It appears that the effect of sulfur per se, without any sea salt present and at the 2000 F operating conditions, is almost negligible with regard to the hot gas corrosion of Inconel 713C and Sierra Metal 200.

C. Sea Salt Corrosion

The effect of sea salt in the combustor air per se, is shown by analyses in the Results section of this report when essentially sulfur free base fuel (2 ppm) was used. This effect is shown visually in Figures 6 and 7 for Inconel 713C. In the bar graphs of metal weight losses the geometric means and the upper and lower confidence limits of the means are shown. The presence of 1.5 ppm sea salt in combustor air did not increase metal weight loss in the essential absence of sulfur; however, at 15.0 ppm of sea salt metal weight loss was markedly increased. As shown in Figure 5, this metal loss was characterized by patches of attack and pitting; i.e., localized corrosion taking the form of cavities at the surface.

Tensile properties for Inconel 713C are shown in Figure 8. At 2 ppm fuel sulfur there is no directional trend of change in tensile properties with increased sea salt. This indicates that the sea salt attack was at the surface of the metal, with no deep intergranular penetration.

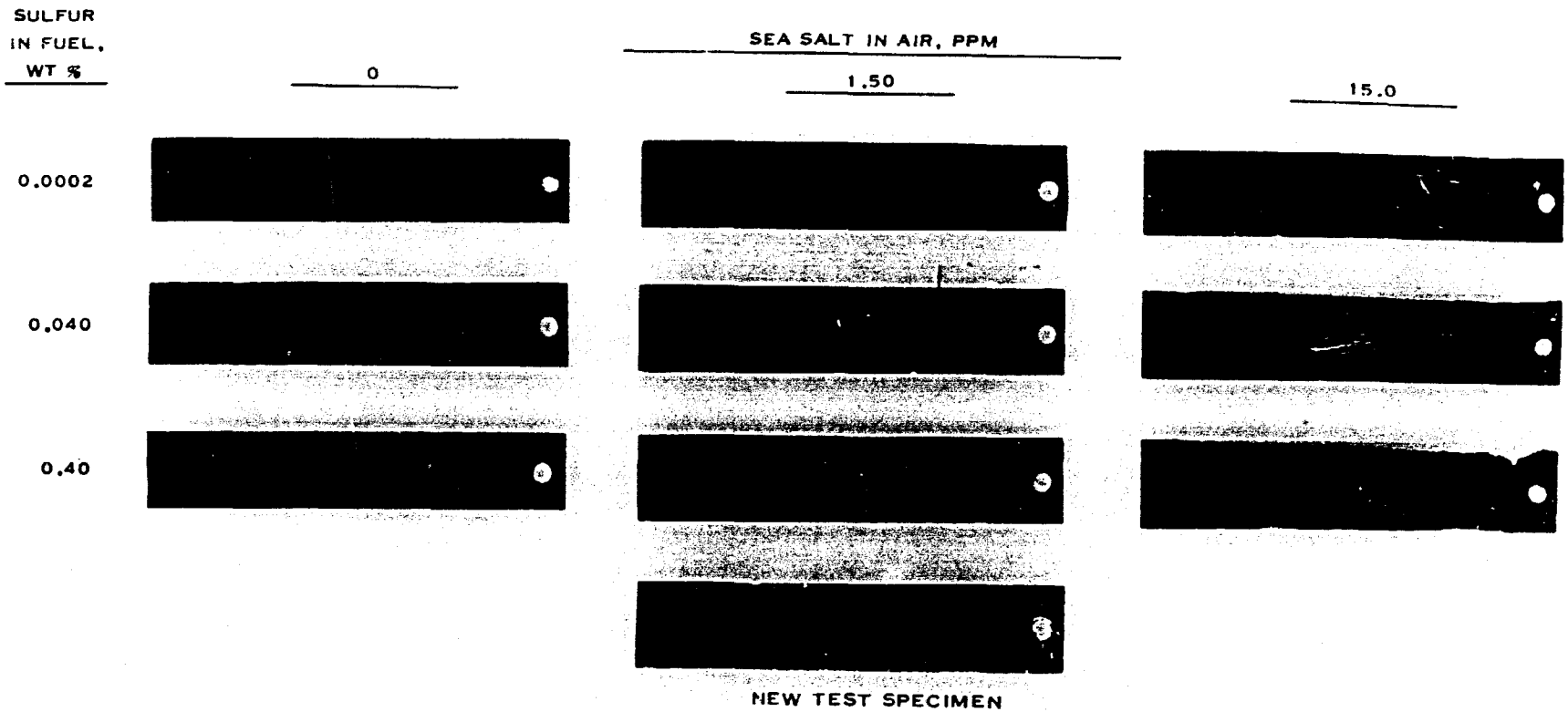

FIGURE 5
PHOTOGRAPH OF INCONEL 713C TEST SPECIMENS
AFTER EXPOSURE TO HOT GAS CORROSION IN PHILLIPS 2-INCH COMBUSTOR
AT 2000 F TEST CONDITION

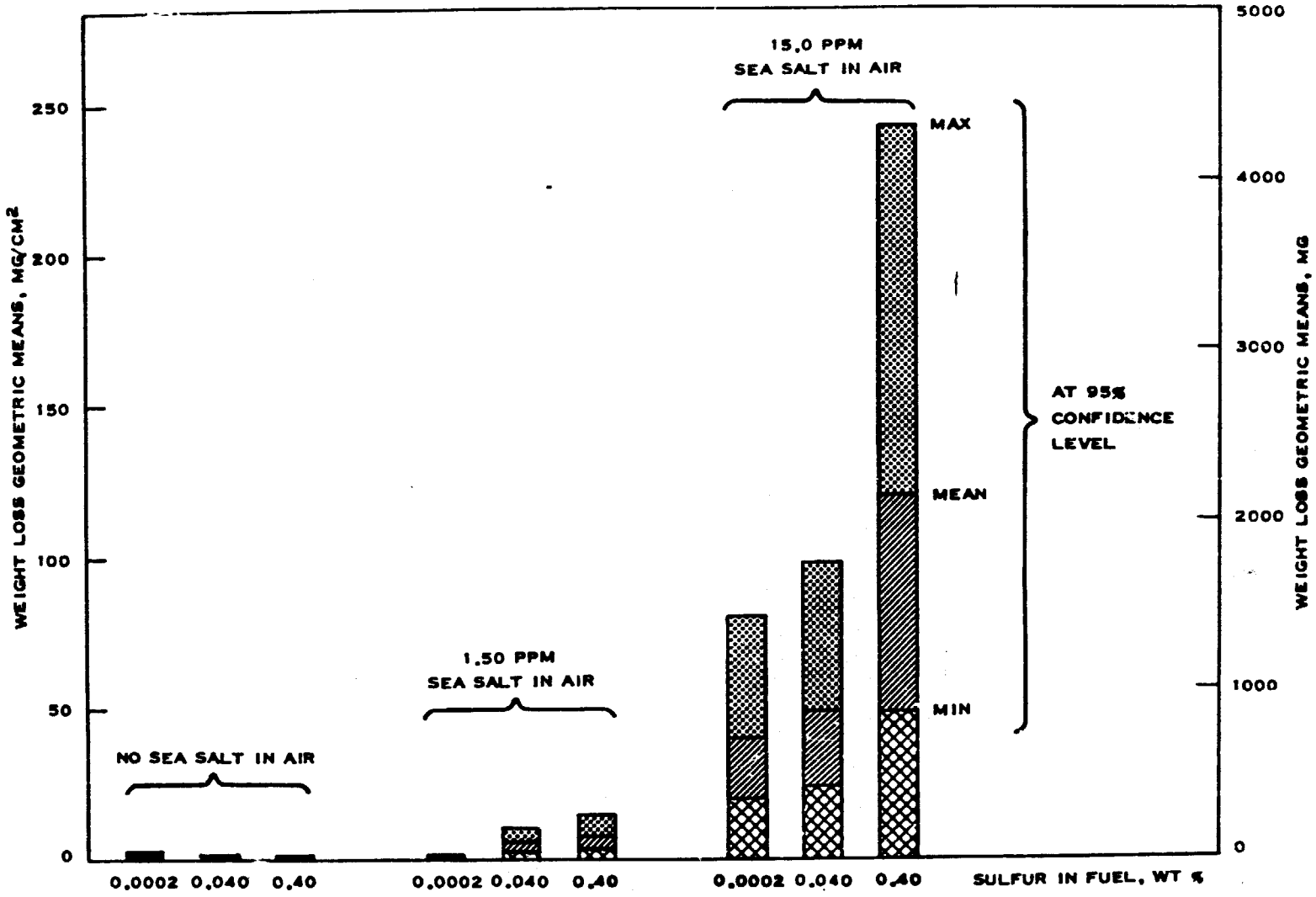

FIGURE 6
EFFECT OF SULFUR AND SEA SALT ON INCONEL 713C TEST SPECIMEN WEIGHT LOSS AS A RESULT OF EXPOSURE TO HOT GAS CORROSION IN PHILLIPS 2-INCH COMBUSTOR AT 2000 F TEST CONDITION

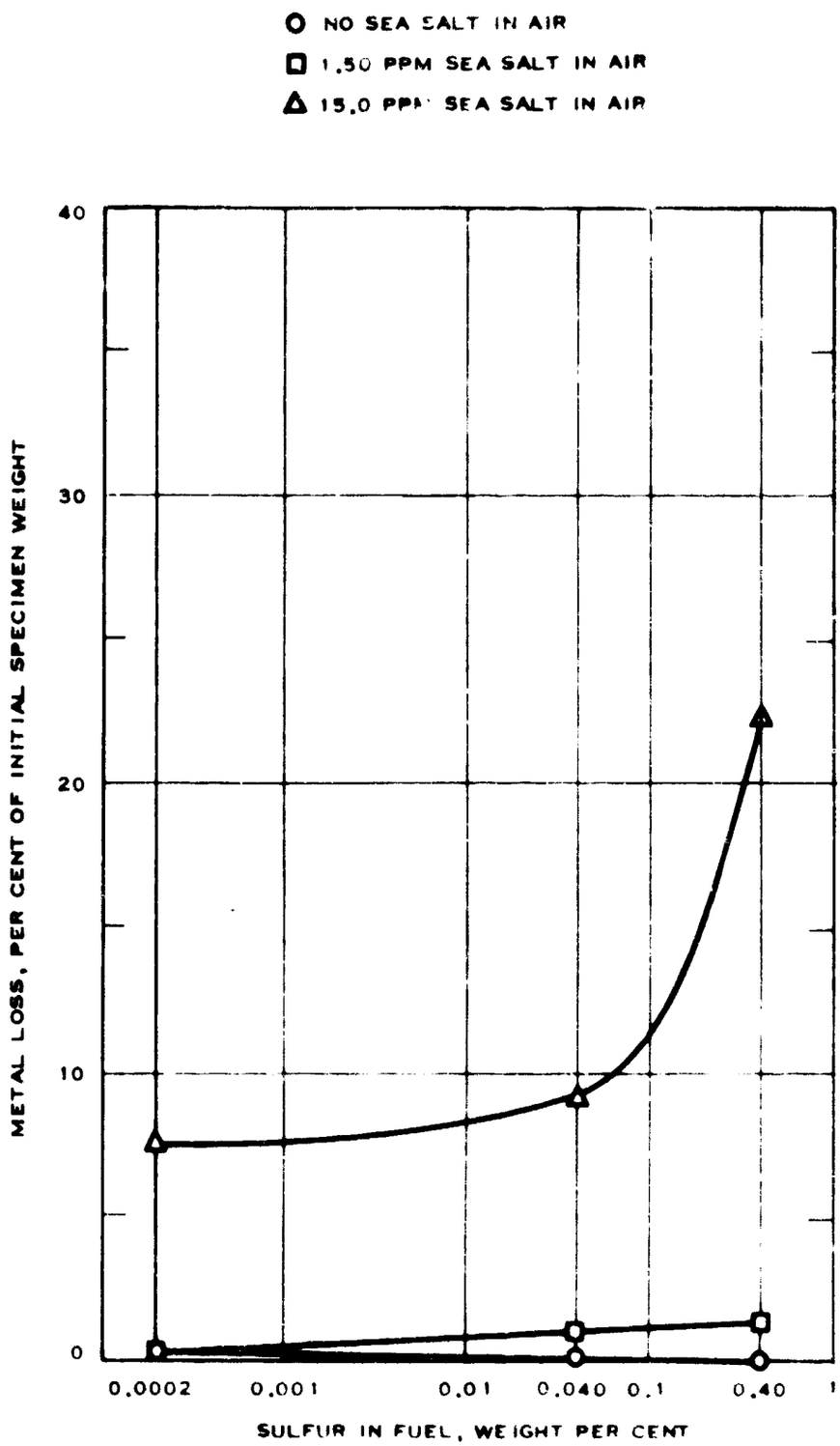

FIGURE 7
EFFECT OF SULFUR AND SEA SALT ON INCONEL 713C TEST SPECIMEN MEAN WEIGHT LOSS AS A RESULT OF EXPOSURE TO HOT GAS CORROSION IN PHILLIPS 2-INCH COMBUSTOR AT 2000 F TEST CONDITION

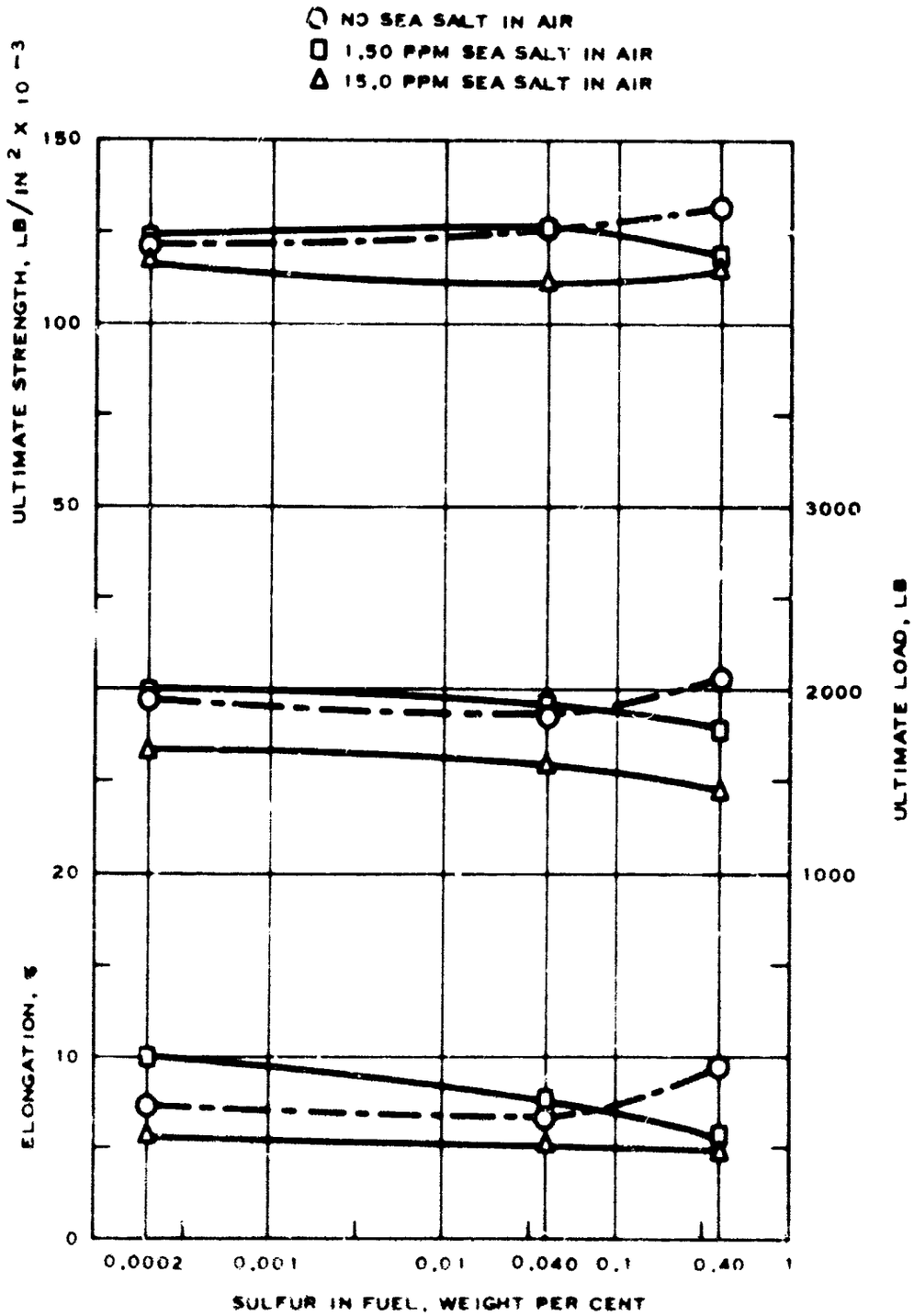

FIGURE 8

EFFECT OF SULFUR AND SEA SALT ON INCONEL 713C TEST SPECIMEN TENSILE PROPERTIES AFTER EXPOSURE TO HOT GAS CORROSION IN PHILLIPS 2 – INCH COMBUSTOR AT 2000 F TEST CONDITION

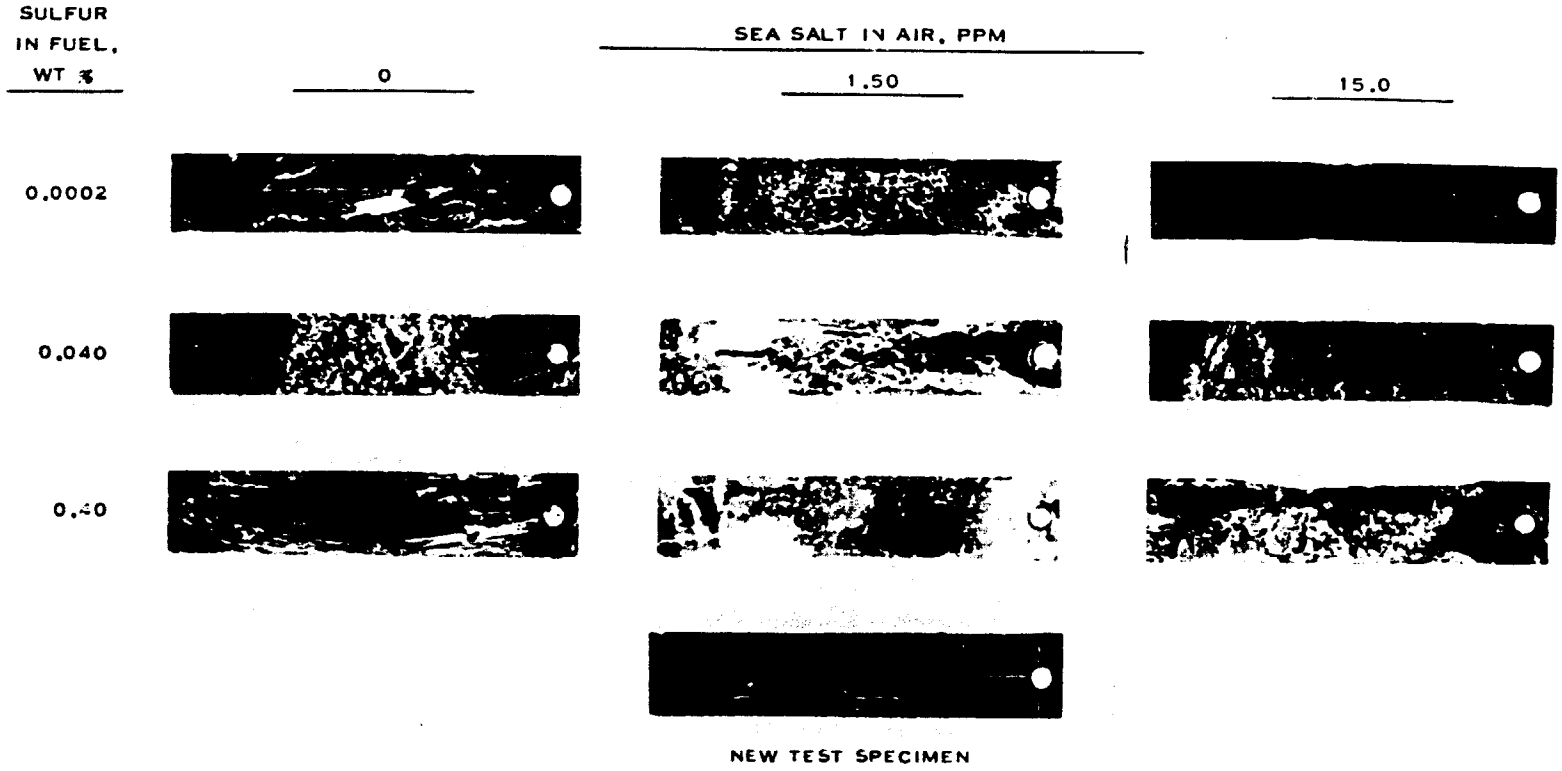

FIGURE 9
PHOTOGRAPHS OF SIERRA METAL 200 TEST SPECIMENS
AFTER EXPOSURE TO HOT GAS CORROSION IN PHILLIPS 2-INCH COMBUSTOR
AT 2000 F TEST CONDITION

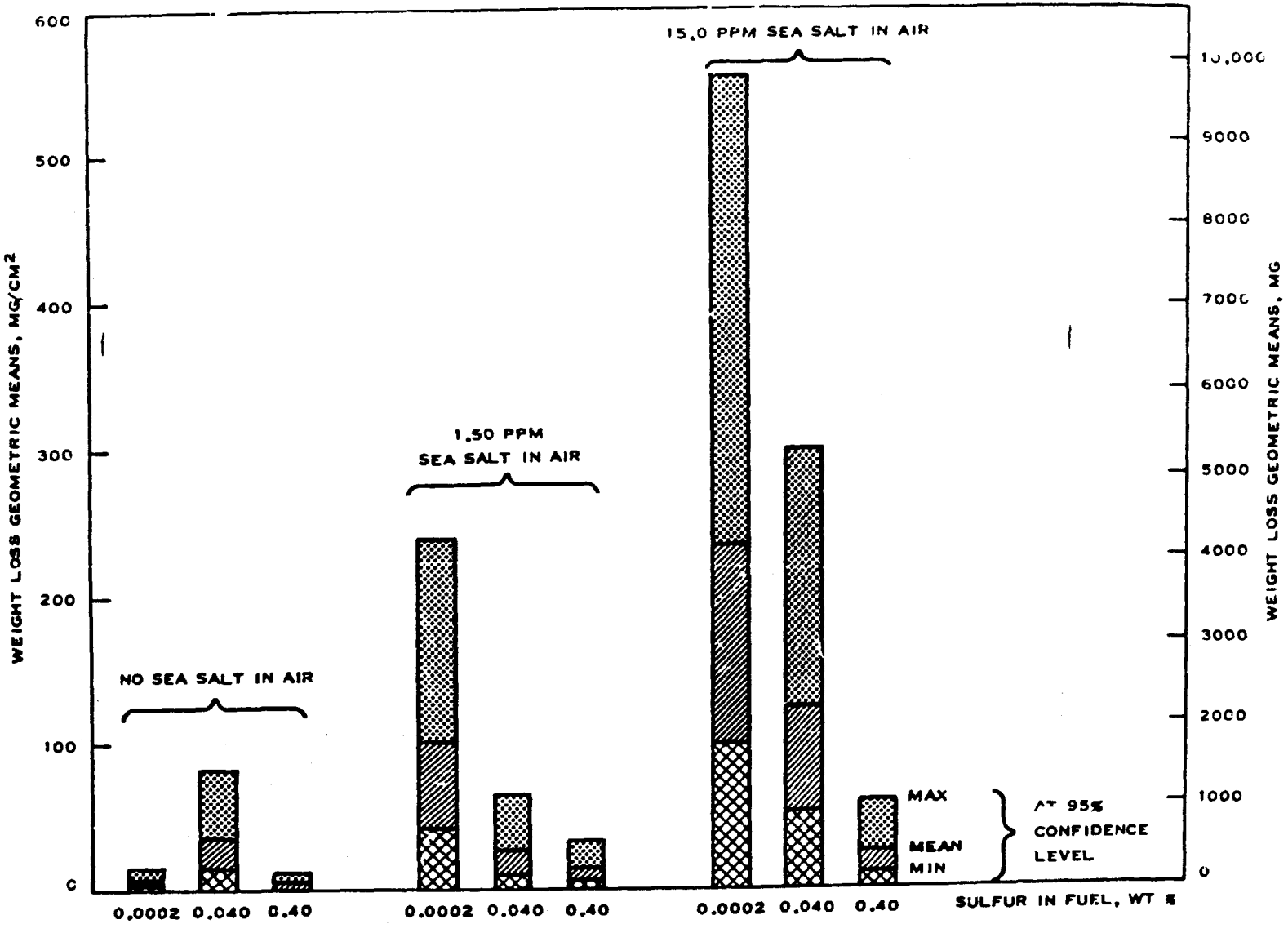

FIGURE 10
EFFECT OF SULFUR AND SEA SALT ON SIERRA METAL 200 TEST SPECIMEN WEIGHT LOSS AS A RESULT OF EXPOSURE TO HOT GAS CORROSION IN PHILLIPS 2-INCH COMBUSTOR AT 2000 F TEST CONDITION

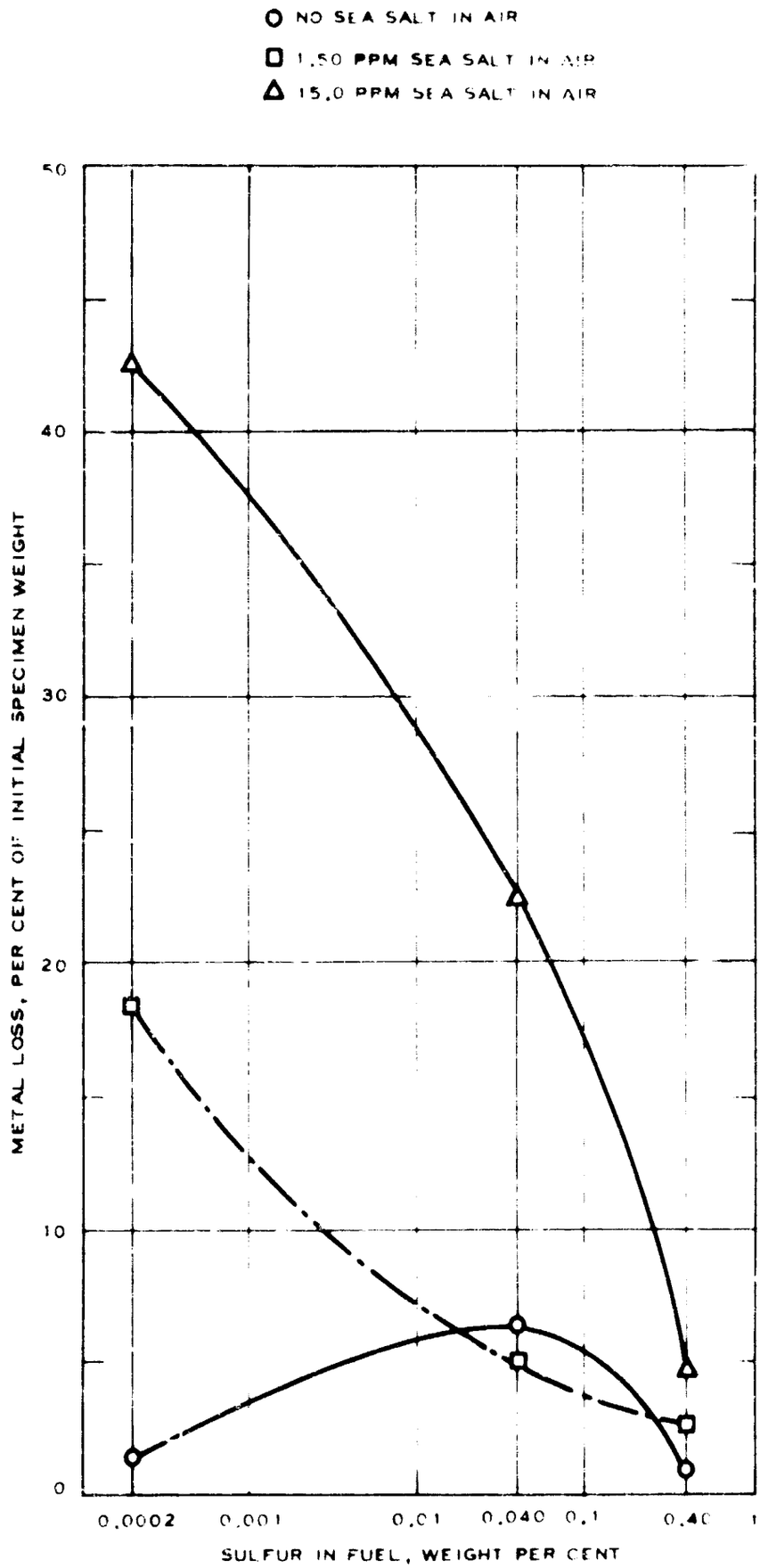

FIGURE 11

EFFECT OF SULFUR AND SEA SALT ON SIERRA METAL 200 TEST SPECIMEN MEAN WEIGHT LO
AS A RESULT OF EXPOSURE TO HOT GAS CORROSION IN PHILLIPS 2 – INCH COMBUSTOR

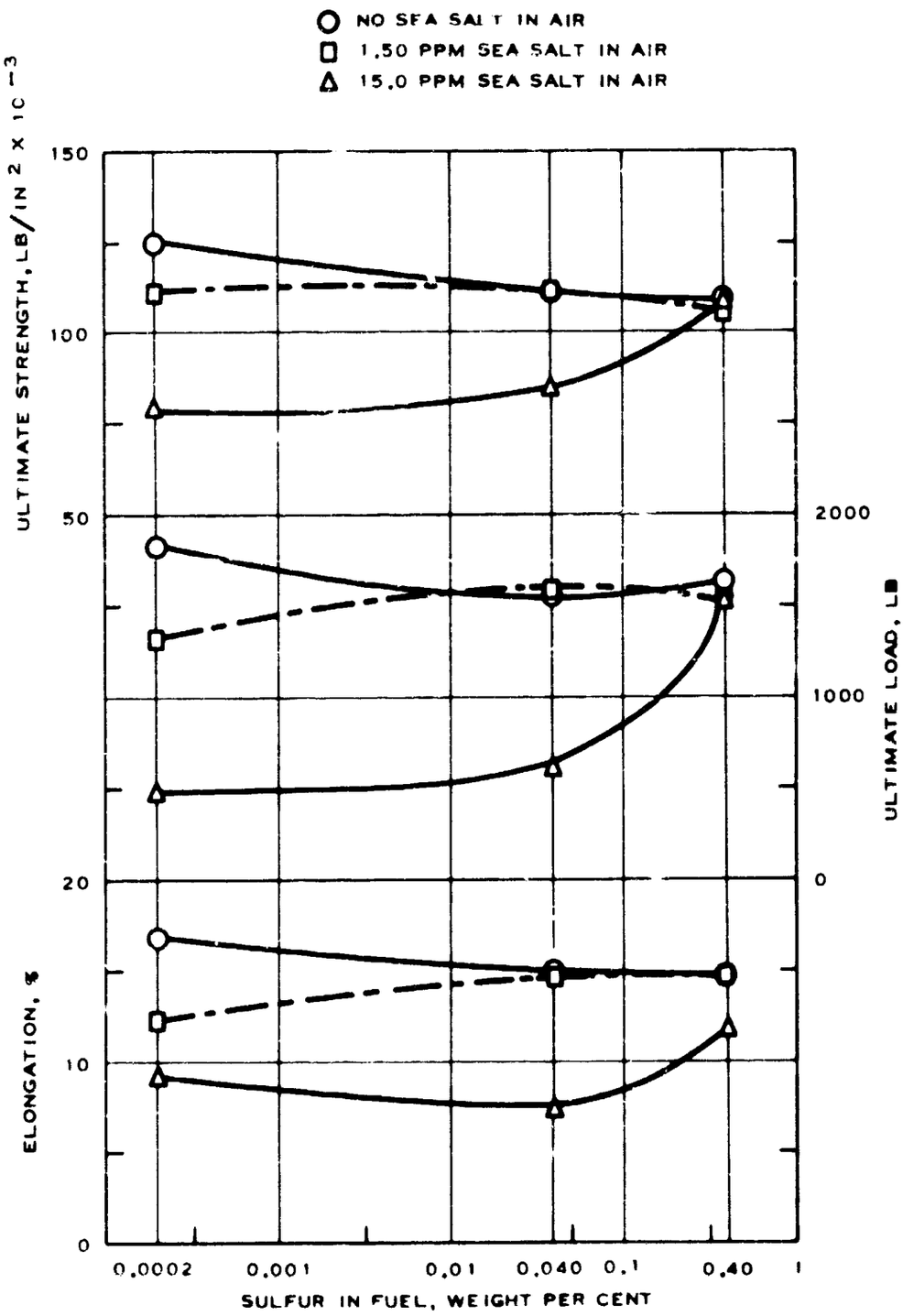

FIGURE 12

EFFECT OF SULFUR AND SEA SALT ON SIERRA METAL 200 TEST SPECIMEN TENSILE PROPERTIES AFTER EXPOSURE TO HOT GAS CORROSION IN PHILLIPS 2-INCH COMBUSTOR AT 2000 F TEST CONDITION

LOSS
R

Metal weight loss data for Sierra Metal 200 are shown in Figures 10 and 11. With only 2 ppm sulfur in the fuel, it can be noted that metal weight loss increases rapidly as sea salt is added to the combustor air. As shown in Figure 9, this metal loss was characterized by metal wastage and thinning of the test specimen, indicating gross attack of the metal on a broad front.

Tensile properties for Sierra Metal 200 are shown in Figure 12. At 2 ppm sulfur in the fuel there is a loss in tensile properties as sea salt is added to the combustor air. This indicates that there was preferential attack at the grain boundaries, introducing localized stress concentrations, resulting in corrosion fatigue.

From the above metal weight loss data it is obvious that high (41 mg/cm^2 or 7.6 per cent for 5 hour test) hot gas corrosion can occur with Inconel 713C, and excessive (235 mg/cm^2 or 42.7 per cent for 5 hour test) hot gas corrosion can occur with Sierra Metal 200 in a saline atmosphere with essentially sulfur-free fuel under 2000 F test conditions.

D. Combined Sulfur-Sea Salt Corrosion

As shown in the Results section there is a significant sulfur x sea salt interaction on metal weight loss of Inconel 713C. Referring to Figure 6 and 7, it is shown that the highest level of hot gas corrosion occurs when sea salt in the combustor air and sulfur in the fuel are both at their maximum concentrations. Metal weight loss is greater than would be predicted from either sulfur or sea salt in the absence of the other.

Figure 8 shows no combined sulfur-sea salt effect on tensile properties of Inconel 713C.

In the Results section it is shown that the sulfur x sea salt interaction on metal weight loss for Sierra Metal 200 is significant. It is shown in Figures 10 and 11 that maximum metal weight loss occurs when sea salt in the combustor air was at a maximum and sulfur in the fuel was at the minimum concentration. The inhibiting effect of sulfur on hot gas corrosion of Sierra Metal 200 would not be predicted from tests with sulfur or sea salt in the absence of the other

In Figure 12 the inhibiting effect of sulfur on tensile properties of Sierra Metal 200 is shown.

From these data it can be observed that sulfur and sea salt can have varied effects on hot gas corrosion with sulfur in the presence of sea salt promoting corrosion of Inconel 713C and inhibiting corrosion of Sierra Metal 200 at the 2000 F operating conditions.

E. Metallography

In order to determine whether hot gas corrosion experienced at the 2000 F test condition was a deep intergranular penetration, photomicrographs of the specimens were made. Photomicrographs of the Inconel 713C specimens

after exposure to the various test conditions, and after cathodic cleaning, are shown in Figures 13 (at 100X) and 14 (at 600X). It may be noted that surface oxidation was general, with no preferential attack on the grain boundaries of the stress-corrosion type. The hot gas corrosion appears to attack the entire surface of the specimen.

Photomicrographs of the Sierra Metal 200 specimens after exposure to hot gas corrosion at the 2000 F test conditions are shown in Figures 15 (at 100X) and 16 (at 600X). It may be noted that with 15.0 ppm sea salt at the various levels of sulfur, hot gas corrosion attack was several grains deep. There is some evidence of sulfur attack, as shown by the gray specks at the metal-scale interface in Figure 16, with the high sea salt ingestion conditions. This material is usually identified as chromium sulfide.

The fact that there is photomicrographic evidence of a deeper attack plus the lower tensile strength values, suggests that the sea salt is affecting the internal structure of the Sierra Metal 200.

F. Mechanism of Corrosion

To obtain information on the mechanism of hot gas corrosion, the statistical program previously described was augmented with a limited number of tests to evaluate the effect of temperature and to study the effect of sodium on hot gas corrosion in an essentially sulfur-free system (2 parts per million sulfur in fuel). In the study of mechanism, X-ray diffraction analysis of the scale from the test specimens was used.

1. Effect of Temperature on Hot Gas Corrosion

Operating conditions used to evaluate the effect of exhaust gas temperature on hot gas corrosion are shown in Table VIII. The supplemental temperatures of 1500 and 1750 F were selected to be below and above the freezing point of sodium sulfate (Na_2SO_4), 1623 F. Selection of these temperatures was based on the premise that it is important whether the corrosive agent is in its solid or liquid phase. The temperatures selected may have been somewhat arbitrary since sodium chloride freezes at 1434 F and the 45/55 percent eutectic mixture of sodium sulfate-sodium chloride freezes at 1153 F.

Using Inconel 713C specimens, 0.40 weight per cent sulfur in the fuel and 15.0 sea salt in the combustor air, the following data were obtained. These data represent single tests where one specimen was cathodically descaled for weight loss determination and the other was used for X-ray diffraction, analysis of scale and photomicrographs.

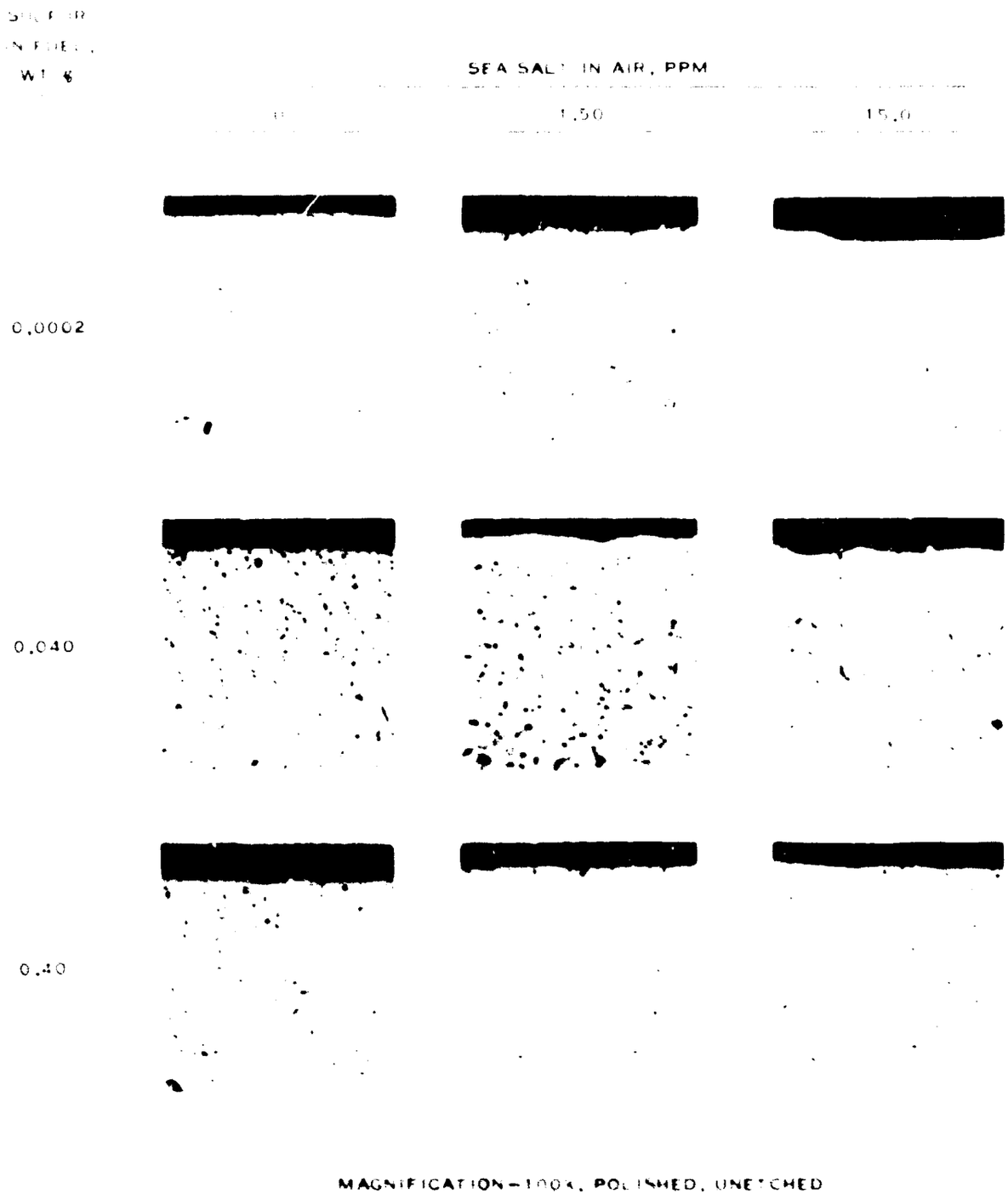

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FIGURE 13
PHOTOMICROGRAPHS AT 100X OF INCONEL 713C TEST SPECIMENS
AFTER EXPOSURE TO HOT GAS CORROSION IN PHILLIPS 2-INCH COMBUSTOR
AT 2000 F TEST CONDITION

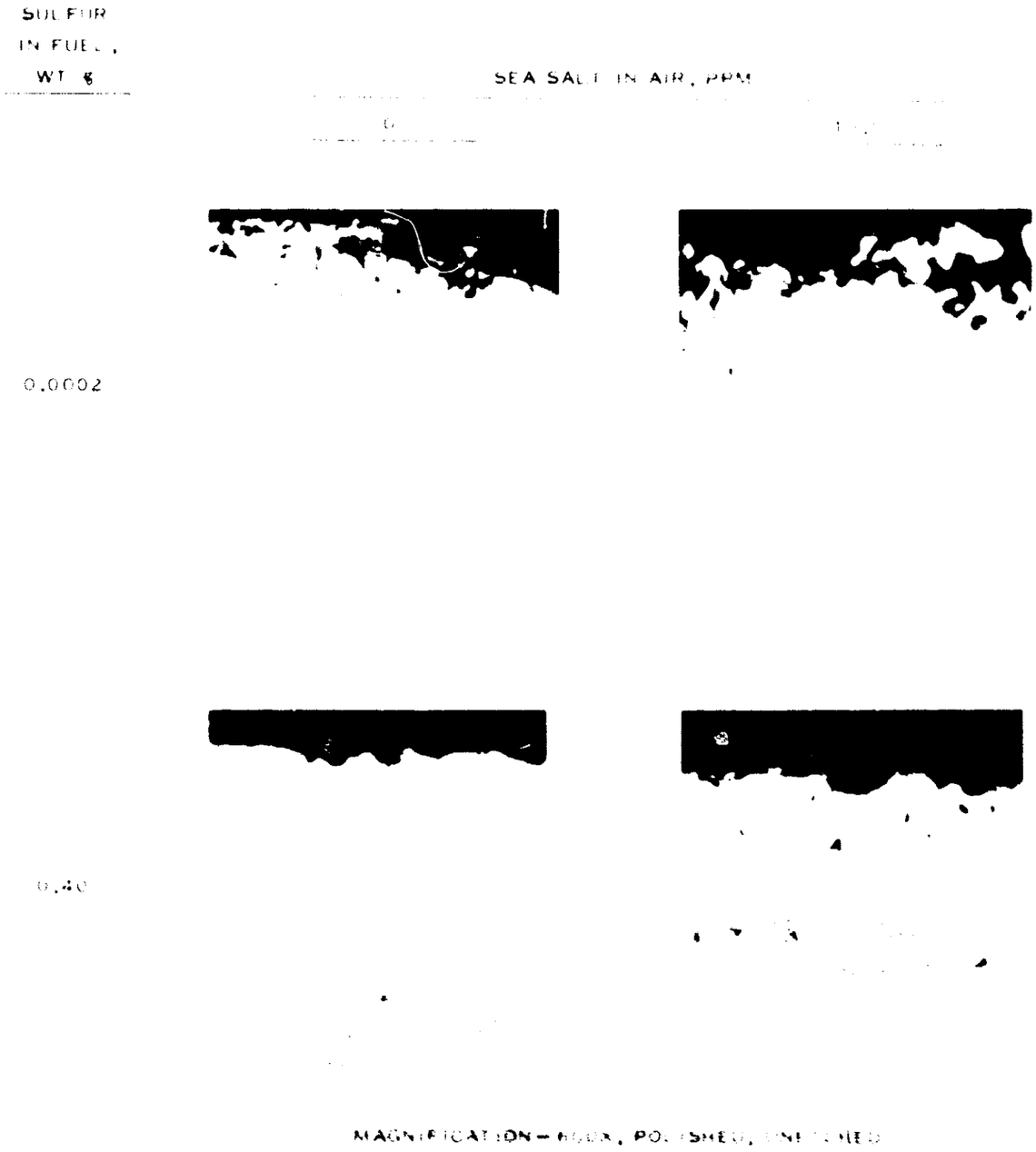

FIGURE 14
PHOTOMICROGRAPHS AT 600X OF INCONEL 713C TEST SPECIMENS
AFTER EXPOSURE TO HOT GAS CORROSION IN PHILLIPS 12 INCH COMBUSTOR
AT 2000°F TEST CONDITION.

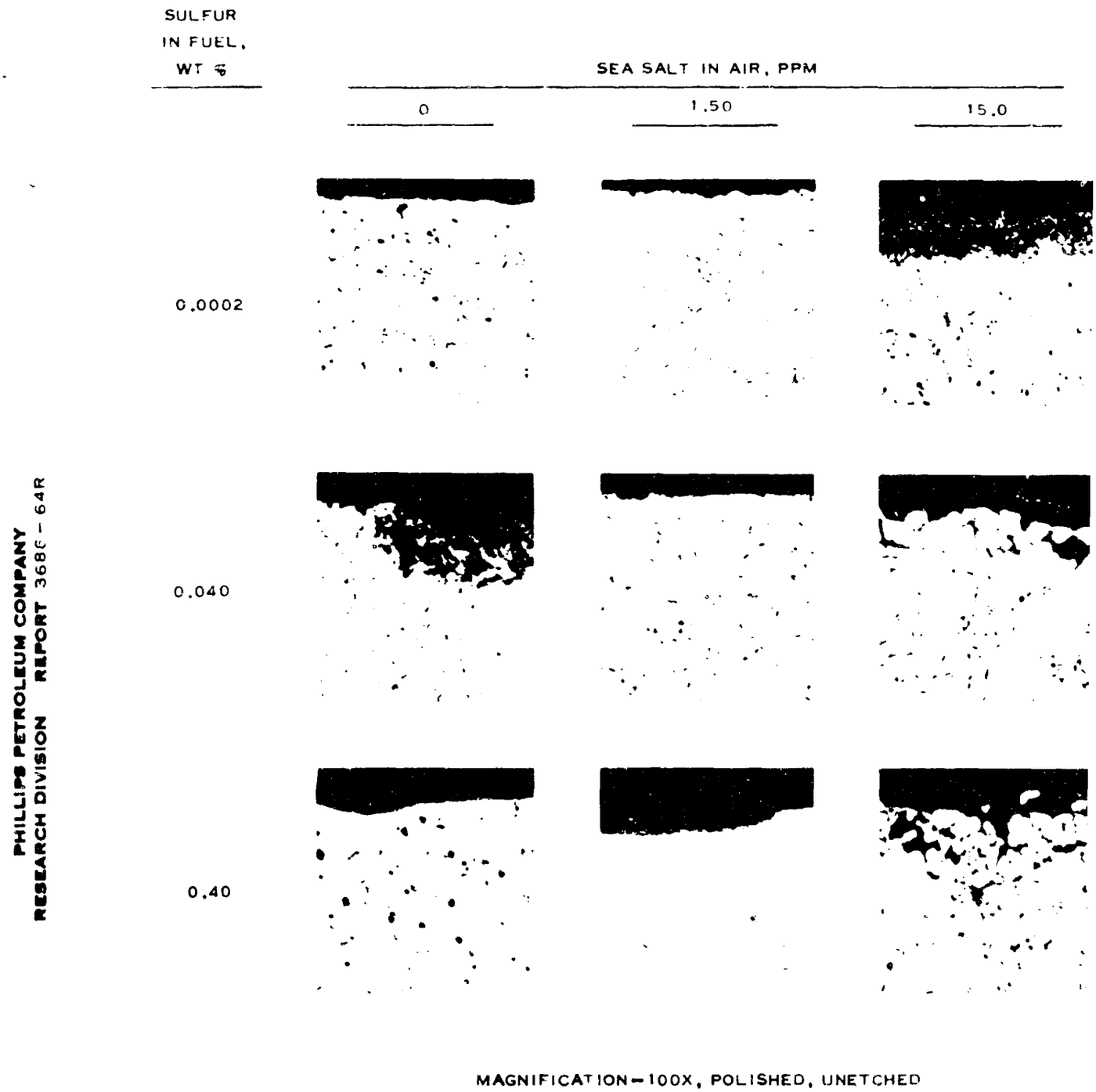

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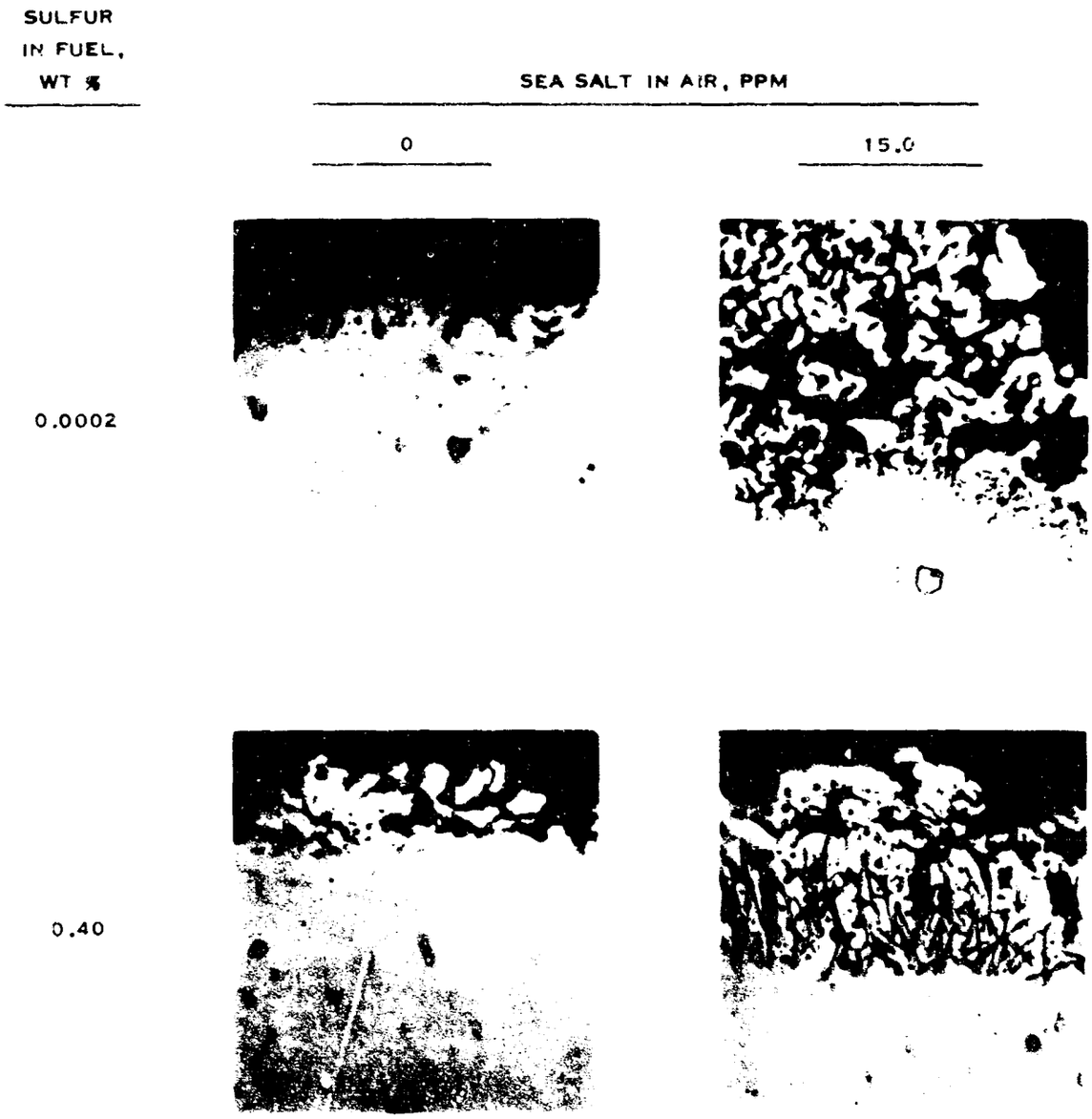
FIGURE 15
PHOTOMICROGRAPHS AT 100X OF SIERRA METAL 200 TEST SPECI
AFTER EXPOSURE TO HOT GAS CORROSION IN PHILLIPS 2-INCH CO
AT 2000 F TEST CONDITION



MAGNIFICATION – 600X, POLISHED, UNETCHED

FIGURE 16
PHOTOMICROGRAPHS AT 600X OF SIERRA METAL 200 TEST SPECIMENS
AFTER EXPOSURE TO HOT GAS CORROSION IN PHILLIPS 2 – INCH COMBUST
AT 2000 F TEST CONDITION

IMENS
MBUST OR

TABLE XLI

EFFECT OF EXHAUST GAS TEMPERATURE ON HOT GAS CORROSION OF INCONEL 713C

Exhaust Gas Temperature, F	X-ray Diffraction Analyses of Scale	Metal Loss, Weight Per Cent
1500	Na_2SO_4 (Thenardite), MgO (Periclase)	10.0
1750	MgO (Periclase), Na_2SO_4 (Thenardite), Ni, Fe (Josephinite)	0.4
2000	NiO (Bunsenite), MgO (Periclase), Na_2SO_4 (Form III)	25.8

As shown in Table XLI, increasing the exhaust gas temperature from 1500 F to 2000 F does not increase metal loss linearly. This suggests that a change in the mechanism of hot gas corrosion is involved. Photographs of the specimens before and after cathodic cleaning are shown in Figure 17. The amount and appearance of the deposits as well as the X-ray diffraction of the scale or corrosion products are different at the low and high exhaust gas temperature. The uncleaned specimen at 1500 F had a thick coating of scale and a 10 per cent weight loss. This temperature is below the freezing point of Na_2SO_4 and MgO which accounts for the heavy build-up of deposits from the sea salt. No material could be identified as a corrosion product. At 1750 F the scale was very thin and the metal loss was very low (0.4 per cent). Since the temperature was above the freezing point of Na_2SO_4 a major portion of molten deposit was washed from the specimen by the high velocity exhaust gases. The minor amount of Josephinite may have resulted from contamination from other metal in the burner system since metal weight loss was low. At 2000 F the coating of scale was thin but the attack on the specimen was heavy as indicated by the weight loss and the photograph of the cathodically cleaned specimen. This high metal weight loss (25.8 per cent) was associated with a change in scale composition with the major constituent being NiO and Na_2SO_4 changing from Thenardite to Form III. The large effect of temperature shown above merits further investigation.

Photomicrographs (at 600 X) of the heavily corroded test specimens at 1500 F and 2000 F are shown in Figure 18. The attack at 1500 F is characteristic of sulfidation, with the formation of randomly distributed internal gray globules of chromium sulfide. However, at 2000 F, where the metal is no longer covered by a heavy layer of sodium sulfate, there is little evidence of sulfidation. Generally, a black oxide lace is found at the surface of the metal; however, in some areas, as shown, the attack is led by a very fine grained structure of sulfides. This 2000 F specimen differs from that shown in Figure 14, in that it was not cathodically cleaned. These findings indicate that the characteristic sulfidation attack at 1500 F gives way to gross oxidation at higher temperatures when the metal is no longer covered by a heavy deposit of sodium sulfate. However, the patches of attack, with pitting, at 2000 F still indicate localized areas of sulfidation.

15.0 PPM SEA SALT IN AIR
0.40 WT % SULFUR IN FUEL

1500 F TEST CONDITION

UNCLEANED

CATHODICALLY CLEANED
925 MG METAL LOSS

1750 F TEST CONDITION

UNCLEANED

CATHODICALLY CLEANED
43 MG METAL LOSS

2000 F TEST CONDITION

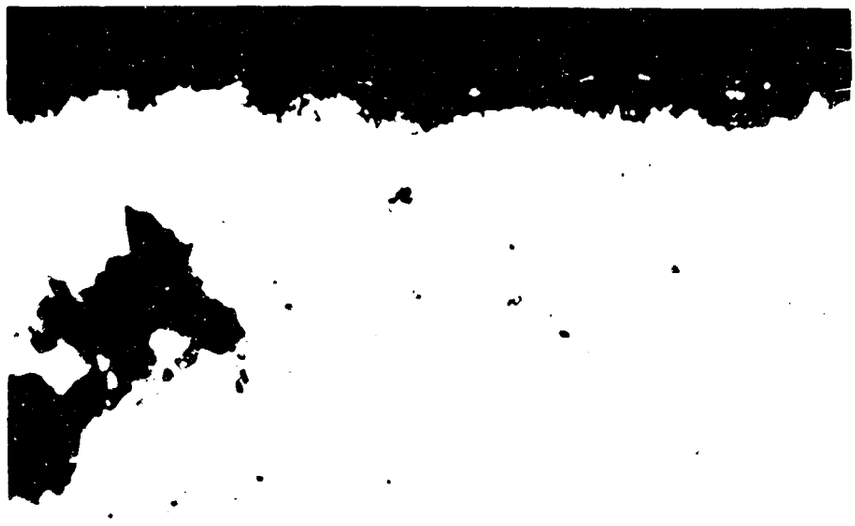
UNCLEANED

CATHODICALLY CLEANED
2766 MG METAL LOSS

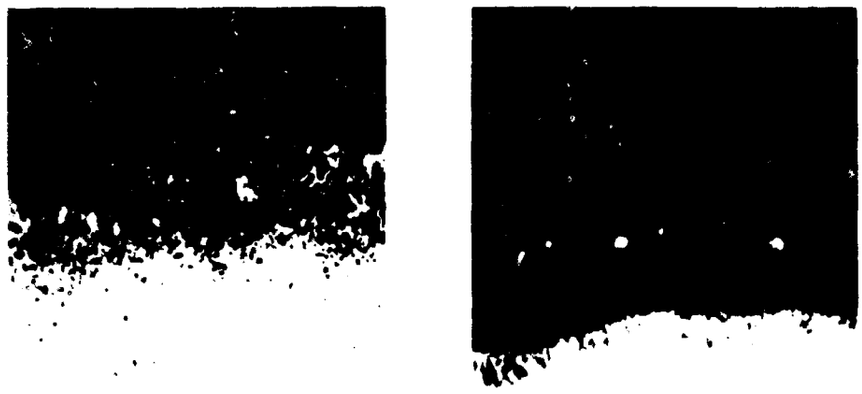
FIGURE 17
PHOTOGRAPHS OF INCONEL 713C TEST SPECIMENS
AFTER EXPOSURE TO HOT GAS CORROSION IN PHILLIPS 2 - INCH COMBUSTOR
AT VARIOUS TEMPERATURES

15.0 PPM SEA SALT IN AIR
0.40 WT % SULFUR IN FUEL

1500 F TEST CONDITION



2000 F TEST CONDITION



MAGNIFICATION – 600 X, POLISHED, UNETCHED

FIGURE 18
PHOTOMICROGRAPHS OF INCONEL 713C TEST SPECIMENS
AFTER EXPOSURE TO HOT GAS CORROSION IN PHILLIPS 2- INCH COMBUSTION
AT VARIOUS TEMPERATURES

2. X-Ray Diffraction Analysis

X-ray diffraction analyses were made of the scale formed on Inconel 713C specimens, under the 2000 F conditions, with (a) 0.040 per cent fuel sulfur, 15.0 ppm sea salt, (b) 0.40 per cent fuel sulfur, 1.50 ppm sea salt and (c) 0.40 per cent fuel sulfur, 15.0 ppm sea salt. X-ray diffraction patterns were identical for each of the three conditions. These deposits were chiefly Bunsenite (NiO). Other weaker diffraction lines were not identified. There was no evidence of sulfide compounds in the corrosion products as indicated by X-ray diffraction. This does not eliminate the possibility of the presence of sulfides since X-ray diffraction techniques are somewhat insensitive to small concentrations of sulfides and sulfides, if present, would be expected only adjacent to the metal where an overlay of deposits provides protection from oxygen in the gas stream. More extensive investigations are needed in this area.

More extensive X-ray diffraction data were obtained on corrosion products from Sierra Metal 200 than from Inconel 713 C. These data are shown in Table XLII.

TABLE XLII

X-RAY DIFFRACTION ANALYSIS OF SIERRA METAL 200 CORROSION PRODUCTS

Sulfur, %	Sea Salt, ppm 0.0	1.50	15.0
0.0002	No XRD Data (2% Wt. Loss)	NiO[a] Na_2WO_4[b] $Fe(AlCr)_2O_4$ [c] (18% Wt. Loss)	NiO Na_2WO_4 $Fe(AlCr)_2O_4$ (42% Wt. Loss)
0.040	No XRD Data (7% Wt. Loss)	NiO $Fe(AlCr)_2O_4$ (5% Wt. Loss)	NiO Na_2WO_4 $Fe(AlCr)_2O_4$ (22% Wt. Loss)
0.40	Ni, Fe[d] γ-Fe_3O_4 (1% Wt. Loss)	NiO $Fe(AlCr)_2O_4$ (2% Wt. Loss)	NiO $Fe(AlCr)_2O_4$ (5% Wt. Loss)

(a) NiO - Bunsenite
(b) Na_2WO_4 - Sodium Tungstate
(c) $Fe(AlCr)_2O_4$ - Aluminian Chromite
(d) Ni, Fe - Josephinite
(e) γ-Fe_3O_4 - Maglemite

Shown with the X-ray diffraction analysis data are metal weight losses which occurred at the sulfur x sea salt test conditions indicated. In all cases where sea salt was present NiO was the major constituent in the scale. Of particular interest is the occurrence of sodium tungstate (Na_2WO_4) whenever the weight loss was 18 per cent or above. It may be postulated that sulfur from the fuel is reacting with sea salt in the combustor air to prevent formation of Na_2WO_4. In the essential absence of sulfur, Na_2WO_4 is formed with 1.50 ppm sea salt in combustor air; however, with 0.040 per cent sulfur in the fuel Na_2WO_4 is not present and metal weight loss is greatly reduced. Likewise with 15.0 ppm sea salt, increasing fuel sulfur from 0.040 to 0.40 per cent eliminated Na_2WO_4 and markedly reduced metal weight loss.

A more complete investigation of corrosion products might show the reason for the obvious differences in sea salt x sulfur attack and elucidate the differences in the mechanism.

3. Sodium Corrosion

In order to study the effect of sodium on hot gas corrosion, a series of tests were conducted at the 2000 F test conditions with an essentially sulfur-free system with regard to fuel. These data are shown in Table XLIII.

TABLE XLIII

EFFECT OF VARIOUS SODIUM CONTAINING COMPOUNDS ON HOT GAS CORROSION

Additive	Additive Concentration	Sulfur In Fuel	Super Alloy	Weight Loss,%	X-Ray Diffraction Analysis of Scale
None[a]	None	2 ppm	713C	0.2	- - -
Sea Salt[a]	15 ppm in air	2 ppm	713C	8.7	- - -
Sodium Chloride[b]	9 ppm in air	2 ppm	713C	14.2	NiO, $Fe(AlCr)_2O_4$
None[a]	None	2 ppm	SM 200	1.2	- - -
Sea Salt[a]	15 ppm in air	2 ppm	SM 200	42.7	NiO,Na_2WO_4,$Fe(AlCr)_2O_4$
Sodium Chloride[b]	9 ppm in air	2 ppm	SM 200	43.7	NiO,Na_2WO_4,$Fe(AlCr)_2O_4$
Sodium Hydroxide[b]	6 ppm in air	2 ppm	SM 200	100[c]	NiO,Na_2WO_4,$Fe(AlCr)_2O_4$

(a) Base line data.
(b) Added at a concentration to obtain a comparable level of sodium as found in sea salt.
(c) About 90 per cent of the specimen was corroded away in four hours at which time the test was terminated.

In these tests sodium was introduced in the form of sea salt, sodium chloride or sodium hydroxide. The concentration of sodium was maintained in all cases equivalent to the sodium in sea salt.

With Inconel 713C it is shown that substitution of sodium chloride for sea salt increases hot gas corrosion. This suggests that materials present in sea salt such as non-aggressive alkaline earths or passive sulfates are retarding the corrosive attack of sodium. Nickel oxide and aluminian chromite [$Fe(AlCr)_2O_4$] were identified by X-ray diffraction of the deposits with sodium chloride, with NiO being the major component.

With Sierra Metal 200 substitution of sodium chloride for sea salt had essentially no effect on metal weight loss; however, sodium hydroxide, NaOH, was much more severe than sea salt or sodium chloride with respect to metal weight loss. It is interesting to note that the same corrosion products were identified by X-ray diffraction for each of the three sodium systems. This suggests that the same mechanism of attack was involved in all three cases. While the mechanism of NaOH attack is unknown, it may be that excessive corrosion was caused by active oxygen from sodium peroxide, Na_2O_2, or it may be that sodium hydroxide forms a fused salt of greater solubility for the protective scale and thus clears the base metal for accelerated attack.

From these data it is quite apparent that catastrophic corrosion may be experienced in an essentially sulfur-free environment.

VII. CONCLUSIONS

The following statements can be made concerning the effects of sulfur in fuel (0.0002, 0.040 and 0.40 weight per cent) and sea salt in air (zero, 1.50 and 15.0 parts per million) on hot gas corrosion of super alloys (Inconel 713C and Sierra Metal 200). They are based on evaluations of metal specimens following exposure to vitiated air from Phillips 2-inch combustor (56 air-fuel ratio) at high temperature (2000 degrees Fahrenheit), high pressure (15 atmospheres), and high velocity (500 feet per second) during a five hour cyclic test (55 minutes fuel-on, 5 minutes fuel-off). The significance of test specimen metal losses and changes in tensile properties were established by analyses of variance, made at a confidence level of 95 per cent.

1. Oxidation and erosion were minor - in the absence of sulfur and sea salt.

2. Sulfur had little, or no, effect on hot gas corrosion - in the absence of sea salt.

3. Sea salt accelerated hot gas corrosion, in some instances to catastrophic levels.

4. Sulfur x sea salt interactions were significant; but, while hot gas corrosion of Inconel 713C was accelerated, hot gas corrosion of Sierra Metal 200 was inhibited.

5. 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.

VIII. RECOMMENDATIONS

This study was made to determine whether the maximum sulfur limit of 0.4 weight per cent, currently allowed in grade JP-5 aviation turbine fuel, is a safe level for the protection of turbine blade alloys used in advanced engines. If not, information was sought to show whether a reduction in the sulfur specification limit would alleviate hot gas corrosion significantly. However, the complex interaction found with ingested sea water does not allow for either recommendation without additional information.

This study does not indicate a need for precipitate action to reduce the maximum sulfur limit of 0.4 weight per cent, currently allowed in grade JP-5 aviation turbine fuel. Both of the nickel-base alloys used in this study showed good resistance to oxidation, erosion, and sulfidation - in the absence of sea salt. However, catastrophic sea salt corrosion was encountered with both super alloys, in some instances. A significant sulfur x sea salt interaction was shown by both super alloys; but, while hot gas corrosion of Inconel 713C was accelerated, hot gas corrosion of Sierra Metal 200 was inhibited. The catastrophic level of corrosion encountered with Inconel 713 C at high sea salt x sulfur concentrations was reduced to a negligible level by a drop in exhaust gas temperature from 2000 to 1750 F, indicating the prime importance of operating temperature. Therefore, it is recommended that this study be extended to obtain more complete data, covering additional super alloys, evaluated over a range in exhaust gas temperature.

IX. FUTURE WORK

A test program to obtain the desired data, concerning the effect of JP-5 sulfur concentration and sea water ingestion on hot gas corrosion of turbine blade and turbine nozzle guide vane materials, would require an estimated 108 five-hour tests. Since this would take about six months of test facility time for completion, it is not feasible under the present contract. Nevertheless, the test program will be detailed to provide an understanding of the authors' intent, and to facilitate future planning.

The test program recommended to obtain additional information differs from that used for the present study in several significant respects, as follows:

1. Exhaust Gas Temperature. Three levels of exhaust gas temperature (1800, 2000, and 2200 F), bracketing the useful limit for nickel-base and cobalt-base alloys, would be investigated to establish whether there are significant changes in the mechanism of corrosion occurring at temperatures above the freezing point of sodium sulfate (1623 F).

2. Sea Salt Concentration. An intermediate concentration of 10 parts per million of sea salt in air, between the 1.50 and 15.0 levels used in this study, has been chosen to reduce the total number of tests.

3. Cascade Testing. The super alloy test specimens would be mounted in pairs, as in the present study; but three test specimen holders would be used, stacked in series and successively rotated 120 degrees to prevent channeling of hot gas flow. This would allow for the exposure of six test specimens during each test, at little additional cost except for the price of the test specimens. Cascade testing subjects downstream test specimens to corrosion products not normally present in the exhaust gas, as would be the case in a multi-stage turbine.

4. Super Alloys. The composition of the five super alloys recommended for future testing are shown in Table XLIV. An attempt has been made to select turbine blade and/or turbine nozzle guide vane materials used in high performance engines of advanced design. INCO 713C and SM-200 have been included because of the background of complex sulfur x sea salt interaction experienced with them during this study. The selection of IN-100 and Udimet 500 has been tempered by a desire to cover a range of from 10 to 20 per cent in chromium content with nickel-base alloys. In addition, it is recommended that a coated super alloy be included to obtain a measure of its increased resistance to corrosion; specifically, Misco Coating No. MDC-1 on INCO 713C. This is an aluminum diffusion type coating for nickel-base alloys, approximately 0.002 inches thick, of sufficient ductility to resist erosion by foreign material.

5. Scale Composition. Studies of the mechanism of corrosion would be enhanced by supplementing present measurements of test specimen weight change, tensile properties, and metallographic evaluation with more comprehensive analysis of all corrosion products; using X-ray diffraction, X-ray fluorescence, and emission spectroscopy.

TABLE XLIV

COMPOSITION OF SUPER ALLOYS RECOMMENDED FOR FUTURE TESTING

	Nominal Composition, per cent				
Alloying Elements	INCO 713C	SM-200	IN-100	Udimet 500	WI-52
Nickel	72	60	60	51	—
Cobalt	—	10	15	19	63
Chromium	13	9	10	19	21
Molybdenum	4.5	—	3	4	—
Tungsten	—	12	—	—	11
Aluminum	6	5	5.5	3	—
Titanium	0.6	2	5	3	—
Iron	1	0.5	0.5	1	2
Columbium	2	1	—	—	2
Vanadium	—	—	1	—	—
Carbon	0.1	0.2	0.2	0.1	0.5

6. Exhaust Gas Composition. Measurements of exhaust gas temperature by thermocouples, and subsequent calculation of combustion efficiency, would be augmented by determination of exhaust gas composition using gas chromatography.

7. Tensile Properties. The flat bar test specimen would be reduced in cross section for tensile evaluation by drilling a 1/8 inch diameter hole in its center. This would allow for retention of its usually more highly corroded edges. The present method of reducing cross section by fillets affords an excellent evaluation of intergranular attack on the base metal. However, the proposed method would provide a more sensitive measure of the over-all damage to the test specimen, reflecting both intergranular attack and loss in cross sectional area.

The experimental lay-out suggested for this test program is shown in Table XLV. It will allow statistical analysis of the data to evaluate the effects, and interactions, of the three test variables - (a) sulfur concentration in fuel, (b) sea salt in air, and (c) exhaust gas temperature. All six of the super alloys would be tested simultaneously, with their locations in the cascade programed so that each super alloy would occupy each of the three stages twice; once in the left position, and once in the right position. This will permit analysis of the effects, and interactions, of the three test variables on each super alloy, as located in each stage of the cascade.

A total of 72 analyses of variance would be made. These would consist of the six super alloys, at each of the three stages, for each of the four response variables - (a) test specimen metal loss, (b) tensile strength, (c) ultimate load, and (d) elongation. Each analysis would be based upon the various means calculated from the data and interactions obtained from an analysis of variance table. An analysis of variance for the recommended test program would have the form shown in Table XLVI.

TABLE XLV

RECOMMENDED TEST PROGRAM

Test Variables (a)			Position of Super Alloy Specimens in Cascade											
Exhaust Temp., F	Sulfur in Fuel, Wt. %	Sea Salt in Air, ppm	Stage 1				Stage 2				Stage 3			
			L	R	L	R	L	R	L	R	L	R	L	R
1800	0.0002	0	X_2	Y_2	Y_2	X_2	X_2	Y_2	Y_2	X_2	X_2	Y_2	Y_2	X_2
1800	0.0002	10.0	X_2	Y_2	Y_2	X_2	X_2	Y_2	Y_2	X_2	X_2	Y_2	Y_2	X_2
1800	0.040	0	X_2	Y_2	Y_2	X_2	X_2	Y_2	Y_2	X_2	X_2	Y_2	Y_2	X_2
1800	0.040	10.0	X_2	Y_2	Y_2	X_2	X_2	Y_2	Y_2	X_2	X_2	Y_2	Y_2	X_2
1800	0.40	0	X_2	Y_2	Y_2	X_2	X_2	Y_2	Y_2	X_2	X_2	Y_2	Y_2	X_2
1800	0.40	10.0	X_2	Y_2	Y_2	X_2	X_2	Y_2	Y_2	X_2	X_2	Y_2	Y_2	X_2
2000	0.0002	0	X_3	Y_1	Y_1	X_3	X_3	Y_1	Y_1	X_3	X_3	Y_1	Y_1	X_3
2000	0.0002	10.0	X_3	Y_1	Y_1	X_3	X_3	Y_1	Y_1	X_3	X_3	Y_1	Y_1	X_3
2000	0.040	0	X_3	Y_1	Y_1	X_3	X_3	Y_1	Y_1	X_3	X_3	Y_1	Y_1	X_3
2000	0.040	10.0	X_3	Y_1	Y_1	X_3	X_3	Y_1	Y_1	X_3	X_3	Y_1	Y_1	X_3
2000	0.40	0	X_3	Y_1	Y_1	X_3	X_3	Y_1	Y_1	X_3	X_3	Y_1	Y_1	X_3
2000	0.40	10.0	X_3	Y_1	Y_1	X_3	X_3	Y_1	Y_1	X_3	X_3	Y_1	Y_1	X_3
2200	0.0002	0	X_1	Y_3	Y_3	X_1	X_1	Y_3	Y_3	X_1	X_1	Y_3	Y_3	X_1
2200	0.0002	10.0	X_1	Y_3	Y_3	X_1	X_1	Y_3	Y_3	X_1	X_1	Y_3	Y_3	X_1
2200	0.040	0	X_1	Y_3	Y_3	X_1	X_1	Y_3	Y_3	X_1	X_1	Y_3	Y_3	X_1
2200	0.040	10.0	X_1	Y_3	Y_3	X_1	X_1	Y_3	Y_3	X_1	X_1	Y_3	Y_3	X_1
2200	0.40	0	X_1	Y_3	Y_3	X_1	X_1	Y_3	Y_3	X_1	X_1	Y_3	Y_3	X_1
2200	0.40	10.0	X_1	Y_3	Y_3	X_1	X_1	Y_3	Y_3	X_1	X_1	Y_3	Y_3	X_1
		Runs 1-36	M_1		M_2		M_3		M_4		M_5		M_6	
		Runs 37-72	M_3		M_4		M_5		M_6		M_1		M_2	
		Runs 73-108	M_5		M_6		M_1		M_2		M_3		M_4	

Notation:

M_1 = Udimet 500
M_2 = WI-52
M_3 = SM-200
M_4 = IN-100
M_5 = INCO 713 C
M_6 = INCO 713 C with Misco Aluminum Diffusion Coating No. MDC-1
L = Left hand position in test specimen holder.
R = Right hand position in test specimen holder.
X = First series of tests.
Y = Second series of tests.

Procedure:

A. Choose to run X's or Y's first at random.
B. Randomize the order of X_1, X_2, X_3 and Y_1, Y_2, Y_3 for each group of 36 runs.
C. Steps (A) and (B) will provide an ordering of runs in groups of six. Randomize the order of runs within each of these groups of six.

TABLE XLV (Continued)

Notes:

(a) Other Phillips 2-inch combustor operating variables to be held constant throughout the test program.

Combustor Inlet Air Pressure, atmospheres	15.0
Air-Fuel Ratio, pounds per pound	60
Exhaust Gas Velocity at Test Specimen, feet per second . .	500
Test Duration, hours	5.00

Inlet air temperature to be varied over a range of from 800 to 1200 F, as required to obtain desired exhaust gas temperature.

Thermal shock to be introduced by an operating cycle of 55 minutes at test conditions, followed by 5 minutes with fuel off.

TABLE XLVI

ANALYSIS OF VARIANCE FOR RECOMMENDED TEST PROGRAM

Source of Variation	Degrees of Freedom
Total	35
Units of Six Runs.	5
Position (Left vs. Right)	1
Temperature	2
Main Plot Error	2
Single Runs in Units of Six.	30
Sea Salt.	1
Sulfur.	2
Sea Salt x Sulfur	2
Sea Salt x Temperature.	2
Sulfur x Temperature.	4
Sea Salt x Sulfur x Temperature . .	4
Split Plot Error.	15

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XI. REFERENCES

1. "The Corrosion of Nickel-Base Material in Gas-Turbine and Boiler Atmospheres," by E. J. Bradbury, P. Hancock and H. Lewis. Metallurgia, January 1963, pp. 3-14.

2. "Gas Turbine Engine Corrosion Control," by M. F. Grandey. Paper 813A presented at SAE Automotive Engineering Congress, Detroit, January 1964.

3. "Design and Fabrication Aspects of Transpiration Air Cooled Turbine Blades for 2500 F Turbine Operation," by S. Lombardo, N. F. Lauziere and D. Kump. Paper 820A presented at SAE Automotive Engineering Congress, Detroit, January 1964.

4. "Fuel Requirements for Supersonic Transport," by E. A. Droegemueller. Section VI - Paper 20, Sixth World Petroleum Congress, Francfort/Main, June 1963.

5. "Comparative Evaluation of Protective Coatings for High Temperature Alloys," by E. S. Nichols, J. A. Burger and D. K. Hanink. Paper 843E presented at SAE Air Transport and Space Meeting, New York, April 1964.

6. "The Corrosion of Nickel-Chromium Alloys in Sulfur-Containing Atmospheres at High Temperatures," by P. Hancock. Proceedings First International Congress on Metallic Corrosion, London, 1961, pp. 290-298.

7. "Sodium Sulfate in Gas Turbines", by E. L. Simons, G. V. Browning and H. A. Liebhafsky. Corrosion, Vol. 11, December 1955, pp. 17-26.

8. "Effects of Sulphate-Chloride Mixtures in Fuel-Ash Corrosion of Steels and High-Nickel Alloys," by H. T. Shirley. Journal of the Iron and Steel Institute, Vol. 82, February 1956, pp. 144-153.

9. Corrosion of High-Temperature Nickel-Base Alloys by Sulphate-Chloride Mixtures," by H. Lewis and R. A. Smith. Proceedings First International Congress on Metallic Corrosion, London, 1961, pp. 326-336.

10. "Sulfur Attack on High Temperature Turbine Materials," prepared by Bristol Siddeley Engines Ltd. Paper presented before the Thirteenth Technical Conference, International Air Transport Association, Lucerne, May 1960.

11. "Gas Turbine and Jet Engine Fuels," by G. D. Kittredge, S. H. Gwinn and W. L. Streets. Summary Report for U. S. Navy Contract NOas 58-310-d. Phillips Petroleum Company Research Division Report 2506-59R.

12. "Gas Turbine and Jet Engine Fuels," by G. D. Kittredge and W. L. Streets. Summary Report for U. S. Navy Contract NOas 60-6009-c. Phillips Petroleum Company Research Division Report 2760-60R.

13. "Gas Turbine and Jet Engine Fuels," by W. L. Streets. Summary Report for U. S. Navy Contract NO(w) 61-0590-d. Phillips Petroleum Company Research Division Report 3185-62R.

14. "Gas Turbine and Jet Engine Fuels," by W. L. Streets and R. M. Schirmer. Summary Report for U. S. Navy Contract N600(19)-58219. Phillips Petroleum Company Research Division Report 3529-63R.

15. "Effects of Fuel Sulfur Contamination and Sea Water Ingestion on the Durability of Jet Engine Hot Section Components," by W. L. Streets. Journal of the Institute of Petroleum, Vol. 48, No. 466, pp. 314-324, October 1962.

16. "Gas Turbine and Jet Engine Fuels," by W. L. Streets. Progress Report No. 1 for U. S. Navy Contract NOw63-0406-d. Phillips Petroleum Company Research Division Report 3559-63R.

17. "Gas Turbine and Jet Engine Fuels," by W. L. Streets. Progress Report No. 2 for U. S. Navy Contract NOw63-0406-d. Phillips Petroleum Company Research Division Report 3615-63R.

18. "Design and Calibration of the Improved Phillips Jet Fuel Testing Facility," by E. H. Fromm. Phillips Petroleum Company Research Division Report 3527-63R.

19. "Chemical Technology of Petroleum," by W. A. Gruse and D. R. Stevens. Third Edition. Published by McGraw-Hill Book Company, Inc., New York, 1960.

20. "Aviation Fuel," by O. C. Blade. Petroleum Products Survey Nos. 4, 9, 14, 19, 24, 29 and 34, Bartlesville, Oklahoma, Petroleum Research Center, Bureau of Mines, U. S. Department of the Interior, 1958, 1959, 1960, 1961, 1962, 1963 and 1964.

21. "The Oceans as a Chemical System," by E. D. Goldberg, p. 4. "The Sea," by M. N. Hill, Volume 2, "The Composition of Sea-Water," Interscience Publishers, John Wiley & Sons, Inc., 1963, New York.

22. "Sampling Atmospheric Sea-Salt Nuclei Over the Ocean," by A. H. Woodstock and M. M. Gifford. Journal of Marine Research, Vol. 8, 1949, p. 194.

23. "Ultrafine Particles in the Atmosphere and Space," by R. D. Cadle, pp. 20-40. "Ultrafine Particles" by W. E. Kuhn, H. Lamprey and C. Sheer. John Wiley & Sons, Inc., 1963, New York.

24. "Gas Turbine for Unconventional Craft", by G. L. Graves and R. S. Carleton. Paper 685A presented at SAE-ASNE National Aero-Nautical Meeting, Washington, April 1963.

25. "Statistical Methods," by G. W. Snedecor. Fifth Edition. The Iowa State University Press, Ames, Iowa, 1956.

26. "Principles and Procedure of Statistics," by R. G. D. Steel and J. H. Torril. Chapter 5. McGraw-Hill Book Company, Inc. New York, 1960.