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

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OCTOBER 2, 1968 TO JANUARY 1, 1969

BY
H. T. QUIGG AND R. M. SCHIRMER

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IDENTIFIERS: NICKEL ALLOY INCONEL 713, NICKEL ALLOY UDIMET 500, ALUMINUM ALLOY MISCO MDC-1, EXPOSURE TESTS, SUPERALLOYS (U)

AN EXPERIMENTAL INVESTIGATION IS BEING CONDUCTED TO EVALUATE THE EFFECT OF VERY-LOW CONCENTRATIONS OF SULFUR IN FUEL ON THE DURABILITY OF TURBINE-BLADE ALLOYS WHEN EXPOSED UNDER CONDITIONS SIMULATING THE ENVIRONMENT IN AN AIRCRAFT-TURBINE ENGINE OPERATING IN A MARINE ENVIRONMENT. THIS HAS REQUIRED DEVELOPMENT OF A NEW TURBINE SIMULATOR, TO INCREASE PRODUCTIVITY BY SIMULTANEOUS EXPOSURE OF SPECIMENS AND TO IMPROVE PRECISION BY ROTATING SPECIMENS FOR UNIFORM EXPOSURE IN THE HIGH-VELOCITY STREAM OF HIGH-TEMPERATURE, HIGH-PRESSURE, CORROSIVE GAS. (AUTHOR) (U)

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

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S U M M A R Y

An experimental investigation is being conducted to evaluate the effect of very-low concentrations of sulfur in fuel on the durability of turbine-blade materials when exposed under conditions simulating the environment in an aircraft-turbine engine operating in a marine environment. This has required development of new test equipment, termed Phillips Turbine Simulator, to increase productivity by simultaneous exposure of 48 specimens and to improve precision by rotating specimens for uniform exposure in the high-velocity stream of high-temperature, high-pressure, corrosive gas.

A preliminary test was conducted to evaluate a cyclic-temperature operating procedure with Phillips Turbine Simulator. Specimens of both bare and aluminum-diffusion coated Inconel 713C were exposed at 15 atmospheres combustor pressure using air with 1 ppm sea salt and fuel with 0.040 weight per cent sulfur to obtain exhaust-gas temperatures which reached a maximum of 2000 F. It was concluded that this test equipment is sufficiently durable, and the test method provides sufficient precision, for conducting our proposed investigation with a reasonable expenditure of time, manpower, and materials.

A comparison of the relative durability of superalloys in an ASTM "round-robin" program was made during this preliminary test. Only single specimens of each superalloy were available from a common exposure, for 44 hours duration. Metallographic measurements of "surface-loss" and "maximum-attack" were made, but the difference between them (subsurface deterioration) was relatively unimportant with these materials at this condition of exposure. This is shown by the following tabulation, which also indicates the linear relationship that was found with specimen weight-loss (after electro-cleaning).

Superalloy	Weight Loss	Surface Loss	Maximum Attack
IN-100	372 mg/cm^2	56 mils	56 mils
Inco 713C	200	31	32
Udimet 700	164	20	21
Mar-M421	126	20	22
IN-738	97	15	16
Udimet 500	56	2	8

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PHILLIPS PETROLEUM COMPANY

BARTLESVILLE, OKLAHOMA

Progress Report No. 3
For
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EFFECT OF SULFUR IN JP-5 FUEL ON HOT CORROSION OF COATED SUPERALLOYS IN MARINE ENVIRONMENT

1. INTRODUCTION

The corrosion of hot-section parts in modern aircraft-turbine engines is one of the factors that determines the time before overhaul. With operation in a marine environment it becomes a major factor in limiting engine life. Various terms have been used to identify this accelerated attack on the superalloys from which hot-section parts are fabricated. We favor the term "hot corrosion" and will use it in this report to indicate the attack by sea salt on superalloys at high temperature.

Considerable metal loss can be sustained by hot-section parts before failure because hot corrosion advances on a broad front. The attack is led by penetration of randomly dispersed light-grey globules of metallic sulfide. The formation of these sulfides is associated with changes, characterized by chromium depletion, in the surface composition of the alloy. Rapid oxidation of the weakened layer of the alloy follows. Because of the prominent band of precipitated sulfides preceding surface oxidation, hot corrosion is frequently identified as "sulfidation." This has focused attention on the sulfur content of the fuel as being the principal causative agent of hot corrosion. If so, hot corrosion could be controlled by fuel specification. More restrictive limitations on the amount of sulfur allowed in aviation-turbine fuels have been proposed for this purpose, particularly for naval operations.

Most specifications for aviation-turbine fuel allow a sulfur content of 0.40 weight per cent. A significant reduction in the sulfur limit would certainly decrease the amount of available fuel. Also, a more restrictive specification carries with it the potential of higher cost, and a modest increase can amount to a substantial sum because of the large volume involved. Therefore, proposals to lower the sulfur content of aviation-turbine fuel must be approached with caution.

Many investigators feel that deposition of sodium sulfate on the metal surface is a normal precursor to hot corrosion. Some have reasoned that sodium from sea salt and sulfur from fuel combine to form the objectionable sodium sulfate. However, compounds other than sodium chloride are present in sea salt. Sea salt contains 11 per cent by weight of sodium sulfate. Control of hot corrosion by reducing sulfur in fuel to remove one of the critical ingredients from the salt-sulfur combination ignores the sulfur in sea salt.

For clarification, the Naval Air Systems Command has supported our work to determine whether the maximum sulfur content of 0.40 weight per cent, currently allowed in grade JP-5 aviation-turbine fuel, is a safe level for protection of turbine alloys in high-performance engines when operated in a marine environment.

In an early experimental investigation, reported in detail by Schirmer and Quigg (1), we attempted to simulate the environment in the turbine section of an aircraft engine with respect to temperature, velocity, pressure, and stoichiometry by use of a high-pressure test facility, originally developed for evaluating the combustion characteristics of aviation-turbine fuels. The test program included the effect of three levels of sulfur in fuel on hot corrosion of six superalloys, one of which had an aluminum-diffusion coating, at five levels of temperature and three levels of sea salt in air. The five hours of test duration used was sufficient for extensive corrosion of all of the uncoated superalloys under some conditions. It was concluded that an order-of-magnitude reduction (0.40 to 0.040 weight per cent) in sulfur content would not decrease hot corrosion and no change in fuel specification was indicated.

The aluminum-rich coating (Misco MDC-1) on Inconel 713C resulted in a material immune to attack under the conditions of exposure used during this early investigation; therefore, no measure of the effect of sulfur in fuel on the hot corrosion of coated superalloys was obtained. Such thin, aluminum-rich, diffusion coatings are generally used now to protect superalloys in a marine environment; however, the need continues for improved coatings applied to more resistant superalloys.

In a subsequent experimental investigation (2) we attempted to measure the effect of sulfur in fuel on the hot corrosion of coated superalloys by extending the duration of exposure from 5 up to 55 hours. The programs included one bare superalloy (Inconel 713C) and three coating-alloy systems (Misco MDC-1 and MDC-9 on Inconel 713C and Misco MDC-1 on Mar M-200). In these programs two levels of sea salt in air (zero and 1.0 ppm) and three levels of sulfur in fuel (<0.0040, 0.040, and 0.40 weight per cent) were used. These programs were confined to exposure of specimens at the 2000F test condition. These studies showed cases where a reduction of sulfur in fuel from the present limit to 0.040 weight per cent significantly decreased hot corrosion, significantly increased hot corrosion and had no significant effect on hot corrosion. This indicated that our previous recommendation, based upon a study of bare superalloys, could be extended to coated superalloys.

Because of the diverse results on hot corrosion from this very-limited investigation, it was recommended that the effect of sulfur in fuel should be studied with other coating-superalloy systems and other exposure temperatures. Also, when sea salt was present in the air during this investigation, it was found that a very-large reduction of sulfur in fuel, from 0.40 to ≤ 0.0040 weight per cent, significantly decreased the relative rate of corrosion on uncoated Inconel 713C and both MDC-1 and MDC-9 coated Inconel 713C. Therefore, it was concluded that it would be

prudent to obtain additional data before making a final recommendation as to the limit of fuel sulfur allowed for protection of turbine-blade materials in a marine environment.

In a recent experimental investigation (3) conducted with one coating-superalloy system (Misco MDC-9 coated Inconel 713C) at one level of sulfur in fuel (0.40 weight per cent) and one sea salt concentration (1.0 ppm sea salt in air) over a range of temperature conditions (1400, 1600, 1800, and 2000 F) it was found that exposure times of up to several hundred hours would be required to evaluate the effect of sulfur in fuel on hot corrosion with coated superalloys at some conditions. It was concluded that the time required for a reasonably complete investigation of the effect of fuel sulfur on hot corrosion would be prohibitive with test equipment in which only six specimens could be exposed at one time. To reduce the time required for the proposed investigation to a practical level, a new test section, termed the Turbine Simulator, was designed which allowed the simultaneous exposure of 48 specimens.

In our most recent experimental investigation (4) this Turbine Simulator was used to expose both bare and MDC-1 coated Inconel 713C specimens in an aggressive-thermal environment. Gas temperature, pressure, velocity, and composition were adjusted to simulate the environment in an aircraft-turbine engine operating in a marine environment. The mechanical durability of our Turbine Simulator and the precision of weight-loss values for specimens exposed in this test equipment were demonstrated to be adequate for the proposed investigation. However, an exposure time of about 350 hours was indicated to obtain coating failure, with 1 ppm sea salt in air and 0.040 weight per cent sulfur in fuel, at a specimen temperature of 1650 F. It was recommended that the Turbine Simulator be operated with rapid temperature cycling, which is characteristic of aircraft-turbine-engine operation, to accelerate coating breakdown (by spalling). Also, to further reduce the time required for the proposed investigation, it was recommended that rather than separate tests at 1600, 1800 and 2000 F, specimen exposure at these temperatures be combined by spacing with exposure at 1000 F during the cyclic test.

The purpose of our current investigation is to evaluate the performance of the Turbine Simulator, using a cyclic-temperature procedure, with respect to its mechanical durability, its severity of operating conditions, and its uniformity of specimen exposure. If satisfactory, the Turbine Simulator will be used with a cyclic temperature procedure to evaluate the durability of turbine-blade materials in a marine environment during our investigation which will place emphasis on the effect of very low concentrations of sulfur in fuel.

2. CONCLUSIONS

An experimental investigation has been conducted to evaluate the performance of the Phillips Turbine Simulator, using a cyclic temperature procedure, with respect to (a) mechanical durability, (b) severity of operating conditions, and (c) uniformity of specimen exposure. An adequate supply of bare and Misco MDC-1 coated Inconel 713C specimens were available from our most recent study and a limited number of specimens of six superalloys (IN-100, Inconel 713C, U-700, Mar-M421, IN-738, and U-500) were available as a part of a "round-robin" testing program of the ASTM Hot-Corrosion Task-Force. Specimens were exposed at 15 atmospheres combustor pressure using air with 1 ppm sea salt and fuel with 0.040 weight per cent sulfur to obtain exhaust-gas temperatures that were rapidly cycled from 1000 F to a maximum of 2000 F. Testing was halted at 11-hour intervals to allow visual inspection of specimens. Specimens of each superalloy and coating-superalloy system were removed at the first sign of significant hot corrosion or coating breakdown. Replacements were made to obtain four different levels of attack on each test material. Exposure of specimens varied from 11 to 143 hours.

A linear relationship was found between specimen weight-loss and depth of penetration with these superalloys at this condition of exposure, and either measurement provides an evaluation of hot-corrosion attack. Since weight-loss measurements are the most economical, they should be used for evaluation whenever shown to be feasible by metallographic inspection.

Test precision was improved during this investigation by modification of the flame-tube configuration, which improved combustor durability and supplied a hot gas of more uniform temperature to the Turbine Simulator.

It was concluded from this investigation that:

A. The wear and corrosion on test-section components was not excessive, and the Turbine Simulator has satisfactory mechanical durability for use in conducting future test programs at temperatures to 2000 F, the highest level attempted.

B. This simulated-environmental test now represents a satisfactory compromise for conducting our proposed investigation with a reasonable expenditure of time, manpower, and materials. An exposure time of about 55 hours was indicated to obtain failure of an aluminum-diffusion coating with rapid-temperature cycling from 1000 F to a maximum of 2000 F. This is roughly a reduction to 1/6 the time required for coating breakdown during the initial trial of the Turbine Simulator when exposure was at 1650 F.

C. The improved precision obtained by the more uniform specimen exposure in the Turbine Simulator should facilitate a satisfactory evaluation of the effect of very-low levels of sulfur in JP-5 fuel on the durability of turbine alloys in high-performance engines when operated in a marine environment. The standard deviation of weight-loss data for coated specimens (as represented by Misco MDC-1 coated Inconel 713C) is significantly greater than for non-coated specimens (as represented by bare Inconel 713C); but, both values are less than obtained with the flat specimens used previously.

3. RECOMMENDATIONS

The purpose of this investigation was to evaluate the performance of the Phillips Turbine Simulator, using a cyclic-temperature procedure, with respect to its mechanical durability, its severity of operating conditions, and its uniformity of specimen exposure.

Mechanical durability of the Phillips Turbine Simulator, severity of the cyclic-temperature procedure, and precision of the test method have been demonstrated to be adequate for use in proposed test programs.

It is recommended that we proceed with our investigation to determine whether the maximum sulfur limit of 0.40 weight per cent, currently allowed in grade JP-5 aviation-turbine fuel, is a safe level for the protection of turbine materials in engines of advanced design. Emphasis should be placed on very-low levels of sulfur by evaluating the effect of 0.040, 0.0040, and 0.00040 weight per cent sulfur in JP-5 fuel. The Phillips Turbine Simulator should be used with a rapid-cycling procedure, having a maximum temperature of 2000 F, and with 1.0 ppm sea salt in air. The hot corrosion of turbine materials, covering a broad range in composition, should be studied by using eight different nickel-base superalloys with two different coatings (aluminum and chromium-aluminum diffusion) and four different cobalt-base superalloys with one coating (chromium-aluminum diffusion).

4. RESULTS AND DISCUSSION

In a previous study (4) the Phillips Turbine Simulator, operated at 1650 F specimen temperature, was demonstrated to have adequate mechanical durability and precision for use in evaluating the effect of sulfur in fuel on hot corrosion; however, exposure times of 50 to 100 hours for bare superalloys and over 350 hours for some coating-superalloy systems were indicated. It was recommended that a cyclic-temperature procedure be considered, using the Phillips Turbine Simulator, to span the range of temperatures of interest in a single test and thus reduce the time required to complete the investigation of the effect of sulfur in fuel on hot corrosion of coated superalloys in a marine environment.

The primary purpose of the current investigation was to evaluate the performance of the Phillips Turbine Simulator, using a cyclic-temperature procedure, with respect to mechanical durability and precision, and to obtain an estimate of the severity of the operating conditions. An adequate supply of bare and Misco MDC-1 coated Inconel 713C specimens was available from batches used in the previous investigation (4). In addition, a limited number of specimens of six superalloys (IN-100, Inconel 713C, Udimet 700, Mar-M421, IN-738, and Udimet 500) were available as part of a "round-robin" testing program of the ASTM Hot-Corrosion Task-Force, and these superalloys were included in the investigation. Except for Mar-M421 and Udimet 500 these superalloys are scheduled for inclusion in our future test programs. Specimens were exposed at 15 atmospheres combustor pressure in the presence of 1.0 ppm sea salt in air and 0.04 weight per cent sulfur in fuel at nominal exhaust-gas temperatures from 1000 to 2000 F. The cyclic testing was halted at 11-hour intervals to allow visual inspection of specimens. Specimens of each superalloy and coating-superalloy system were removed at the first sign of significant hot corrosion or coating breakdown. Replacements were made to obtain four different levels of attack on each test material.

Details concerning the test equipment, test materials, and test procedures are presented in Appendicies 1, 2, and 3, respectively, which are Sections 8., 9., and 10. of this report.

The test plan proposed for use in the future evaluation of the effect of sulfur in fuel on hot corrosion of superalloys in a marine environment was used in this investigation and is described in Section 10.4. of Appendix 3. This test plan provides for the use of a block of four positions in a row of the specimen retainer to expose two test specimens at each of four different lengths of time, with the duplicate specimens exposed during different test periods.

The availability of only four test specimens of four of the six superalloys in the ASTM portion of the program required a modification of the test plan for some of the blocks.

4.1. Test-Section Durability

Some mechanical problems were encountered with the specimen drive section during this investigation. Wear and attack on components exposed to hot gases was greater than during the previous test (4) at lower exhaust-gas temperature; however, the problems were corrected by replacement of parts and it was concluded that this design for a test section has satisfactory durability for use in conducting future test programs.

During the first 10 periods (110 hours) the temperature profile of the gas from the combustor showed an increased spread with time during each period, and at the end of each period the first internal deflector skirt was damaged to the extent that the flame tube required removal for replacement and repair. At 110 hours the flame tube was changed from Configuration 15 to Configuration 20 of Table 13 (Appendix 1, Section 8.2.). This change in the combustor configuration eliminated the flame-tube-durability problem and improved the temperature profile.

4.2. Deposit-Weight Data

After exposure to the hot gases for the desired periods of time specimens were removed from the retainer, weighed with deposits in place, electro-cleaned to remove the surface deposits and scale and then reweighed. The weight-of-deposit on a specimen, after exposure, was calculated as the difference in the weight of the specimen as removed from the test rig and the weight after electro-cleaning. The deposit-weight and deposit-weight per unit-area of new specimens are shown for each specimen in Tables 1, 2, and 3. Deposits were observed to flake off the specimens as they cooled and thus the amount of deposits on the specimens during exposure may be greater than the indicated weights.

The deposit-weight per unit-area data were examined, and for those cases where duplicate measurements were available an Analysis-of-Variance technique was used to determine the effect of time-of-exposure on the amount of deposit on a specimen. It was concluded that there was no statistically-significant effect of time-of-exposure on weight-of-deposits on any of the superalloys or the coating-alloy system.

An examination of the deposit-weight data for the six superalloys in the ASTM portion of the program (Table 1) indicated no statistically-significant difference in the amount of deposits; however, as shown in the following tabulation, the maximum weight-of-deposits increased with an increase in chromium content of the superalloys.

Superalloy	Chromium Content, %	Maximum Deposit Weight, mg/cm^2
IN-100	10.1	19.17
Inconel 713C	13.0	20.85
Udimet 700	14.3	29.88
IN-738	15.0	25.46
Mar-M421	15.1	28.72
Udimet 500	18.9	37.30

TABLE 1

DEPOSIT AND WEIGHT LOSS DATA FOR ASTM SPECIMENS

(a) Position	Specimen Number	Exposure Time, Hours	Exposure Period Numbers	Deposits		Total Specimen Weight Loss	
				mg	mg/cm2	mg	mg/cm2
			IN-100				
3A	45	11	1	86.4	11.13	977.2	125.92
3F	46	22	1-2	133.3	17.18	1782.7	229.72
3K	47	33	1-3	148.8	19.17	2397.6	308.96
3P	48	44	1-4	49.8	6.42	2883.6	371.58
			Inconel 713C				
2A	25	11	1	65.2	8.40	496.5	63.98
2M	76	11	5	82.2	10.59	407.2	52.47
2C	26	22	1-2	99.6	12.83	912.4	117.57
2K	75	22	4-5	76.1	9.81	365.5	45.94
2K	27	33	1-3	97.0	12.50	1272.5	163.97
2C	74	33	3-5	161.8	20.85	461.1	59.42
2M	28	44	1-4	120.1	15.48	1552.6	200.07
2A	73	44	2-5	159.7	20.58	1381.2	177.98
			Udimet 700				
3D	33	22	1-2	137.2	17.68	547.7	70.58
3H	34	44	1-4	72.8	9.38	1273.1	164.05
3Q	35	66	1-6	231.9	29.88	2458.2	316.76
3R	36	88	1-8	121.8	15.70	2539.8	327.28
			Mar M421				
3E	41	22	1-2	113.9	14.68	537.0	69.20
3G	42	44	1-4	134.6	17.34	979.0	126.15
3L	43	66	1-6	222.9	28.72	1838.3	236.88
3M	44	88	1-8	206.2	26.57	2238.0	288.39
			IN-738				
2D	29	22	1-2	80.4	10.36	333.2	42.94
2G	30	44	1-4	148.1	19.08	754.0	97.16
2J	31	66	1-6	173.9	22.41	1176.3	151.58
2R	32	88	1-8	197.7	25.46	1479.0	190.58
			Udimet 500				
3B	37	44	1-4	46.0	5.93	435.6	56.13
3P	80	44	5-8	65.4	8.43	158.2	20.39
3C	38	77	1-7	75.6	9.74	1070.0	137.88
3K	79	77	4-10	235.7	30.37	918.1	118.31
3J	39	110	1-10	208.7	26.89	1761.4	226.97
3F	78	110	3-12	133.1	17.15	1760.4	226.84
3N	40	143	1-13	289.5	37.30	3339.9	430.38
3A	77	143	2-14	141.3	18.21	3194.8	411.68

(a) Position in holder: 1 = Front Row, 2 = Second Row, 3 = Rear Row. Letter = Position in Row.

TABLE 2

DEPOSIT AND WEIGHT LOSS DATA FOR
MISCO MDC-1 COATED INCONEL 713C SPECIMENS

(a) Position	Specimen Number	Exposure Time, Hours	Exposure Period Number	Deposits		Total Specimen Weight Loss	
				mg	mg/cm²	mg	mg/cm²
1H	1	55	1-5	54.1	6.97	311.8	40.18
1P	170	55	12-16	35.0	4.51	222.2	28.63
1K	2	77	1-7	31.7	4.08	900.6	116.05
1L	166	77	10-16	47.1	6.07	1090.8	140.56
1L	3	99	1-9	46.4	5.98	1513.7	195.06
1K	150	99	8-16	44.0	5.67	1844.9	237.73
1P	4	121	1-11	53.9	6.95	2336.8	301.12
1H	146	121	6-16	32.5	4.19	2884.7	371.72
1E	5	55	1-5	47.0	6.06	275.4	35.49
1R	171	55	12-16	33.4	4.30	297.6	38.35
1F	6	77	1-7	34.5	4.45	799.2	102.98
1Q	167	77	10-16	47.1	6.07	1261.4	162.54
1Q	7	99	1-9	62.8	8.09	1372.8	176.90
1F	151	99	8-16	36.7	4.73	2155.5	277.76
1R	8	121	1-11	59.4	7.65	2135.5	275.18
1E	147	121	6-16	37.2	4.79	3132.7	403.68
1B	9	55	1-5	45.2	5.82	328.1	42.28
1M	172	55	12-16	45.3	5.84	643.3	82.90
1C	10	77	1-7	42.3	5.45	872.9	112.48
1D	168	77	10-16	39.8	5.13	428.7	55.24
1D	11	99	1-9	61.1	7.87	1343.2	173.08
1C	152	99	8-16	35.1	4.52	1610.6	207.54
1M	12	121	1-11	62.7	8.08	2399.1	309.15
1B	148	121	6-16	39.8	5.13	2689.2	346.53
1A	13	55	1-5	53.5	6.89	208.8	26.91
1N	173	55	12-16	31.9	4.11	643.0	82.86
1G	14	77	1-7	42.2	5.44	821.9	105.91
1J	169	77	10-16	38.0	4.90	920.5	118.62
1J	15	99	1-9	65.5	8.44	1414.3	182.25
1G	153	99	8-16	37.8	4.87	2025.5	261.01
1N	16	121	1-11	57.2	7.37	2358.6	303.93
1A	149	121	6-16	45.9	5.91	2697.1	347.55

(a) Position in holder: 1 = Front Row, 2 = Second Row, 3 = Rear Row.
Letter = Position in Row.

TABLE 2

DEPOSIT AND WEIGHT LOSS DATA FOR MISCO MDC-1
COATED INCONEL 713C SPECIMENS (CONTINUED)

(a) Position	Specimen Number	Exposure Time Hours	Exposure Period Number	Deposits		Total Specimen Weight Loss	
				mg	mg/cm^2	mg	mg/cm^2
2D	174	55	11-15	32.1	4.14	798.6	102.91
2G	175	55	11-15	36.9	4.76	900.4	116.03
2J	176	55	11-15	35.5	4.57	570.0	73.45
2R	177	55	11-15	35.0	4.51	272.8	35.15
3E	182	55	11-15	33.0	4 25	551.0	71.00
3G	183	55	11-15	32.8	4.23	486.2	62.65
3L	184	55	11-15	32.6	4.20	422.6	54.46
3M	185	55	11-15	37.1	4.78	357.7	46.09

(a) Position in holder: 1 = Front Row, 2 = Second Row, 3 = Rear Row. Letter = Position in Row.

TABLE 3

DEPOSIT AND WEIGHT LOSS DATA FOR

INCONEL 713C SPECIMENS

(a) Position	Specimen Number	Exposure Time Hours	Exposure Period Numbers	Deposits mg	Deposits mg/cm^2	Total Specimen Weight Loss mg	Total Specimen Weight Loss mg/cm^2
			Block 1				
2E	17 (b)	11	1	90.7	11.69	259.7	33.46
2L	52 (b)	11	5	112.4	14.48	225.0	28.99
2F	18 (b)	22	1-2	95.1	12.25	662.9	85.42
2H	51 (b)	22	4-5	97.5	12.56	225.1	29.01
2H	19 (b)	33	1-3	102.5	13.21	1125.3	145.01
2F	50 (b)	33	3-5	155.4	20.02	496.4	63.97
2L	20 (b)	44	1-4	74.6	9.61	1326.0	170.87
2E	49 (b)	44	2-5	152.8	19.69	1058.1	136.35
			Block 2				
2E	81 (b)	11	6	89.4	11.52	110.7	14.26
2L	72 (b)	11	10	123.0	15.85	203.2	26.18
2F	82 (b)	22	6-7	78.4	10.10	417.8	53.84
2H	71 (b)	22	9-10	127.1	16.38	630.6	81.26
2H	83 (b)	33	6-8	89.8	11.57	619.9	79.88
2F	70 (b)	33	8-10	171.8	22.14	780.4	100.56
2L	84 (b)	44	6-9	129.6	16.70	1114.5	143.61
2E	69 (b)	44	7-10	168.1	21.66	732.5	94.36
			Block 3				
2B	21 (c)	11	1	91.8	11.83	180.0	23.19
2Q	56 (c)	11	5	117.0	15.08	210.6	27.14
2N	22 (c)	22	1-2	98.0	12.63	590.0	76.10
2P	55 (c)	22	4-5	99.5	12.82	246.0	31.70
2P	23 (c)	33	1-3	118.6	15.28	1084.7	139.77
2N	54 (c)	33	3-5	113.8	17.24	463.1	59.68
2Q	24 (c)	44	1-4	64.4	8.30	1131.2	145.77
2B	53 (c)	44	2-5	124.9	16.09	1139.3	146.81
			Block 4				
2B	85 (c)	11	6	95.5	12.31	134.1	17.28
2Q	92 (c)	11	10	142.5	18.36	201.0	25.90
2N	86 (c)	22	6-7	83.5	10.76	340.2	43.84
2P	91 (c)	22	9-10	105.6	13.61	656.2	84.56
2P	87 (c)	33	6-8	80.1	10.32	443.4	57.14
2N	90 (c)	33	8-10	137.1	17.67	788.2	101.57
2Q	88 (c)	44	6-9	126.4	16.29	948.2	122.18
2B	89 (c)	44	7-10	167.0	21.52	673.7	86.81

(a) Position in holder: 1 = Front Row, 2 = Second Row, 3 = Rear Row. Letter = Position in Row.
(b) Heat No. RW 437. (c) Heat No. RW 424.

TABLE 3

DEPOSIT AND WEIGHT LOSS DATA FOR INCONEL 713C SPECIMENS (Continued)

(a) Position	Specimen Number	Exposure Time, Hours	Exposure Period Numbers	Deposits		Total Specimen Weight Loss	
				mg	mg/cm^2	mg	mg/cm^2
			Block 5				
2A	93 (c)	11	6	77.4	9.97	64.5	8.31
2M	100 (c)	11	10	111.5	14.37	153.4	19.77
2C	94 (c)	22	6-7	135.0	17.40	286.4	36.91
2K	99 (c)	22	9-10	126.3	16.28	559.0	72.03
2K	95 (c)	33	6-8	69.4	8.94	609.3	78.51
2C	98 (c)	33	8-10	123.8	15.95	762.0	98.19
2M	96 (c)	44	6-9	126.2	16.26	926.6	119.40
2A	97 (c)	44	7-10	175.2	22.58	809.4	104.30
2D	57 (b)	44	3-6	108.4	13.97	938.1	120.88
2G	58 (b)	33	5-7	71.8	9.25	805.2	103.76
2J	59 (b)	22	7-8	43.4	5.59	40.4	5.21
2R	60 (b)	11	9	101.3	13.05	181.0	23.32
2D	105 (c)	44	7-10	169.4	21.83	866.5	111.66
2G	106 (c)	33	8-10	113.8	14.66	737.2	95.00
2J	107 (c)	22	9-10	117.2	15.10	692.2	89.20
2R	108 (c)	11	10	136.9	17.64	220.4	28.40
			Block 6				
2B	125 (c)	11	11	91.5	11.79	305.9	39.42
2Q	132 (c)	11	15	71.4	9.20	303.9	39.16
2N	126 (c)	22	11-12	112.4	14.48	775.7	99.96
2P	131 (c)	22	14-15	78.4	10.10	891.5	114.88
2P	127 (c)	33	11-13	103.0	13.27	1163.7	149.95
2N	130 (c)	33	13-15	95.4	12.29	1475.5	190.13
2Q	128 (c)	44	11-14	84.4	10.88	1532.0	197.41
2B	129 (c)	44	12-15	93.3	12.02	1771.6	228.29
			Block 7				
2E	117 (c)	11	11	103.3	13.31	294.9	38.00
2L	124 (c)	11	15	66.6	8.58	254.3	32.77
2F	118 (c)	22	11-12	110.2	14.20	893.9	115.19
2H	123 (c)	22	14-15	93.4	12.04	830.6	107.03
2H	119 (c)	33	11-13	129.4	16.67	1258.2	162.13
2F	122 (c)	33	13-15	80.1	10.32	1417.7	182.68
2L	120 (c)	44	11-14	99.4	12.81	1853.9	238.89
2E	121 (c)	44	12-15	74.4	9.59	1613.7	207.94

(a) Position in holder: 1 = Front Row, 2 = Second Row, 3 = Rear Row. Letter = Position in Row.

(b) Heat No. RW 437. (c) Heat No. RW 424.

TABLE 3

DEPOSIT AND WEIGHT LOSS DATA FOR INCONEL 713C SPECIMENS (Continued)

Position	Specimen Number	Exposure Time, Hours	Exposure Period Number	Deposits		Total Specimen Weight Loss	
				mg	mg/cm^2	mg	mg/cm^2
			Block 8				
2A	133 (c)	11	11	114.6	14.77	211.1	27.20
2M	140 (c)	11	15	65.0	8.38	283.0	36.47
2C	134 (c)	22	11-12	95.6	12.32	813.8	104.87
2K	139 (c)	22	14-15	65.5	8.44	859.9	110.81
2K	135 (c)	33	11-13	84.4	10.88	1387.5	178.79
2C	138 (c)	33	13-15	81.0	10.44	1269.9	163.64
2M	136 (c)	44	11-14	83.4	10.75	1816.0	234.00
2A	137 (c)	44	12-15	71.3	9.19	1705.6	219.78
3D	61 (b)	44	3-6	138.2	17.81	850.0	109.53
3H	62 (b)	33	5-7	93.9	12.10	665.3	85.73
3Q	63 (b)	22	7-8	68.6	8.84	63.1	8.13
3R	64 (b)	11	9	35.7	4.60	143.5	18.49
3D	109 (c)	44	7-10	172.1	22.18	639.6	82.42
3H	110 (c)	33	8-10	157.4	20.28	511.9	65.96
3Q	111 (c)	22	9-10	169.3	21.82	445.4	57.39
3R	112 (c)	11	10	100.0	12.89	159.4	20.54
3E	65 (b)	44	3-6	121.5	15.66	620.8	80.00
3G	66 (b)	33	5-7	77.3	9.96	785.7	101.24
3L	67 (b)	22	7-8	59.4	7.65	54.7	7.05
3M	68 (b)	11	9	99.3	12.80	293.9	37.87
3E	113 (c)	44	7-10	164.4	21.18	502.0	64.69
3G	114 (c)	33	8-10	164.4	21.18	532.2	68.58
3L	115 (c)	22	9-10	131.2	16.91	567.9	73.18
3M	116 (c)	11	10	136.9	17.64	155.6	20.05
			Block 9				
3D	141 (c)	11	11	98.8	12.73	215.3	27.74
3R	192 (c)	11	15	74.3	9.57	153.9	19.83
3H	142 (c)	22	11-12	122.1	15.73	699.3	90.11
3Q	191 (c)	22	14-15	71.5	9.21	649.5	83.69
3Q	143 (c)	33	11-13	100.8	12.99	912.7	117.61
3H	190 (c)	33	13-15	102.0	13.14	1166.5	150.32
3R	144 (c)	44	11-14	90.3	11.64	1447.0	186.46
3D	145 (c)	44	12-15	81.3	10.48	1265.8	163.11

(a) Position in holder: 1 = Front Row, 2 = Second Row, 3 = Rear Row. Letter = Position in Row.
(b) Heat No. RW 437. (c) Heat No. RW 424.

TABLE 3

DEPOSIT AND WEIGHT LOSS DATA FOR INCONEL 713C SPECIMENS (Continued)

(a) Position	Specimen Number	Exposure Time, Hours	Exposure Period Numbers	Deposits		Total Specimen Weight Loss	
				mg	mg/cm^2	mg	mg/cm^2
3B	193 (c)	44	13-16	132.6	17.09	1592.2	205.17
3C	194 (c)	33	14-16	94.3	12.15	1124.6	144.92
3J	195 (c)	22	15-16	93.4	12.04	556.4	71.70
3N	196 (c)	11	16	78.8	10.15	121.7	15.68
3B	162 (c)	44	9-12	165.2	21.29	1454.0	187.36
3C	163 (c)	33	11-13	104.1	13.41	923.9	119.05
3J	164 (c)	22	13-14	121.7	15.68	489.1	63.02
3N	165 (c)	11	15	106.1	13.67	193.1	24.88
3P	154 (c)	44	9-12	151.1	19.47	1526.7	196.73
3K	155 (c)	33	11-13	88.5	11.40	975.8	125.74
3F	156 (c)	22	13-14	113.7	14.65	539.4	69.51
3A	157 (c)	11	15	62.0	7.99	156.8	20.20
3P	158 (c)	44	13-16	115.3	14.86	1469.9	189.41
3K	159 (c)	33	14-16	90.4	11.65	1151.2	148.34
3F	160 (c)	22	15-16	109.3	14.08	527.2	67.94
3A	161 (c)	11	16	82.9	10.68	108.5	13.98
3B	101 (c)	44	5-8	103.6	13.35	742.7	95.70
3C	102 (c)	33	8-10	150.0	19.33	529.4	68.22
3J	103 (c)	22	11-12	91.4	11.78	689.3	88.82
3N	104 (c)	11	14	100.8	12.99	184.7	23.80

(a) Position in holder: 1 = Front Row, 2 = Second Row, 3 = Rear Row. Letter = Position in Row.

(b) Heat No. HW 437.

(c) Heat No. HW 424.

4.3. Metal-Weight-Loss Data

The metal weight-loss of a specimen was calculated as the difference between the weight of a new specimen and the weight of the electro-cleaned specimen following exposure to hot gases. The weight-loss and weight-loss per unit-area for specimens in the ASTM program, Misco MDC-1 coated Inconel 713C specimens, and bare Inconel 713C specimens are shown in Tables 1, 2, and 3 respectively.

4.3.1. Standard Deviation

In previous studies (1) of weight-loss from hot corrosion, using a cascade-specimen holder mounting two flat specimens in each of three stages and exposing specimens to hot gases in a 5-hour test, a uniform coefficient-of-variation of the data was found and logarithms of weight-loss per unit-area were used to provide a basis of uniform variance. The standard deviations for the two programs were estimated to be 0.355 and 0.288 in terms of logarithms or coefficients-of-variation of 116 and 94 per cent. In a recent investigation (4), round specimens of bare Inconel 713C were exposed at temperatures of 1650 F for periods of up to 150 hours. An estimate of the standard deviation from the data was 0.022 in terms of logarithms or a coefficient-of-variation of 5.3 per cent; however, this value is an estimate of specimen-to-specimen variability within a test while the previous estimate represented differences between tests.

An analysis of the raw data (mg/cm^2) for the current investigation indicated a uniform coefficient-of-variation of the data and logarithms of weight-loss per unit-area have been used in the following analyses.

Because only four specimens were available for each of four superalloys in the ASTM portion of the investigation, it was necessary to modify the test plan for some positions in the specimen retainer and duplicate specimens were tested consecutively in the same position rather than in blocks as planned. During the initial portion of the test, flame-tube durability was poor and it was necessary to replace the flame tube at each eleven-hour inspection. After ten test-periods (110 hours) a change was made from flame tubes of Configuration No. 15 to No. 20 (Table 13). Test specimens in the ASTM portion of the program were formed by plunge grinding while the remaining specimens were formed as investment castings. Therefore, the ASTM specimens have been considered separately because of the difference in surface finish.

For each pair of weight-loss values an estimate of experimental variance can be obtained with one degree-of-freedom. Appropriate values have been pooled and a summary of the estimates of experimental variance, based on logarithms of weight-loss per unit-area, are shown in Table 4. All estimates of standard deviations for the current program are equal to, or lower than, the estimates from the previous programs using flat specimens (0.355 and 0.288); however, as expected, the values are not as small as the within-test variability of the most recent test (0.022).

TABLE A

ESTIMATES OF PRECISION FROM HOT CORROSION TEST DATA

A. Inconel 713C

1. Before combustor modification: Duplicate data from blocks and between duplicates with no overlap.

$\hat{\sigma}_B^2 = 0.07273$ (d.f. = 32)

$\hat{\sigma}_B = 0.26968$

Coefficient of Variation, % = 86.1

2. After combustor modification: Duplicate data from blocks and between duplicates with no overlap.

$\hat{\sigma}_A^2 = 0.00407$ (d.f. = 24)

$\hat{\sigma}_A = 0.06380$

Coefficient of Variation, % = 15.8

B. ASTM Metals

1. Inconel 713C: Duplicate data from block before combustor modification.

$\hat{\sigma}^2 = 0.04635$ (d.f. = 4)

$\hat{\sigma} = 0.21529$

Coefficient of Variation, % = 64.2

2. Udimet 500: Duplicate data from block before and after combustor modification.

$\hat{\sigma}^2 = 0.02478$ (d.f. = 4)

$\hat{\sigma} = 0.15742$

Coefficient of Variation, % = 43.7

3. Pooled estimates for ASTM metals: Inconel 713C and Udimet 500.

$\hat{\sigma}^2 = 0.03556$ (d.f. = 8)

$\hat{\sigma} = 0.18857$

Coefficient of Variation, % = 54.4

TABLE 4

ESTIMATES OF PRECISION FROM HOT CORROSION TEST DATA (Continued)

C. Misco MDC-1 Coated Inconel 713C

Estimates using duplicates from blocks before and after combustor modification.

$\hat{\sigma}^2$ = 0.01904 (d.f. = 16)

$\hat{\sigma}$ = 0.13799

Coefficient of Variation, % = 37.4

Data on bare Inconel 713C specimens exposed prior to, and after, the combustor modification are available and permit an evaluation of the effect of the combustor modification on the precision of the weight-loss data. As shown in Table 4, the estimate of the standard deviation of weight-loss per unit-area for bare Inconel 713C specimens exposed prior to the combustor modification was $\sigma_B = 0.26969$, or a coefficient-of-variation of 86.1%, and the estimate of the standard deviation of data after the combustor modification was $\sigma_A = 0.06360$, or a coefficient-of-variation of 15.8%. The reduction in experimental variance from the combustor modification is statistically significant, and the combustor modification improves test precision.

In the ASTM portion of the investigation only two of the superalloys were evaluated in duplicate (Inconel 713C and Udimet 500) and the experimental variance to judge metal comparisons was estimated from these duplicates. Data on Inconel 713C were obtained prior to the combustor modification, and data on Udimet 500 were obtained both before and after the combustor modification. The pooled value for the estimate of the standard deviation ($\hat{\sigma} = 0.18857$) includes the effect of the combustor modification on precision.

The estimate of the standard deviation for the Misco MDC-1 coated Inconel 713C specimens is $\hat{\sigma} = 0.13799$, or a coefficient-of-variation of 37.4%. The modification of the combustor occurred such that the effects of the modification are confounded between duplicates, and the estimate of the standard deviation could be higher than would have been estimated had all data been obtained after the combustor modification. Based on the data available, the estimate of the standard deviation for coated superalloys (as represented by Misco MDC-1 coated Inconel 713C) is isgnificantly higher than the estimate of the standard deviation for the bare Inconel 713C exposed after the combustor modification. While this indication of poorer precision may be characteristic of coating deterioration, additional data are needed to establish more valid error estimates for comparison of coated superalloys.

From these data it is concluded that Phillips Turbine Simulator, with present operating procedures, provides improved precision and should allow a satisfactory evaluation of the effect of sulfur in JP-5 fuel on hot corrosion of coated superalloys in a marine environment.

4.3.2. Magnitude of Weight Loss

Data for bare Inconel 713C specimens exposed in blocks in Row 2 of the specimen retainer (Blocks 1 - 8, Table 3) were examined for an effect of the combustor modification on the magnitude of weight-loss. A summary of an Analysis-of-Variance (AOV) of these weight-loss data are shown in Table 5. Statistically-significant effects of Time-of-exposure and Blocks-of-data on weight-loss are shown in the analysis, but the Time-by-Block interaction was not statistically significant. In the absence of a Time-by-Block interaction, comparisons can be made of mean weight-losses, averaged over time for each block. From the estimate of standard deviation a Tolerable-Spread-between-Means (TSM) for comparisons of means of weight-loss for Blocks-of-data was calculated to be 0.14111 in terms of logarithms. In

TABLE 5

EFFECTS OF TIME, HEAT, AND COMBUSTOR MODIFICATION ON HOT CORROSION
(Bare Inconel 713C - Row 2)

A. Analysis of Variance

Source of Variation	Degrees of Freedom	Sum of Squares	Mean Square
Total	63	7.54642	
Time (T)	3	5.59292	1.86431 *
Blocks (B)	7	1.13192	0.16170 *
T x B	21	0.18413	0.00877
Error	32	0.63745	0.01992

Asterisk (*) indicates a significant effect at 95 per cent confidence level.

B. Effect of Alloy Heats and Combustor Modification

Block	Heat No.	Combustor Modification	Mean Weight Loss, log mg/cm^2	Geometric Mean Weight Loss, mg/cm^2
5	RW 424	Before	1.70282	50.44
4	RW 424	Before	1.75114	56.38
2	RW 437	Before	1.78124	60.42
3	RW 424	Before	1.80413	63.69
1	RW 437	Before	1.83945	69.09
8	RW 424	After	2.02990	107.13
7	RW 424	After	2.04423	110.71
6	RW 424	After	2.04469	110.84

TSM = 0.14111 (log mg/cm^2)

Table 5 the mean weight-loss for each Block of data is listed in increasing order and the data are separated, on the basis of the TSM, into two groups that differ significantly. From this separation it can be concluded that the combustor modification significantly increased the magnitude of the weight-loss of bare Inconel 713C specimens. It can also be concluded that there is no statistically-significant difference in the resistance to hot corrosion attack of the two heats of Inconel 713C.

The limited data available does not provide a firm evaluation of the effect of rows in the specimen retainer, if any, on hot corrosion; however, in future experiments a design will be used that will eliminate the effect of rows in any desired comparisons.

All meaningful comparisons of the relative resistance to hot corrosion, as measured by weight-loss, of the six superalloys in the ASTM portion of the program have been made. Summaries of three AOV are shown in Table 6, and comparisons of mean weight-losses are shown in Table 7. The experimental error (0.03556) to judge metal comparisons was estimated from duplicates on Inconel 713C and Udimet 500.

Specimens of IN-738, Udimet 700 and Mar-M421 were exposed for 22, 44, 66, and 88 hours, and an AOV indicated no statistically-significant difference in weight-loss between these three superalloys.

Specimens of IN-100 and Inconel 713C were exposed for 11, 22, 33, and 44 hours and an AOV indicates a significant difference in weight-loss for the two superalloys. A comparison of the means shows that the weight-loss for IN-100 is larger than for Inconel 713C.

Using only two of the time points for each of the five superalloys above (22 and 44 hours) and AOV indicates a statistically-significant effect of superalloy on weight-loss. Comparisons of means indicated that the weight-loss with IN-100 was significantly greater, and the weight-loss with Inconel 713C was the same as the weight-loss with IN-738, Mar-M421 and Udimet 700.

The six superalloys in the ASTM program were exposed at only one common time, 44 hours, and statistically-significant differences between their mean weight-losses are indicated in Table 7-D. In making these multiple-comparison tests, the significance of differences between individual means were determined at the 95 per cent confidence level. If two or more means have the same line drawn underneath them, they are not statistically different. If two or more means do not have the same line underneath them, they are said to be statistically different. Thus, at 44 hours exposure, no significant difference is shown between Udimet 500 and IN-738, but their weight-loss is significantly less than for IN-100. Also, the weight-loss for Udimet 500 is significantly less than for Mar-M421, Udimet 700, Inconel 713C, and IN-100.

TABLE 6

ANALYSIS OF VARIANCE OF WEIGHT LOSS DATA
(ASTM Program)

A. IN-738, Mar M421, and U-700 at 22, 44, 66, and 88 hours.

Source of Variation	Degrees of Freedom	Sum of Squares	Mean Square
Total	11	0.90630	
Metals (M)	2	0.13092	0.06546
Time (H)	3	0.76839	0.25613 *
M x H	6	0.00699	0.00117
Error (a)	8		0.03556

Asterisk (*) indicates a significant effect at 95 per cent confidence level.
(a) Pooled value from Inconel 713C and U-500 data.

B. IN-100 and Inconel 713C at 11, 22, 33, and 44 hours.

Source of Variation	Degrees of Freedom	Sum of Squares	Mean Square
Total	11	1.04601	
Metals (M)	1	0.43843	0.43843 *
Time (H)	3	0.40003	0.13334 *
M x H	3	0.02215	0.00738
Error (a)	8		0.03556

Asterisk (*) indicates a significant effect at 95 per cent confidence level.
(a) Pooled value from Inconel 713C and U-500 data.

C. IN-738, U-700, Mar M421, IN-100, and Inconel 713C at 22 and 44 hours.

Source of Variation	Degrees of Freedom	Sum of Squares	Mean Square
Total	11	0.90884	
Metals (M)	4	0.47073	0.11768 *
Time (H)	1	0.33654	0.33654 *
M x H	4	0.01700	0.00425
Error (a)	8		0.03556

Asterisk (*) indicates a significant effect at 95 per cent confidence level.
(a) Pooled value from Inconel 713C and U-500 data.

TABLE 7

COMPARISONS OF SUPERALLOYS IN ASTM PROGRAM

A. IN-738, Mar M421, and U-700 at 22, 44, 66, and 88 hours.

No statistically significant difference indicated in weight loss of these three superalloys.

B. IN-100 and Inconel 713C at 11, 22, 33, and 44 hours.
(TSM = 0.23095)

Superalloy	Mean Weight Loss, log mg/cm^2	Geometric Mean Weight Loss, mg/cm^2
IN-100	2.38031	239.99
Inconel 713C	1.97484	94.36
Difference	0.40547 *	

*Significant at 95 per cent confidence level.

C. IN-738, U-700, Mar M421, IN-100 and Inconel 713C at 22 and 44 hours.
(TSM = 0.37715 except TSM = 0.32662 for Inconel 713C)

Superalloy	Mean Weight Loss, log mg/cm^2	Geometric Mean Weight Loss, mg/cm^2
IN-738	1.81017	64.60
Mar M421	1.97050	93.43
U-700	2.03183	107.60
Inconel 713C	2.07101	117.76
IN-100	2.46562	292.15

D. Comparison of all metals at 44 hours.

TSM_1 = 0.53398

TSM_2 = 0.46191 (Compare U-500 and Inconel 713C with others)

TSM_3 = 0.37715 (Compare U-500 with Inconel 713C)

Superalloy	U-500	IN-738	Mar-M421	U-700	Inconel 713C	IN-100
MWL (a)	1.52930	1.98748	2.10089	2.21498	2.27578	2.57005
GMWL (b)	33.83	97.16	126.2	164.1	188.7	371.5

Notes:

(a) MWL = Mean Weight Loss, log mg/cm^2

(b) GMWL = Geometric Mean Weight Loss, mg/cm^2

On the basis of all of the data, IN-100 would be rated the least resistant to hot corrosion attack; Inconel 713C, Udimet 700, Mar-M421, and IN-738 equal in resistance to hot-corrosion attack, and more resistant than IN-100; and Udimet 500 and IN-738 equal in resistance to hot corrosion attack.

4.3.3. Regression Equations

Estimated relationships between weight-loss and time-of-exposure are given in Table 8 for the various comparisons in this study. All data points in this experiment were used to estimate these relationships. Comparisons were made of the variance for each relationship, using the residual mean-square to estimate the adequacy of the model. On the basis of these data, the estimated relationships adequately describe the data. Graphical summaries of the data are shown in Figures 1 and 2, where the lines represent the calculated relationships.

A comparison of the relative durability of superalloys in the ASTM program can be made by using these regression equations. The weight-loss for the least resistant superalloy (IN-100) was 127 mg/cm^2 at the first 11-hour inspection point. Such a loss amounts to about 12 per cent of the original specimen weight, and this is estimated to be near that suffered by many turbine blades when finally removed from service. By using this level of attack, comparisons can be made without extrapolating the regressions, and the time-of-exposure for the other materials in the program were calculated to be:

Superalloy	Source	Exposure Time for 127 mg/cm^2
IN-100	ASTM	11 Hours
Udimet 700	ASTM	35
Inconel 713C	ASTM	37
Mar-M421	ASTM	42
IN-738	ASTM	55
Udimet 500	ASTM	81
Inconel 713C	Misco	48
Inconel 713C (Modified Combustor)	Misco	28
Misco MDC-1 Coated Inconel 713C	Misco	81

These data can provide an indication of exposure times that will be required for destruction of coating-alloy systems in our proposed investigation to determine the effect of very-low-sulfur fuels on hot corrosion. Comparing the data for bare Inconel 713C (after combustor modification) and the MDC-1 coated Inconel 713C specimens, the coating increases time-of-exposure by a factor of three. Assuming that this ratio would apply to the more resistant superalloys, comparable to Udimet 500, an exposure time of over 200 hours may be required for some materials.

TABLE 8

REGRESSION EQUATIONS FOR WEIGHT-LOSS VS TIME

Regression Equation (a)	S.E.E.	R^2
Inconel 713C Before Combustor Modification		
$\text{Log } Y = 0.94844448 + 0.03783375\ T - 0.00028621\ T^2$	0.231835	0.60
Inconel 713C After Combustor Modification		
$\text{Log } Y = 0.74124979 + 0.07199467\ T - 0.00083225\ T^2$	0.102269	0.92
Misco MDC-1 Coated Inconel 713C		
$\text{Log } Y = 0.65460615 + 0.02282596\ T - 0.00006114\ T^2$	0.145423	0.84
IN-100, ASTM		
$\text{Log } Y = 1.76947536 + 0.03455002\ T - 0.00037387\ T^2$	0.0187488	0.99
Inconel 713C, ASTM		
$\text{Log } Y = 1.7222542 + 0.0002783\ T^2$	0.177044	0.61
Udimet 700, ASTM		
$\text{Log } Y = 1.25857825 + 0.03038993\ T - 0.00018187\ T^2$	0.042714	0.99
Mar-M421, ASTM		
$\text{Log } Y = 1.4413978 + 0.01965855\ T - 0.00009056\ T^2$	0.044956	0.99
IN-738, ASTM		
$\text{Log } Y = 1.16757935 + 0.02420308\ T - 0.00013181\ T^2$	0.0151498	0.99
Udimet 500, ASTM		
$\text{Log } Y = 0.62962998 + 0.02395764\ T - 0.00007084\ T^2$	0.1490505	0.92

(a) Y = Weight Loss, mg/cm^2 and T = Exposure Time, hours.

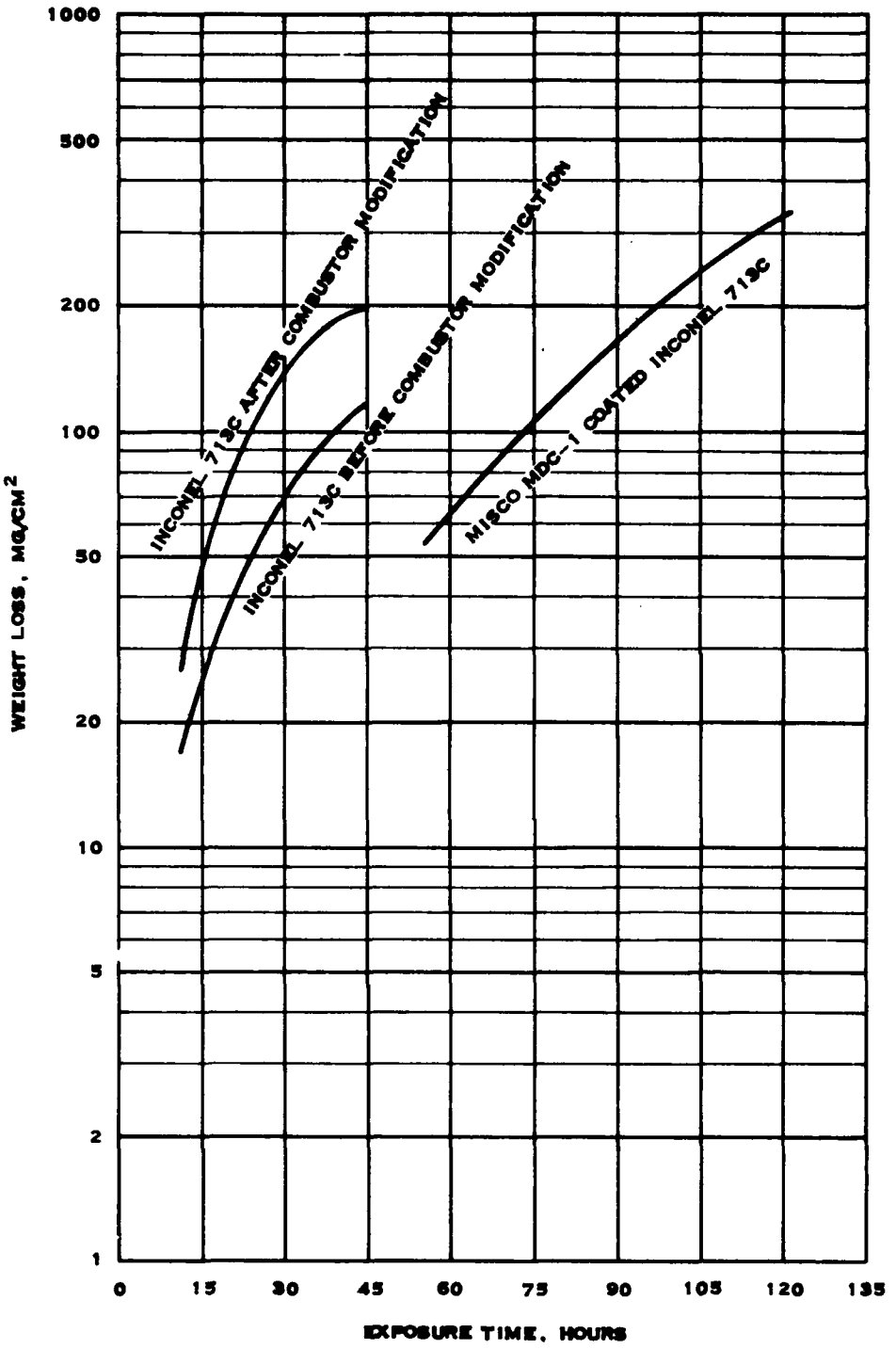


FIGURE 1
RELATIONSHIP BETWEEN WEIGHT LOSS AND EXPOSURE TIME FOR BARE AND COATED INCONEL 713C

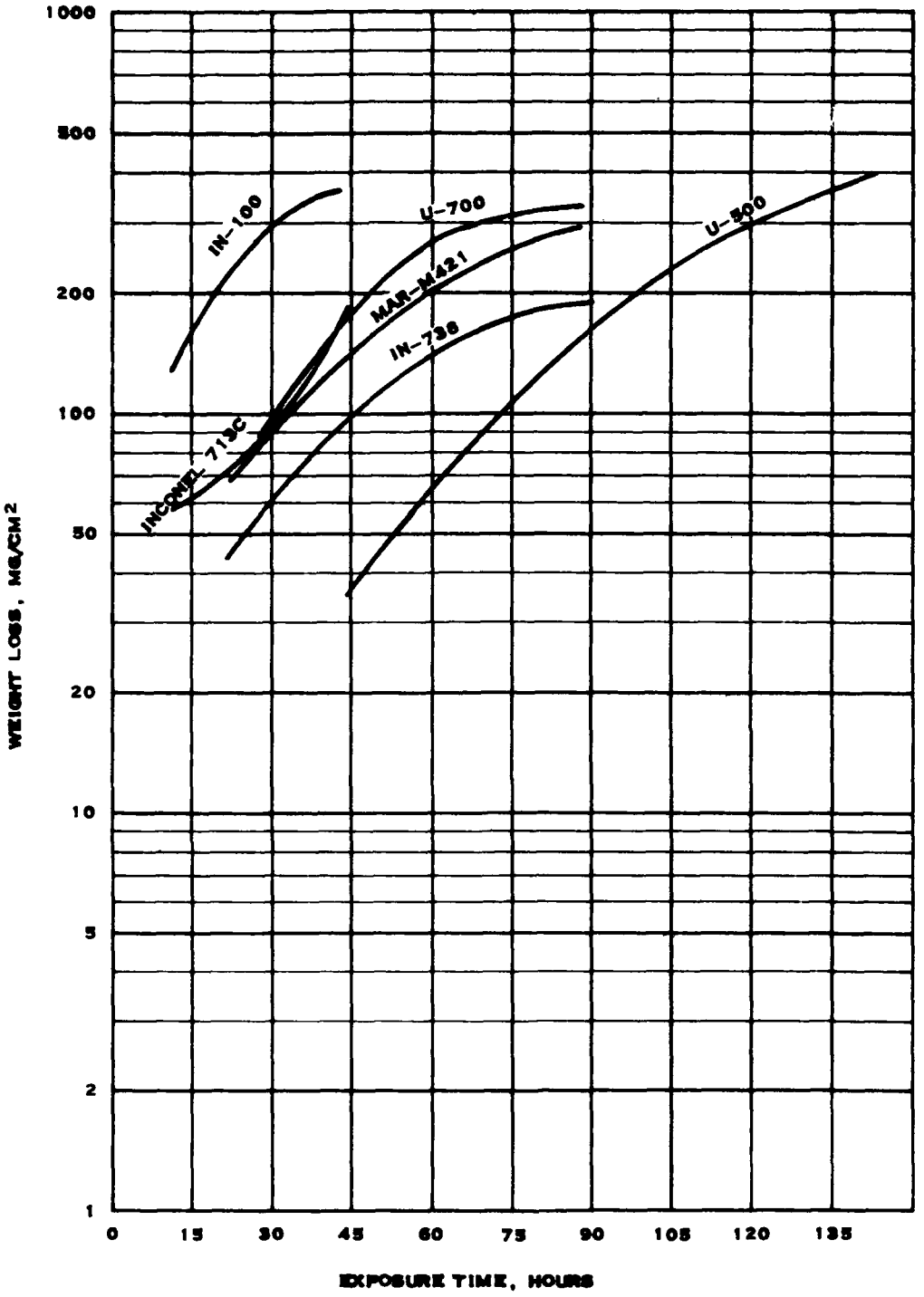


FIGURE 2
RELATIONSHIP BETWEEN WEIGHT LOSS AND EXPOSURE TIME FOR SUPERALLOYS IN ASTM PROGRAM

4.4 Penetration Data

In the ASTM "round-robin" test program of the Hot-Corrosion Task Force the requested technique for evaluating hot-corrosion attack was by metallographic measurement of "surface-loss" and "maximum-attack". This technique and terms are described in Section 10.3. of Appendix 3. Penetration data obtained on the ASTM specimens are shown in Table 9. One block of Misco MDC-1 coated Inconel 713C and one block of bare Inconel 713C specimens were examined, also, and the data are shown in Table 10. These data will permit a comparison of the relative merits of the weight-loss and metallographic techniques for evaluating hot-corrosion attack on superalloys.

A constant coefficient-of-variation was found only for the penetration data with the Misco MDC-1 coated Inconel 713C specimens; therefore, the raw data have been used in the following analyses.

The difference in maximum-attack and surface-loss is a measure of the extent of subsurface deterioration of the superalloy by hot-corrosion attack. An Analysis-of-Variance (AOV) was made of the penetration data for each of the superalloys, and the coating-alloy system, to determine any statistically-significant difference between surface-loss and maximum-attack. The error sum-of-squares for the six superalloys in the ASTM portion of the program were pooled, and the error term was used in the evaluation of the ASTM alloys. From the analyses, the difference between surface-loss and maximum-attack for Udimet 500 was the only difference indicated to be statistically significant. The AOV and the comparison for the Udimet 500 data are shown in Table 11. The indicated difference in maximum-attack and surface-loss for the Udimet 500 of only 5 mils, and the lack of a statistically-significant difference for the other materials evaluated, suggests that subsurface deterioration of these materials, at this test condition, is relatively unimportant.

Means of penetration (average of surface-loss and maximum-attack) for the six superalloys in the ASTM program after 44 hours of exposure are shown in the following multiple-comparison test. The TSMs for comparison were calculated at the 95 per cent confidence level.

Udimet 500	IN-738	Udimet 700	Mar-M421	Inconel 713C	IN-100
6	16	20	21	30	56

$TSM_1 = 5$

$TSM_2 = 4$ (Udimet 500 vs Inconel 713C)

$TSM_3 = 4$ (Udimet 500 or Inconel 713C vs Others)

If two or more means have the same line drawn underneath them, they are not statistically different. If two or more means do not have a common line underneath them, they are said to be statistically different. With one slight reversal, between Udimet 700 and Mar-M421, which is not statistically significant, these superalloys are ranked in the same order

TABLE 9

PENETRATION DATA FOR ASTM SPECIMENS

Spec. No.	Superalloy	Exposure Time hr	Surface Loss			Maximum Attack		
			Average Visual Attack, mils	Maximum Visual Attack, mils	Mean, mils	Average Visual Attack, mils	Maximum Visual Attack, mils	Mean, mils
45	IN 100	11	19 & 21 (B)	21 & 23 (B)	21	20 & 21 (B)	21 & 23 (B)	21
46	IN 100	22	37 & 38 (B)	39 & 40 (B)	38	37 & 38 (B)	39 & 40 (B)	38
47	IN 100	33	43 & 45 (B)	49 & 53 (B)	48	43 & 45 (B)	49 & 53 (B)	48
48	IN 100	44	53 & 56 (B)	57 & 58 (B)	56	53 & 56 (B)	57 & 58 (B)	56
25	Inco 713C	11	9 & 13 (B)	12 & 13 (B)	12	9 & 15 (B)	12 & 13 (B)	12
26	Inco 713C	22	20 & 22 (B)	20 & 23 (B)	21	22 & 23 (B)	23 & 23 (B)	23
27	Inco 713C	33	24 & 30 (B)	28 & 35 (B)	29	24 & 30 (B)	28 & 35 (B)	29
28	Inco 713C	44	30 & 30 (B)	32 & 32 (B)	31	30 & 32 (B)	32 & 32 (B)	32
76	Inco 713C	11	6 & 9 (B)	9 & 11 (B)	9	6 & 9 (B)	9 & 11 (B)	9
75	Inco 713C	22	7 & 11 (B)	10 & 10 (B)	10	7 & 12 (B)	10 & 10 (B)	10
74	Inco 713C	33	6 & 12 (B)	9 & 10 (B)	9	7 & 12 (B)	9 & 10 (B)	10
73	Inco 713C	44	24 & 30 (B)	29 & 34 (B)	29	24 & 30 (B)	29 & 34 (B)	29
33	Udimet 700	22	7 & 7 (B)	11 & 15 (B)	10	10 & 11 (B)	12 & 16 (B)	12
34	Udimet 700	44	18 & 18 (B)	18 & 19 (B)	20	18 & 19 (B)	23 & 25 (B)	21
35	Udimet 700	66	18 & 33 (B)	44 & 46 (B)	35	19 & 35 (B)	47 & 48 (B)	37
36	Udimet 700	88	37 & 43 (B)	45 & 47 (B)	43	38 & 45 (B)	47 & 51 (B)	45
41	Mar M421	22	10 & 10 (B)	12 & 15 (B)	12	13 & 15 (B)	17 & 18 (B)	16
42	Mar M421	44	19 & 19 (B)	21 & 22 (B)	20	21 & 21 (B)	22 & 24 (B)	22
43	Mar M421	66	32 & 34 (B)	37 & 37 (B)	35	35 & 35 (B)	37 & 37 (B)	36
44	Mar M421	88	33 & 38 (B)	38 & 46 (B)	39	33 & 38 (B)	40 & 46 (B)	39

Notes : (B) = Attack on both sides of cross-sectioned area.

TABLE 9

PENETRATION DATA FOR ASTM SPECIMENS (CONTINUED)

Spec. No.	Superalloy	Exposure Time, hr	Surface Loss			Maximum Attack		
			Average Visual Attack, mils	Maximum Visual Attack, mils	Mean, mils	Average Visual Attack, mils	Maximum Visual Attack, mils	Mean, mils
29	IN - 738	22	7 & 9 (B)	10 & 11 (B)	9	13 & 13 (B)	12 & 14 (B)	13
30	IN - 738	44	15 & 15 (B)	14 & 16 (B)	15	15 & 17 (B)	16 & 18 (B)	16
31	IN - 738	66	22 & 23 (B)	27 & 30 (B)	26	26 & 29 (B)	28 & 32 (B)	29
32	IN - 738	88	30 & 33 (B)	30 & 34 (B)	32	33 & 35 (B)	32 & 37 (B)	34
37	Udimet 500	44	0 & 2 (B)	3 & 4 (B)	2	6 & 9 (B)	(& 10 (B)	8
38	Udimet 500	77	26 & 27 (B)	27 & 27 (B)	27	28 & 29 (B)	30 & 31 (B)	30
39	Udimet 500	110	30 & 32 (B)	34 & 35 (B)	33	38 & 39 (B)	38 & 40 (B)	39
40	Udimet 500	143	62 & 66 (B)	70 & 71 (B)	67	67 & 71 (B)	74 & 77 (B)	72
80	Udimet 500	44	1 & 5 (B)	4 & 6 (B)	4	7 & ((B)	10 & 11 (B)	9
79	Udimet 500	77	11 & 12 (B)	16 & 24 (B)	16	18 & 20 (B)	20 & 27 (B)	21
78	Udimet 500	110	30 & 41 (B)	39 & 43 (B)	38	33 & 54 (B)	43 & 43 (B)	43
77	Udimet 500	143	67 & 68 (B)	69 & 70 (B)	68	71 & 72 (B)	71 & 73(B)	72

Notes: (B) = Attack on both sides of cross-sectioned area.

TABLE 10

PENETRATION DATA FOR SELECTED TEST SPECIMENS

Spec. No.	Exposure Time, hr	Surface Loss			Maximum Attack		
		Average Visual Attack, mils	Maximum Visual Attack, mils	Mean, mils	Average Visual Attack, mils	Maximum Visual Attack, mils	Mean, mils
			MDC-1 Coated Inconel 713C				
1	55	3 & 4 (B)	8 & 12 (B)	7	5 & 6 (B)	10 & 14 (B)	9
2	77	14 & 15 (B)	24 & 27 (B)	20	17 & 18 (B)	26 & 29 (B)	22
3	99	42 & 49 (B)	43 & 49 (B)	46	42 & 51 (B)	45 & 51 (B)	47
4	121	27 & 28 (B)	61 & 74 (B)	48	28 & 29 (B)	62 & 76 (B)	49
170	55	1 & 7 (B)	15 & 16 (B)	10	2 & 7 (B)	16 & 17 (B)	10
166	77	9 & 17 (B)	18 & 32 (B)	19	10 & 20 (B)	19 & 35 (B)	21
150	99	17 & 25 (B)	54 & 70 (B)	42	19 & 27 (B)	56 & 72 (B)	44
146	121	95 & 107(B)	105 & 109(B)	104	97 & 109(B)	106 & 111(B)	106
			Inconel 713C				
17	11	1 & 5 (B)	4 & 5 (B)	4	1 & 8 (B)	4 & 8 (B)	5
18	22	7 & 13 (B)	12 & 18 (B)	12	9 & 14 (B)	13 & 20 (B)	14
19	33	12 & 20 (B)	18 & 24 (B)	18	13 & 22 (B)	20 & 24 (B)	20
20	44	21 & 23 (B)	28 & 30 (B)	26	22 & 24 (B)	29 & 32 (B)	27
52	11	0 & 7 (B)	2 & 5 (B)	4	3 & 7 (B)	3 & 9 (B)	6
51	22	0 & 3 (B)	4 & 4 (B)	3	3 & 5 (B)	3 & 6 (B)	4
50	33	4 & 7 (B)	5 & 6 (B)	6	6 & 9 (B)	6 & 9 (B)	8
49	44	15 & 18 (B)	11 & 23 (B)	17	17 & 20 (B)	13 & 25 (B)	19

Notes: (B) = Attack on both sides of cross-sectioned area.

TABLE 11

ANALYSIS OF VARIANCE OF PENETRATION DATA
(Udimet 500)

Source of Variation	Degrees of Freedom	Sum of Squares	Mean Square
Total	63	36843.00	
Type of Attack (T)	1	380.25	380.25 *
Time (H)	3	35315.62	11771.88 *
Severity of Attack (S)	1	150.06	150.06 *
T x H	3	9.12	3.04
T x S	1	10.56	10.56
H x S	3	10.56	3.52
T x H x S	3	11.81	3.94
Error	48	955.02	19.90
Pooled Error (a)	160	3982.47	24.89

Asterisk (*) indicates a significant effect at 95 per cent confidence level.

(a) Pooled error for the six alloys in the ASTM portion of the program.

Comparison of Penetration Data
(TSM = 2.0)

Maximum Attack, mils minus Surface Loss, mils
37.0 - 32.0 = 5.0 mils

of resistance to hot-corrosion attack by the penetration data as was shown previously by the weight-loss data.

A comparison of weight-loss and average penetration was made using data for each specimen and it was found that weight-loss correlates linearly with penetration data. The estimated linear relationship describing this correlation is:

$$P = 0.2101957 + 0.16627034\ W,$$

where P = Penetration, mils, and

W = Weight-Loss, mg/cm^2.

The data are shown graphically in Figure 3, and the line on the figure represents the calculated relationship. This linear relationship indicates that either weight-loss or penetration could be used to evaluate hot-corrosion attack for these superalloys at this condition of exposure.

4.5. Metallography

Two different types of measurement, loss-in-weight and depth-of-penetration, were used to evaluate the extent of metal damage during this preliminary test with the Turbine Simulator. The results of these measurements have been discussed in the preceding sections of this report. Both methods require some metallographic examination of specimens, but they differ appreciably in the extent of this requirement. Both methods suffer from inherent weaknesses.

The weight-loss method assumes a uniform attack over the surface of the specimen, with the absence of subsurface deterioration by deep sulfide penetration or intergranular oxidation. Also, it requires complete removal of corrosion products from the specimen without further damage to its surface. Both of these potential faults in the weight-loss method can be aggravated by the application of coatings to the superalloy specimens, and such materials will be employed extensively in our proposed investigation to determine the effect of very-low concentrations of sulfur in fuel on hot corrosion.

The penetration method avoids the weaknesses in the weight-loss method by using metallographic cross-sections; however, it assumes that the location of maximum subsurface deterioration, for taking the cross-section, can be identified by a visual inspection of the specimen's exterior. Also, it presumes that precise measurements of penetration can be made with a reasonable expenditure of time and effort. The demand for metallography by the penetration method can be quite formidable in a program having the productivity of specimens, 768, projected for our proposed investigation to determine the effect of very-low concentrations of sulfur in fuel on hot corrosion.

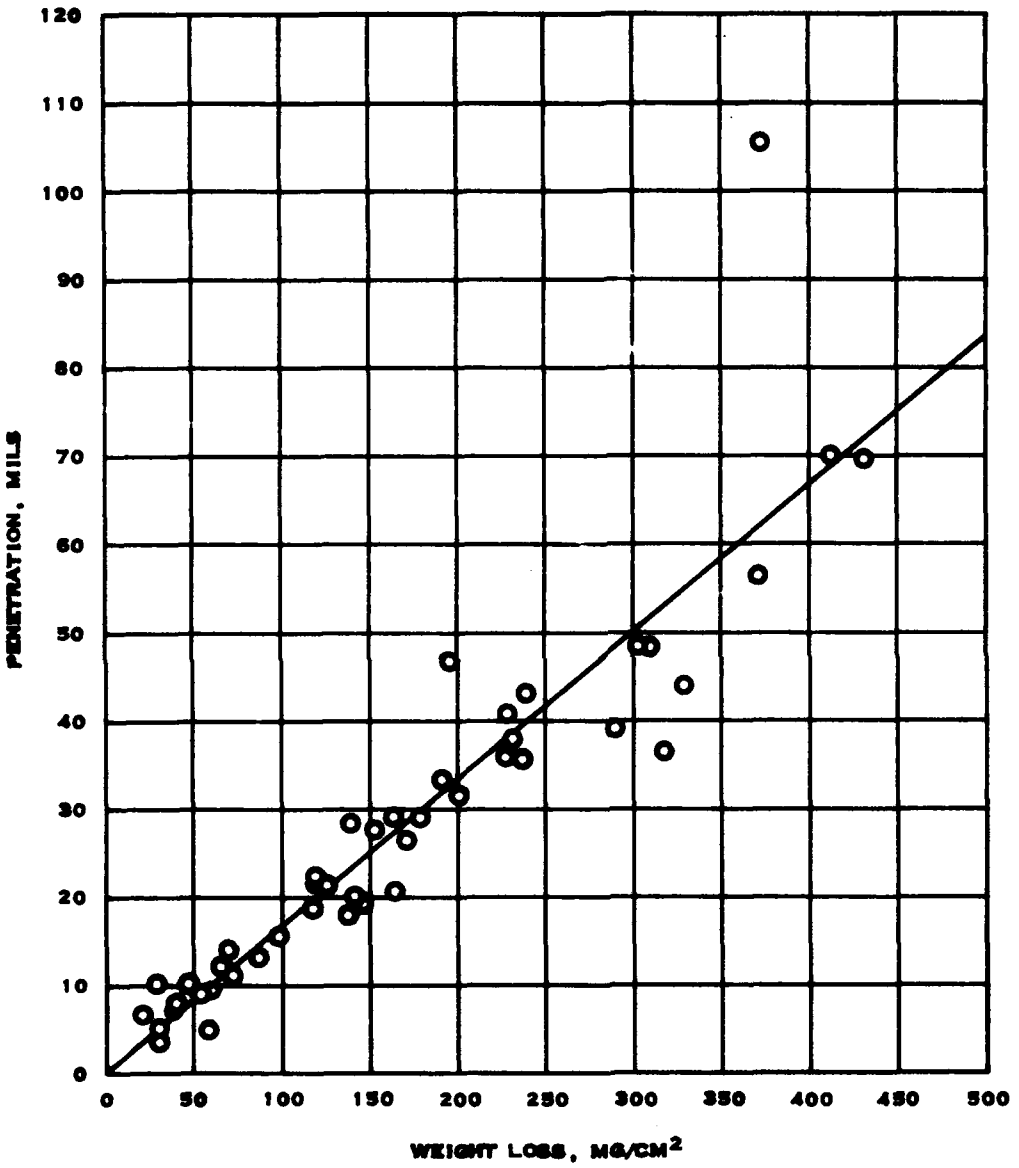


FIGURE 3
RELATIONSHIP BETWEEN PENETRATION AND WEIGHT LOSS

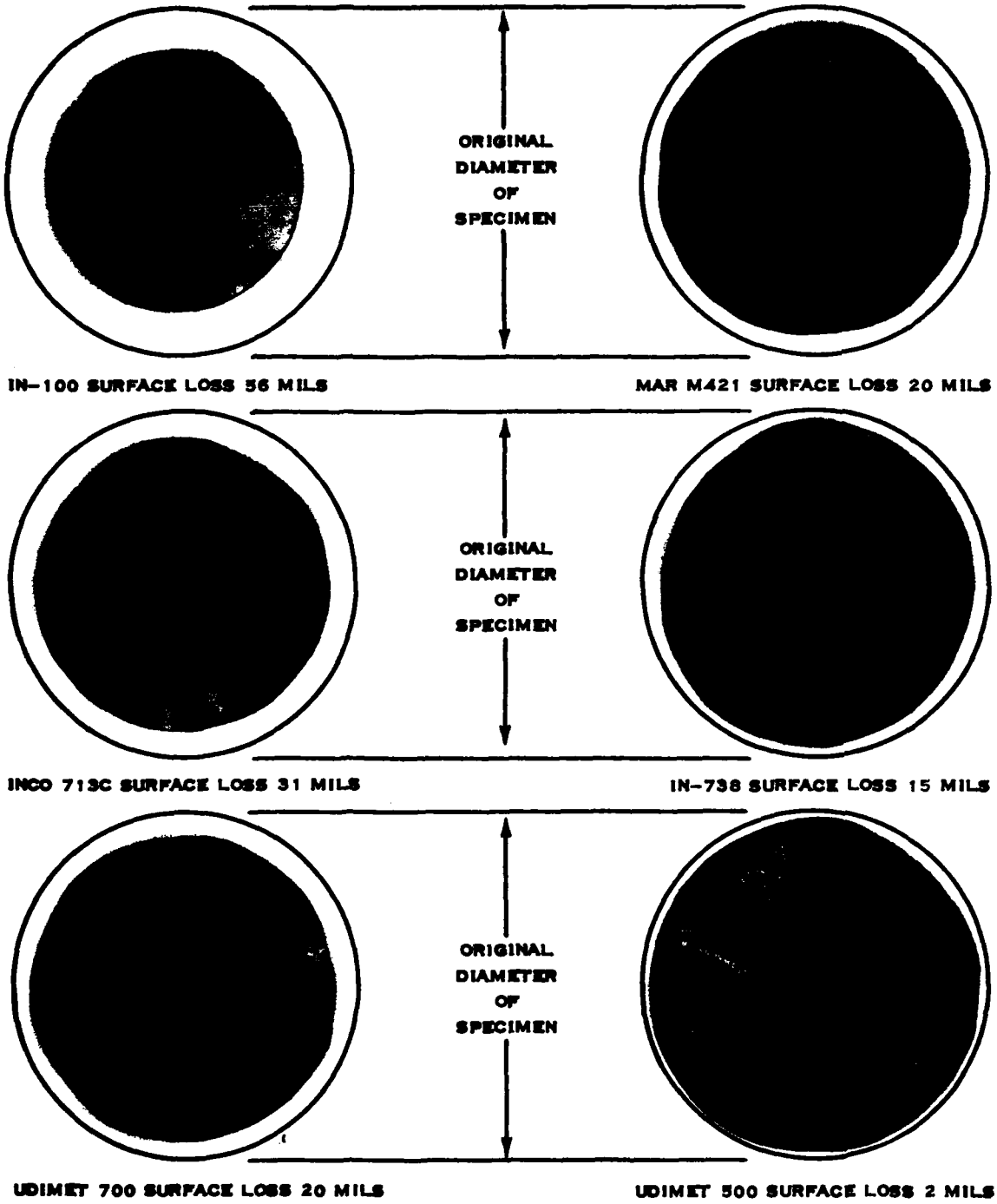
Choice of Weight-Loss Method - Since measurements of weight-loss are simpler, faster, more economical, and less subjective (selection of locations for cross-sectioning specimens), they should be used to evaluate the extent of metal damage whenever feasible. However, the validity of using weight-loss data for evaluation of hot-corrosion attack should be established by showing the absence of any unusual amount of subsurface deterioration. This can be done by metallographic examination of a few representative specimens of each superalloy, or coating-superalloy system, selected from over the range of exposure conditions.

Mode of Attack - Previous studies (1,2) have shown hot corrosion to advance on a broad front without deep-intercrystalline penetration by sulfides or oxides. The attack is led by penetration of randomly dispersed, light-grey, globules of metallic sulfides. Their formation is associated with changes in the surface composition of the alloy, which is characterized by chromium depletion. Rapid oxidation of the weakened layer of alloy follows. The depth of subsurface deterioration ranges from less than one to a maximum of about five mils. As in the current investigation, a linear relationship has been found between measurements of weight-loss and corrosion penetration. In the past, these observations have served to justify our use of specimen weight-loss data as a valid measurement of metal damage.

Uniformity of Attack - As noted in the preceding section of this report, metallographic measurements of corrosion penetration were made on all 32 specimens from the ASTM "round-robin" program. The general appearance of metallographic cross-sections of the six superalloys are shown in Figure 4. These cross-sections were made at the zone of maximum-visual attack on specimens that had been exposed for 44 hours, which was the only common time for all the superalloys. While this figure does serve to illustrate the differences found between these superalloys in their resistance to hot-corrosion attack at our conditions of exposure, its primary purpose is to show that the depth-of-penetration was remarkably uniform all-around these specimens exposed in the Turbine Simulator.

Detailed Examinations - The general mode and intensity of corrosion attack, sustained by specimens exposed in the Turbine Simulator at operating conditions used during this investigation, was appraised while making metallographic measurements of the depth-of-penetration. This inspection included Misco MDC-1 coated Inconel 713C, in addition to the six superalloys from the ASTM "round-robin" program. From these, typical specimens of Udimet 500, Inconel 713C, and Misco MDC-1 coated Inconel 713C were selected for a more detailed metallographic examination. Photomicrographs were made to show the condition of the corrosion interface on these specimens. Emphasis was placed on illustrating the extent of subsurface deterioration by alloy depletion, or intercrystalline penetration by corrosion products.

Udimet 500 was the only superalloy in the ASTM "round-robin" program which evidenced a statistically-significant difference between surface-loss and maximum-attack, as noted in the preceding section of this



METALLOGRAPHIC CROSS-SECTION OF SPECIMENS FROM 2000 F CYCLIC TEST IN PHILLIPS TURBINE SIMULATOR WITH 1.0 PPM SEA SALT IN AIR AND 0.040 WT % SULFUR IN FUEL. ELECTRO-CLEANED. MARBLE'S REAGENT ETCHED. 9X MAGNIFICATION.

FIGURE 4
HOT CORROSION OF SUPERALLOYS AFTER 44 HOURS EXPOSURE

report. This subsurface deterioration, which reached a maximum depth of about five mils, can be seen in Figure 5. It should be remembered that the heavy surface scale has been removed from the test specimen by electro-cleaning. While the specimen after maximum exposure (143 hours) is shown, this surface attack appeared to be the same as that on the other Udimet 500 specimens with shorter exposure times (44, 77, and 110 hours). Micro-features of the accelerated surface oxidation, which are characteristic of hot corrosion, as already described, are shown in Figure 6.

Inconel 713C is less resistant to hot corrosion than Udimet 500, and the extent of subsurface deterioration on the Inconel 713C specimens was only about one mil, as illustrated in Figure 7. However, the mode of attack did not change, as shown in Figure 8. It is assumed that this results from more-rapid oxidation of the depleted surface layer with lower-chromium-content superalloys. A similar increase in the intensity of attack was observed with the other superalloys in the ASTM "round-robin" program; i.e., IN-100, Udimet 700, Mar-M421, and IN-738.

Misco MDC-1 coated Inconel 713C was the only coating-superalloy system evaluated during this investigation. This aluminum-rich coating had a total depth of approximately 2 mils, which was divided about equally between an outer-layer with non-metallic dispersions and a diffusion-layer. The coating was very resistant to hot corrosion, but coating breakdown was observed after 55 hours exposure in the Turbine Simulator at operating conditions used during this investigation. At this time of incipient failure, coating breakdown was not completely uniform over the specimens. This is illustrated by the cross-section, made at the location of maximum attack on a specimen, shown in Figure 9. The outer-layer was destroyed by oxidation and aluminum depletion, as indicated in Figure 10; however, it is of interest to note that the attack on the diffusion-layer was led by sulfide penetration. Once the coating was penetrated, the mode and intensity of attack observed was similar to that on the bare Inconel 713C.

A cross-section made near the cooler base of a specimen, where the coating had survived after 121 hours exposure, is shown in Figure 11. Penetration of the coating is similar to that after 55 hours exposure, shown in Figures 9 and 10; however, it is of interest to note the heavier oxidation of the coating remnant after 121 hours exposure.

In general, the extent of subsurface attack on superalloys and coatings was limited to only shallow penetration by corrosion products, despite catastrophic rates of metal loss, during this preliminary test with the Turbine Simulator. This serves to justify our use of metal-loss data as a valid measurement of the damage which these materials suffer under these conditions of exposure.

4.6. Test Severity

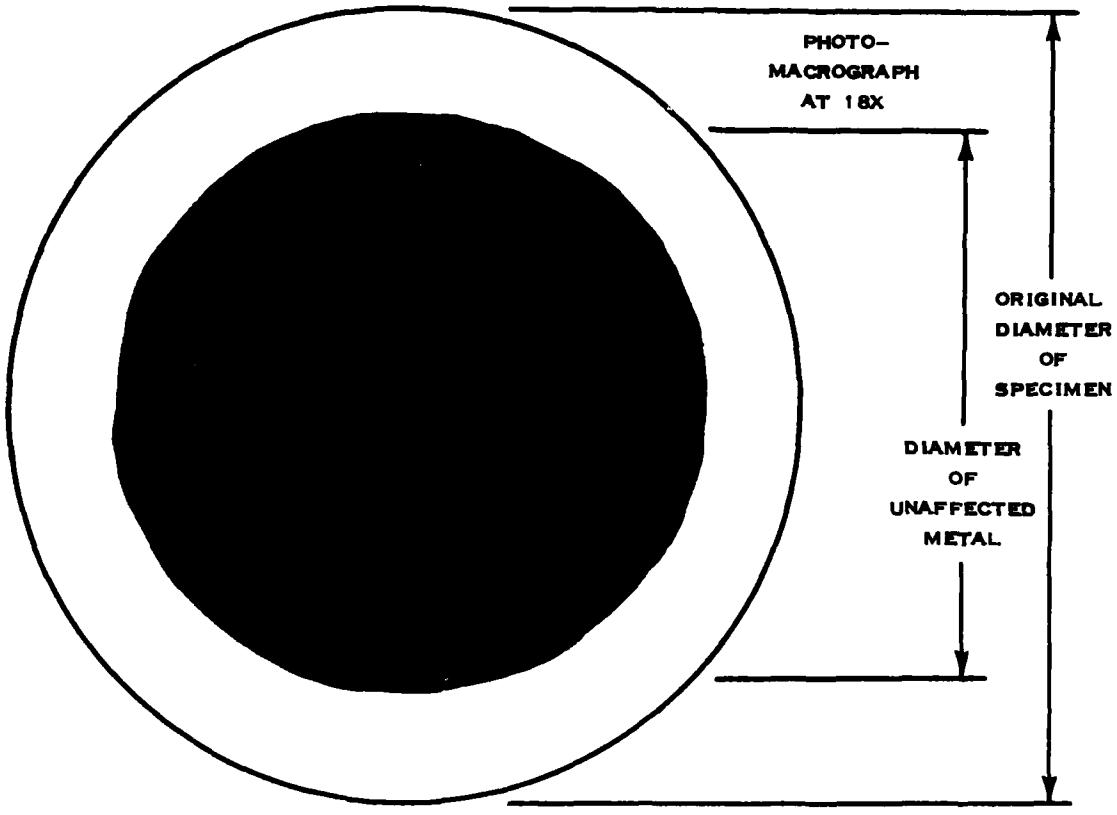
The severity of hot-corrosion attack on the coating-alloy system exposed during this investigation was approximately six times greater than

that experienced during the initial trial of the Turbine Simulator; i.e., the weight-loss for Misco MDC-1 coated Inconel 713C specimens after 55 hours of exposure in the current program was as great as the weight-loss after 350 hours of exposure during the previous program (4). This increase in test severity was obtained by changing temperature conditions. The specimens were exposed to gas at higher temperatures to accelerate aluminum depletion and oxidation of the coating, and to rapid-temperature cycling to promote spalling of the protective scale.

Photomacrographs (2X magnification) of representative specimens from the current program are presented in Figure 12. Bare Inconel 713C specimens, exposed for periods of up to 44 hours, show a relatively uniform attack over their entire surface and a gradual decrease in diameter with exposure time. A similar, uniform, attack was observed on specimens of the other superalloys in the ASTM "round-robin" program; but, the superalloys differed in their resistance to hot corrosion, as previously illustrated by Figure 4. Also, such uniform attack over the entire surface was observed on bare Inconel 713C specimens in the previous program (4), where the exposure temperature was lower and was not rapidly cycled.

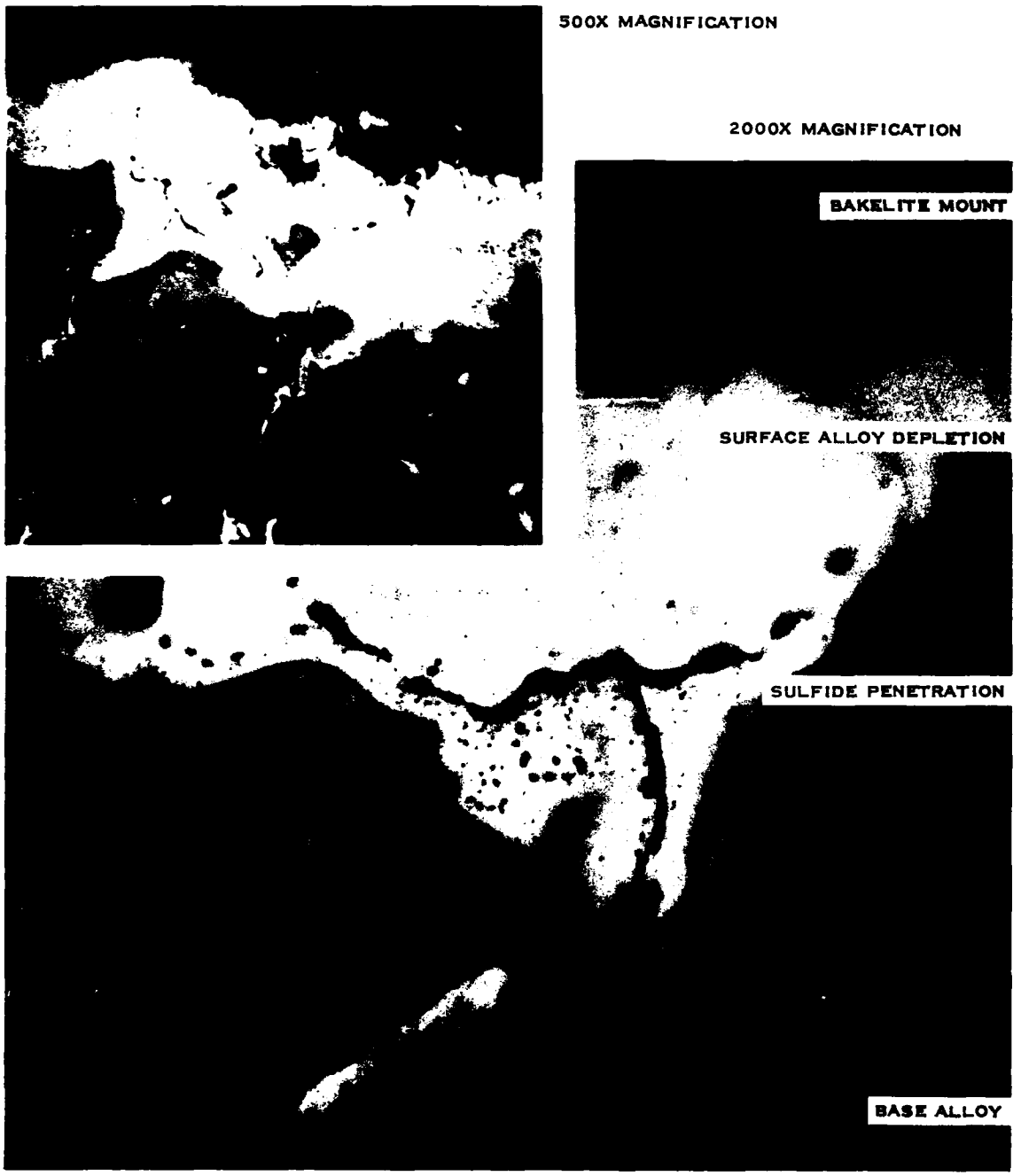
In contrast, the attack on Misco MDC-1 coated Inconel 713C exhibited appreciable sensitivity to specimen temperature during this preliminary program, as illustrated in Figure 12. The aluminum-diffusion coating failed progressively along the length of the specimens, starting after 55 hours at the tips where the temperature is highest and moving to their base after 121 hours exposure. Such temperature sensitivity was not found during the previous program (4), where the coating failed at random over the length of the specimen.

The temperature sensitivity of Misco MDC-1 coated Inconel 713C to hot-corrosion attack during this preliminary test with the Turbine Simulator raised a question as to whether the maximum temperature of 2000F in its operating cycle is excessively severe for coating-superalloy systems. The answer is pertinent to our proposed investigation, where the effect of very-low concentrations of sulfur in fuel on hot corrosion will be studied by evaluating the durability of a wide variety of coating-superalloy systems. A reduction in the maximum temperature, to 1900F or less, to obtain a milder environment, would significantly increase the expenditure of time, manpower, and materials for the proposed investigation. After careful consideration, it was concluded that the temperature sensitivity of this particular coating-superalloy system did not indicate an abnormal mode-of-failure, that it did not warrant a reduction in the severity of the operating conditions used for the Turbine Simulator, and that this simulated-environmental test now represents a satisfactory compromise for conducting our proposed investigation.



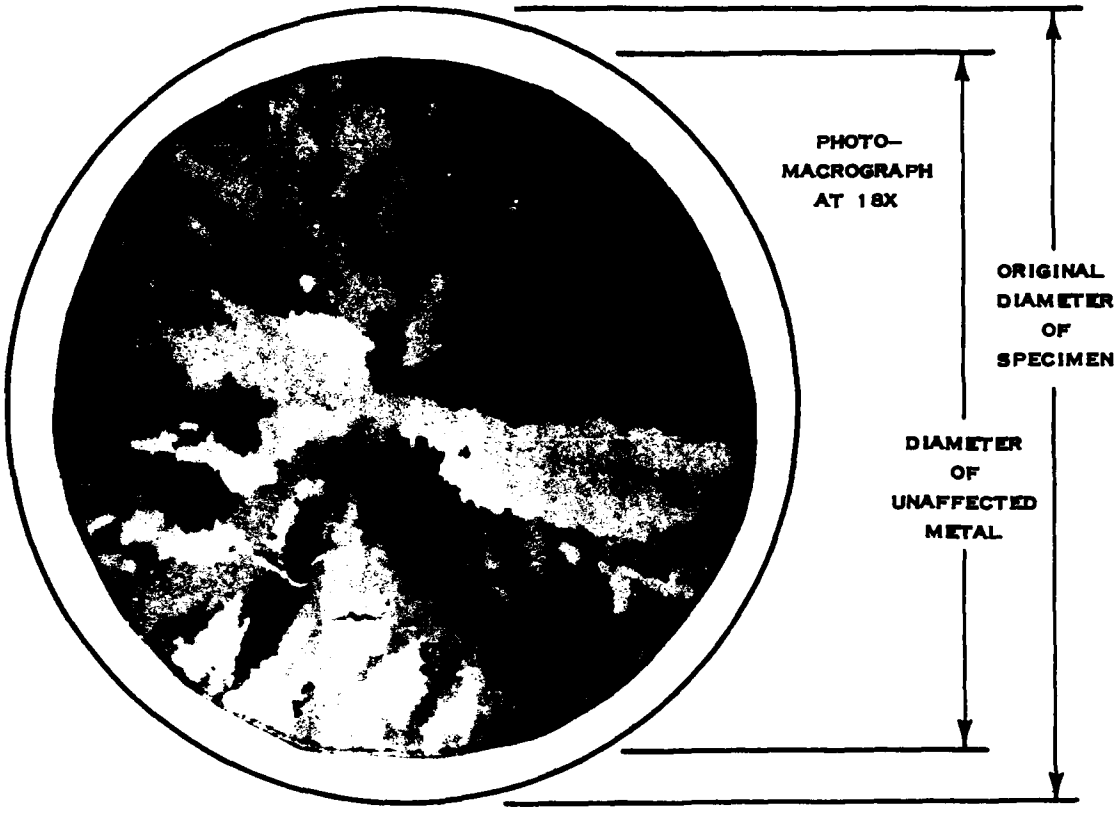
METALLOGRAPHIC CROSS-SECTION OF SPECIMEN FROM 2000 F CYCLIC TEST IN PHILLIPS TURBINE SIMULATOR WITH 1.0 PPM SEA SALT IN AIR AND 0.040 WT % SULFUR IN FUEL. ELECTRO-CLEANED. 2% SULFURIC ACID-ELECTROLYTIC ETCHED. 200X MAGNIFICATION.

FIGURE 5
HOT CORROSION OF UDIMET 500 SPECIMEN AFTER 143 HOURS



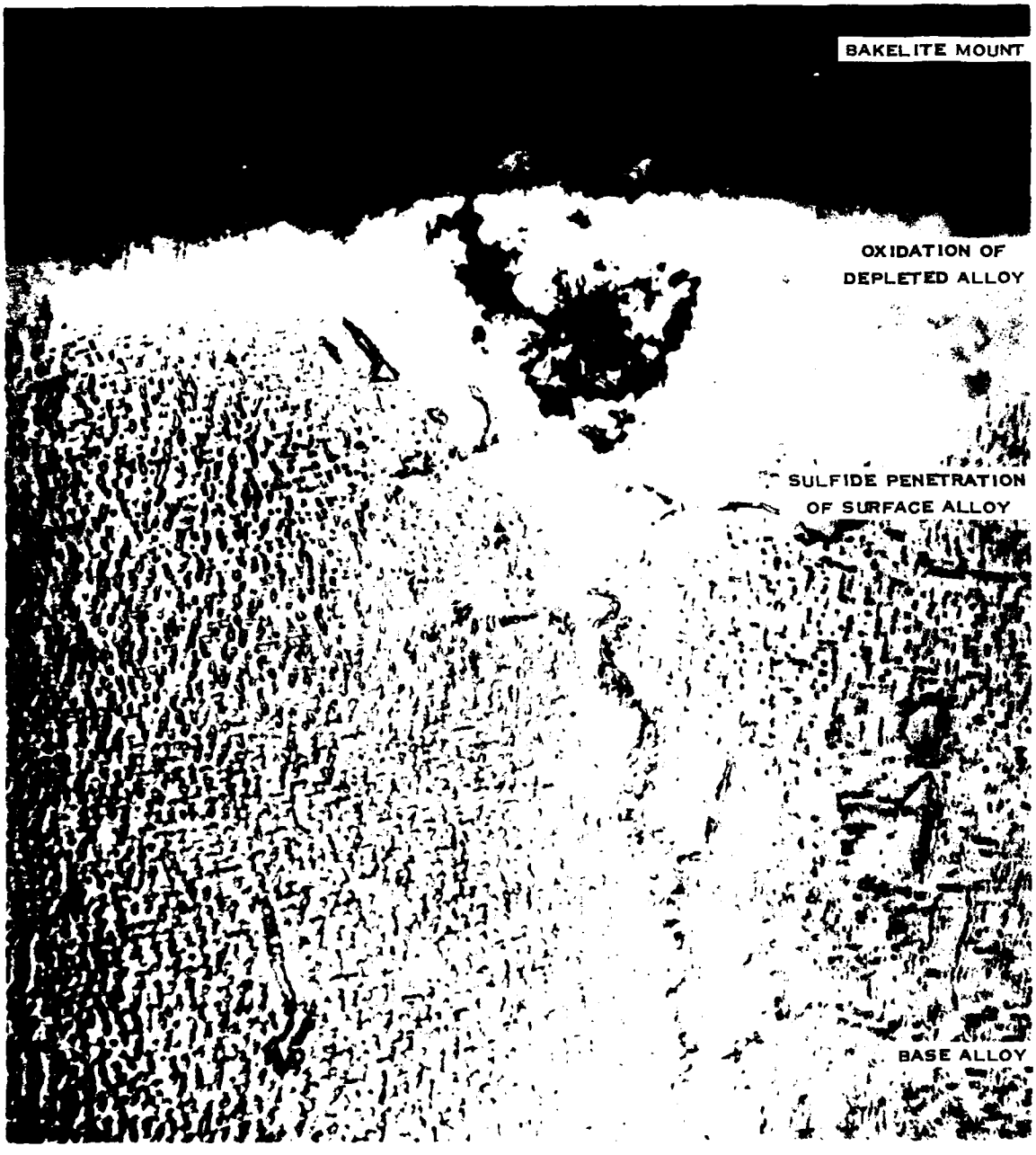
METALLOGRAPHIC CROSS-SECTION OF SPECIMEN FROM 2000 F CYCLIC TEST IN PHILLIPS TURBINE SIMULATOR WITH 1.0 PPM SEA SALT IN AIR AND 0.040 WT % SULFUR IN FUEL. ELECTRO-CLEANED. 2% SULFURIC ACID-ELECTROLYTIC ETCHED.

FIGURE 6
ACCELERATED SURFACE OXIDATION OF UDIMET 500
SPECIMEN AFTER 143 HOURS



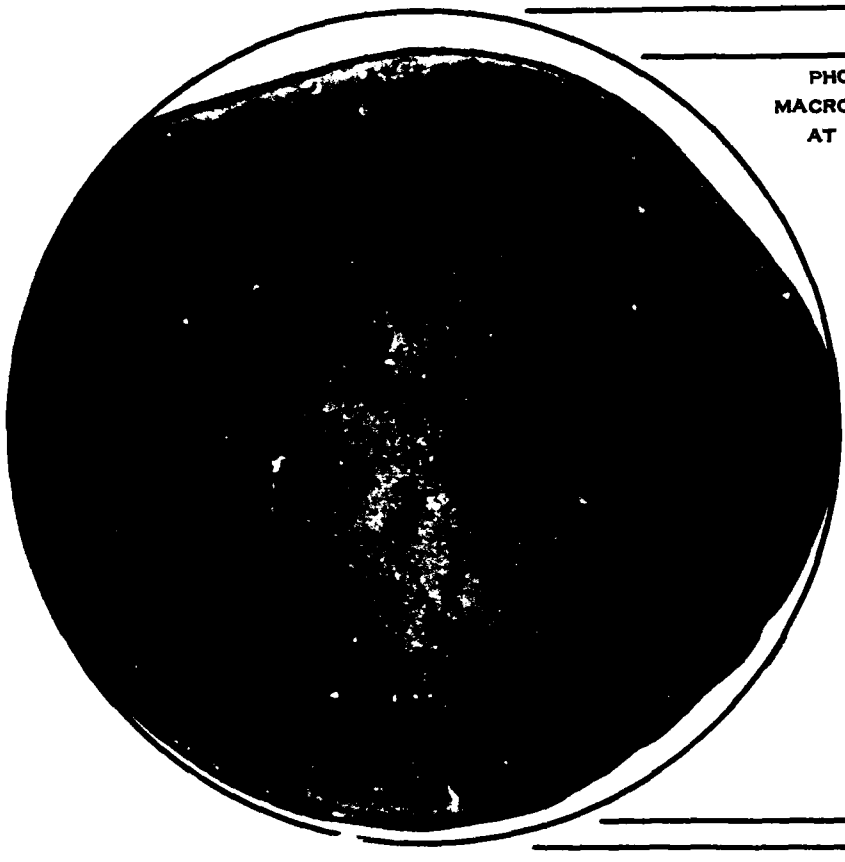
METALLOGRAPHIC CROSS-SECTION OF SPECIMEN FROM 2000 F CYCLIC TEST IN PHILLIPS TURBINE SIMULATOR WITH 1.0 PPM SEA SALT IN AIR AND 0.040 WT % SULFUR IN FUEL. ELECTRO-CLEANED. MARBLE'S REAGENT ETCHED. 200X MAGNIFICATION.

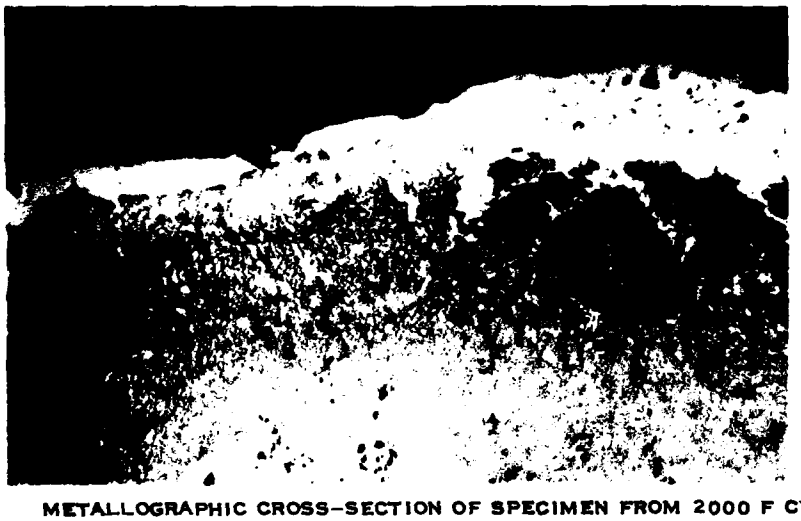
FIGURE 7
HOT CORROSION OF INCONEL 713C SPECIMEN AFTER 44 HOURS



METALLOGRAPHIC CROSS-SECTION OF SPECIMEN FROM 2000 F CYCLIC TEST IN PHILLIPS TURBINE SIMULATOR WITH 1.0 PPM SEA SALT IN AIR AND 0.040 WT % SULFUR IN FUEL. ELECTRO-CLEANED. MARBLE'S REAGENT ETCHED. 2000X MAGNIFICATION.

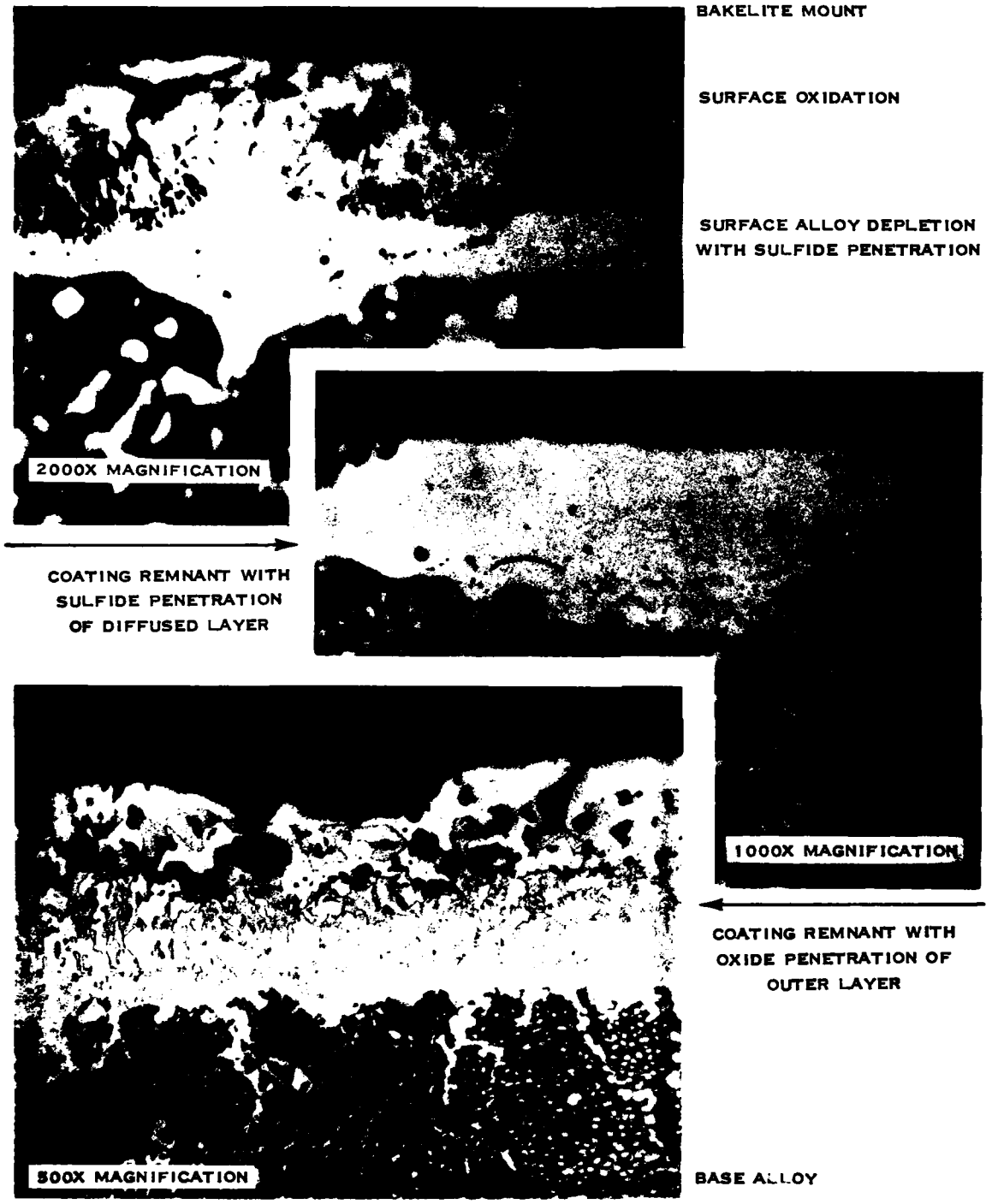
FIGURE 8
ACCELERATED SURFACE OXIDATION OF
INCONEL 713C SPECIMEN AFTER 44 HOURS





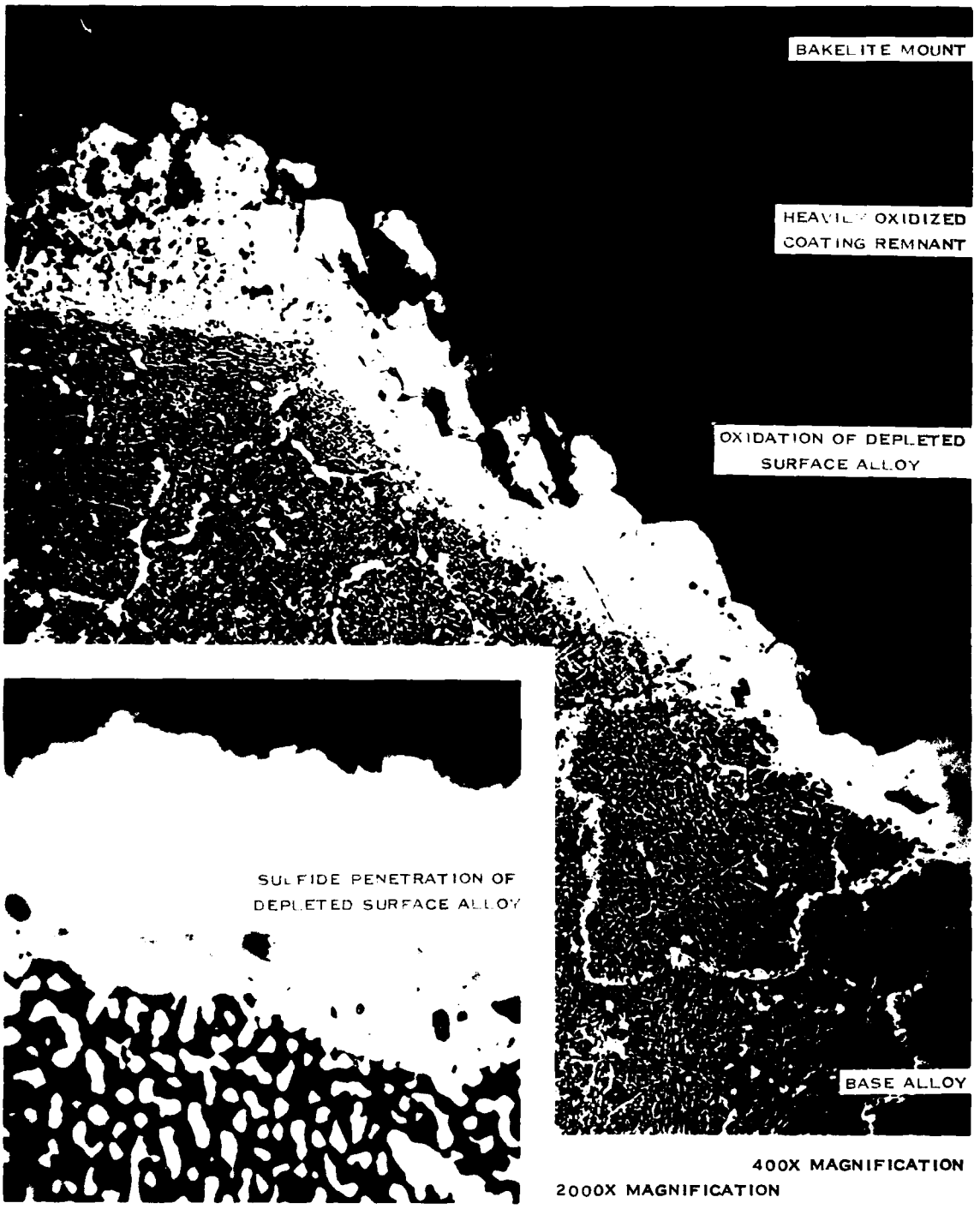
METALLOGRAPHIC CROSS-SECTION OF SPECIMEN FROM 2000 F CYCLIC TEST IN PHILLIPS TURBINE SIMULATOR WITH 1.0 PPM SEA SALT IN AIR AND 0.040 WT % SULFUR IN FUEL. ELECTRO-CLEANED. 2% SULFURIC ACID-ELECTROLYTIC ETCHED. 200X MAGNIFICATION.

FIGURE 9
HOT CORROSION OF MISCO MDC-1 COATED
INCONEL 713C SPECIMEN AFTER 55 HOURS



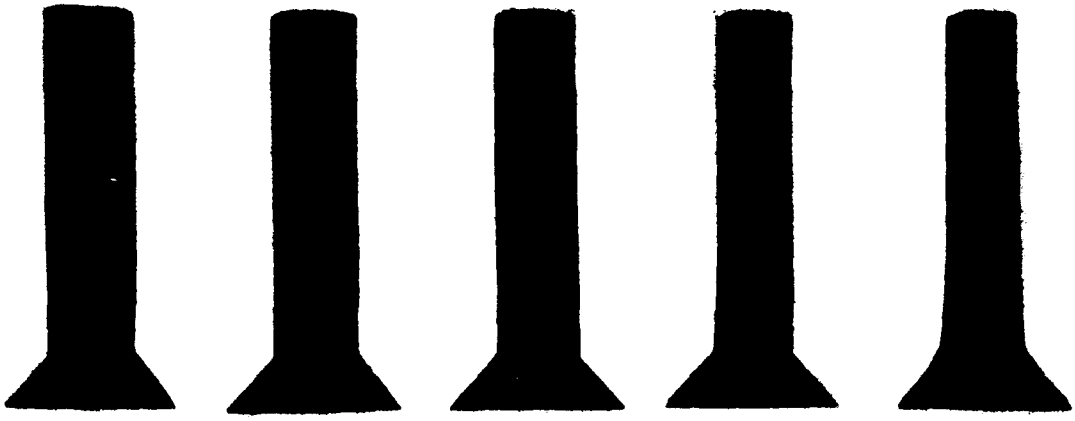
METALLOGRAPHIC CROSS-SECTION OF SPECIMEN FROM 2000 F CYCLIC TEST IN PHILLIPS TURBINE SIMULATOR WITH 1.0 PPM SEA SALT IN AIR AND 0.040 WT % SULFUR IN FUEL. ELECTRO-CLEANED. 2% SULFURIC ACID-ELECTROLYTIC ETCHED.

FIGURE 10
PENETRATION OF MISCO MDC-1 COATING ON INCONEL 713C SPECIMEN AFTER 55 HOURS



METALLOGRAPHIC CROSS-SECTION OF SPECIMEN FROM 2000 F CYCLIC TEST IN PHILLIPS TURBINE SIMULATOR WITH 1.0 PPM SEA SALT IN AIR AND 0.040 WT % SULFUR IN FUEL. ELECTRO-CLEANED. 2% SULFURIC ACID-ELECTROLYTIC ETCHED.

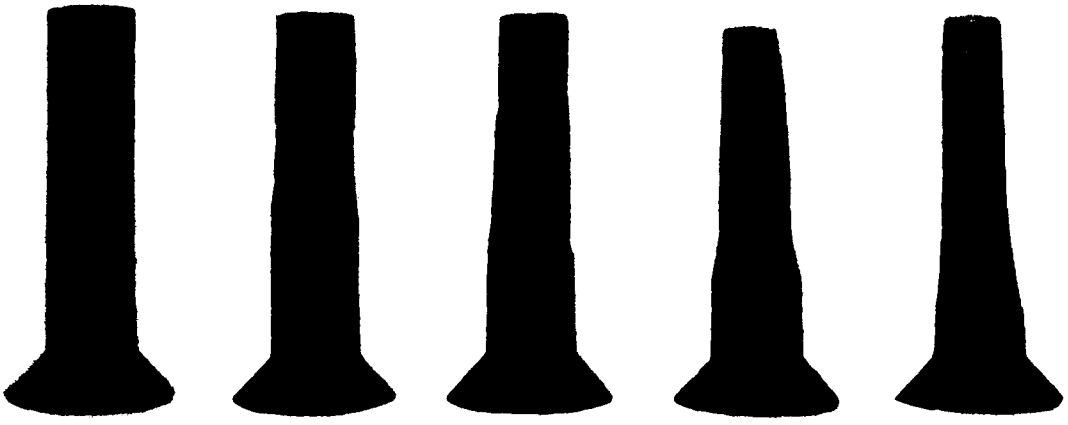
FIGURE 11
PENETRATION OF MISCO MDC-1 COATING ON INCONEL 713C
SPECIMEN AFTER 121 HOURS



0 11 22 33 44

EXPOSURE TIME, HOURS

BARE INCONEL 713C



0 55 77 99 121

EXPOSURE TIME, HOURS

MISCO MDC-1 COATED INCONEL 713C

PHOTOMACROGRAPHS AT 2X MAGNIFICATION OF ELECTRO-CLEANED SURFACES FROM 2000F CYCLIC TEST WITH 1 PPM SEA SALT IN AIR AND 0.04 WT % SULFUR IN FUEL

FIGURE 12
REPRESENTATIVE SPECIMENS FROM PHILLIPS TURBINE SIMULATOR

5. FUTURE WORK

The effect of sulfur in fuel on hot corrosion was found to vary among the superalloys used in previous programs (1); i.e., a reduction in fuel sulfur had no effect on hot corrosion with some superalloys, and increased hot corrosion with two superalloys under some conditions. Programs with three coating-alloy systems (2) at 2000F conditions showed that a reduction in fuel sulfur from the specification maximum of 0.40 to 0.040 weight per cent increased, decreased or had no effect on hot corrosion; however, a reduction in sulfur to a very low level (0.0040 weight per cent) was found to decrease hot corrosion.

In previous programs (1) it was found that the effect of sulfur in fuel on hot corrosion varied with temperature. Statistically significant decreases in hot corrosion were found with reductions in fuel sulfur at temperatures of 1600F and below with superalloys that were unaffected by changes in sulfur content at higher temperatures. Evaluations with coated superalloys have been confined to 2000F exposure temperatures.

To complete the investigation of the effect of sulfur in fuel on hot corrosion of bare and coated superalloys in a marine environment, additional superalloys and coatings should be evaluated over a range of temperatures and emphasis should be given to very low concentrations of sulfur in fuel. Plans for a test program to complete this investigation are presented in the following paragraphs.

5.1. Coating-Alloy Systems

In consultation with U. S. Navy personnel, engine builders and alloy suppliers, a group of eight nickel-base and four cobalt-base superalloys have been selected for inclusion in our program to evaluate the effect of sulfur in fuel on hot corrosion. As shown in Table 12, these alloys cover a broad range in composition. Test specimens of these twelve superalloys have been prepared as investment castings. Specimens of each of the twelve superalloys are now being coated with Misco MDC-9 coating and specimens of the eight nickel based superalloys are being coated with Misco MDC-1 coating. The test program will include the evaluation of each of the bare and coated superalloys at each level of sulfur in fuel.

5.2. Sulfur in Fuel

Previous investigations have indicated that a reduction in concentration of sulfur in fuel from the specification maximum of 0.40 to 0.04 weight per cent would not reduce hot corrosion significantly. In the investigation to be conducted, we have selected three levels of sulfur (0.040, 0.0040, and 0.00040 or less weight per cent) which represent approximately the median of current production, approximately the minimum of current production and an essentially sulfur free fuel that could be met with modern refining technology.

TABLE 12

SUPERALLOYS FOR HOT-CORROSION STUDIES IN PHILLIPS ROTATING-SPECIMEN RIG

Nominal Chemical Composition, wt %

Alloying Elements	Nickel-Base Alloys								Cobalt-Base Alloys			
	Udimet 710	IN-738X	Udimet 700	Inco 713C	IN-100	MAR-M200	MAR-M246	B-1900	X-40	MAR-M509	MAR-M302	WI-52
Ni	Bal.	Bal.	Bal.	Bal.	Bal.	Bal.	Bal.	Bal.	10.5	10.0	...	...
Co	15.0	8.5	18.5	...	15.0	10.0	10.0	10.0	Bal.	Bal.	Bal.	Bal.
Cr	18.0	16.0	15.0	12.5	10.0	9.0	9.0	8.0	25.5	21.5	21.5	21.0
Al	2.5	3.4	4.3	6.1	5.5	5.0	5.5	6.0	...	...	...	...
Ti	5.0	3.4	3.5	0.8	4.7	2.0	1.5	1.0	...	0.2	...	...
Mo	3.0	1.75	5.2	4.2	3.0	...	2.5	6.0	...	...	...	...
Ta	...	1.75	...	...	...	...	1.5	4.0	...	3.5	9.0	...
W	1.5	2.6	...	...	...	12.5	10.0	...	7.5	7.0	10.0	11.0
Zr	...	0.10	...	0.10	0.06	0.05	0.05	0.10	...	0.5	0.20	...
V	...	...	...	...	1.0	...	...	...	...	...	...	...
C	0.07	0.17	0.08	0.12	0.18	0.15	0.15	0.10	0.50	0.60	0.85	0.45
B	0.02	0.01	0.03	0.012	0.014	0.015	0.015	0.015	...	...	0.005	...
Cb	...	0.90	...	2.0	...	1.0	...	...	...	...	...	2.0
Fe	...	...	...	...	...	0.25	0.15	...	...	...	1.0	2.0
Mn	...	...	...	...	...	...	0.10	...	0.75	...	0.05	0.25
Si	...	...	...	...	...	...	0.05	...	0.75	...	0.30	0.25

5.3. Sea Salt

Previous investigations have shown that sea salt in air is a primary cause of hot corrosion; therefore, the test program will be initiated using 1.0 ppm sea salt in combustor air to simulate a marine environment. If time and funds permit, some evaluation in the absence of sea salt will be included in the program.

5.4. Operating Conditions

For this investigation, the Phillips 2-inch combustor will be operated at 15 atmospheres combustor pressure with air flows of 6660 pounds per hour for combustor air and 324 pounds per hour for test specimen retainer purge air. These values represent the maximum capabilities of the test facility and are selected to give maximum test severity.

It is proposed to operate the Phillips Turbine Simulator using a temperature cycle of 30 minutes, with a maximum temperature in the cycle of 2000F.

5.5. Test Plan

The experimental design will provide information for comparisons of the hot corrosion attack on the superalloys and the coating-alloy systems in addition to the primary evaluation of the effect of sulfur in fuel on hot corrosion. The design has been selected to provide the strongest comparisons of the effect of sulfur in fuel on hot corrosion.

The 16 positions in each of the three rows of the specimen retainer will be divided into four groups of four positions each with the positions selected at random. Once the groups have been selected, they will be maintained throughout the program. All evaluations with a given superalloy or coating-alloy system will be made in the same four positions of the specimen retainer. At the start of a test, the four positions in a group will be filled with specimens of a coating-alloy system and specimens will be removed, with replacement, on the basis of visual inspection to provide duplicate four-point curves of weight-loss with time of exposure with the level of attack being significant at each point. The time for removal of the first specimen of a group will be determined by visual inspection of the four specimens in the group to be sure the representative level of attack is at a significant level. The time periods for removal of specimens need not be uniform; however, the times should be adjusted so that the sums of the times of exposure of the initial and replacement specimens in each of the four positions in the group are equal. The hours of exposure selected for each system with the first level of sulfur tested will set the hours of exposure for the other two levels of sulfur so that comparisons of the effect of fuel sulfur on hot corrosion for each system can be made at fixed times. It is anticipated that the resistance to hot corrosion attack and, therefore, the time of exposure required to obtain a significant level of

attack on the superalloys and coating-alloy systems will vary widely. On the basis of any available information, the 20 coating-alloy systems and the 12 superalloys will be arranged in order of estimated decreasing resistance to hot corrosion attack. The 12 materials estimated to be the most resistant to corrosion will be used at the start of the test and as evaluations are completed, the next most resistant material will be selected. The estimates of the relative resistance to hot corrosion attack are not critical but they will aid in completing the evaluations in the 12 groups of positions at as nearly the same time as possible.

6. ACKNOWLEDGEMENTS

THIS WORK WAS ADMINISTERED UNDER THE DIRECTION OF C. C. SINGLETERRY, HEAD, FUELS AND LUBRICANTS BRANCH, PROPULSION DIVISION, NAVAL AIR SYSTEMS COMMAND, DEPARTMENT OF THE NAVY, WITH S. M. COLLEGEMAN AS PROJECT ENGINEER.

THE AUTHORS WISH TO EXPRESS THEIR APPRECIATION FOR GUIDANCE AND ASSISTANCE IN THE AREAS OF:

TEST EQUIPMENT CALIBRATION AND OPERATION BY E. H. FROMM.

STATISTICAL ANALYSIS BY M. R. GOSS AND LYNN JONES.

METALLOGRAPHIC ANALYSIS BY E. H. BORGMAN AND VELMA GOOCH.

E. H. FROMM

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1. Schirmer, R. M. and Quigg, H. T., "Effect of JP-5 Sulfur Content on Hot Corrosion of Superalloys in Marine Environment." Progress Report No. 3 for Naval Air Systems Command Contract NOw 65-0310-d. Phillips Petroleum Company Research Division Report 4370-66R. Bartlesville, Oklahoma, July, 1966, 173 pp.

2. Quigg, H. T. and Schirmer, R. M., "Effect of Sulfur in JP-5 Fuel on Hot Corrosion of Coated Superalloys in Marine Environment." Final Summary Report for Naval Air Systems Command Contract N00019-67-C-0275. Phillips Petroleum Company Research Division Report 5058-68R. Bartlesville, Oklahoma, May, 1968, 114 pp.

3. Quigg, H. T. and Schirmer, R. M., "Effect of Sulfur in JP-5 Fuel on Hot Corrosion of Coated Superalloys in Marine Environment." Progress Report No. 2 for Naval Air Systems Command Contract N00019-67-C-0275. Phillips Petroleum Company Research Division Report 4886-67R. Bartlesville, Oklahoma, October, 1967, 68 pp.

4. Quigg, H. T. and Schirmer, R. M., "Effect of Sulfur in JP-5 Fuel on Hot Corrosion of Coated Superalloys in Marine Environment." Progress Report No. 2 for Naval Air Systems Command Contract N00019-68-C-0252. Phillips Petroleum Company Research and Development Report 5226-68. Bartlesville, Oklahoma, November, 1968, 54 pp.

5. Fromm, E. H., "Design and Calibration of the Improved Phillips Fuel Testing Facilities." Phillips Petroleum Company Research Division Report 3527-63R. Bartlesville, Oklahoma, July 2, 1963.

6. Hlade, O. C., "Aviation Turbine Fuels, 1966." Petroleum Products Survey No. 49. Bartlesville, Oklahoma, Petroleum Research Center, Bureau of Mines, U. S. Department of the Interior, March, 1967.

7. Method of Test for Rust-Preventing Characteristics of Steam-Turbine Oil in the Presence of Water (D 665), 1966 Book of ASTM Standards, Part 17.

8. Hall, N. A., "Mean Specific Heats for the Working Media of Gas Turbine Powerplants." SAE Quarterly Transactions, Vol. 1 No. 3, pp 490 - 497, July, 1947.

8. APPENDIX 1
(Test Equipment)

8.1. Test Facility

Phillips research facility for testing jet fuel, pictured in part in Figure 13 has been described in detail by Fromm (5).

Air is supplied by rotary Fuller compressors and filtered by a Selas Vape-Sorber, both of which can be seen in the foreground. This air is preheated just before it enters the burner by a Thermal Research heat-exchanger. Both fuel and sea water are supplied by nitrogen pressurization of their respective tanks. A portion of the metering and automatic control equipment can be seen in Figure 14.

The burners operate with air-flow rates up to 2.0 lb/sec at inlet-air pressure up to 240 psi and inlet-air temperatures up to 1400F.

8.2. Phillips 2-Inch Combustor

A scale diagram of the 2-inch combustor used in this study is shown in Figure 15. Design details of the combustor are presented in Table 13. Basically, it embodies the principal features of combustors used in modern aircraft-turbine engines. It was a straight-through can-type, combustor with fuel atomization by a single, simplex-type, nozzle. The combustor liner was fabricated from 2-inch, Schedule 40, Inconel pipe, with added internal deflector skirts for film cooling of surfaces exposed to the flame.

Combustor configuration number 15 was used during the first 10 periods (110 hours) of the investigation and combustor configuration number 20 was used during the remainder of the investigation. This change was made to improve the durability of the flame tube and distribution of the flame.

8.3. Test Rig

The Phillips Turbine Simulator is shown in Figure 16. A scale diagram of the rig is shown in Figure 17. Its design permits easy access to the fuel nozzle, combustor liner, test specimens, etc. The combustor installation was disassembled, inspected, and reconditioned after every shutdown.

Four chromel-alumel thermocouples were mounted at 90 degree intervals, with the tips 3/8-inch upstream from the centers of the first row of specimens, for measurement of gas temperature. The thermocouples were housed in 1/4-inch diameter Inconel sheaths for protection.

A look-box, Figure 16, permitted observation of the test specimens during operation to obtain temperature measurements with an optical pyrometer.

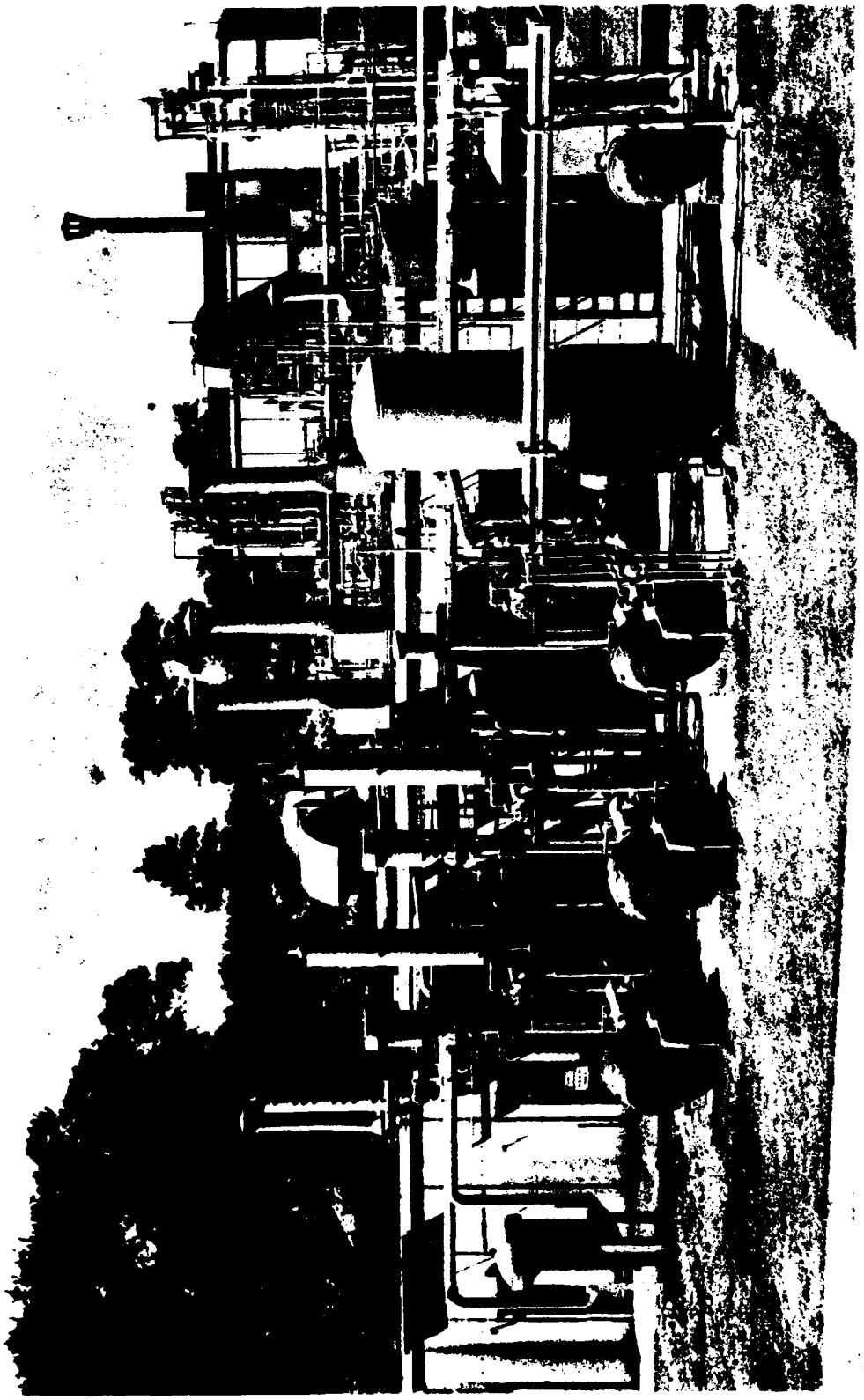


FIGURE 13
PHILLIPS RESEARCH FACILITY FOR JP FUELS

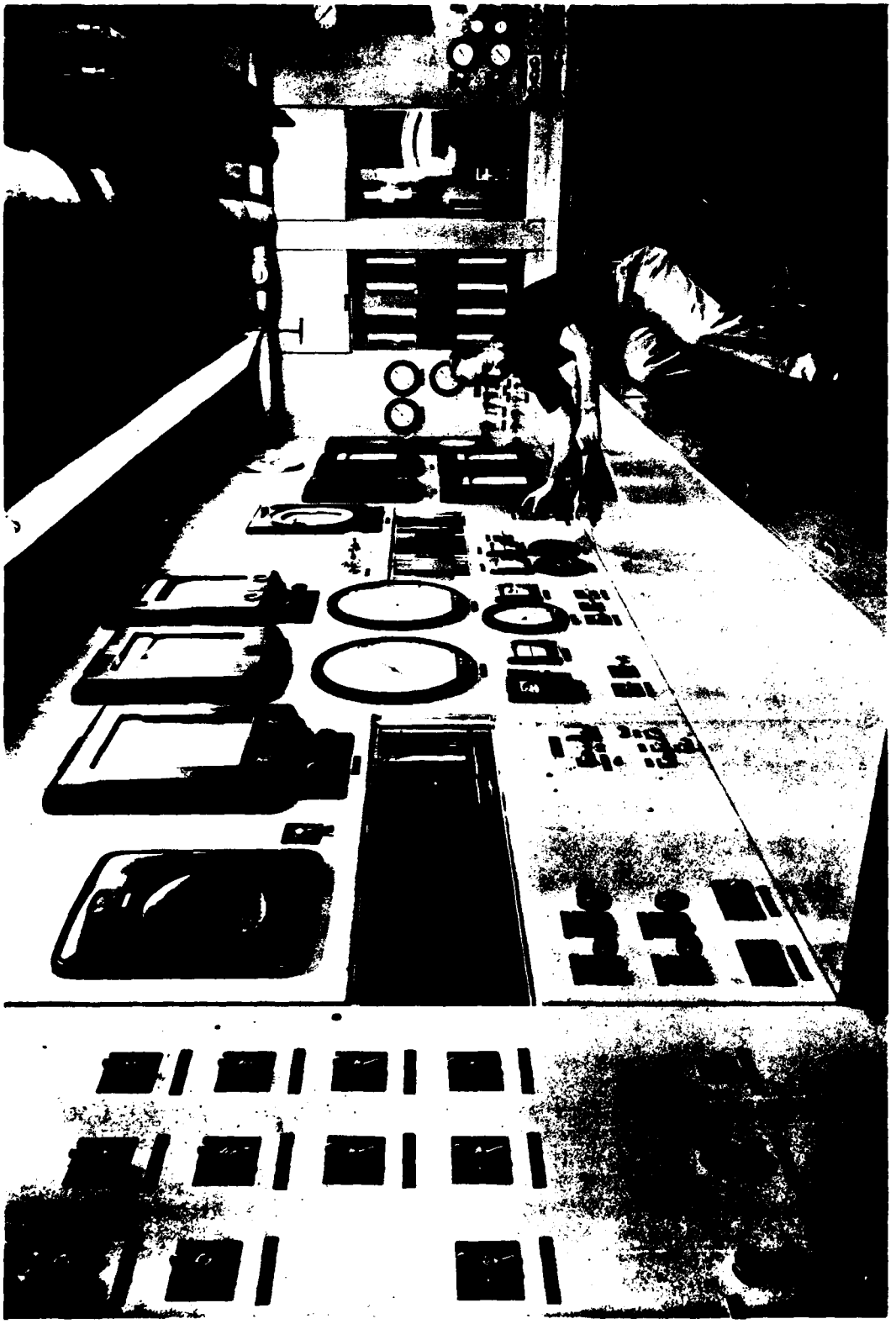


FIGURE 14
CONTROL ROOM FOR HIGH-PRESSURE COMBUSTOR

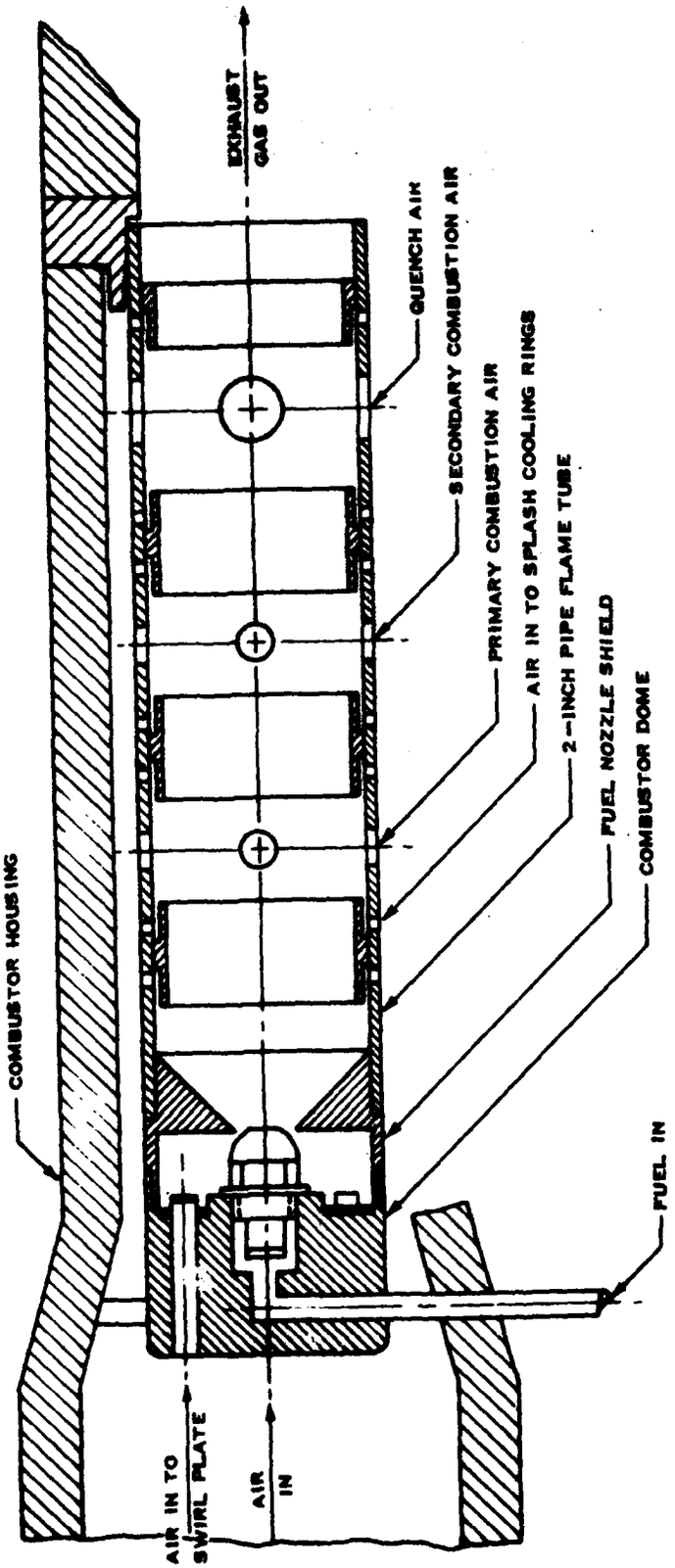


FIGURE 15
PHILLIPS 2 - INCH COMBUSTOR

TABLE 13

DESIGN DETAILS OF PHILLIPS 2-INCH COMBUSTOR

Combustor Configuration Number	15	20
Fuel Nozzle		
Type	Simplex (Monarch)	Simplex (Monarch)
Spray Pattern	Semi-Solid Cone (PLP)	Semi-Solid Cone (PLP)
Spray Angle, degrees	50	50
Capacity, gph of No. 2 Fuel Oil @ 100 psi	10.5	10.5
Combustor Dome		
Air Inlet Type	Tangential Swirl	Tangential Swirl
Shield Hole Diameter, in.	0.625	0.500
Total Hole Area, sq. in.	0.307	0.196
% Total Combustor Hole Area	8.7	5.1
Splash Cooling Air		
Hole Diameter, in.	0.125	0.125
Holes/Station	16	16
Number of Stations	7	7
Total Number of Holes	112	112
Total Hole Area, sq. in.	1.374	1.374
% Total Combustor Hole Area	38.7	35.7
Primary Combustion Air		
Hole Diameter, in.	0.250	0.312
Total Number of Holes	4	8
Total Hole Area, sq. in.	0.196	0.612
% Total Combustor Hole Area	5.5	15.9
Secondary Combustion Air		
Hole Diameter, in.	0.375	0.375
Total Number of Holes	4	4
Total Hole Area, sq. in.	0.442	0.442
% Total Combustor Hole Area	12.5	11.5
Quench Air		
Hole Diameter, in.	0.625	0.625
Total Number of Holes	4	4
Total Hole Area, sq. in.	1.227	1.227
% Total Combustor Hole Area	34.6	31.8
Total Combustor Hole Area, sq. in.	3.546	3.851
% Cross Section Area	133.4	144.8

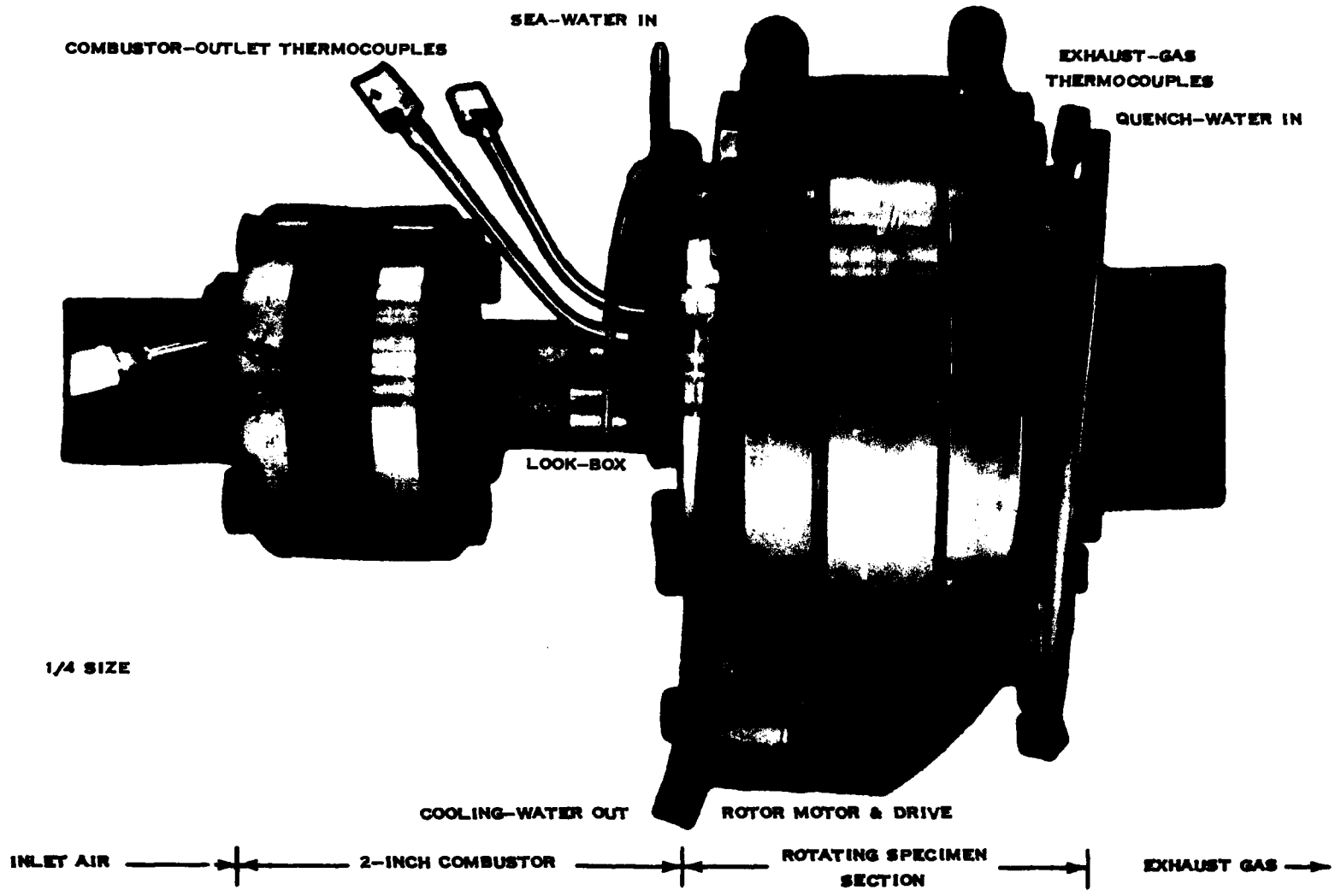


FIGURE 16
PHILLIPS TURBINE SIMULATOR FOR HOT-CORROSION STUDIES

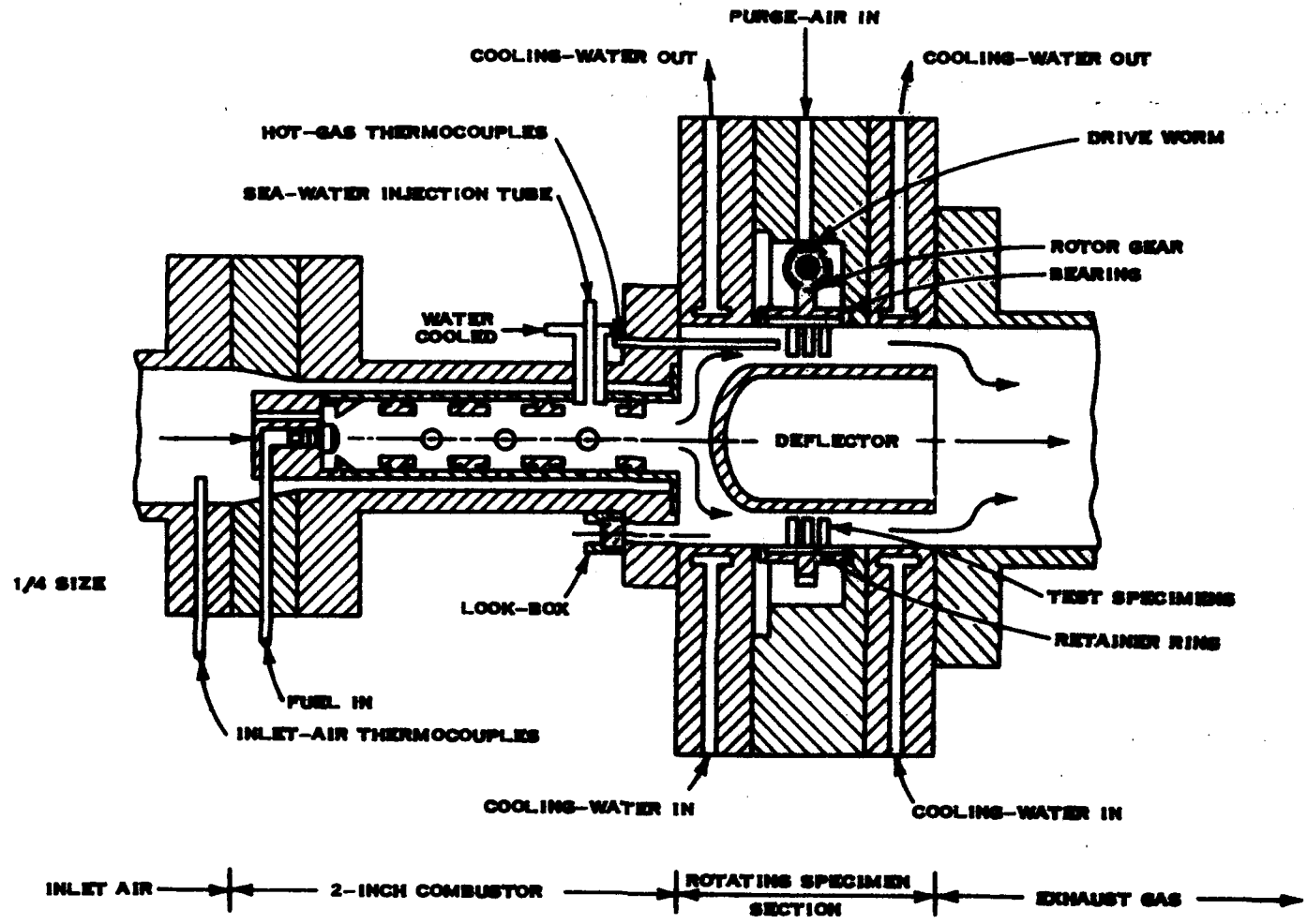


FIGURE 17
SCHEMATIC OF PHILLIPS TURBINE SIMULATOR FOR HOT-CORROSION STUDIES

Sea water was injected in the quench zone of the combustor, as indicated in Figure 17, rather than upstream of the combustor or in the primary-combustion zone. Injection in the quench zone avoided a severe corrosion problem with the combustor liner, and also insured exposure of test specimens to the desired sea-salt concentrations. The sea water was divided into two metered portions and introduced through opposing jets to obtain uniform distribution of sea salt in air by impingment of the jet streams.

The test section was water jacketed to obtain the desired durability of operation with high-temperature gases.

The investment cast test specimens are round pins of the form shown in Figure 18. The design of the retainer for mounting 48 specimens in the hot gas stream from the Phillips 2-inch combustor is shown in Figure 19. The general location of the test specimens in the exhaust gas from the 2-inch combustor is shown in Figure 17. A deflector is centered in the gas stream by four equally spaced, thin plates downstream of the specimens to provide an annulus for hot gas flow on the specimens. A view of the specimen retainer, with specimens, mounted in the test rig is shown in Figure 20.

A worm-gear drive is provided to slowly rotate the specimens around the annulus and provide exposure of each test specimen to an average gas temperature. The specimens are mounted in the retainer in three rows of 16 specimens each, with each row rotated 7 1/2 degrees from the prior row and thus no specimen is in the shadow of another specimen.

Purge air is introduced into the cavity containing the worm gear drive (Figure 17) at a pressure slightly higher than combustion pressure to prevent exposure of the gear drive and bearings to the corrosive atmosphere from the combustor.

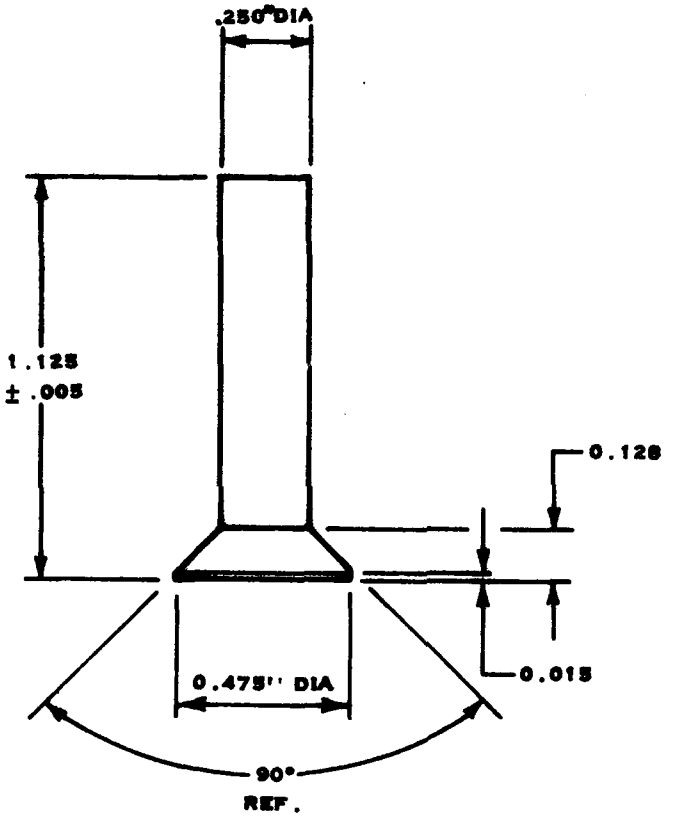


FIGURE 18
TEST SPECIMEN

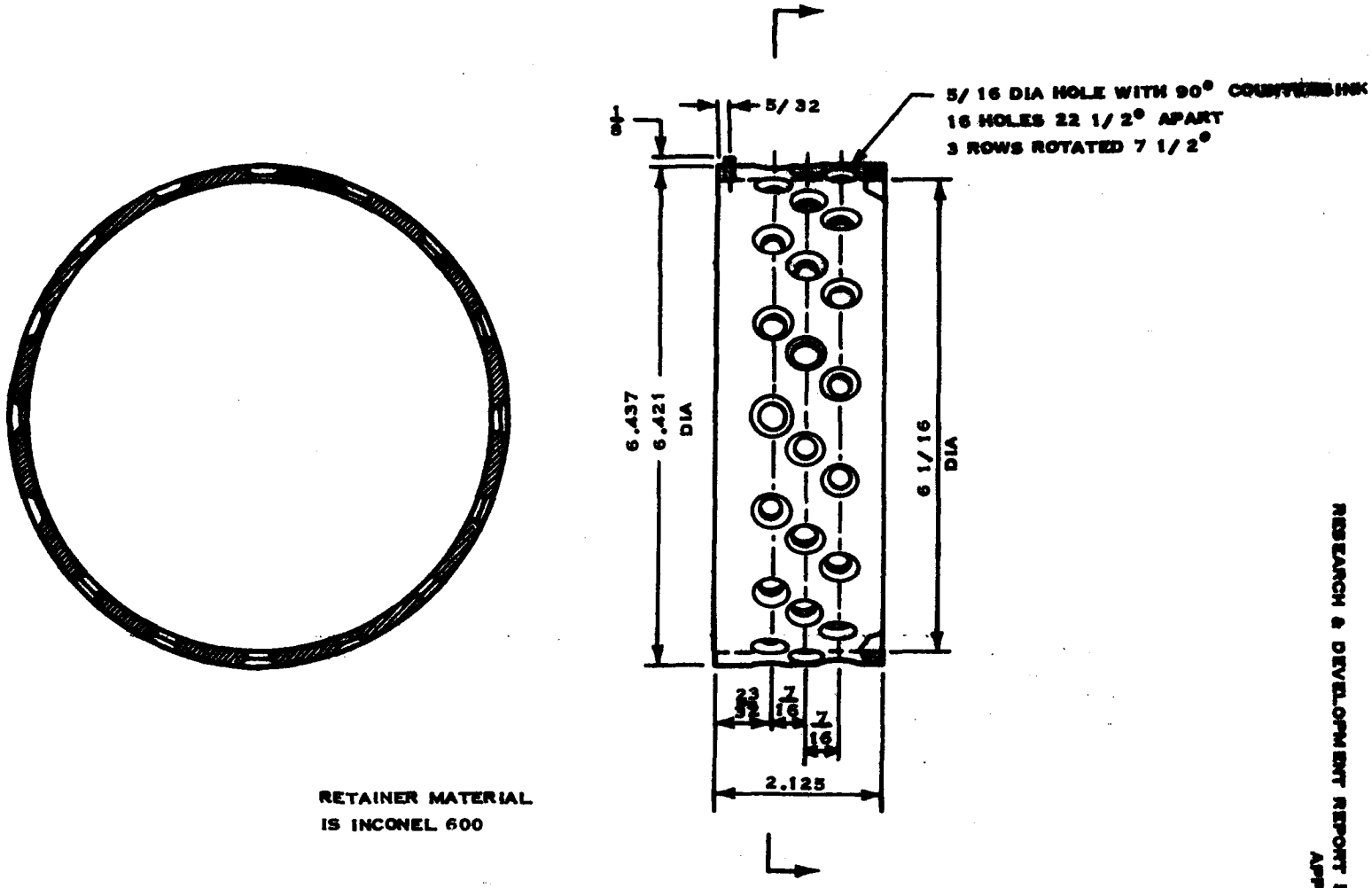
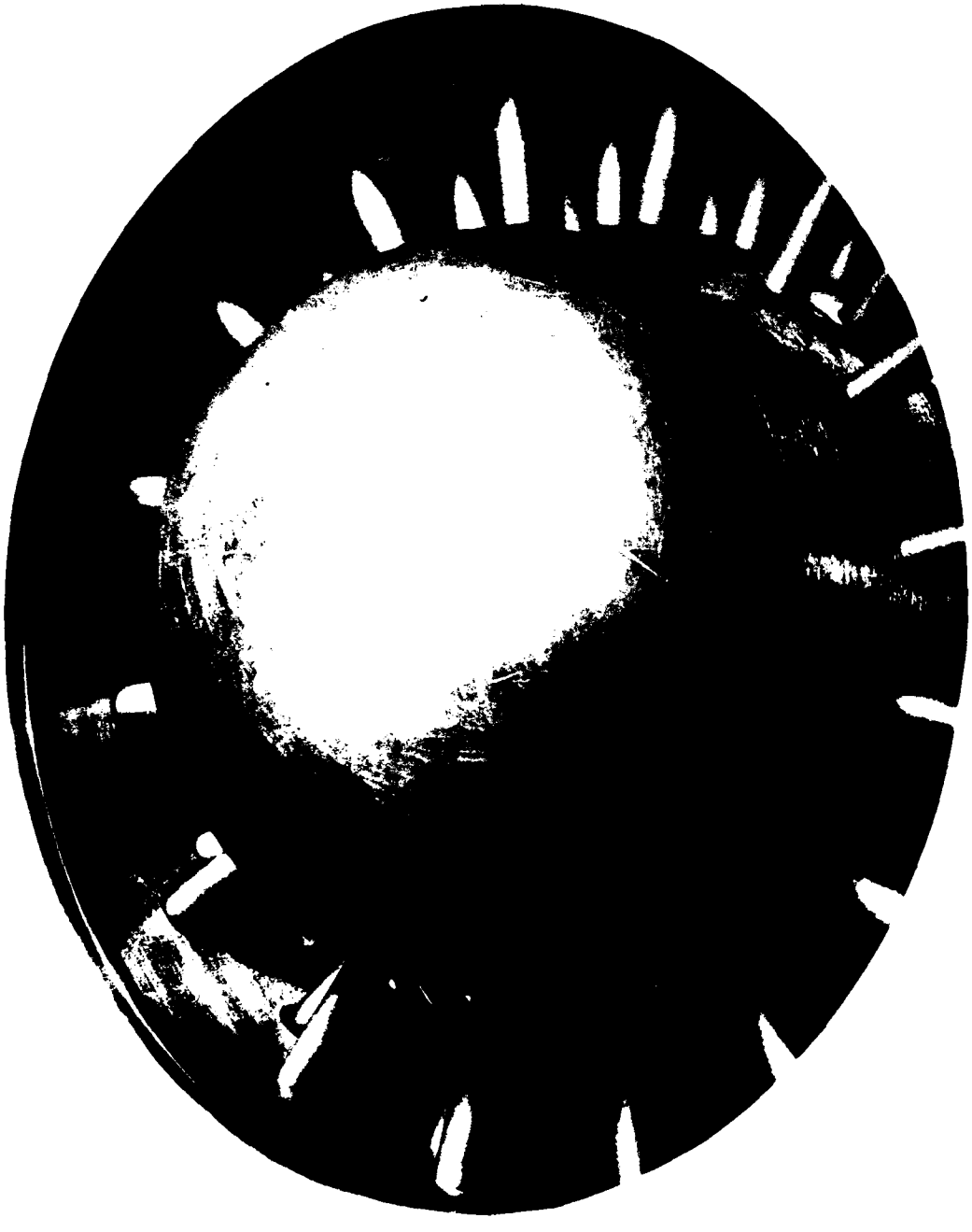


FIGURE 19
TEST SPECIMEN RETAINER



FULL SIZE

FIGURE 20
ROTATING-SPECIMEN SECTION OF PHILLIPS HOT-CORROSION TEST RIG

9. APPENDIX 2 (Materials)

9.1. Test Fuel

The base fuel selected for use in this investigation was a segregated sample of production ASTM Type A aviation-turbine fuel. The physical and chemical properties of interest to the investigation are presented in Table 14. The average values of pertinent properties from the Bureau of Mines Product Survey (6) over a period of the past ten years are also shown for grade JP-5 aviation-turbine fuel. The physical and chemical properties of the base fuel closely approximate the average 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 contents were negligible; if present, they would concentrate as ash and might alter the scale composition on the test specimens exposed to the exhaust gases.

The base fuel contains less than 0.0040 per cent by weight of sulfur. The test fuel of higher sulfur content was produced by blending to 0.040 per cent by weight of sulfur using ditertiary butyl disulfide.

9.2. Sea Water

A synthetic sea water was used in this study. Its formulation was taken from ASTM Method D 665 (7). The components and their concentrations are shown in Table 15. The abundance of various elements in the synthetic formula compares very favorably with the average sea water composition (1).

9.3. Test Specimens

Two groups of superalloys were used in this investigation. The Inconel 713C base alloy used in the primary portion of the investigation is a nickel-base alloy used in previous studies. Investment-cast specimens of this superalloy were obtained from Misco Division, Howmet Corporation. The chemical analysis for the heats of Inconel 713C, furnished by the supplier, is shown in Table 16.

The dimensions of the test specimens are shown in Figure 18. The investment castings were finished by the supplier by grinding the base and inspected to provide specimens having a smooth, uniform, finish and uniform dimensions with a tolerance of ± 0.005 inches. The specimens were inspected by fluorescent penetrant (Zyglo) and X-ray to insure freedom from cracks, porosity and inclusions.

Specimens of Inconel 713C, from Heat HW437, were coated with MDC-1 coating by the Reactive Metal Products Division, Howmet Corporation. The coating is characterized by the following description.

TABLE 14

PHYSICAL AND CHEMICAL PROPERTIES OF TEST FUEL

Distillation Temperature, F	Test Fuel Base (a)	Average JP-5 (b)
Initial Boiling Point	346	-
5 Volume per cent evaporated	365	-
10 Volume per cent evaporated	369	-
20 Volume per cent evaporated	379	382
30 Volume per cent evaporated	385	-
40 Volume per cent evaporated	395	-
50 Volume per cent evaporated	405	413
60 Volume per cent evaporated	417	-
70 Volume per cent evaporated	432	-
80 Volume per cent evaporated	447	-
90 Volume per cent evaporated	466	455
95 Volume per cent evaporated	477	-
End Point	501	-
Gravity, degrees API	46.1	41.9
Gum, milligrams per 100 mls.	0.7	1.1
Smoke Point, millimeters	26.2	22.6
Hydrogen Content, weight per cent	14.1	13.6
Composition, parts per million		
Sulfur	27 (c)	1030
Metals		
Iron	< 0.2	-
Vanadium	< 0.2	-
Nickel	< 0.2	-
Copper (d)	< 0.01	-
Hydrocarbon Types, volume per cent		
Normal Paraffins	27	-
Isoparaffins	23	-
Cycloparaffins	36	-
Olefins	0.20	1.6
Aromatics	12.2	15.1

Notes:

(a) Values for segregated sample of Phillips Kansas City production ASTM Type-A aviation turbine fuel, finished by hydrotreating.

(b) U. S. Bureau of Mines Petroleum Product Survey, 1957 - 1966 (6).

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

(d) Spectro-photometric analysis.

TABLE 15

COMPOSITION OF SYNTHETIC SEA WATER (a)

Salt (b)	Formula	Grams per liter (c)
Sodium Chloride	NaCl	24.54
Magnesium Chloride	$MgCl_2 \cdot 6H_2O$	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	$SrCl_2 \cdot 6H_2O$	0.04
Sodium Fluoride	NaF	0.003
	TOTAL	41.953

(a) ASTM D665 (7).

(b) Use cp chemicals.

(c) Use distilled water.

TABLE 16

COMPOSITION OF INCONEL 713C TEST SPECIMENS

	Chemical Analysis, Per Cent				
	Specification		Investment Cast by Misco		Cast Bars
Alloying Elements	Min.	Max.	Heat RW437	Heat RW424	from ASTM
Nickel + Cobalt	Remainder		Remainder	Remainder	Remainder
Cobalt	- - -	1.0	0.1	0.1	- - -
Chromium	12.0	14.0	13.50	13.35	13.0
Molybdenum	3.8	5.2	4.31	4.53	4.0
Aluminum	5.5	6.5	5.78	6.35	6.3
Titanium	0.50	1.0	0.85	0.90	0.9
Manganese	0.08	0.20	0.1	0.1	- - -
Iron	- - -	2.5	0.10	0.14	- - -
Zirconium	0.05	0.15	0.115	0.080	<0.01
Silicon	- - -	0.50	0.1	0.1	- - -
Boron	0.005	0.015	0.008	0.008	0.014
Sulfur	- - -	0.015	0.003	0.003	- - -
Carbon	0.08	0.20	0.11	0.11	0.15
Copper	- - -	0.50	0.1	0.1	- - -
Columbium + Tantalum	1.8	2.8	2.38	2.16	- - -
Columbium	- - -	- - -	- - -	- - -	2.1
Tantalum	- - -	- - -	- - -	- - -	<0.1
Tungsten	- - -	- - -	- - -	- - -	< 0.1

MDC-1 is an aluminum coating, which was applied by a pack-diffusion process to obtain a total thickness of approximately 2 mils. This coating is divided about equally between an outer layer which contains non-metallic dispersions and a diffused zone.

From specification dimensions, the surface area was calculated to be 7.76 square centimeters. The average weight from a random sample of bare Inconel 713C specimens is 8251 mg and for MDC-1 coated Inconel 713C is 8306 mg.

Specimens of six superalloys (Inconel 713C, Udimet 500, IN-100, IN-738, Udimet 700, and Mar-M-421) were received for evaluation as part of a "Round Robin" hot corrosion test program being conducted by the Hot Corrosion Task Force of ASTM. The specimens, as received, were cast round bars approximately one half inch in diameter and three inches in length. Four specimens each of Inconel 713C and Udimet 500 and two specimens each of IN-100, Inconel 738, Udimet 700 and Mar-M-421 were received. Two test specimens for the Phillips Turbine Simulator were prepared from each test bar by Plunge Grinding to conform to the dimensions shown in Figure 18. The chemical analyses, furnished by International Nickel Company, are shown in Table 16 for Inconel 713C and in Table 17 for the other five superalloys.

TABLE 17

COMPOSITION OF ROUND ROBIN NICKEL-BASE ALLOYS

Alloy	Values	C	Cr	Co	Al	Ti	Mo	W	Cb	Ta	B	Zr
IN-100	nominal	.18	10.0	15.0	5.5	4.7	3.0	-	-	-	.014	.06
	analyzed	.22	10.1	14.4	5.5	4.7	3.0	<.1	<.1	<.1	.015	<.01
Inco 713C	nominal	.12	12.5	-	6.1	.8	4.2	-	2.0	-	.012	.10
	analyzed	.15	13.0		6.3	.9	4.0	<.1	2.1	<.1	.014	<.01
Udimet 700	nominal	.08	15.0	18.5	4.3	3.5	5.2	-	-	-	.030	-
	analyzed	.08	14.3	14.6	4.0	3.0	3.7	<.1	<.1	<.1	.035	<.01
Mar-M421	nominal	.15	15.5	9.5	4.3	1.8	2.0	3.8	2.0	-	.015	.05
	analyzed	.18	15.1	9.8	5.4	2.7	1.6	3.7	2.1	.3	.045	<.01
IN-738	nominal	.17	16.0	9.0	3.3	3.3	1.8	2.5	.9	1.8	.020	.10
	analyzed	.23	15.0	8.9	3.5	3.5	1.5	2.6	.9	1.3	.013	.12
Udimet 500	nominal	.07	19.0	19.0	3.0	3.0	4.2	-	-	-	.007	.05
	analyzed	.10	18.9	18.4	3.2	3.0	3.5	<.1	.1	.1	.004	<.01

10. APPENDIX 3 (Procedures)

10.1. Pre-Test Cleaning

New specimens were cleaned by vapor degreasing with trichloroethylene, using the apparatus shown diagrammatically in Figure 21. Cleaned specimens were handled with degreased stainless-steel tongs. The initial weight of each specimen was determined following degreasing.

10.2. Post-Test Cleaning

After exposure, cleaning was necessary to remove the frequently heavy accumulation of surface deposit or scale, to allow for the measurement of metal loss by the specimens from hot corrosion. After weighing following removal from the test rig, specimens of bare superalloys and MDC-1 coated Inconel 713C were immersed in molten sodium hydroxide at 750 to 790F with 1/3 amp/sq cm passing through the specimens for a period of 10 minutes. The specimens were scrubbed with a stainless-steel wire brush during a water quench, rinsed in acetone, dried, and reweighed. The apparatus used for electro-cleaning is shown diagrammatically in Figure 22.

10.3. Metallographic Examination

Following exposure, specimens were cleaned for determination of their loss in weight, and subsequently specimens were chosen for metallographic examination. All specimens from the ASTM portion of the investigation and one set of eight specimens each of Inconel 713C and MDC-1 coated Inconel 713C from the remainder of the investigation were selected for metallographic examination.

Two cross-sectional areas of each selected specimen were mounted for examination. One represents a zone of maximum visual attack and the other represents a zone of average attack. For a specimen corroded over half or less of its length, the sections were taken in a corroded zone and a non-corroded zone. Each of the cross-sectioned areas were measured for hot-corrosion effects across two diameters approximately 90 degrees apart as shown in Figure 23; thus, each specimen was measured in four places. Two types of hot-corrosion effects were determined; i.e., gross (massive surface oxidation) attack and maximum attack, as illustrated in Figure 23. All values were reported as loss in diameter (mils).

Surface Loss (Gross Attack) is a measurement of all material loss, plus any massive oxidation and sulfidation; it does not factor in other types of subsurface attack, such as intergranular attack. For each specimen, four measurements are reported to show the consistency of attack. The measurements are averaged together, and this new value reported as the gross attack.

Maximum attack is a measurement which includes gross attack plus the depth of penetration of all sulfides and oxides, which may be scattered

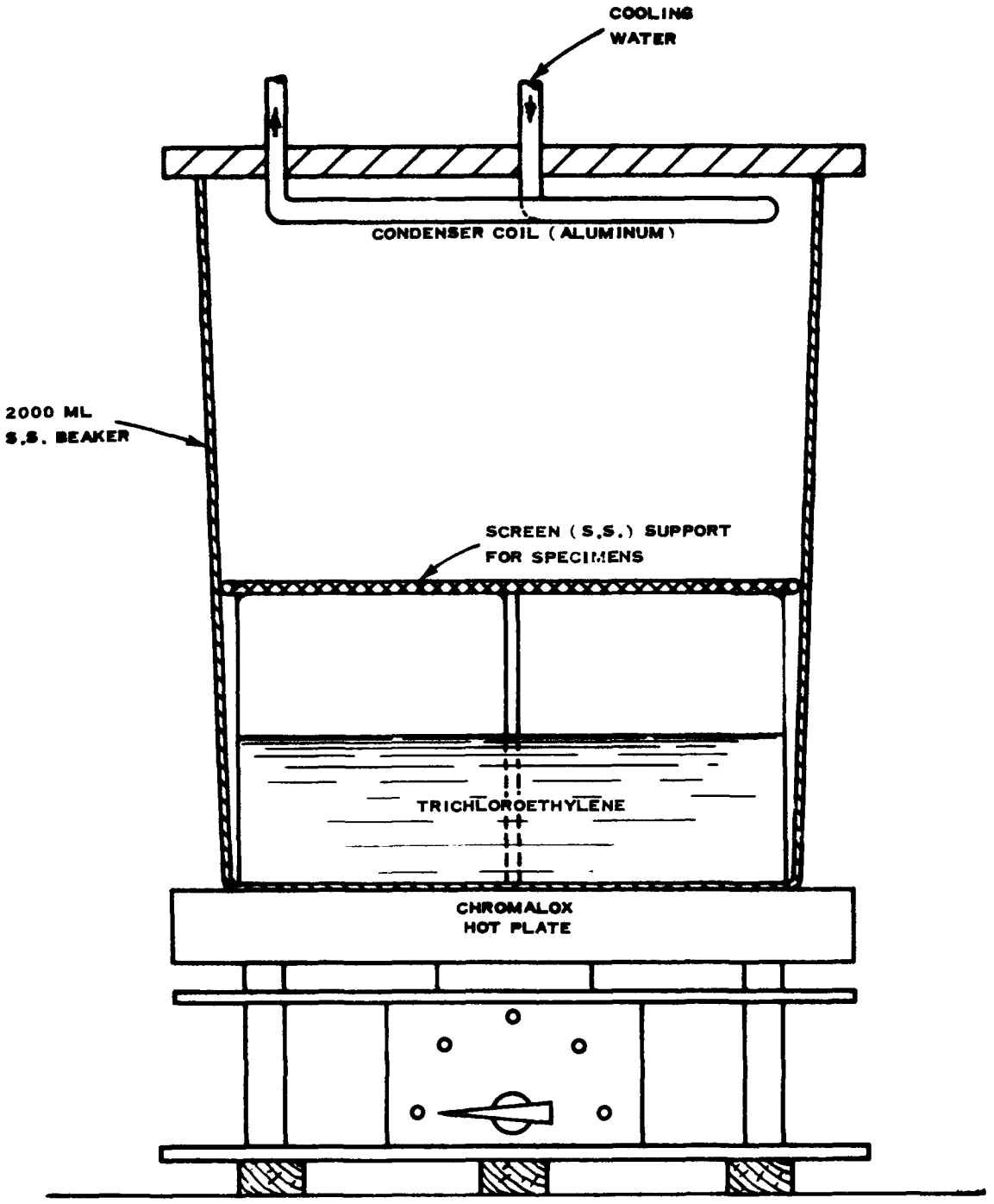


FIGURE 21
VAPOR-DEGREASING APPARATUS FOR PRE-TEST CLEANING OF SPECIMENS

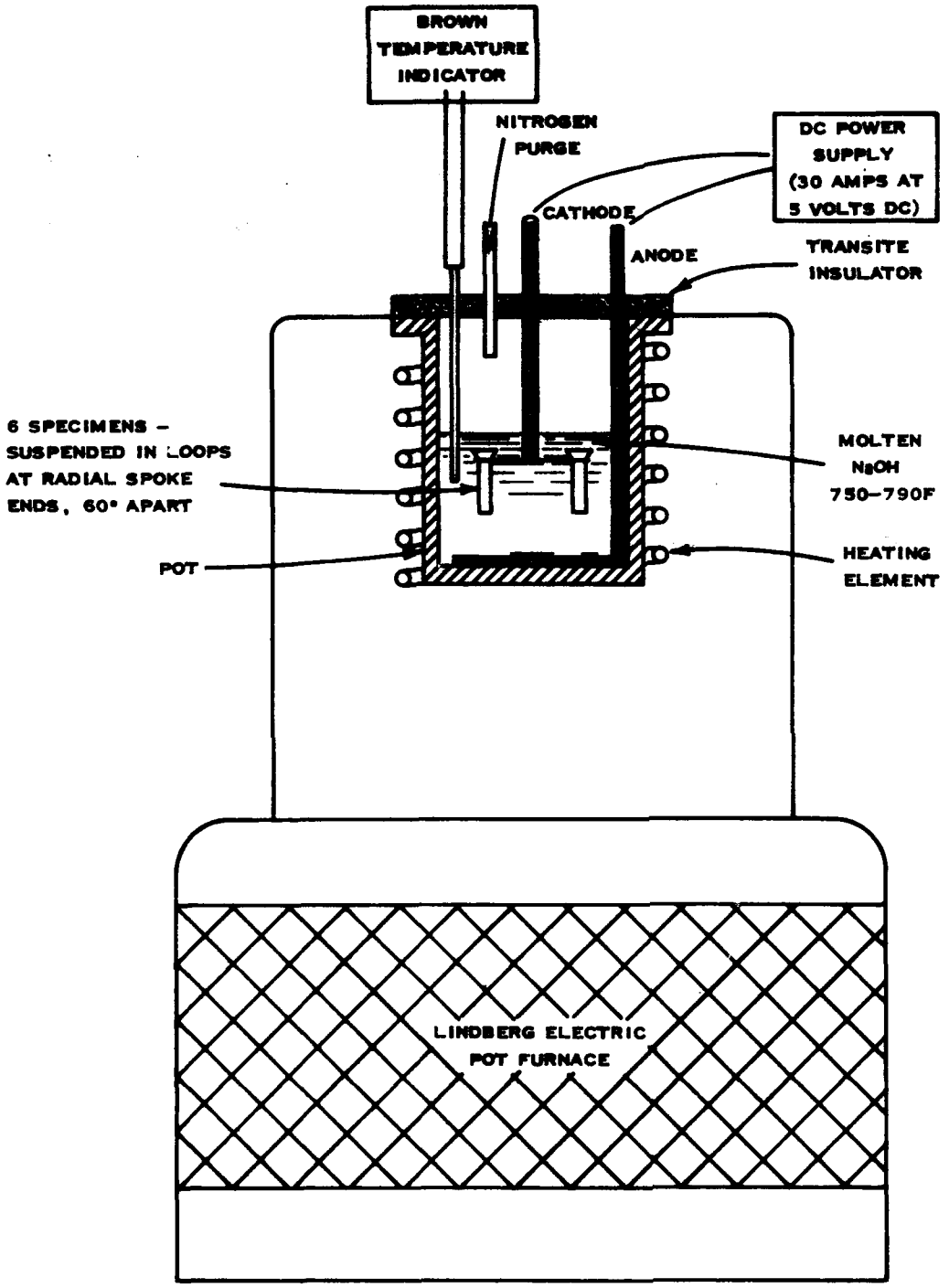
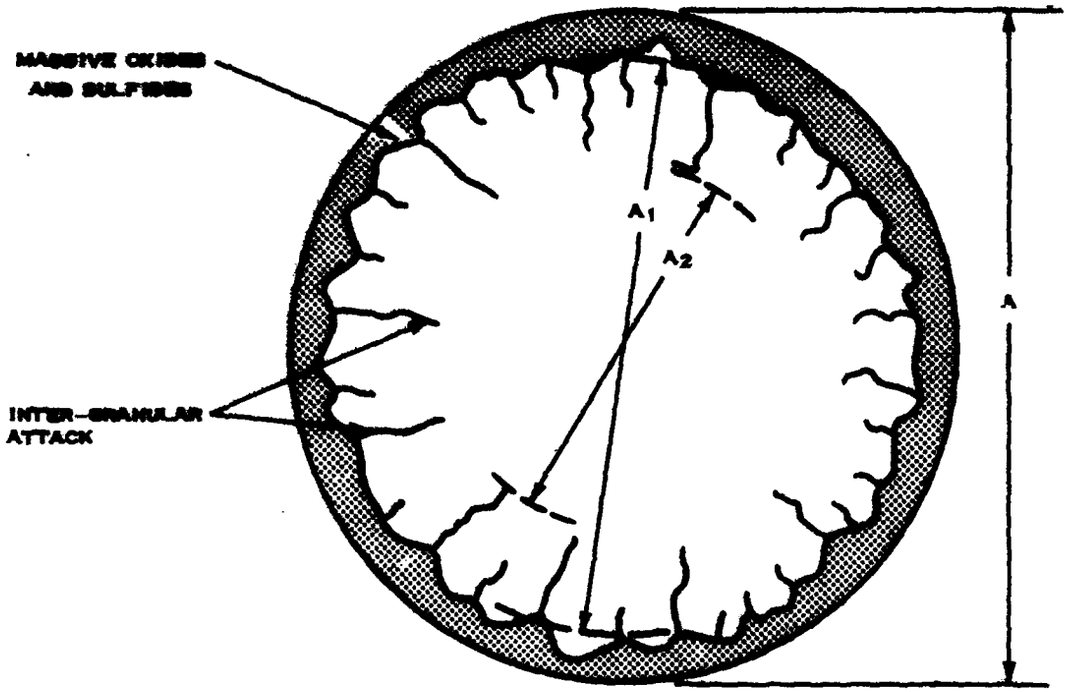


FIGURE 22
ELECTRO-DESCALING APPARATUS
FOR POST-TEST CLEANING OF SPECIMENS



A = ORIGINAL DIAMETER, MEASURED WITH A MICROMETER
A_1 = DIAMETER OF STRUCTURALLY USEFUL METAL, MEASURED AT 100X
A_2 = DIAMETER OF METAL UNAFFECTED BY OXIDES AND SULFIDES, MEASURED AT 100X
SURFACE LOSS: $A-A_1$ LOSS IN DIAMETER DUE TO MASSIVE OXIDES AND SULFIDES
MAXIMUM ATTACK: $A-A_2$ LOSS IN DIAMETER DUE TO ALL FORMS OF OXIDATION AND SULFIDATION

FIGURE 23
METHOD OF MEASURING HOT-CORROSION ATTACK

or in local concentrations; e.g., grain boundaries. The depth of subsurface oxides and sulfides may vary from one side of the specimen to the other side; thus, measurements in terms of loss in diameter are qualified as attack on both sides (B), mostly on one side (M), and on one side (O). A measurement for each cross-sectioned area is reported to indicate the consistency of attack, but only the greatest value is considered the maximum attack for the alloy.

In mounting the specimen, the two pieces were placed in a 1-inch red Bakelite mold with the coupons centered inside pieces of 3/8-inch diameter steel tubing. This arrangement aided in obtaining a flat surface on the coupon during polishing. The following eight step technique was used to polish the coupons.

(a) Dry ground on an 8", 180 grit Carbimet disc turned at 570 rpm.
(b) Dry ground on an 8", 120 grit Carbimet disc turned at 570 rpm.
(c) Hand lapped, wet, on 240 grit silicon carbide paper.
(d) Hand lapped, wet, on 320 grit silicon carbide paper.
(e) Hand lapped, wet, on 400 grit silicon carbide paper.
(f) Hand lapped, wet, on 600 grit silicon carbide paper.
(g) 6-micron diamond paste on nylon lap with polishing oil.
(h) AB micro cloth with Linde B polishing compound.

The polished coupons were etched to facilitate identification of the extent of alloy depletion at the corrosion interface. Generally this was done electrolytically with 2 per cent sulfuric acid; however, in some cases, Marble's Reagent was used to obtain a less severe etch.

Photomicrographs were made of each specimen using a Bausch & Lomb Research Metallograph at 9X and 500X magnification, and in some cases, at other magnifications.

10.4. Turbine Simulator Operation

The Phillips Test Facility and Test Rig used for this investigation are described in Sections 8.1. and 8.2. of Appendix 1.

Flow through the sea water injection jets (Figure 17) was maintained at 1.75 pounds per hour per jet or 3.5 pounds per hour total. Sea water injection was maintained during the fuel-off as well as the fuel-on portions of the cycle. The sea water (Table 15) was diluted with deionized water to give a concentration of 1.0 ppm sea salt in combustor air.

Purge-air was introduced into the cavity surrounding the test specimen retainer, at the location indicated in Figure 17, at a pressure slightly greater than combustor pressure, to minimize contact of combustion products with the specimen retainer bearings and worm gear drive.

The test specimen retainer (Figure 19) mounts 48 specimens in three rows of 16 specimens each. For identification of positions in the retainer, the rows are designated 1, 2, and 3 from front to rear and the positions in each row are identified in alphabetical order omitting I and O. For each superalloy or coating-alloy system, the basic test plan was to expose specimens in duplicate for four different lengths of time using four positions in a row in the specimen retainer. The 16 positions in each row were divided into four groups of four positions each with the positions selected at random and the grouping was maintained throughout the test. The grouping of positions in the specimen retainer are shown in Table 18.

Superalloys or coating-alloy systems were assigned to each group of four locations and the specimen retainer was filled with new cleaned and weighed specimens prior to the start of a test. The Phillips Turbine Simulator was operated under the cyclic temperature conditions of Table 19 for periods of 11 hours each. At the completion of each 11 hour period, the test rig was disassembled and the specimen retainer removed for visual inspection. The time for removal, with replacement, of the first specimen of a group was determined by visual inspection of the four specimens in the group to be sure that the representative level of attack was at a significant level. The times for the remaining specimen removals, with replacement, were selected to provide a considerable spread in weight-loss with time. The specimens in each group were removed in alphabetical order. The time periods for removal of specimens need not be uniform; however, the times should be adjusted so that the sums of the times of exposure of the initial and replacement specimens for each of the four positions in the group are equal. Examples of removals at equal and unequal times are shown in the following test schedules.

EXAMPLE I

Position	Exposure Time, Hours: Initial Specimen	Exposure Time, Hours: Replacement Specimen	Total Hours
1A	11	44	55
1C	22	33	55
1G	33	22	55
1R	44	11	55

EXAMPLE II

Position	Exposure Time, Hours: Initial Specimen	Exposure Time, Hours: Replacement Specimen	Hours
2F	55	121	176
2J	77	99	176
2L	99	77	176
2Q	121	55	176

TABLE 18

GROUPS OF POSITIONS IN SPECIMEN RETAINER

Group	Positions (a)			
I	1H	1K	1L	1P
II	1E	1F	1Q	1R
III	1B	1C	1D	1M
IV	1A	1G	1J	1N
V	2E	2F	2H	2L
VI	2B	2N	2P	2Q
VII	2A	2C	2K	2M
VIII	2D	2G	2J	2R
IX	3D	3H	3Q	3R
X	3B	3C	3J	3N
XI	3E	3G	3L	3M
XII	3A	3F	3K	3P

(a) Positions in each row assigned at random.

TABLE 19

OPERATING CONDITIONS FOR PHILLIPS TURBINE SIMULATOR

Test Variables	Test Condition					
Temperature, deg F						
Nominal Gas	1600	1000	1800	1000	2000	1000
Exhaust Gas (a)	1600		1800		2000	
Combustor Inlet Air	1000	1000	1000	1000	1000	1000
Pressure, atmospheres						
Combustor Inlet Air	15	15	15	15	15	15
Mass Flow Rate, lb/hr						
Purge Air	324	324	324	324	324	324
Combustor Air	6660	6660	6660	6660	6660	6660
Fuel	63.0		85.0		108.0	
Water (b)		49		49		49
Air-Fuel Ratio	106		78		62	
Flow Velocity, ft/sec						
at Test Specimen (c)	230	163	253	163	275	163
Cycle Time, minutes	8	2	8	2	8	2

Notes:

(a) Value calculated using mean specific heats (8) for 100 per cent combustion efficiency.

(b) Water flow through nozzle while fuel is off.

(c) Calculated value based on unblocked area in specimen retainer of 4.00 square inches.

The availability of only four test specimens for four of the six superalloys in the ASTM portion of the program required a modification of the scheduling of specimen removal for some of the groups during the investigation.

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13. ABSTRACT An experimental investigation is being conducted to evaluate the effect of very-low concentrations of sulfur in fuel on the durability of turbine-blade alloys when exposed under conditions simulating the environment in an aircraft-turbine engine operating in a marine environment. This has required development of new test equipment, termed Phillips Turbine Simulator, to increase productivity by simultaneous exposure of 48 specimens and to improve precision by rotating specimens for uniform exposure in the high-velocity stream of high-temperature, high-pressure, corrosive gas.

A preliminary test was conducted to evaluate a cyclic-temperature operating procedure with Phillips Turbine Simulator. Specimens of both bare and aluminum-diffusion coated Inconel 713C were exposed at 15 atmospheres combustor pressure using air with 1 ppm sea salt and fuel with 0.040 weight per cent sulfur to obtain exhaust gas temperatures which reached a maximum of 2000 F. It was concluded that this test equipment is sufficiently durable, and the test method provides sufficient precision for conducting our proposed investigation with a reasonable expenditure of time, manpower, and materials.

A comparison of the relative durability of superalloys in an ASTM "round-robin" program was made during this preliminary test. At this condition of exposure, the materials ranked in the following order: Udimet 500 (best), IN-738, Mar-M421, Udimet 700, Inconel 713C, and IN-100 (poorest). Metallographic measurements of "surface-loss" and "maximum-attack" were made, but the difference between them (subsurface deterioration) was relatively unimportant. Also, a linear relationship was found between depth-of-penetration and specimen weight-loss.

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KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
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OXIDATION						
SULFATION						
TURBINE BLADES						
GAS TURBINE BLADES						
ALLOYS						
NICKEL ALLOYS						
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