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

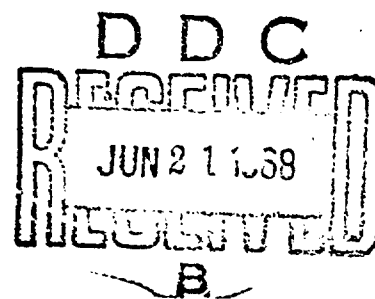
FINAL SUMMARY REPORT
APRIL 1, 1967 TO MARCH 31, 1968

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

PREPARED UNDER CONTRACT N00019-67-C-0275 FOR THE
NAVAL AIR SYSTEMS COMMAND, DEPARTMENT OF THE NAVY
BY PHILLIPS PETROLEUM COMPANY
BARTLESVILLE, OKLAHOMA

MAY 1968

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Final Summary Report
Naval Air Systems Command Contract N00019-C-67-0275

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

by

H. T. Quigg and R. M. Schirmer

S U M M A R Y

An experimental investigation is in progress to determine whether the 0.4 per cent by weight of sulfur which is currently allowed in JP-5 fuel is a safe level for protection of coated turbine blades in high-performance engines when operated in a marine environment. Tests have been conducted at 15 atmospheres pressure and 2000 F gas temperature using specimens of two different nickel-base alloys (Inconel 713C and Mar M-200) with aluminum-diffusion coating (Misco MDC-1) and with aluminum-chromium-diffusion coating (Misco MDC-9). Comparisons are made which show the effect of three different levels of sulfur in fuel (0.4, 0.04, and <0.004 per cent), both with and without 1.0 ppm sea salt in air. A significant decrease in the relative rate of corrosion when sea salt was removed from the air shows it to be the primary cause of hot corrosion. These data indicate that little or no benefit would result from a reduction in the sulfur limit for JP-5 fuel.

A screening program was conducted to evaluate the effect of vanadium on hot corrosion of a turbine-blade alloy in a marine environment. Specimens of Inconel 713C were exposed to exhaust gases from Phillips 2-Inch Combustor operated at 2000 F conditions with 0.40 weight per cent sulfur in JP-5 fuel, zero or 1.0 ppm sea salt in air and vanadium in air equivalent to either 3.8 or 1.0 ppm vanadium in fuel. The presence of vanadium equivalent to 3.8 ppm vanadium in fuel more than doubled the weight-loss of specimens with either zero or 1.0 ppm sea salt in air. The presence of vanadium equivalent to 1.0 ppm vanadium in fuel in the absence of sea salt in air had no significant effect on hot corrosion of Inconel 713C.

Specimens of one coated superalloy were exposed in a screening program using one level of sulfur in fuel, one level of sea salt in air, and four temperatures. This program was conducted to obtain information as to the length of exposure necessary to evaluate the effect of sulfur in fuel on hot-corrosion of coated superalloys in a marine environment over a range of temperatures. Exposure times were insufficient to provide a direct estimate of exposure times required at the lower temperatures to obtain meaningful evaluations; however, indirect comparisons indicate that up to several hundred hours or more of exposure will be required.

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PHILLIPS PETROLEUM COMPANY

BARTLESVILLE, OKLAHOMA

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IN MARINE ENVIRONMENT

1. INTRODUCTION

1.1. Turbine Durability

The corrosion of hot-section parts in modern aircraft-turbine engines is one of the factors that determines time before overhaul. In a marine environment corrosion becomes a major factor in limiting engine life. Various terms, including "black plague" and "sulfidation," 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 we will use it in this report to indicate the attack by sea salt on superalloys at high temperature.

1.1.1. High Temperature Oxidation

A typical example of the oxidation structure at the surface of a turbine blade after long exposure is shown in Figure 1. This photomicrograph was taken at the convex surface of a cross section from the center of a first-stage turbine blade that had completed 1800 hours in domestic-airline service since overhaul. An oxide scale covers the surface to a depth of about 0.001 inch. Alloy depletion and some intergranular oxidation, which precede surface oxidation by about 0.001 inch, are attributed to the diffusion of more reactive constituents, such as chromium and aluminum, to the surface where they combine with oxygen to form the protective scale.

1.1.2. Catastrophic Oxidation

The accelerated attack on aviation-turbine engines when operated in a marine environment is shown in Figure 2. This photomicrograph was taken at the leading edge of a cross-section from the center of a first-stage turbine blade recovered from a scrap drum at a U.S. Navy engine-overhaul shop. Deep sub-surface oxidation produced a mixed-phase zone composed of metal and corrosion products; however, considerable metal loss can be sustained without blade failure because the attack advances on a broad front without deep-intercrystalline penetration of sulfides or oxides. The attack is led by penetration of randomly dispersed, light-grey globules of metallic sulfides. Formation of these sulfides is associated with changes in the surface composition of the alloy, which is characterized by chromium depletion. The mechanism of hot corrosion is complex and not fully understood.



METALLOGRAPHIC CROSS-SECTION. MARBLE'S REAGENT ETCHED. 2000X MAGNIFICATION.

FIGURE 1
OXIDATION OF UHMET 500 TURBINE BLADE



METALLOGRAPHIC CROSS-SECTION. MARBLE'S REAGENT ETCHED. 4000X MAGNIFICATION.

FIGURE 2
ACCELERATED OXIDATION OF INCONEL 713C TURBINE BLADE

1.2. Sulfur in Fuel

The prominence of the band of precipitated sulfides preceding surface oxidation has focused attention on the sulfur content of the fuel as a principal cause of hot corrosion. If so, hot corrosion could be controlled by fuel specification. However, proposals to lower the sulfur content of aviation-turbine fuels must be approached with caution.

Most specifications for aviation-turbine fuel allow a sulfur content of 0.40 per cent by weight. The median value of the sulfur content of 197 samples of grade JP-5 aviation turbine fuel, representative of domestic and foreign purchases by the United States Navy from September 1966 to February 1967, was 0.04 weight per cent. The cumulative distribution for West Coast, East and Gulf Coasts, foreign, and total are shown in Figure 3. These data indicate that a substantial reduction in the sulfur limit for JP-5 fuel could drastically curtail availability, unless accompanied by incentives for modernization of manufacturing techniques.

1.3. Sea Salt

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.

Required aircraft operational patterns exclude control over seawater or sea salt ingestion by the engine with operation in a marine environment. However, establishing a realistic level for the concentration of sea salt in air ingested by an aircraft-turbine engine is difficult because the level is highly dependent upon the weather and aircraft mission. In general, satisfactory operation and durability has been sought for marinized gas-turbine engines with a sea-salt-ingestion rate of one part per million sea salt in air.

1.4. Vanadium

The detrimental effects of vanadium in fuel on fire-side corrosion in boilers has been well established. The U. S. Navy is considering a specification for a multi-purpose fuel to be used in marine boilers and gas-turbine engines. The proposed fuel will be slightly heavier than No. 2 Diesel fuel and it is proposed to allow 2 to 5 parts per million of vanadium and from 1.3 to 1.5 per cent by weight of sulfur. While the proposed fuel may create problems because it is heavier and allows more sulfur than JP-5, its vanadium content is the primary concern for gas-turbine engines.

1.5. New Test Section

Tests with a number of alloys and coatings over a range of temperatures from 1400 to 2000 F are needed to complete the study of the

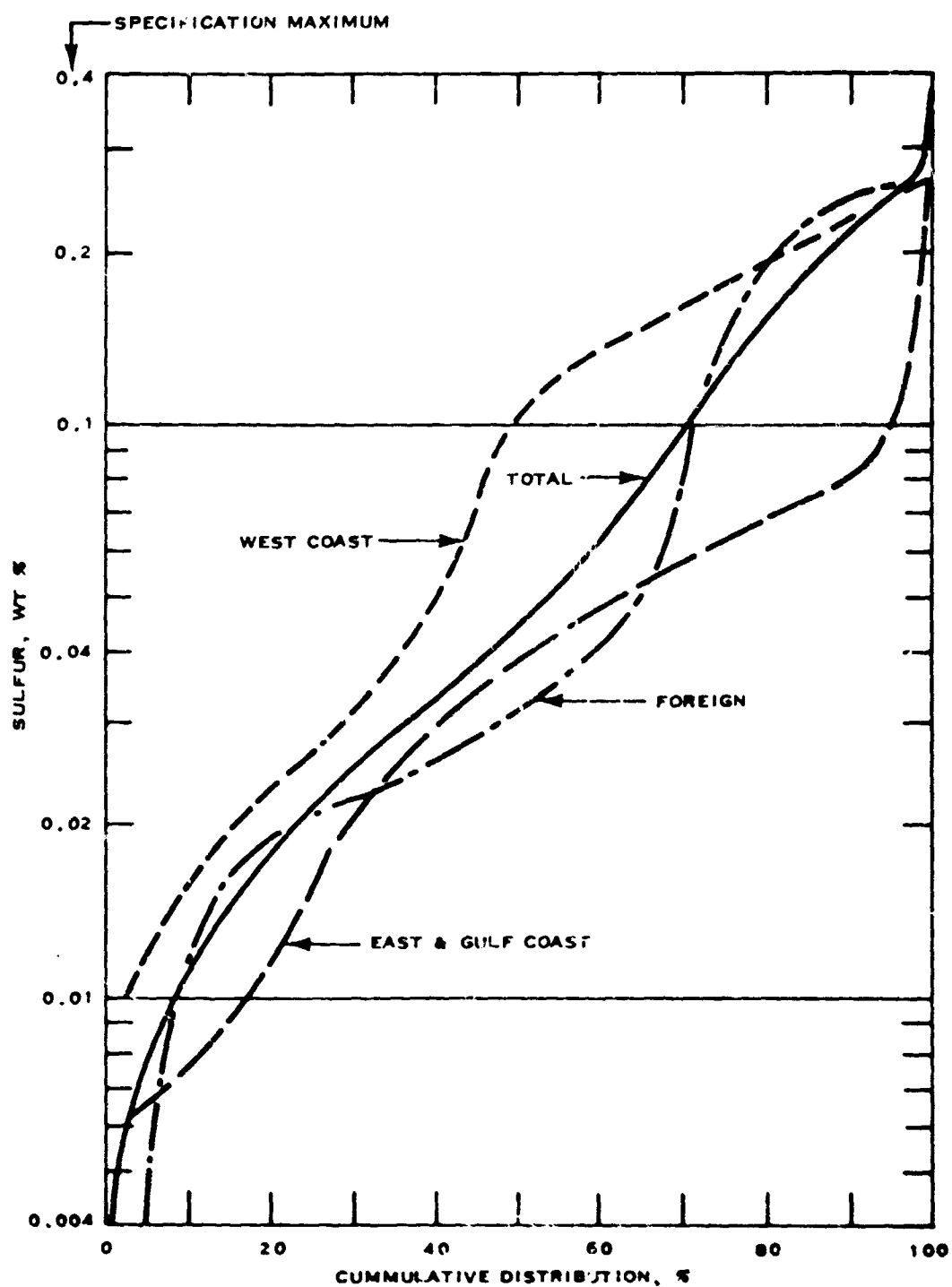


FIGURE 3
SULFUR CONTENT OF JP-5 FUEL PURCHASES
(SEPTEMBER 1966 - FEBRUARY 1967)

effect of sulfur in fuel on hot corrosion in a marine environment. Screening tests have indicated that exposure times of up to several hundred hours will be required at the lower temperatures (1400 and 1600 F) to stress some coated superalloys to the point of coating failure. The current test rig, exposing only six test specimens simultaneously, would require an excessive expenditure of time and materials for completion of the desired program. Development of a new test section which will expose a larger number of specimens simultaneously will permit completion of the proposed program with a realistic expenditure of time and materials.

1.6. Purpose

The Naval Air Systems Command has supported our work to determine whether the maximum sulfur content of 0.40 per cent by weight, 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 previous studies (1) aluminum-coated specimens (Misco MDC-1 on Inconel 713C) were immune to attack under all combinations of temperature, sulfur in fuel, and sea salt in air with the standard 5-hour test duration. During the last contract (NOW 66-0263-d) with the Naval Air Systems Command, experiments were designed to stress the coating to failure by extending test duration at 2000 F conditions and thereby, permit evaluating the effect of sulfur in fuel on the durability of coated superalloys. Experiments were completed (2) with Misco MDC-1 and MDC-9 coatings on Inconel 713C and on the bare alloy for comparison and the program was extended under the present contract to include another coating-alloy system (Misco MDC-1 coated Mar M-200).

Screening tests were conducted to evaluate the effect of another fuel impurity (vanadium) on hot corrosion of a bare superalloy (Inconel 713C). Specimens were exposed to exhaust gases from the Phillips 2-Inch combustor operated at 2000 F conditions with 0.4 weight per cent sulfur in fuel, zero or 1.0 ppm sea salt in air, and vanadium in air equivalent to 3.8 ppm vanadium in fuel (0.063 ppm vanadium in air). Specimens were also exposed at the same conditions with zero sea salt in air and vanadium in air equivalent to 1.0 ppm vanadium in fuel (0.017 ppm vanadium in air).

To complete the evaluation of the effect of sulfur in fuel on hot corrosion of coated superalloys the investigation needs to be extended to include a number of superalloys and coatings over a range of temperatures. A screening program has been conducted to aid in designing a program to complete this investigation.

2. CONCLUSIONS

2.1. Hot Corrosion of Coated Superalloys at 2000 F Condition

Experiments have been conducted, using a high-pressure burner rig, to evaluate the effect of sulfur in JP fuel on the hot corrosion of coated turbine-blade materials. The durability of two diffusion-type coatings, differing in composition, and two nickel-base superalloys, have been investigated at a 2000 F test condition, which simulates exposure in a high-performance engine when operated in a marine environment. Also, for reference purposes, data have been obtained in the absence of sea salt and with one of the uncoated superalloys.

The use of weight loss by test specimens as a measure of hot-corrosion attack was validated by metallographic examination, which showed corrosion advancing on a broad front without deep intercrystalline penetration. Electrocleaning in a hot-caustic bath was used to remove accumulated metal-oxidation products and sea-salt residues from Misco MDC-1 coated Inconel 713C and Mar M-200 specimens, and also from uncoated Inconel 713C specimens; however, it was found to be too severe for the Misco MDC-9 coated Inconel 713C specimens, and they were sonically cleaned in a water bath.

Exponential equations of weight-loss with time were developed which permit evaluating the effects of three levels of sulfur in fuel (< 0.0040 , 0.04 , and 0.40 weight per cent) and two levels of sea salt in air (zero and 1.0 ppm). The following conclusions can be drawn at a 95 per cent confidence level.

- A. In all comparisons, a significant decrease in the relative rate of corrosion in the absence of sea salt indicated that sea salt is a primary cause in hot corrosion of both coated and bare superalloys in a marine environment.
- B. In the absence of sea salt in air, a reduction of sulfur in fuel from 0.40 to either 0.040 or < 0.0040 weight per cent:
 - (a) significantly decreased the relative rate of corrosion on Misco MDC-1 coated Inconel 713C;
 - (b) had no significant effect on the relative rate of corrosion for Misco MDC-9 coated Inconel 713C but significantly increased the level of attack on this coated superalloy;
 - (c) had no significant effect on the relative rate of corrosion for Misco MDC-1 coated Mar M-200 but significantly decreased the level of attack on this coated superalloy; and
 - (d) had no significant effect on the relative rate of corrosion for uncoated Inconel 713C.

While statistically significant differences in the relative rates of corrosion were detected, the levels of attack were very low and may not be of practical importance.

- C. In the presence of 1.0 ppm sea salt in air, a reduction of sulfur in fuel from 0.40 to 0.040 weight per cent:
- (a) had no significant effect on the relative rate of corrosion for either Misco MDC-1 or MDC-9 coated Inconel 713C or MDC-1 coated Mar M-200; and
 - (b) had no significant effect on the relative rate of corrosion for uncoated Inconel 713C but significantly decreased the level of attack on this uncoated superalloy.

However, a further reduction of sulfur in fuel from 0.40 to <0.0040 weight per cent significantly decreased the relative rate of corrosion on both Misco MDC-1 and MDC-9 coated Inconel 713C, as well as on the uncoated Inconel 713C.

Results of this experimental investigation show that a reduction in sulfur content to 0.040 weight per cent would not reduce hot corrosion significantly. No change in the sulfur limit for JP-5 to alleviate hot corrosion of coated turbine alloys is recommended.

2.2. Vanadium

A screening program has been conducted, using a high-pressure burner rig, to evaluate the effect of vanadium on hot corrosion of a turbine-blade alloy in a marine environment. The evaluation was made using Inconel 713C specimens exposed at the 2000 F condition with 0.40 weight per cent sulfur in fuel. Tests were conducted with zero and 1.0 ppm sea salt in air and 0.063 ppm vanadium in air (equivalent to 3.8 ppm vanadium in fuel at the 2000 F condition) and with zero sea salt in air and 0.017 ppm vanadium in air (equivalent to 1.0 ppm vanadium in fuel at the 2000 F condition). Data from a previous investigation served for comparison. Visual appearance, specimen weight-loss and metallographic examinations were used in evaluating the effect of vanadium on hot corrosion. The following conclusions can be drawn from the data.

- A. From visual examination of the Inconel 713C specimens, an increase in attack was apparent with 0.063 ppm vanadium in air, but not with 0.017 ppm vanadium in air for up to the maximum exposure of 55 hours.
- B. Metallographic examination of representative specimens showed evidence of vanadium attack by penetration of the surface layer of depleted alloy; however, the vanadium attack did not progress along grain boundaries to a sufficient depth to preclude the use of weight-loss for evaluation although it may underestimate the severity of vanadium attack.
- C. The presence of 0.063 ppm vanadium in air increased specimen weight-loss significantly (95 per cent confidence level) with both zero and 1.0 ppm sea salt in air. Weight-loss in the presence of vanadium was 3.77 times as large in the absence of sea salt and 2.24 times as large in the presence of 1.0 ppm sea salt in air as in the absence of vanadium.

- D. The presence of 0.017 ppm vanadium in air had no significant effect on hot corrosion in the absence of sea salt in air. Weight-loss in the presence of 0.017 ppm vanadium in air was 1.04 times larger, but this increase was not statistically significant at a 95 per cent confidence level.

2.3. Effect of Temperature on Hot Corrosion

Experiments have been conducted, using a high-pressure burner rig, to examine 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) to aid in designing a program to evaluate the effect of sulfur in fuel on hot corrosion of coated superalloys.

The phase relationship for sodium sulfate over a range of temperatures and pressures was developed from thermodynamic equilibrium data. Predictions of the presence or absence of a condensed phase of sodium sulfate on test specimens at the various levels of test temperature agreed with data obtained by analysis of deposits.

No direct estimate of the exposure time required to measure coating life was obtained at the 1400 and 1600 F test conditions; however, indirect comparisons indicated that exposures of up to several hundred hours or more could be required under some conditions. From this investigation it was concluded.

- A. With the present cascade test section, mounting only six test specimens, the time required for the proposed program would be prohibitive and a revised test section should be designed to reduce the time required for the program.
- B. The condensation of sodium sulfate on the metal surface is not, as some investigators have proposed, a requisite for hot corrosion.
- C. Specimen weight-loss, in the presence of sulfur in fuel and sea salt in air, increased consistently and rapidly from a very low level with an increase in temperature from 1400 to 2000 F.

3. RECOMMENDATIONS

The primary objective of this investigation is to determine whether the 0.4 weight per cent sulfur currently allowed in grade JP-5 fuel is a safe level for the protection of turbine blades in aircraft engines of advanced design.

Programs have been completed which allowed an evaluation of the effect of sulfur concentration in fuel on the durability of bare superalloys (1). It was concluded that a reduction in sulfur content by an order of magnitude, to 0.04 weight per cent, would not reduce hot corrosion significantly. Therefore, it was recommended that no change in the sulfur limit for JP-5 be made to alleviate hot-corrosion attack on turbine blades.

That recommendation was tempered by another to extend this investigation to evaluate the effect of fuel sulfur on the protection afforded by coatings. Those data were sought because superalloys having the most desirable physical properties are more susceptible to hot corrosion and require coatings for satisfactory durability.

Programs have been completed which allow an evaluation of the effect of sulfur concentration in fuel on the durability of coated superalloys at 2000 F conditions. Our analysis of the data from these programs show that a reduction in fuel sulfur concentration from 0.40 to 0.040 weight per cent increased, decreased or had no effect on hot corrosion. This indicates that our previous recommendation based upon a study of bare superalloys, can be extended to coated superalloys.

The evaluations with coated superalloys have been confined to 2000 F exposure temperatures and the investigations should be extended to cover lower temperatures before a final recommendation is made. Also indications have been found that reductions in fuel sulfur concentrations to very low levels (<0.0040 weight per cent) reduce hot corrosion. Before making a final recommendation as to whether a reduction in fuel sulfur content would alleviate hot corrosion of bare and coated superalloys used in the hot section of turbine engines additional tests should be conducted over a range of temperatures and include one or more low levels of fuel sulfur.

It has been shown that the presence of 0.063 ppm vanadium in air (equivalent to 3.8 ppm vanadium in fuel at the 2000 F test conditions) more than doubles hot corrosion of Inconel 713C specimens with either zero or 1.0 ppm sea salt in air. The presence of 0.017 ppm vanadium in air (equivalent to 1.0 ppm vanadium in fuel at the 2000 F test conditions) did not significantly increase hot corrosion of Inconel 713C in the absence of sea salt in air. Additional data are needed before a firm conclusion can be drawn as to the effect of vanadium on hot corrosion of superalloys used in the hot section of gas turbine engines. The effect of 0.017 ppm vanadium in air in the presence of 1.0 ppm sea salt in air on hot corrosion of Inconel 713C, and the effect of 0.017 ppm vanadium in air with zero and 1.0 ppm sea salt in air on hot corrosion of several coating-alloy systems, should be obtained to complete the evaluation.

4. RESULTS AND DISCUSSION

4.1. Hot Corrosion of Coated Superalloys at 2000 F Condition

This experimental investigation was designed to permit comparisons showing the effect of sulfur concentration in JP-5 fuel on hot corrosion of coated superalloys in a marine environment. To characterize the principal levels of interest, data were obtained with three concentrations of sulfur in fuel (0.40, 0.040, and <0.0040 weight per cent), representing specification maximum, production median, and essentially sulfur-free fuels. A marine environment was simulated by the addition of sea salt to the air (1.0 ppm). Results have been reported previously (2) on the effect of sulfur on hot corrosion of bare Inconel 713C and Inconel 713C with Misco MDC-1 and MDC-9 coatings. During the present contract the investigation was extended to include Misco MDC-1 coated Mar M-200. To provide a summary of the effect of sulfur in fuel on hot corrosion of coated superalloys in a marine environment the previous data will be included along with that obtained during the present contract.

To simulate the environment in the turbine section of an aircraft engine, a cascade holder supporting six test specimens was mounted in the exhaust section of Phillips 2-inch combustor. The facility was operated to obtain exposure of the test specimens at high pressure (15 atmospheres), high temperature (2000 F), high velocity (745 feet per second), and realistic stoichiometry (60 air-fuel ratio). While 2000 F is a higher temperature than normally considered desirable for operation of engine-hot-section parts, it is not considered to be unrealistic when viewed from the standpoint of maximum temperature in the distribution encountered during engine operation with lower mean temperatures.

Further details concerning the test equipment, test materials, and test program are presented in Appendices 1, 2, and 3, respectively, which are Sections 8., 9., and 10. of this report.

No generally-accepted criterion is available for determining the effective life of a coating on a superalloy. Evaluations have been based on visual appearance of specimens, weight loss by specimens, and combinations of appearance and weight loss. During this investigation specimens were examined following exposure for visual evidence of the extent of attack; also, specimens were weighed to determine metal loss. While it is not characteristic of hot corrosion of superalloys, representative specimens were sectioned for metallographic examination to establish whether deep intercrystalline penetration of corrosion products had weakened the remaining metal.

4.1.1. Visual

Typically, the specimens are quite clean in the absence of sea salt; however, the ingestion of sea salt accelerated the attack and increased deposits. The deposits tended to flake-off the specimens during cooling to room temperature, indicating a significant difference in their coefficients of expansion. Analysis of deposits accumulated with sea salt present in the combustor exhaust gas showed them to be alloy oxidation.

products with little evidence of sea-salt residue or reaction products. This is as expected, for sodium sulfate should not be present in the condensed state to deposit on the specimens at these conditions, and is pertinent because it shows that condensed sodium sulfate is not a requisite for hot corrosion.

Following removal from the holder, specimens were cleaned for examination of the extent of attack. Photomicrographs (2X magnification) were taken of the impact face of specimens which had been exposed for 25 hours without sea salt and with 1.0 ppm sea salt in air, using three levels of sulfur in fuel (0.40, 0.040 and <0.0040 per cent). Representative uncoated Inconel 713C specimens are shown in Figure 4, Misco MDC-1 coated Inconel 713C in Figure 5, Misco MDC-9 coated Inconel 713C in Figure 6, and Misco MDC-1 coated Mar M-200 in Figure 7. It is evident that the coatings protect the superalloys from hot corrosion. Increased attack on both bare and coated specimens is evident in the presence of 1.0 ppm sea salt in air, over that in the absence of sea salt.

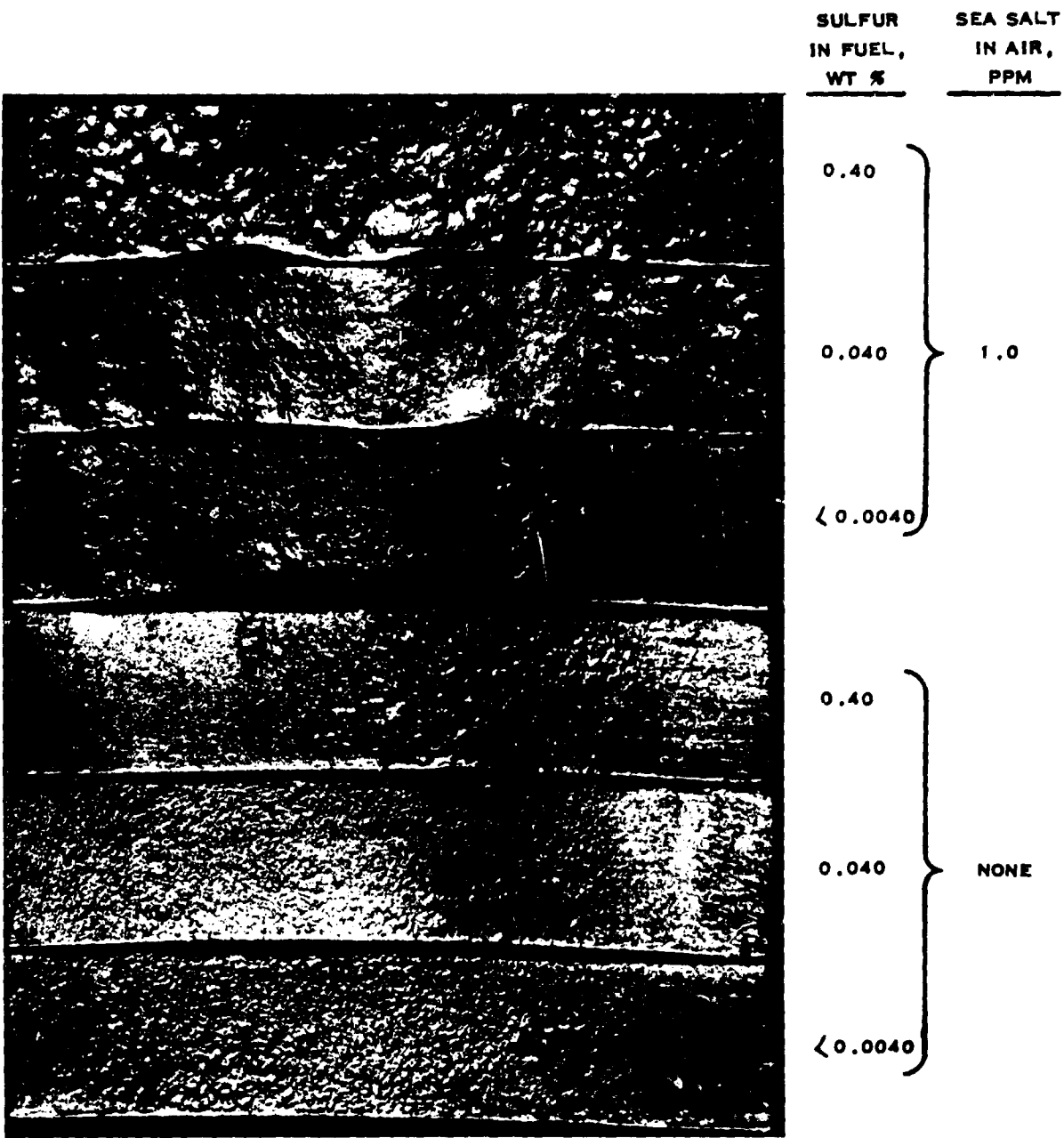
A systematic technique for numerically rating the extent of attack was applied in evaluating the condition of some coated specimens following exposure. Because visual rating is subjective and less accurate than measurement of weight loss, weight-loss data were used in evaluating the effect of sulfur in fuel and sea salt in air on hot corrosion.

The Misco MDC-9 coated Inconel 713C specimens developed visible surface cracks as the result of exposure to hot gases. Similar cracks were not observed with the Misco MDC-1 coated Inconel 713C or Mar M-200 specimens. The cracks usually developed on the leading edge of the specimens, and became more evident with increasing exposure time. This is illustrated in Figure 8 by the set of specimens accumulated from the test with medium-sulfur fuel. The accelerated rate of corrosion which accompanied the addition of sea salt to this environment appeared to have little effect on the occurrence and development of cracks.

4.1.2. Metallography

The mode and intensity of corrosive attack sustained by the superalloy and coatings was appraised by metallographic examination of selected specimens as received from Misco and following exposure over a range in conditions of this investigation. In particular, evidence of deep intercrystalline corrosion was sought to establish the validity of weight-loss data as a measure of metal damage.

Uncoated Inconel 713C - A metallographic cross-section of a typical uncoated Inconel 713C specimen as received from Misco is shown in Figure 9. This photomicrograph is included for reference purposes to emphasize that prior to exposure the specimens were free from any unusual surface scale or impurities. The uniformity of the etch, to the very edge of the coupon, indicates that the surface alloy was not depleted.



2X MAGNIFICATION AFTER ELECTRO-CLEANING

FIGURE 4
INCONEL 713C SPECIMENS AFTER 25 HOURS
AT 2000 F TEST CONDITION

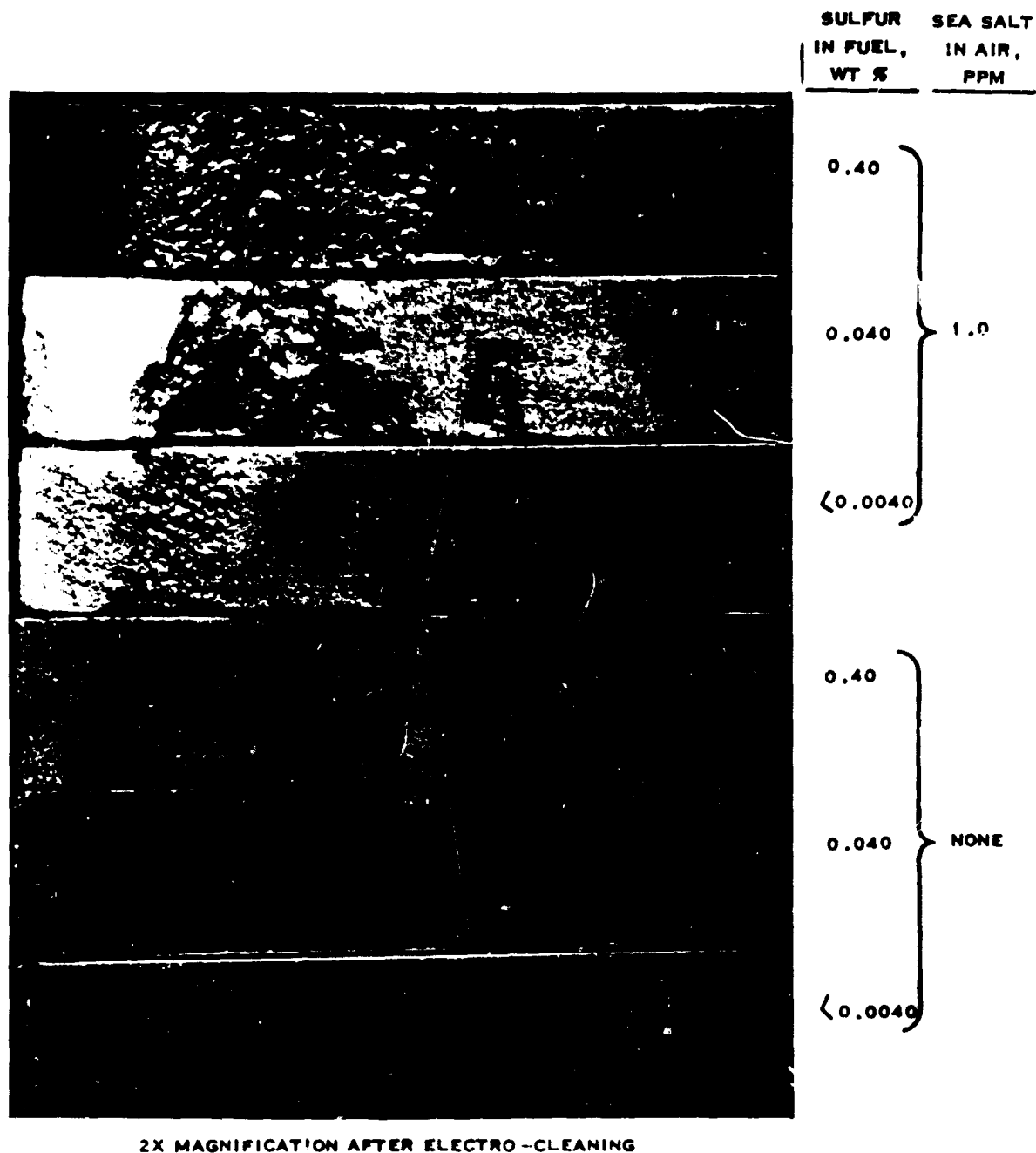


FIGURE 5
MISCO MDC-1 COATED INCONEL 713C SPECIMENS
AFTER 25 HOURS AT 2000 F TEST CONDITION

THIS ONE SPECIMEN WAS
ELECTRO-CLEANED TO SHOW
DEPOSIT-FREE SURFACE



SULFUR IN FUEL, WT %	SEA SALT IN AIR, PPM
----------------------------	----------------------------

0.40

0.040

<0.0040

0.40

0.040

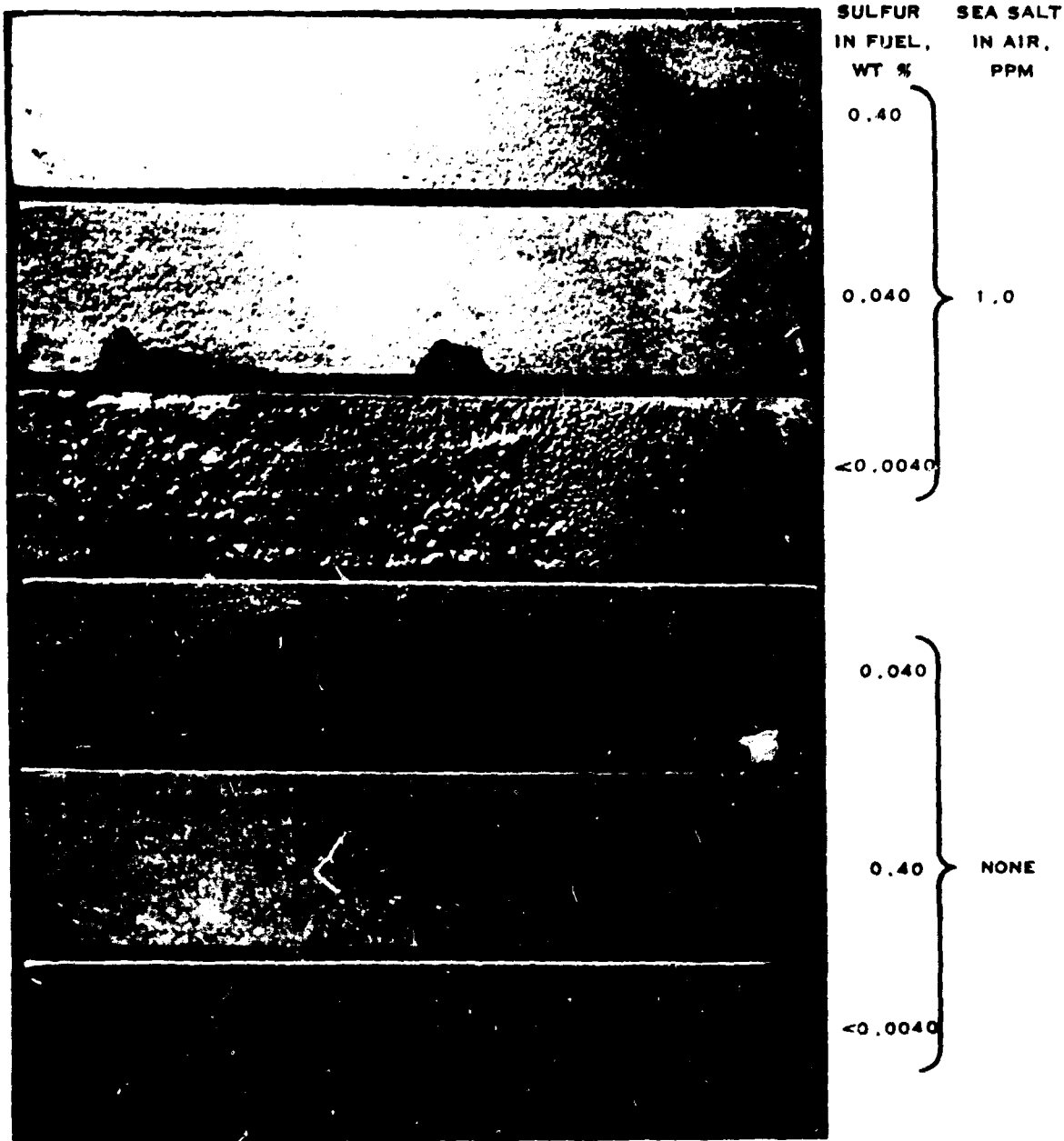
<0.0040

1.0

NONE

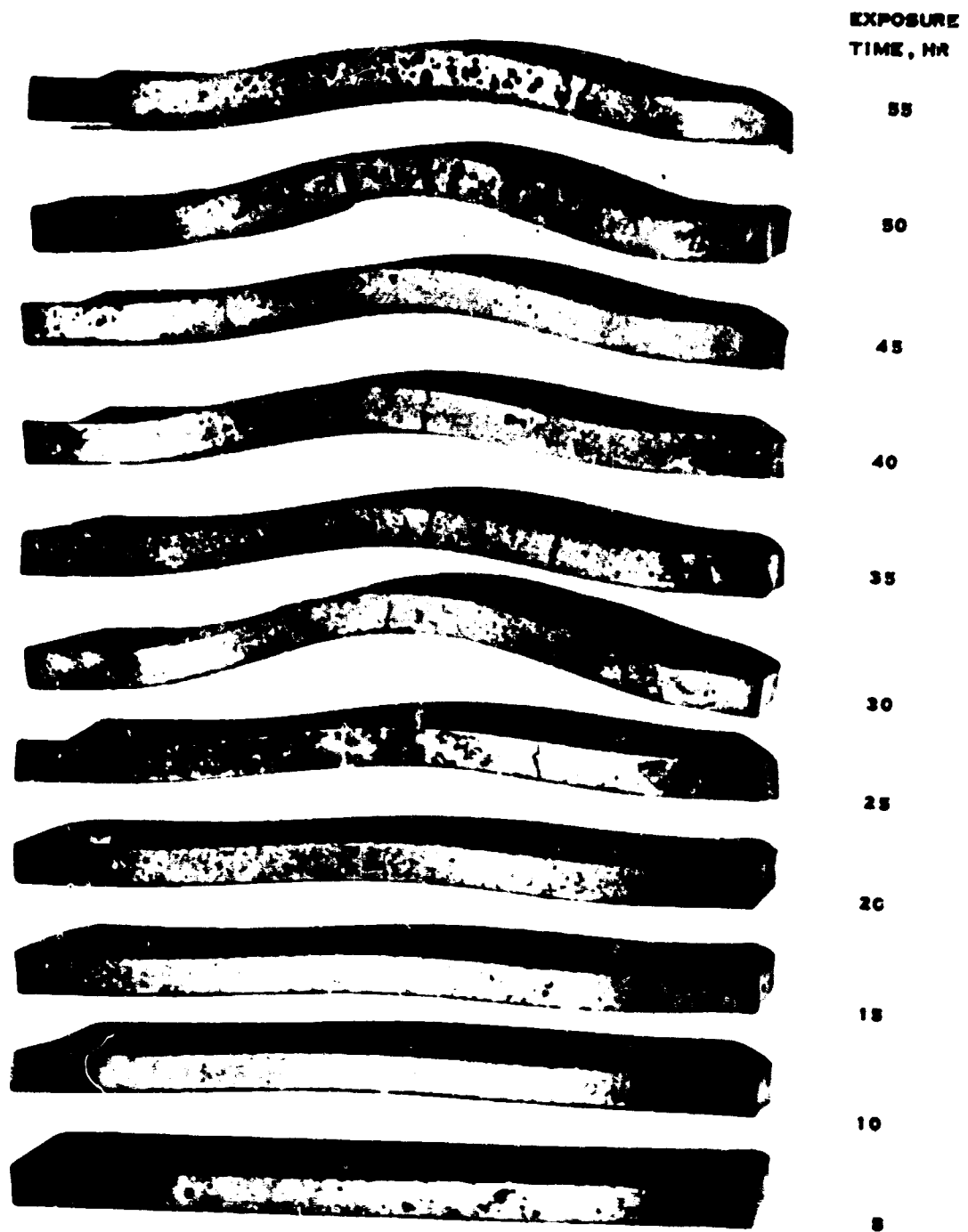
2X MAGNIFICATION AFTER SONIC-CLEANING

FIGURE 6
MISCO MDC-9 COATED INCONEL 713C SPECIMENS
AFTER 25 HOURS AT 2000 F TEST CONDITION



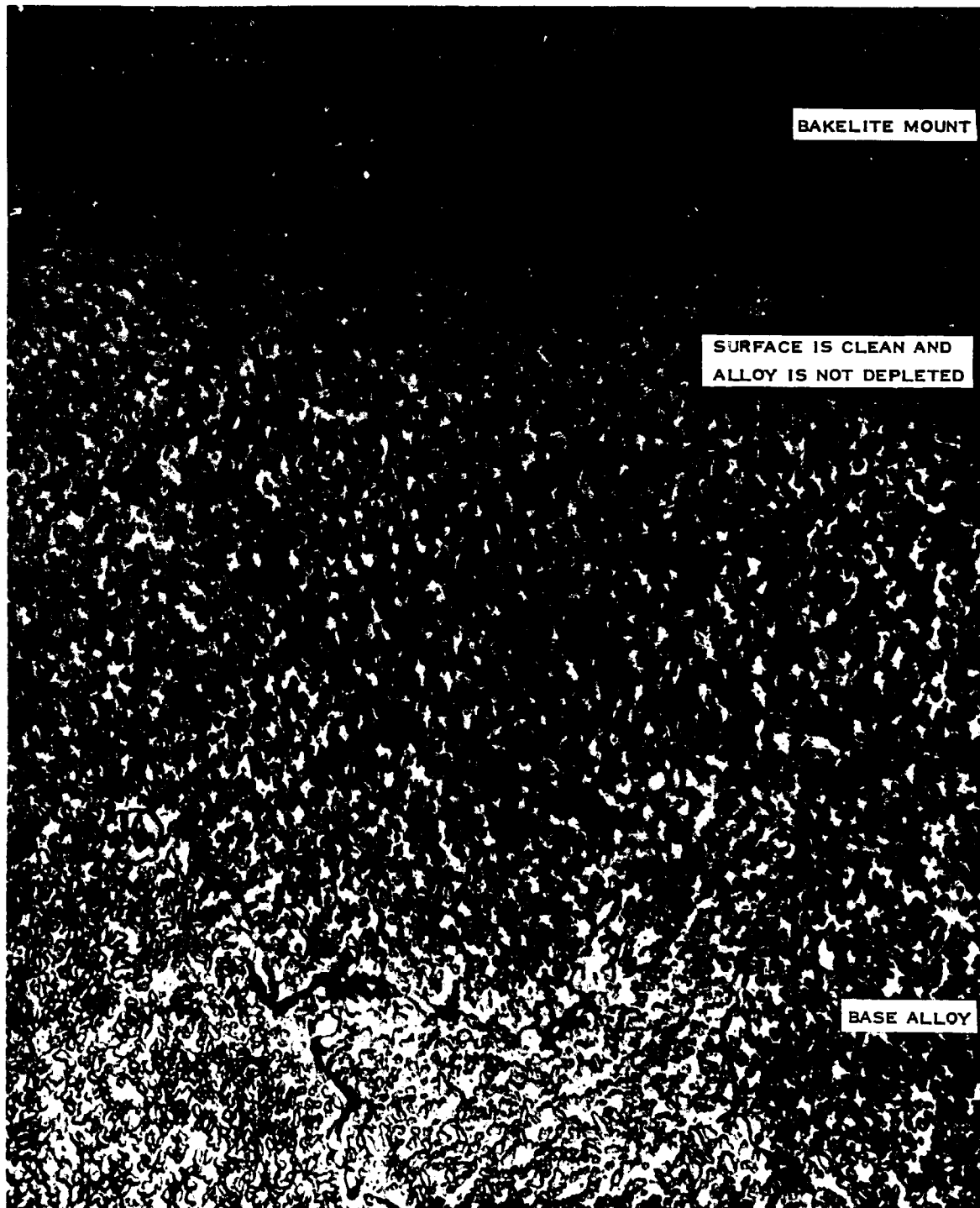
2X MAGNIFICATION AFTER ELECTRO-CLEANING

FIGURE 7
MISCO MDC-1 COATED MAR M200 SPECIMENS
AFTER 25 HOURS AT 2000F TEST CONDITION



2X MAGNIFICATION OF SONICALLY-CLEANED SPECIMENS
AFTER EXPOSURE AT 2000 F TEST CONDITION
WITH NO SEA SALT IN AIR AND 0.040 WT % SULFUR IN FUEL

FIGURE 8
DEVELOPMENT OF CRACKS IN LEADING EDGE
OF MISCO MDC-9 COATED INCONEL 713C SPECIMENS



METALLOGRAPHIC CROSS-SECTION. MARBLE'S REAGENT ETCHED. 1000X MAGNIFICATION.

FIGURE 9
UNEXPOSED INCONEL 713C SPECIMEN

Exposure of uncoated Inconel 713C in an environment free of sea salt resulted in only a slight, uniform surface oxidation. This was preceded by a limited zone of alloy depletion, which increased in depth with increasing exposure time. The addition of sulfur, via the fuel, to this environment (2000 F) did not result in the formation of metal sulfides at the corrosion interface, nor in accelerated oxidation attack on the depleted surface alloy, as illustrated in Figure 10. The similarity between the micro-features of specimens exposed in Phillips test rig can be seen by comparison with Figure 1. When making this comparison, it should be remembered that the heavy surface scale has been removed from the test specimen by electrocleaning.

With the addition of sea salt, via the air, to this environment, characteristic hot corrosion was encountered on uncoated Inconel 713C. Only the extent of the attack increased with increasing exposure time. The further addition of sulfur, via the fuel, to this sea salt environment affected neither the mode nor the intensity of the attack, which is shown in Figure 11. The micro-features of hot corrosion on Inconel 713C specimens exposed in Phillips test rig are quite similar to those evident on Inconel 713C turbine blades from service engines, which can be seen by comparison with Figure 2.

Misco MDC-1 Coated Inconel 713C - A metallographic cross-section of a typical MDC-1 coated Inconel 713C specimen as received from Misco is shown in Figure 12. The aluminum-type coating had a total depth of approximately 2 mils, which is divided about equally between an outer-layer with non-metallic dispersions and a diffusion-layer. Previous experience (1) has shown that the appearance of this coating is not altered by the electrocleaning procedure used to remove deposits and scale following exposure.

In those areas where the MDC-1 coating remained intact following exposure, evidence was found of alloy depletion in the diffused layer, as shown in Figure 13. This modification of the coating remnant became increasingly evident with increasing exposure time, but was not favored by any other exposure variable in this program.

Once the MDC-1 coating was penetrated, the mode and intensity of attack observed was similar to that experienced by the bare Inconel 713C at comparable conditions of exposure. Characteristic micro-features of hot corrosion were found when the coating was penetrated by prolonged exposure in the presence of sea salt, as shown in Figure 14.

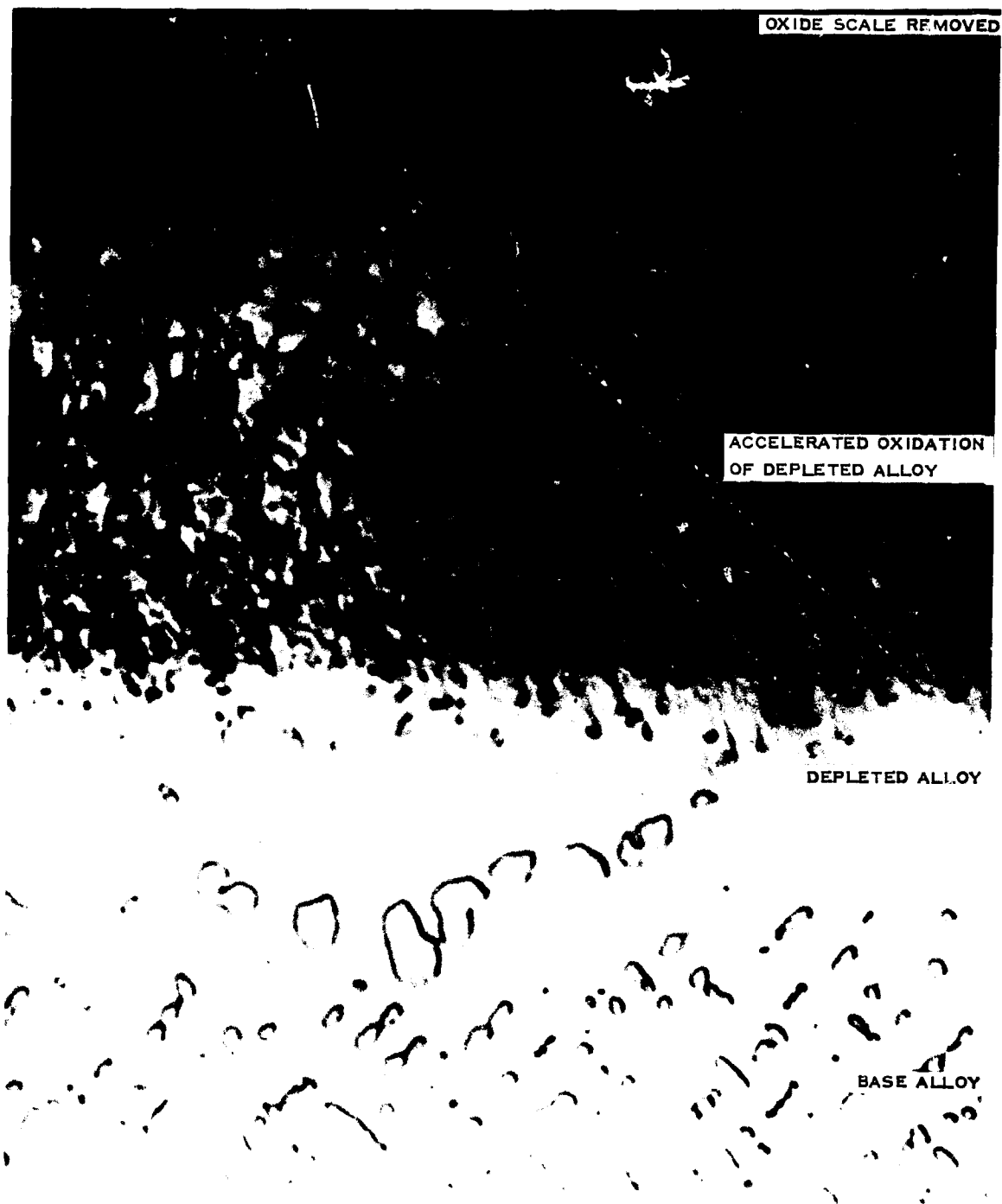
Misco MDC-9 Coated Inconel 713C - A metallographic cross-section of a typical MDC-9 coated Inconel 713C specimen as received from Misco is shown in Figure 15. The aluminum-chromium type coating is about 2 mils thick and is divided about equally between an outer-layer and a diffused-layer.

Specimens of Inconel 713C were inspected by Misco using fluorescent penetrant (Zyglo) and certified to be free from cracks. Similar inspection by Phillips following application of the MDC-9 coating by Misco revealed no cracks. However, it is of interest to note that a few cracks were found in coating of a new, unexposed, specimen when sectioned for metallographic study. One such crack is shown in Figure 15, and it is pertinent to point out that characteristically the crack does not extend into the base alloy.



METALLOGRAPHIC CROSS-SECTION OF INCONEL 713C SPECIMEN AFTER 55 HOURS AT 2000F TEST CONDITION.
ELECTRO-CLEANED. MARBLE'S REAGENT ETCHED. 1000X MAGNIFICATION.

FIGURE 10
SURFACE OXIDATION



METALLOGRAPHIC CROSS-SECTION OF INCONEL 713C SPECIMEN AFTER 25 HOURS AT 2000F TEST CONDITION WITH 1.0 PPM SEA SALT IN AIR. ELECTRO-CLEANED. CARPELLA'S REAGENT ETCHIED. 4000X MAGNIFICATION.

FIGURE 11
ACCELERATED SURFACE OXIDATION



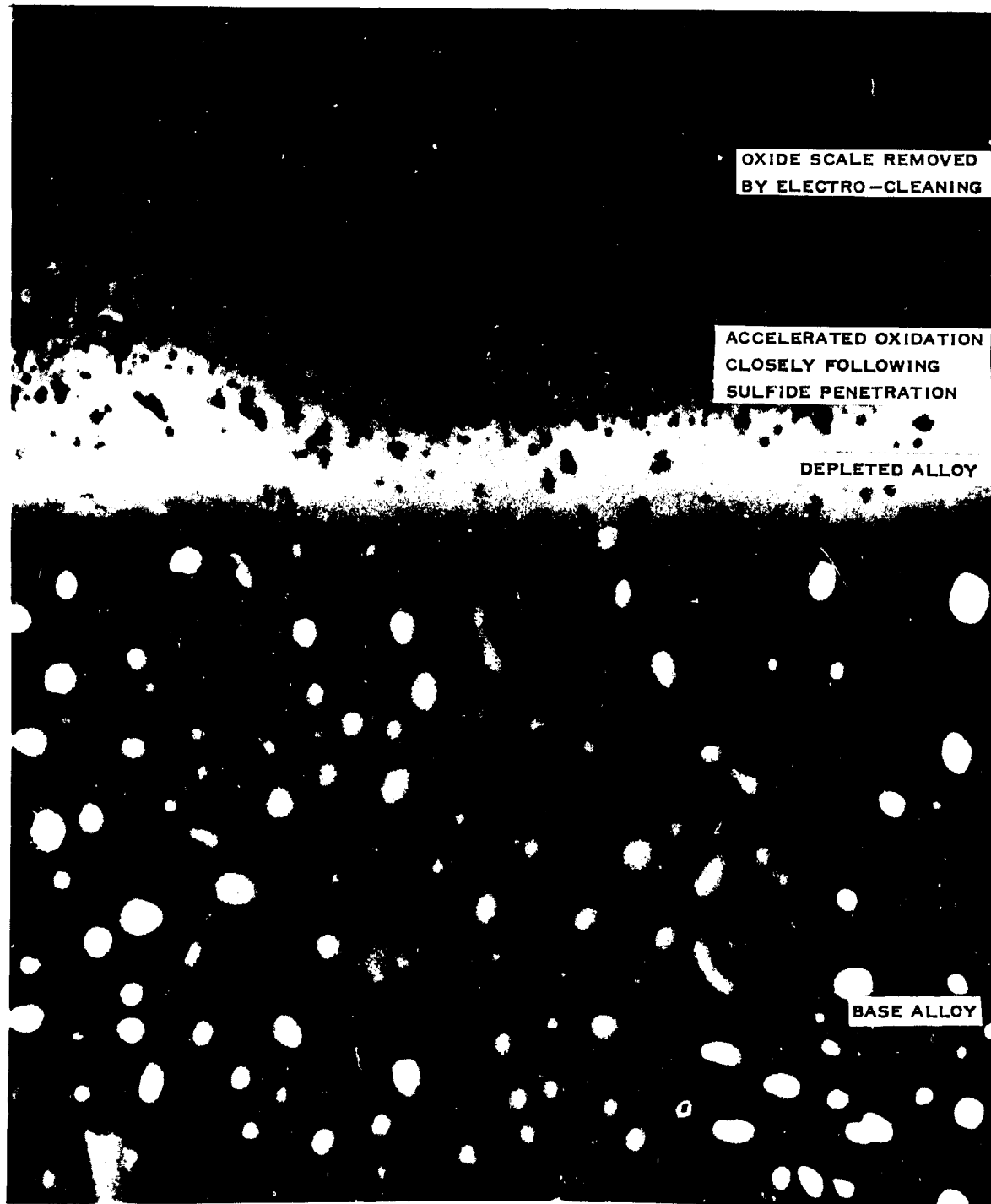
METALLOGRAPHIC CROSS-SECTION, 3% SULFURIC ACID-ELECTROLYTIC ETCHED,
1000X MAGNIFICATION.

FIGURE 12
UNEXPOSED MISCO MDC-1 COATED INCONEL 713C SPECIMEN



METALLOGRAPHIC CROSS-SECTION OF SPECIMEN AFTER 20 HOURS AT 2000 F TEST
CONDITION WITH 1.0 PPM SEA SALT IN AIR AND 0.040 WT % SULFUR IN FUEL.
2% CHROMIC ACID-ELECTROLYTIC ETCHED. 1000X MAGNIFICATION.

FIGURE 13
DEPLETION OF MISCO MDC-1 COATED INCONEL 713C SPECIMEN



METALLOGRAPHIC CROSS-SECTION OF SPECIMEN AFTER 20 HOURS AT 2000 F TEST
CONDITION WITH 1.0 PPM SEA SALT IN AIR AND 0.040 WT % SULFUR IN FUEL,
2% CHROMIC ACID-ELECTROLYTIC ETCHED. 3000X MAGNIFICATION.

FIGURE 14
ACCELERATED OXIDATION OF MISCO MDC-1 COATED INCONEL 713C SPECIMEN



METALLOGRAPHIC CROSS-SECTION, 3% SULFURIC ACID-ELECTROLYTIC ETCHED,
1000X MAGNIFICATION,

FIGURE 15
UNEXPOSED MISCO MDC-9 COATED INCONEL 713C SPECIMEN

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It is speculated that these cracks were developed in the coating when the specimens were sectioned for the metallographic examination, and do not represent the as-received specimen condition. Indications are that the MDC-9 coating as applied by Misco to the Inconel 713C specimens used in this program was subject to cracking under stress.

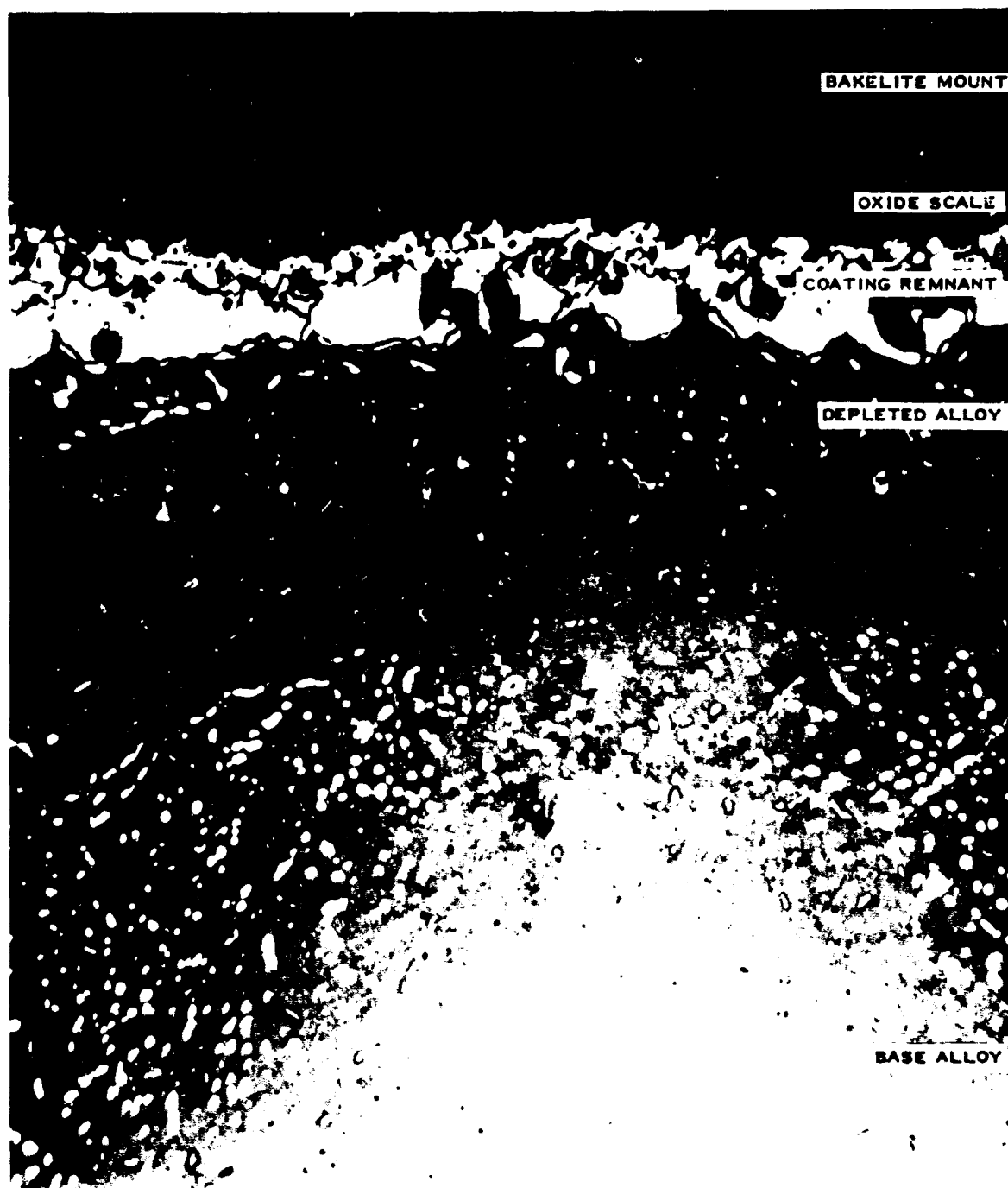
In those areas where the MDC-9 coating remained intact following exposure, evidence was found of alloy depletion in the diffused layer, as shown in Figure 16. This modification of the coating remnant is comparable to that experienced with the Misco MDC-1 coated Inconel 713C specimens, as previously described. Similarly, it was concluded that once the MDC-9 coating was penetrated, the corrosive attack was like that experienced by the bare Inconel 713C at comparable conditions of exposure.

In those areas where the MDC-9 coating was penetrated by hot corrosion the mode and intensity of attack was similar to that observed with bare Inconel 713C, as already described. Thus, it was surprising to find little or no evidence of sulfide penetration associated with the cracks which were found in the Misco MDC-9 coated Inconel 713C specimens following exposure. While there was evidence of alloy depletion around the cracks, as shown in Figure 17, it was not accompanied by the accelerated oxidation associated with hot corrosion. This indicates that the cracks do not serve as a focal point for the attack by corrosive materials over the range in conditions of exposure investigated.

Misco MDC-1 Coated Mar M-200 - The uniformity and thickness of aluminum-diffusion coating was appraised by metallographic examination of several specimens, as received from Misco. Similarly, the mode and intensity of corrosive attack was evaluated following exposure over the range in conditions of this investigation.

A metallographic cross-section of a satisfactory MDC-1 coated Mar M-200 specimen as received from Misco is shown in Figure 18. The aluminum-type coating has a total depth of approximately 2 mils, which is divided about equally between an outer-layer with non-metallic dispersions and a diffusion-layer. Unfortunately, the coating thickness varied from approximately 0.5 to 2.0 mils on the unexposed specimens. A typical example of defective coating is shown in Figure 19. This variability in coating thickness could easily account for the relatively large experimental error found with this coating-alloy system.

It is not known whether the variability on coating thickness is a characteristic of the coating-alloy system, or the specific heat of Mar M-200, or the specific pack of MDC-1. Consultation with Misco revealed that their experience with the application of MDC-1 coating to Mar M-200 was very limited; however, they felt that the variability in coating thickness was not a characteristic of the coating-alloy system. Also, they did not think that it could be attributed to segregation in alloy composition during casting. Rather, they felt that it probably was the result of uneven heating of the pack during the vapor-coating process. This suspicion is based upon the use of a small-research furnace for coating our specimens.



METALLOGRAPHIC CROSS-SECTION OF SPECIMEN AFTER 25 HOURS AT 2000 F TEST
CONDITION WITH 1.0 PPM SEA SALT IN AIR AND 0.040 WT % SULFUR IN FUEL.
2% CHROMIC ACID-ELECTROLYTIC ETCHED. 400X MAGNIFICATION.

FIGURE 16
DEPLETION OF MISCO MDC-9 COATED INCCNEL 713C SPECIMEN



METALLOGRAPHIC CROSS-SECTION OF SPECIMEN AFTER 25 HOURS AT 2000 F TEST
CONDITION WITH 1.0 PPM SEA SALT IN AIR AND 0.040 WT % SULFUR IN FUEL.
3% SULFURIC ACID-ELECTROLYTIC ETCHED. 400X MAGNIFICATION.

FIGURE 17
CRACK IN MISCO MDC-9 COATED INCONEL 713C SPECIMEN



METALLOGRAPHIC CROSS-SECTION. 3% SULFURIC ACID-ELECTROLYTIC ETCHED.
2000X MAGNIFICATION.

FIGURE 18
UNEXPOSED MISCO MDC-1 COATED MAR M-200 SPECIMEN



METALLOGRAPHIC CROSS-SECTION OF UNEXPOSED SPECIMEN,
3% SULFURIC ACID-ELECTROLYTIC ETCHED, 2000X MAGNIFICATION.

FIGURE 19
DEFECTIVE MISCO MDC-1 COATING ON MAR M-200 SPECIMEN

Field experience by the Navy has indicated that variability in coating thickness on turbine blades is not unusual, and is a production problem. In this light, the data obtained from our investigation with this coating-alloy system has been treated as valid.

The fact that the appearance of MDC-1 coating is not altered by the electro-cleaning procedure used to remove deposits and scale was confirmed by metallographic examination of cross-section of coupons from several MDC-1 coated Mar M-200 specimens which had been electro-cleaned.

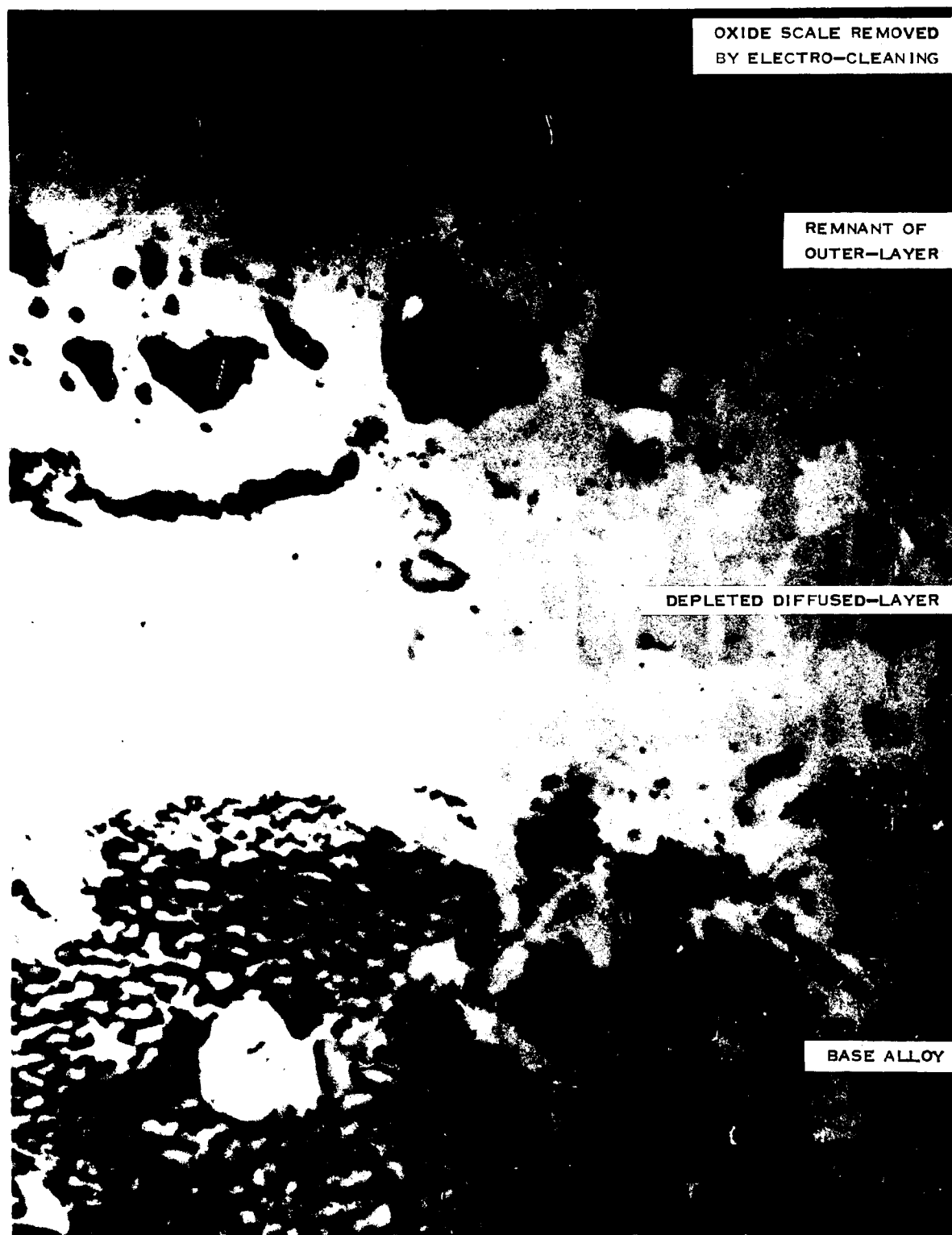
In those areas where the MDC-1 coating remained intact following exposure, evidence was found of gradual diffusion of the aluminum into the base alloy. This depletes the diffused-layer and increases its depth, as shown in Figure 20. This modification of the coating remnant became increasingly evident with increasing exposure time. It was not favored by any other exposure variable in this program.

Exposure of Misco MDC-1 coated Mar M-200 in an environment free of sea salt resulted in only slight, uniform, surface oxidation. In some areas oxide penetration progressed into the depleted, diffused layer of the coating, as shown in Figure 21. A few instances of complete coating failure were observed, as shown in Figure 22, with surface oxidation preceded by a limited zone of alloy depletion. In this environment (2000 F) with the high-sulfur fuel (0.40 wt %) no evidence was found of sulfide penetration in either the depleted diffused-layer of the coating (Figure 21) or the depleted alloy (Figure 22), despite oxide penetration.

With the addition of sea salt, via the air, to this environment, characteristic hot corrosion occurred. Typical attack on the MDC-1 coating is shown in Figure 23; separation of the outer-layer from the diffused layer by corrosion products frequently resulted in loss of the outer-layer of the coating. Also, penetration of the diffused-layer of the coating by randomly-dispersed, light-grey globules of metallic sulfides was usual (Figure 23). The addition of sulfur, via the fuel, to this environment did not preceptively alter the attack.

In those areas where the MDC-1 coating was penetrated by hot corrosion, the mode and intensity of attack was similar to that observed on the bare alloy. In general, the attack was less severe on the rear-face of the specimens, which is shown in Figure 24. The accelerated-oxidation attack, which is typical of hot corrosion, was evident on the impact-face of specimens, which is shown in Figure 25. Characteristically, the attack advances on a broad front without deep-intercrystalline penetration of sulfides or oxides.

Limited evidence of intercrystalline oxidation, to a depth of up to 8 mils, was found in a few specimens at the point of flexure from support by the holder, as shown in Figure 26. This photomicrograph is presented to illustrate the type of attack which would vitiate the use of loss in weight by the specimens as a measure of metal damage. However, such intercrystalline corrosion was not found to be extensive enough under the conditions of our investigation to be of practical importance.



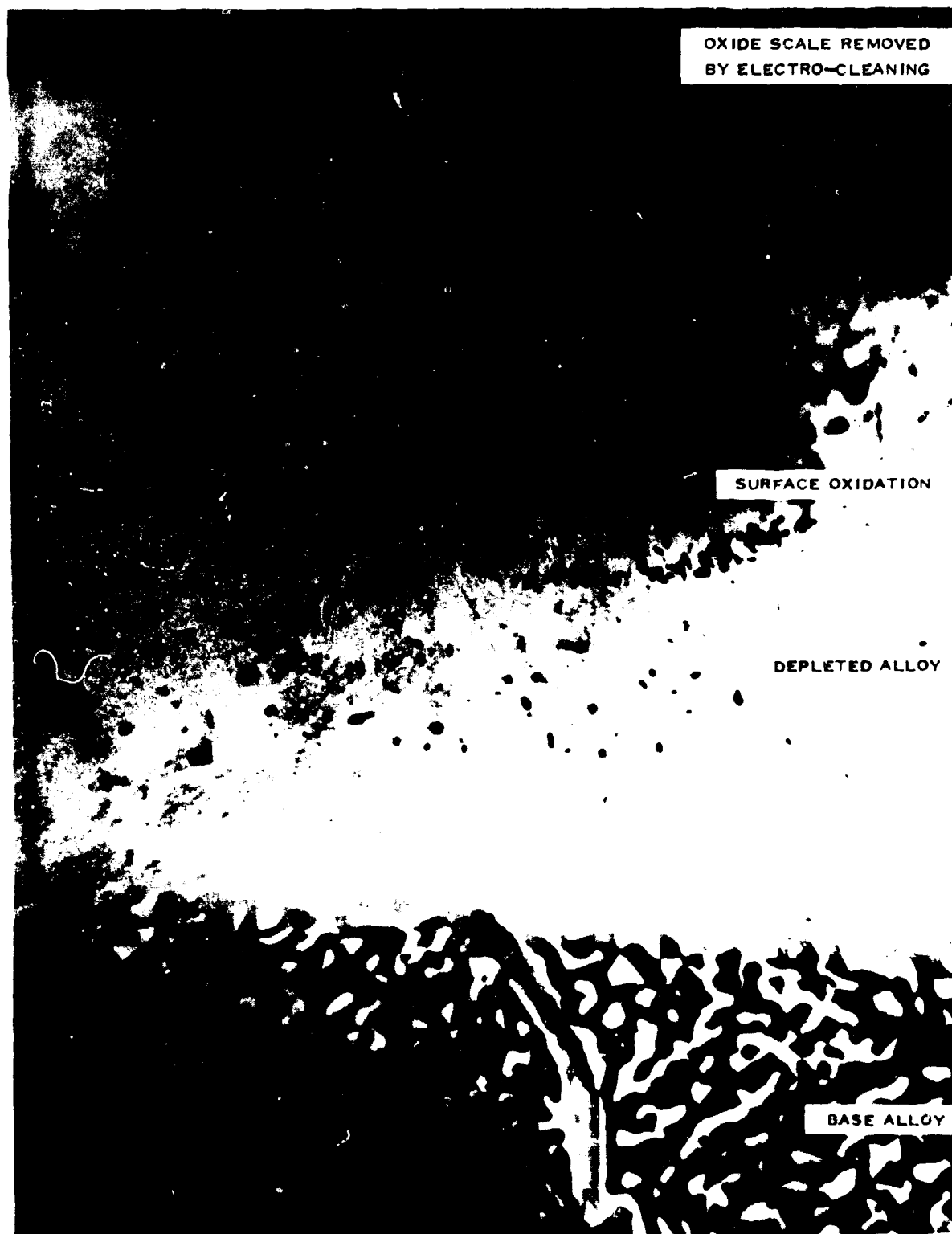
METALLOGRAPHIC CROSS-SECTION OF SPECIMEN AFTER 50 HOURS AT 2000 F TEST CONDITION
WITH NO SEA SALT IN AIR AND <0.0040 WT % SULFUR IN FUEL.
3% SULFURIC ACID-ELECTROLYTIC ETCHED. 2000X MAGNIFICATION.

FIGURE 20
DEPLETION OF MISCO MDC-1 COATING ON MAR M-200 SPECIMEN



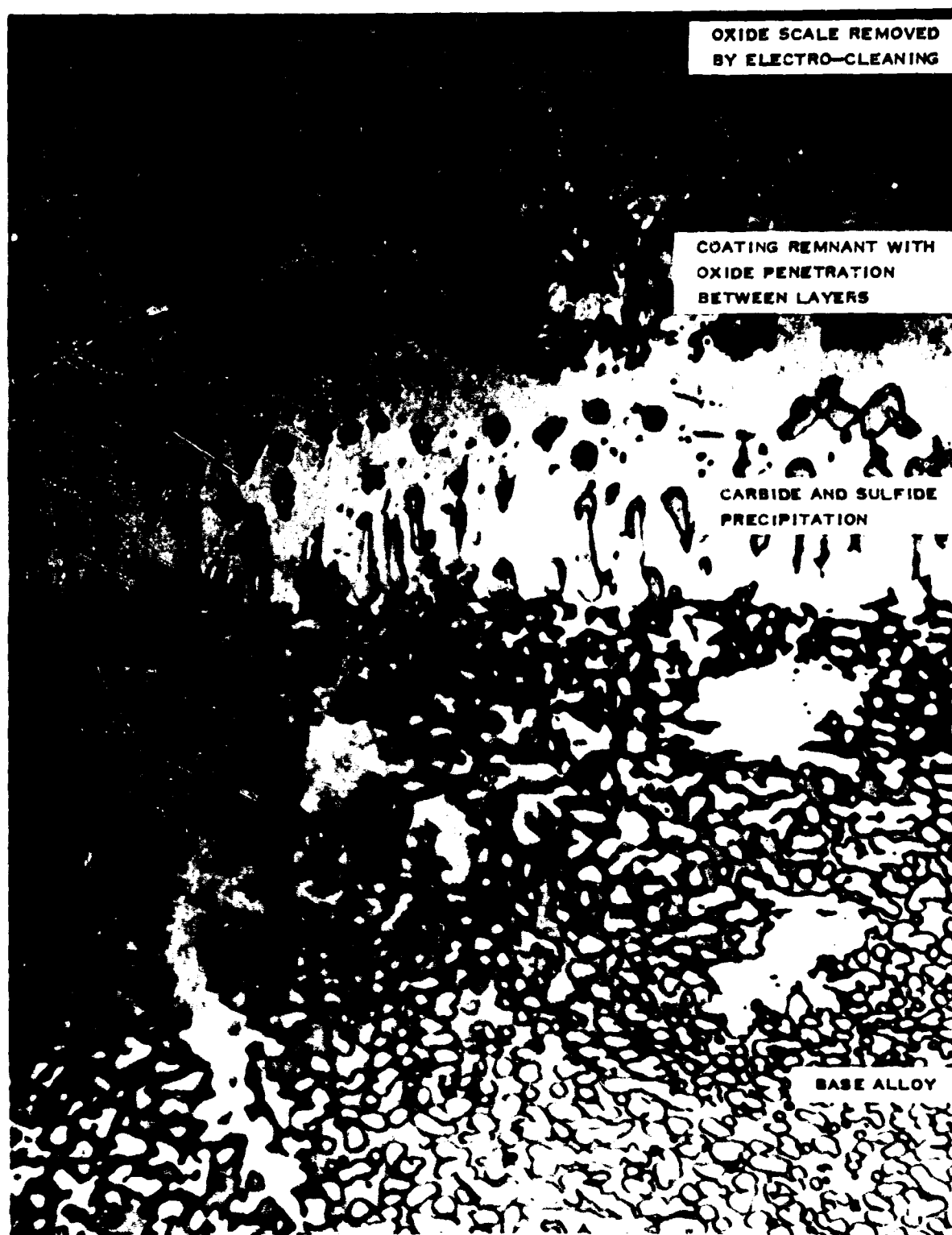
METALLOGRAPHIC CROSS-SECTION OF SPECIMEN AFTER 45 HOURS AT 2000 F TEST CONDITION
WITH NO SEA SALT IN AIR AND 0.40 WT % SULFUR IN FUEL.
3% SULFURIC ACID-ELECTROLYTIC ETCHED. 2000X MAGNIFICATION.

FIGURE 21
OXIDATION OF MISCO MDC-1 COATING ON MAR M-200 SPECIMEN



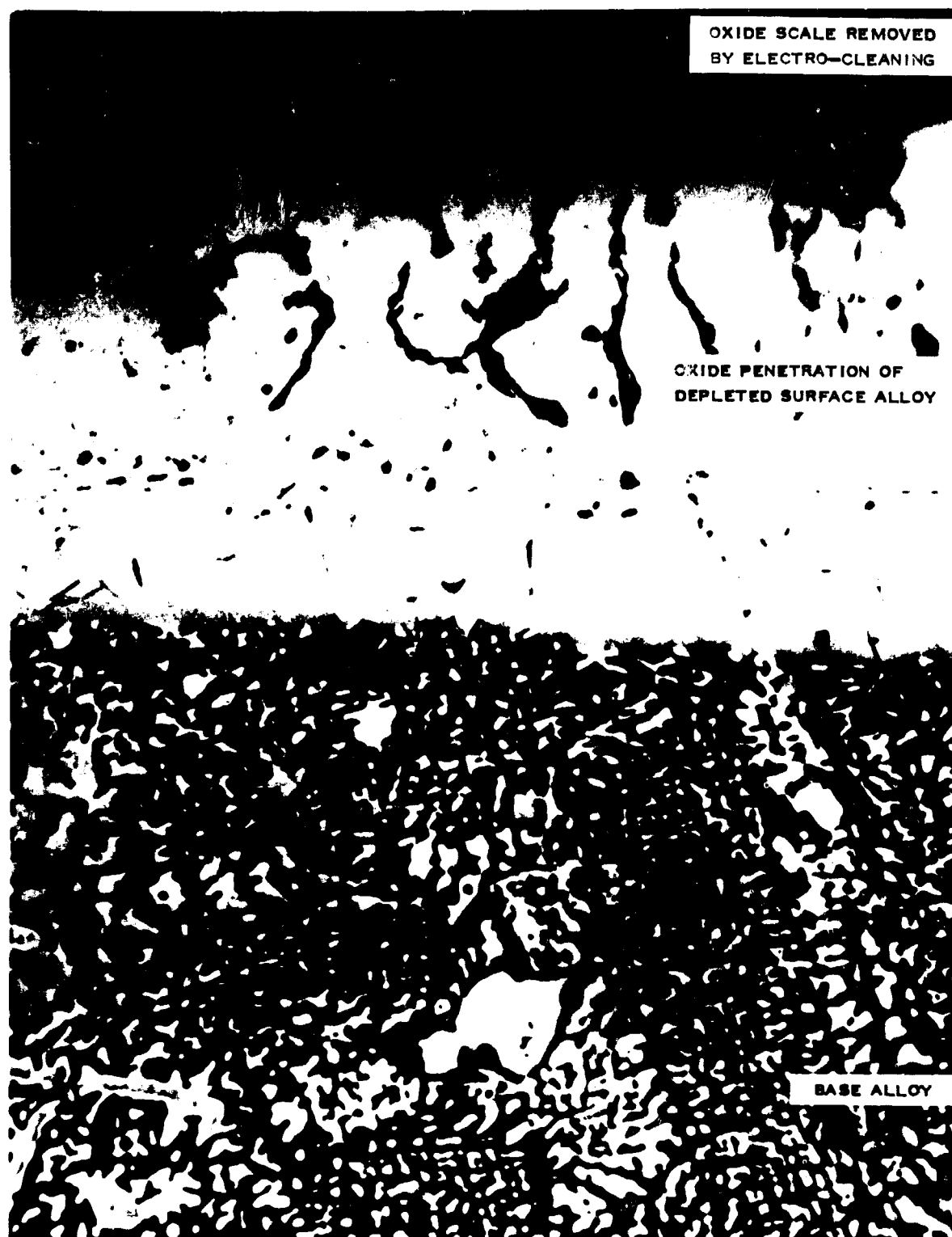
METALLOGRAPHIC CROSS-SECTION OF SPECIMEN AFTER 50 HOURS AT 2000 F TEST CONDITION
WITH NO SEA SALT IN AIR AND 0.40 WT % SULFUR IN FUEL.
3% SULFURIC ACID-ELECTROLYTIC ETCHED. 2000X MAGNIFICATION.

FIGURE 22
OXIDATION OF MISCO MDC-1 COATED MAR M-200 SPECIMEN



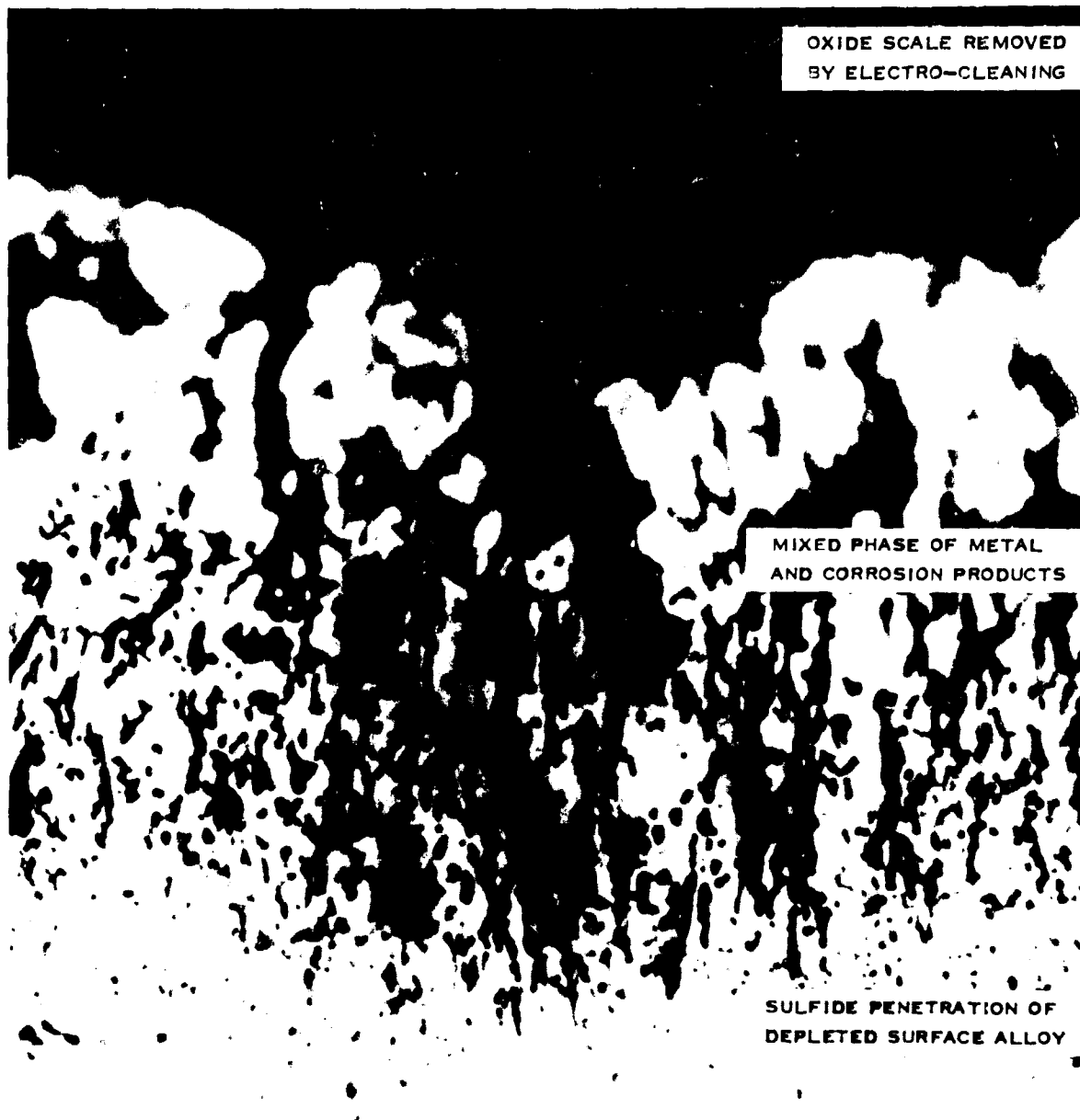
METALLOGRAPHIC CROSS-SECTION OF SPECIMEN AFTER 50 HOURS AT 2000 F TEST CONDITION
WITH 1 PPM SEA SALT IN AIR AND <0.0040 WT % SULFUR IN FUEL.
3% SULFURIC ACID-ELECTROLYTIC ETCHED. 2000X MAGNIFICATION.

FIGURE 23
OXIDATION OF MISCO MDC-1 COATING ON MAR M-200 SPECIMEN



METALLOGRAPHIC CROSS-SECTION OF SPECIMEN AFTER 45 HOURS AT 2000 F TEST CONDITION
WITH 1 PPM SEA SALT IN AIR AND 0.40 WT % SULFUR IN FUEL,
3% SULFUR ACID-ELECTROLYTIC ETCHED. 2000X MAGNIFICATION.

FIGURE 24
OXIDATION OF MISCO MDC-1 COATED MAR M-200 SPECIMEN



METALLOGRAPHIC CROSS-SECTION OF SPECIMEN AFTER 25 HOURS AT 2000 F TEST CONDITION
WITH 1 PPM SEA SALT IN AIR AND 0.40 WT % SULFUR IN FUEL.
3000X MAGNIFICATION.

FIGURE 25
ACCELERATED OXIDATION OF MISCO MDC-1 COATED MAR M-200 SPECIMEN



METALLOGRAPHIC CROSS-SECTION OF SPECIMEN AFTER 45 HOURS AT 2000 F TEST CONDITION
WITH 1 PPM SEA SALT IN AIR AND 0.4 WT % SULFUR IN FUEL.
3% SULFURIC ACID-ELECTROLYTIC ETCHED. 500X MAGNIFICATION.

FIGURE 26

INTERCRYSTALLINE OXIDATION OF MISCO MDC-1 COATED MAR M-200 SPECIMEN

Several specimens which had been exposed under comparable conditions but differed greatly in weight loss, were examined. No basis differences in either the coating or base alloy were observed which would explain their response.

In general, the corrosive attack on the superalloy and coatings during these investigations resulted in only shallow penetration of corrosion products, despite the catastrophic rates of metal loss in the presence of sea salt. The depth of surface penetration was usually less than one mil. This serves to justify the use of the metal-loss data as a valid measurement of the extent of corrosive attack on the superalloy and coatings over the range of conditions investigated.

4.1.3. Weight Loss

The weight-loss per unit area for specimens of the four coating-alloy systems (Inconel 713C, MDC-1 and MDC-9 coated Inconel 713C and MDC-1 coated Mar M-200) resulting from exposure at the 2000 F test condition, without sea salt and with 1.0 ppm sea salt in air and with three levels of sulfur in fuel (0.40, 0.040 and <0.0040 per cent), are presented in Table 1. In general, weight-loss data are available at 5-hour intervals for exposure durations up to 55 hours; however, some tests were stopped short because the severity of attack made completion of 55 hours improbable without loss of specimens. In those cases duplicate specimens are available for some exposure times because of the plan followed in loading the cascade with specimens during a test.

The weight-loss data for MDC-9 coated Inconel 713C specimens after exposure and sonic-cleaning were adjusted by the addition of 1.0 mg/cm² to eliminate negative weight losses, and these values were used in our analyses.

In evaluating oxidation with time, many forms of equations have been proposed and used in the literature to express the relationship. These forms include linear, cubic, parabolic, and exponential equations depending upon the system being investigated. Data from this investigation were plotted using linear coordinates and an example is shown in the lower portion of Figure 27 for Misco MDC-1 coated Inconel 713C exposed to 0.040 weight per cent sulfur in fuel and 1.0 ppm sea salt in air. The line is a visual fit of the data. The shape of the curve suggests the use of the exponential growth curve which is commonly used in the form

$$W = (A)(B)^X, \quad (1)$$

where W is weight loss per unit area, X is exposure time, and A and B are constants to be evaluated. Applying logarithms to the equation,

$$\log W = \log A + (\log B) X \quad (2)$$

$$\text{or } Y = a + bX, \quad (3)$$

where $Y = \log W$, $a = \log A$, and $b = \log B$. Equation 3 is the equation for a straight line. The data in the upper portion of Figure 27 were fitted using this form of equation and the calculated line is shown. The fit of these data by Equation 3 indicates that the logarithm of weight-loss increases at a uniform rate over the time-period studied. Similar fits were obtained with other data from the current investigation.

TABLE 1TEST SPECIMEN WEIGHT LOSS DATA

Total Time, hours	Weight Loss, mg/cm ²								
	<u>Zero Sea Salt in Air</u>			<u>1.0 ppm Sea Salt in Air</u>					
	<u>Sulfur in Fuel, wt %</u>			<u>Sulfur in Fuel, wt %</u>					
	<u><0.0040</u>	<u>0.040</u>	<u>0.40</u>	<u><0.0040</u>		<u>0.040</u>		<u>0.40</u>	
				<u>Run 1</u>	<u>Run 2</u>	<u>Run 1</u>	<u>Run 2</u>	<u>Run 1</u>	<u>Run 2</u>
<u>Inconel 713C</u>									
5	3.78	5.07	5.65	8.67	-	15.41	-	27.67	41.85
5	-	-	-	-	-	15.64	-	28.63	66.14
10	8.88	10.05	13.76	21.63	-	47.52	-	73.10	-
10	-	-	-	-	-	19.72	-	132.31	-
15	15.30	17.91	15.52	57.03	-	85.91	-	146.88	-
15	-	-	-	67.66	-	70.72	-	134.69	-
20	18.35	25.38	24.71	71.97	-	95.84	-	182.85	-
20	-	-	-	72.40	-	115.74	-	199.50	-
25	23.08	42.58	30.92	100.93	-	242.25	-	366.48	-
25	-	24.81	-	114.50	-	220.54	-	299.46	-
30	42.29	30.35	46.82	93.40	-	203.15	-	-	-
35	46.90	21.12	31.55	130.37	-	-	-	-	-
40	91.13	35.64	68.92	127.11	-	-	-	-	-
45	43.35	68.16	48.35	-	-	-	-	-	-
50	74.81	67.61	80.93	-	-	-	-	-	-
55	62.63	-	81.67	-	-	-	-	-	-

MDC-1 Coated Inconel 713C

5	2.19	2.05	1.90	2.79	8.55	2.29	3.50	2.13	2.34
5	-	-	-	-	2.95	-	-	-	2.87
10	1.57	1.86	1.44	2.03	12.56	2.28	3.06	2.77	2.98
10	-	-	-	-	4.80	-	-	-	3.09
15	1.74	1.96	1.29	3.60	11.53	2.87	6.49	5.83	6.24
15	-	-	-	-	3.76	-	3.22	-	2.97
20	2.25	2.27	2.12	3.25	17.52	2.98	11.11	23.87	7.13
20	-	-	-	-	7.00	-	32.52	6.09	13.34
25	2.10	2.11	2.45	10.57	55.61	3.57	39.95	44.14	22.56
25	-	-	-	-	27.89	-	76.87	16.44	78.15
30	1.99	2.38	2.56	25.30	-	7.74	16.79	76.77	-
35	1.83	2.26	1.62	5.59	-	5.82	83.02	58.49	-
40	2.48	3.07	4.21	49.01	-	4.15	244.95	97.85	-
45	1.99	2.08	5.29	15.23	-	5.15	-	226.78	-
50	2.55	2.44	3.93	34.23	-	12.78	-	-	-
55	2.98	3.22	10.19	56.75	-	9.89	-	-	-

TABLE 1 (Continued)

Total Time, hours	Weight Loss, mg/cm ²					
	Zero Sea Salt in Air			1.0 ppm Sea Salt in Air		
	Sulfur in Fuel, wt %			Sulfur in Fuel, wt %		
	0.0040	0.040	0.40	0.0040	0.040	0.40
	Run 1	Run 2		Run 1	Run 2	Run 3

MDC-9 Coated Inconel 713C (a)

5	0.91	0.91	0.46	0.91	-	0.72	0.76	-	-
10	0.92	1.24	0.34	1.34	-	1.17	1.25	-	-
10	-	-	-	-	-	1.58	-	-	-
15	1.42	2.11	0.37	2.75	-	2.45	3.04	-	-
15	-	-	-	-	-	3.47	-	-	-
20	1.28	1.69	0.59	3.03	-	3.48	0.64	-	-
20	-	-	-	2.79	-	5.34	11.80	-	-
25	2.21	1.43	0.98	1.54	-	10.14	3.82	-	-
25	-	-	-	1.74	-	9.39	66.50	-	-
30	1.88	2.44	2.35	3.55	-	39.22	90.15	-	-
35	2.69	3.20	2.89	6.73	-	36.97	46.15	-	-
40	2.88	2.48	1.98	21.05	-	-	66.94	-	-
45	2.91	4.54	1.21	10.41	-	-	115.66	-	-
50	4.29	4.40	1.65	-	-	-	-	-	-
55	2.10	5.38	3.11	-	-	-	-	-	-

MDC-1 Coated Mar M-200

5	2.09	2.13	2.18	3.70	-	2.16	2.22	-	-
10	1.56	2.18	2.44	4.87	-	4.73	2.79	-	-
15	2.18	2.25	2.17	7.55	28.56	3.39	3.23	4.48	13.58
15	-	-	-	-	4.15	-	-	3.45	4.73
15	-	-	-	-	3.87	-	-	13.72	5.30
20	2.34	4.81	6.62	5.63	-	3.30	5.26	-	-
25	2.37	1.85	4.35	61.49	-	11.58	5.43	-	-
25	-	-	-	-	-	-	97.31	-	-
25	-	-	-	-	-	-	7.34	-	-
30	1.91	3.63	3.86	4.85	3.45	5.95	15.64	15.48	88.12
30	-	-	-	-	3.02	-	-	143.89	10.87
30	-	-	-	-	13.08	-	-	77.57	183.13
35	2.07	2.08	4.07	48.59	-	199.49	3.98	-	-
40	2.49	3.95	5.73	233.10	-	3.60	5.87	-	-
45	4.44	2.81	4.13	15.08	155.66	371.78	4.33	262.56	161.25
45	-	-	-	-	87.39	-	-	313.35	240.67
45	-	-	-	-	171.66	-	-	157.29	135.12
50	2.50	3.09	10.39	65.24	-	111.48	-	-	-
55	2.16	2.53	2.94	245.32	-	199.66	4.87	-	-

(a) Weight loss adjusted by adding 1.0 mg/cm².

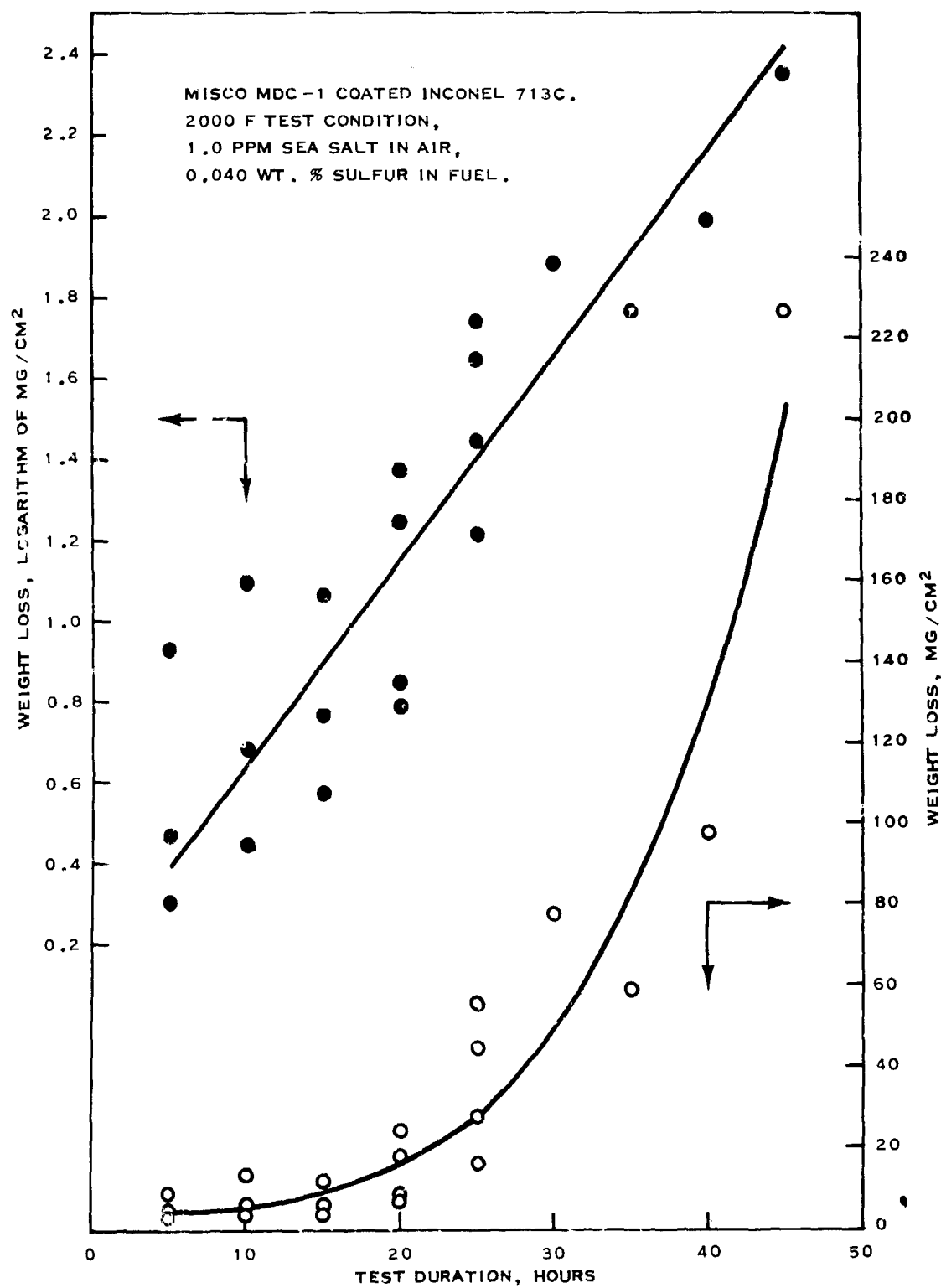


FIGURE 27
CURVE FITTING TO HOT CORROSION DATA

In previous studies (1) of weight loss from hot corrosion, an estimate of the variance was found to be a percentage of the value, and logarithms of weight-loss per unit area were used to provide a basis of uniform variance. Since Equation 3 permits the use of logarithms of weight-loss, it provides for uniform variance in analysis of the data.

The slope, b , of Equation 3 is the relative rate of corrosion with units of reciprocal hours.

The data for each combination of sulfur in fuel and sea salt in air for the superalloy and the three coating-alloy systems shown in Table 1 were fitted to equations of the form of Equation 3. The constants, b , for the three equations with zero or 1.0 ppm sea salt in air describing the superalloy and the three coating-alloy systems, were compared by statistical "t" tests for significant differences at a 95 per cent confidence level. Where data for two or more levels of sulfur in fuel showed no statistically significant difference in the relative corrosion rates, at a given level of sea salt in air, the data were combined by fitting a single equation. In combining the data a sulfur term was included in the equation and the sulfur term was tested for statistical significance. If the sulfur term was not significant it was deleted and the equation was recalculated. The final equations which were calculated to represent the data are shown in Table 2.

Variability for the separate equations differ in magnitude, with nine of the equations having a Standard Error of Estimate (S.E.E.) of less than the standard deviation of 0.288 found in previous 5-hour test on a variety of superalloys, and three of the equations having a S.E.E. larger than the previous standard deviation. The size of the S.E.E. for most of the equations indicate that the variability remaining after removal of the effect of time is essentially the same as in the previous investigations.

The equations for hot corrosion with time of exposure were calculated to establish statistically significant differences among concentrations of sulfur in fuel. The effect of sulfur in fuel on hot corrosion, with the level of sea salt in air and the material of the test specimen fixed can be obtained from the equations.

Inconel 713C - A single equation was obtained with uncoated Inconel 713C specimens for three levels of sulfur in fuel, in the absence of sea salt in air, indicating no significant effect of the concentration of sulfur in fuel on the superalloy. In the presence of 1.0 ppm sea salt in air, a single equation was obtained for uncoated Inconel 713C with 0.40 and 0.040 weight per cent sulfur in fuel. A sulfur term indicates that the relative rates of corrosion for the two concentrations of sulfur in fuel are the same, but the levels of attack are significantly different. A second equation was obtained for data with <0.0040 weight per cent sulfur in fuel. From these equations it can be concluded that a reduction in sulfur concentration from the specification maximum of 0.40 weight per cent sulfur to either 0.040 or <0.0040 weight per cent sulfur will significantly decrease hot corrosion of Inconel 713C.

TABLE 2

REGRESSION EQUATIONS FOR WEIGHT-LOSS VS TIME

<u>Sulfur</u> <u>in Fuel,</u> <u>wt %</u>	<u>Sea Salt</u> <u>in Air,</u> <u>ppm</u>	<u>Regression Equations (a)</u>	<u>S.E.E.</u> <u>(b)</u>
<u>Inconel 713C</u>			
<0.0040 0.040 0.40	0.0 0.0 0.0	$Y = 0.8198599 + 0.02159729 X$	0.153549
<0.0040 0.040 0.40	1.0 1.0 1.0	$Y = 1.167399 + 0.02911474 X$	0.198405
		$Y = 1.012591 + 0.9080433 S + 0.048222 X$	0.138587
<u>Misco MDC-1 Coated Inconel 713C</u>			
<0.0040 0.040 0.40	0.0 0.0 0.0	$Y = 0.2478212 + 0.003211451 X$	0.059775
		$Y = 0.02214815 + 0.01390193 X$	0.153822
<0.0040 0.040 0.40	1.0 1.0 1.0	$Y = 0.1883316 + 0.02158372 X$	0.296952
		$Y = 0.1377441 + 0.05072006 X$	0.261855
<u>Misco MDC-9 Coated Inconel 713C</u>			
<0.0040 0.040 0.40	0.0 0.0 0.0	$Y = -0.09654819 - 0.7288955 S + 0.0144935 X$	0.154956
<0.0040 0.040 0.40	1.0 1.0 1.0	$Y = -0.1946977 + 0.02867117 X$	0.212553
		$Y = -0.4717499 + 0.06061021 X$	0.344121
<u>Misco MDC-1 Coated Mar M-200</u>			
<0.0040 0.040 0.40	0.0 0.0 0.0	$Y = 0.260973 + 0.539306 S + 0.004127 X$	0.144662
<0.0040 0.040 0.40	1.0 1.0 1.0	$Y = 0.260424 + 0.034674 X$	0.552336

(a) $Y = a + cS + bX$ or $Y = a + bX$, where Y = logarithm of weight loss per unit area (mg/cm²), S = weight per cent sulfur in fuel, and X = test duration, hours.

(b) Standard Error of Estimate in terms of logarithms.

Misco MDC-1 Coated Inconel 713C - In the absence of sea salt in air, a reduction in sulfur concentration, from the specification maximum of 0.40 weight per cent for JP-5 fuel to either 0.040 or <0.0040 weight per cent significantly decreased the relative rate of corrosion for Misco MDC-1 coated Inconel 713C; however, this may not be of practical significance because of the very low level of attack. In the presence of 1.0 ppm sea salt in air, a reduction in sulfur concentration from the specification maximum of 0.40 to 0.040 weight per cent did not significantly change the relative corrosion rate; however, a reduction from 0.40 to <0.0040 weight per cent decreased the relative rate of corrosion significantly.

Misco MDC-9 Coated Inconel 713C - A single equation indicates that, in the absence of sea salt in air, the relative rate of corrosion did not differ significantly with Misco MDC-9 coated Inconel 713C for the three concentrations of sulfur in fuel. A sulfur term in the equation indicates a significant effect of fuel sulfur on the level of attack. Further testing showed no statistically significant difference in the level of attack with <0.0040 or 0.040 weight per cent sulfur. Thus, a reduction in sulfur concentration from the specification maximum of 0.40 to either 0.040 or <0.0040 weight per cent sulfur significantly increased hot corrosion; however, the differences may not be of practical significance because of the low level of attack. In the presence of 1.0 ppm sea salt in air, a single equation was obtained with 0.40 and 0.040 weight per cent sulfur in fuel, and a second equation was obtained for data with <0.0040 weight per cent sulfur in fuel. These equations indicate no significant difference in the hot corrosion of Misco MDC-9 coated Inconel 713C with a reduction in fuel sulfur from the specification maximum of 0.40 to 0.040 weight per cent sulfur; however, a reduction in fuel sulfur from the specification maximum of 0.40 to <0.0040 weight per cent sulfur significantly decreased hot corrosion.

Misco MDC-1 Coated Mar M-200 - A single equation indicates that, in the absence of sea salt in air, the relative rates of corrosion did not change significantly for the three concentrations of sulfur in the fuel. A sulfur term in the equation indicates a significant effect of sulfur on the level of attack. Further testing shows a statistically significant reduction in weight-loss for a reduction in sulfur in fuel from the specification maximum of 0.40 to either 0.040 or <0.0040 weight per cent sulfur. This reduction may be of little practical importance because of the very low weight losses in all cases. In the presence of 1.0 ppm sea salt in air, a single equation was obtained for Misco MDC-1 coated Mar M-200 indicating no significant effect of the concentration of sulfur in fuel on hot corrosion of the coating-alloy system.

In initial tests with 1.0 ppm sea salt in air, the variability with Misco MDC-1 coated Inconel 713C specimens was higher than experienced with other superalloys or coating-alloy systems. A reference specimen of uncoated Inconel 713C was mounted at the rear of the cascade for one test and the specimen was removed, with replacement, at each 5-hour period. The variability of the reference specimens was of the same order as found in previous 5-hour tests and much lower than found with Misco MDC-1 coated Mar M-200 specimens. These results indicate that the greater variability with the MDC-1 coated Mar M-200 is not a result of variability of test rig operation but rather is inherent in the coating alloy-system as indicated by metallographic examination of unexposed specimens.

Sea Salt - The relative rate of corrosion from each of the regression equations in Table 2, and their 95 per cent confidence limits, are shown in Table 3. From these, comparisons can be made showing the effect of sea salt on the rate of attack with the concentration of sulfur in fuel and test specimen material fixed. In comparisons where the relative rate of corrosion for one condition is not included in the confidence limits for another condition, it can be concluded with 95 per cent confidence that the rates are significantly different. With both bare and coated superalloys, in all comparisons, a reduction from 1.0 to zero sea salt in air decreased the rate of attack.

Similarly, comparisons can be made of the relative rates of corrosion for bare and coated Inconel 713C with the concentration of sulfur in fuel fixed. In the absence of sea salt, the rate of attack decreased on the coated sample; but with 1.0 ppm sea salt in air the rate of attack was the same for both the bare and coated Inconel 713C.

These findings serve to indicate that sea salt is a primary cause in the hot corrosion of both bare and coated superalloys in a marine environment.

4.1.4. Coating Life

Comparisons of the "life" of the coating may be of interest to some readers. A measure of coating life can be obtained by selecting a criterion for failure based on weight loss and calculating hours to failure using each of the equations in Table 2.

Whitfield and Parzuchowski (3) established a weight loss of 0.100 grams as the criterion for failure of coated specimens. Using dimensions of the corrosion bars given by Daneek (4), the surface area of their specimens is calculated to be 20.00 cm². From these figures we calculated the criterion for failure of the coating as 5.0 mg/cm². A weight loss 5 mg/cm² falls in the range of weight losses obtained for visual ratings of specimens showing light attack during our investigation, and represents a reasonable criterion for coating failure.

Using this value as a criterion for coating failure, the hours to failure for the Misco MDC-1 and MDC-9 coated Inconel 713C and the MDC-1 coated Mar M-200 at each of the combinations of sulfur in fuel and sea salt in air were calculated, and are shown in Table 4. Confidence limits have not been established for these values. Comparisons of the effect of sulfur in fuel and sea salt in air on life of the coatings will not be made, since they would be the same as drawn previously from relative rates of corrosion and levels of attack. One item of interest in Table 4 is that coating-alloy systems are affected differently by combinations of sulfur in fuel and sea salt in air. For the three levels of sulfur in fuel, with either zero or 1.0 ppm sea salt in air, the range in hours to coating failure for the MDC-1 coated Inconel 713C is 129 hours (from 140 to 11 hours), and for MDC-1 coated Mar M-200 the range is 93 hours (from 106 to 13 hours), and for MDC-9 coated Inconel 713C the range is 56 hours (from 75 to 19 hours). Thus the MDC-9 coating appears to be less affected than the MDC-1 coating.

TABLE 3

RELATIVE CORROSION RATES FROM REGRESSION EQUATIONS

Sulfur in Fuel, wt %	Sea Salt in Air, ppm	Relative Corrosion Rate (a)	95% Confidence Limits	
			Lower (a)	Upper (a)
Inconel 713C				
<0.0040 0.040 0.40	0.0 0.0 0.0	0.02159729	0.01800603	0.02518855
<0.0040 0.040 0.40	1.0 1.0 1.0	0.02911474 0.04822200	0.01566623 0.04041496	0.04256325 0.05602904
Misco MDC-1 Coated Inconel 713C				
<0.0040 0.040 0.40	0.0 0.0 0.0	0.003211451 0.01390193	0.001530135 0.00726687	0.004892767 0.02053699
<0.0040 0.040 0.40	1.0 1.0 1.0	0.02158372 0.05072006	0.01312873 0.04272244	0.03003871 0.05871768
Misco MDC-9 Coated Inconel 713C				
<0.0040 0.040 0.40	0.0 0.0 0.0	0.0144935	0.01100982	0.01797718
<0.0040 0.040 0.40	1.0 1.0 1.0	0.02867117 0.06061021	0.01635011 0.04633117	0.04099223 0.07488925
Misco MDC-1 Coated Mar M-200				
<0.0040 0.040 0.40	0.0 0.0 0.0	0.004127	0.000874	0.007380
<0.0040 0.040 0.40	1.0 1.0 1.0	0.034675	0.024491	0.044857

(a) Values and 95 per cent confidence limits for exponential equations in Table 2.

TABLE 4
PREDICTED COATING LIFE

<u>Sulfur in Fuel, wt %</u>	<u>Sea Salt in Air, ppm</u>	<u>Time (a) to Failure, hours</u>
<u>Misco MDC-1 Coated Inconel 713C</u>		
<0.0040	0.0	140
0.040	0.0	140
0.40	0.0	49
<0.0040	1.0	24
0.040	1.0	11
0.40	1.0	11
<u>Misco MDC-9 Coated Inconel 713C</u>		
<0.0040	0.0	55
0.040	0.0	57
0.40	0.0	75
<0.0040	1.0	31
0.040	1.0	19
0.40	1.0	19
<u>Misco MDC-1 Coated Mar M-200</u>		
<0.0040	0.0	106
0.040	0.0	101
0.40	0.0	54
<0.0040	1.0	13
0.040	1.0	13
0.40	1.0	13

(a) Calculated from equations of Table 2 using 5.0 mg/cm² loss in weight as the criterion for failure of coating.

4.2. Vanadium

This investigation was designed to provide a preliminary evaluation of the effect of vanadium in fuel on hot corrosion of a superalloy in a marine environment. The program was initiated because of Navy interest in a proposed multi-purpose fuel for use in marine boilers and gas-turbine engines (Table 5) which will be slightly heavier than No. 2 Diesel fuel and will allow from 1.3 to 1.5 per cent by weight of sulfur and from 2 to 5 ppm vanadium. This fuel may create problems in gas turbine engines because it is heavier and allows more sulfur than JP-5 fuel; however, its vanadium content is the primary concern.

A JP-5 base fuel, a superalloy, and operating conditions from a previous investigation were selected for this study to permit the use of prior data as a basis for comparing the effect of vanadium on hot corrosion in a marine environment.

To simulate the environment in the turbine section of a gas turbine engine, a cascade holder supporting six test specimens was mounted in the exhaust section of Phillips 2-inch combustor. The facility was operated to obtain exposure of the test specimens at high pressure (15 atmospheres), high temperature (2000 F), high velocity (745 feet per second), and realistic stoichiometry (60 air-fuel ratio). With a 1-hour cyclic procedure, test specimens were removed, with replacement, at 5-hour intervals which provided a series of specimens with exposures of up to 55 hours duration.

Further details concerning test equipment, test materials, and test program are presented in Appendices 1, 2, and 3, respectively, which are Sections 8., 9., and 10. of this report.

Initial plans were to use a vanadium naphthenate liquid blended in a JP-5 base fuel as the source of vanadium for the evaluation; however, an insolubility problem and low purity of the vanadium naphthenate sample available made this approach impractical. A water soluble vanadium compound injected into the quench zone of the combustor with the dilute sea water or deionized water was selected as an alternative method for introducing vanadium into the system. This method had the advantage of assuring that the vanadium reached the specimens rather than reacting with the flame tube of the combustor. Blends of vanadyl chloride (VOCl_2) in dilute sea water or deionized water were prepared (Section 9.3., Appendix 2) to provide 0.063 or 0.017 ppm vanadium in air. These concentrations are equivalent to 3.8 and 1.0 ppm vanadium in fuel at the 2000 F condition.

In this report visual appearance, metallographic examination of test specimens, and weight loss will be used to evaluate the effect of vanadium on hot corrosion.

TABLE 5PROPOSED SPECIFICATION FOR NAVY MULTI-PURPOSE FUEL

<u>Properties</u>	<u>Limits</u>
Viscosity at 100°F c.s.	7.0 - 10.0
Gravity °API 60F	Report
Distillation °F	
10%	440 - 465
50%	650 max
90%	725 - 740
Recovery % at 725°F	Report
Heat of Combustion Btu/lb	Report
Sulfur %	1.3 - 1.5
Pour Point °F	35 max
Luminometer Number	Report
Copper Strip Corrosion, ASTM	
Class 2	Pass
Flash Point °F	150 min
Carbon Residue on	
10% Bottoms	0.8 - 1.0
Ash %	.001 max
Neutralization Number	Neutral
Aniline Point °F	Report
Cetane Number	Report
Vanadium ppm	2 - 5
Metals in carbon residue should be trace quantities	

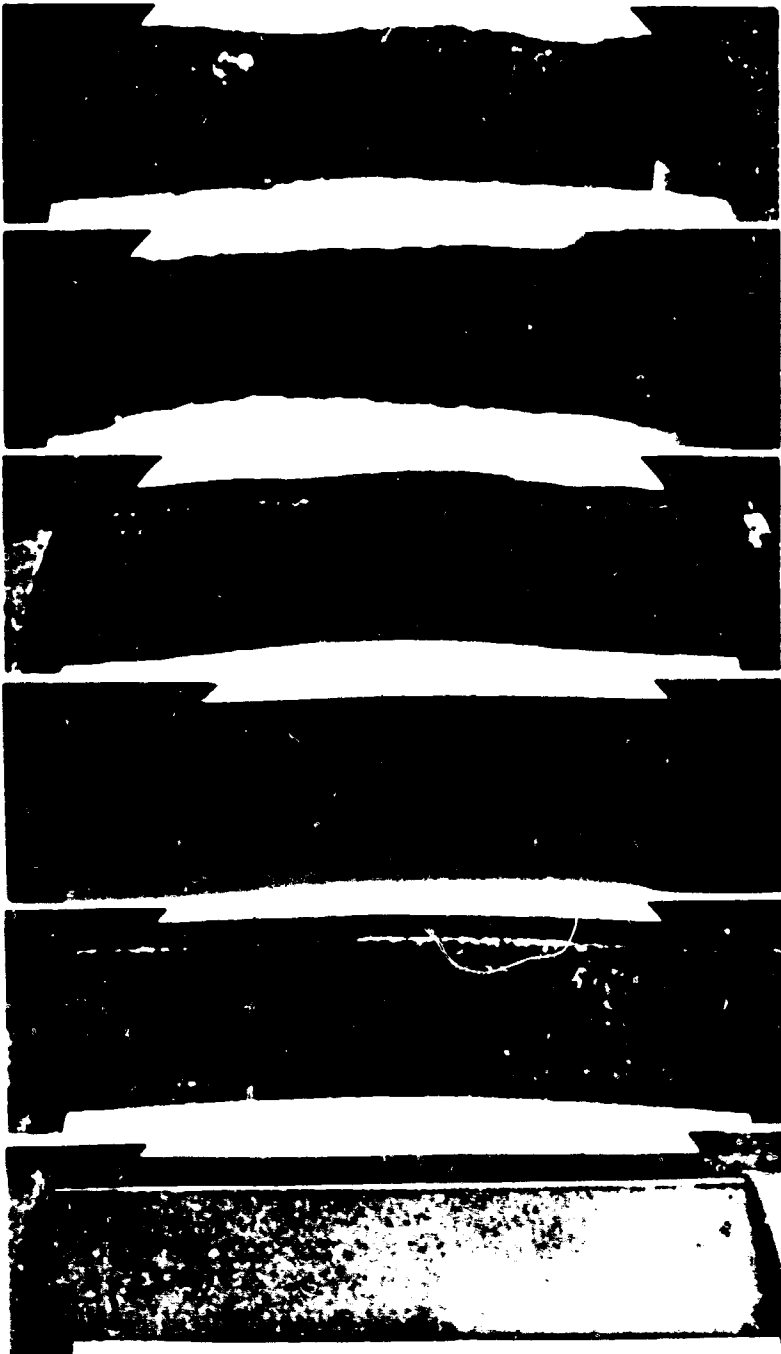
4.2.1. Visual

The condition of the Inconel 713C specimens following extended exposure at the 2000 F test condition, both with and without vanadium and sea salt in air is shown in Figure 28. These photographs were taken with the specimens still mounted in the cascade holder to show the undisturbed accumulation of deposits. In the absence of sea salt or vanadium the specimens are quite clean; however, with the ingestion of vanadium, sea salt, or vanadium and sea salt considerable accumulation of deposits were observed. These deposits tended to flake-off the test specimens during cooling to room temperature, which showed a significant difference in their coefficients of expansion.

Qualitative analyses were obtained of deposits accumulated with 0.063 ppm vanadium in air and zero or 1.0 ppm sea salt in air. X-ray diffraction analyses of the surface of the deposits, in-situ on the specimens, showed them to be primarily metal oxide corrosion products (Bunsenite, NiO). Sea salt residues were not identified on the deposits from the test with sea salt in air. There were some unidentified lines from the analyses. Similarly, x-ray diffraction analyses of the total deposits, removed from the specimens by light scraping, showed them to be primarily metal oxide corrosion products (Bunsenite, NiO), with some unidentified lines and no indication of sea salt residue for the test with sea salt in air. These analyses showing the absence of sea salt residue are in agreement with other data obtained on deposits at this 2000 F condition (Section 4.3.2.). X-ray fluorescence analyses of the total deposits from both tests showed the presence of vanadium.

The weight of deposits for each specimen, for tests with vanadium in air, were obtained as the difference in weight of the specimen as removed from the test and the weight of the specimen after electro-cleaning (Table 6). These data tend to underestimate the amount of deposit on the specimen during exposure in the test rig because of the flaking of deposits during cooling to ambient temperature. The amount of deposit on the specimens reached an equilibrium within the first few hours of the test. Deposits on specimens exposed in the presence of sea salt were heavier than for those exposed in the absence of sea salt.

To illustrate the effect on appearance of the exposure variables in this investigation, photomicrographs (2X magnification) of representative electro-cleaned Inconel 713C specimens exposed for 20 hours in the Phillips test rig at 2000 F conditions with the various combinations of vanadium and sea salt in air are shown in Figure 29. In this figure the detrimental effects of both sea salt and 0.063 ppm vanadium in air are apparent; however, the addition of 0.017 ppm vanadium in air, in the absence of sea salt, did not appear to increase the level of attack.



EXPOSURE TIME, HR	VANADIUM IN AIR, PPM	SEA SALT IN AIR, PPM
20	0.063**	1.0
25	NONE	
55	0.063**	NONE
55	0.017*	
55	NONE	
UNEXPOSED		

2X MAGNIFICATION OF INCONEL 713C SPECIMENS MOUNTED
IN HOLDER AFTER EXPOSURE AT 2000F TEST CONDITION
WITH 0.40 WT % SULFUR IN FUEL .

*CONCENTRATION EQUIVALENT TO 1.0 PPM VANADIUM IN FUEL AT 2000F TEST CONDITION .
**CONCENTRATION EQUIVALENT TO 3.8 PPM VANADIUM IN FUEL AT 2000F TEST CONDITION .

FIGURE 25
ACCUMULATION OF DEPOSITS ON SPECIMENS

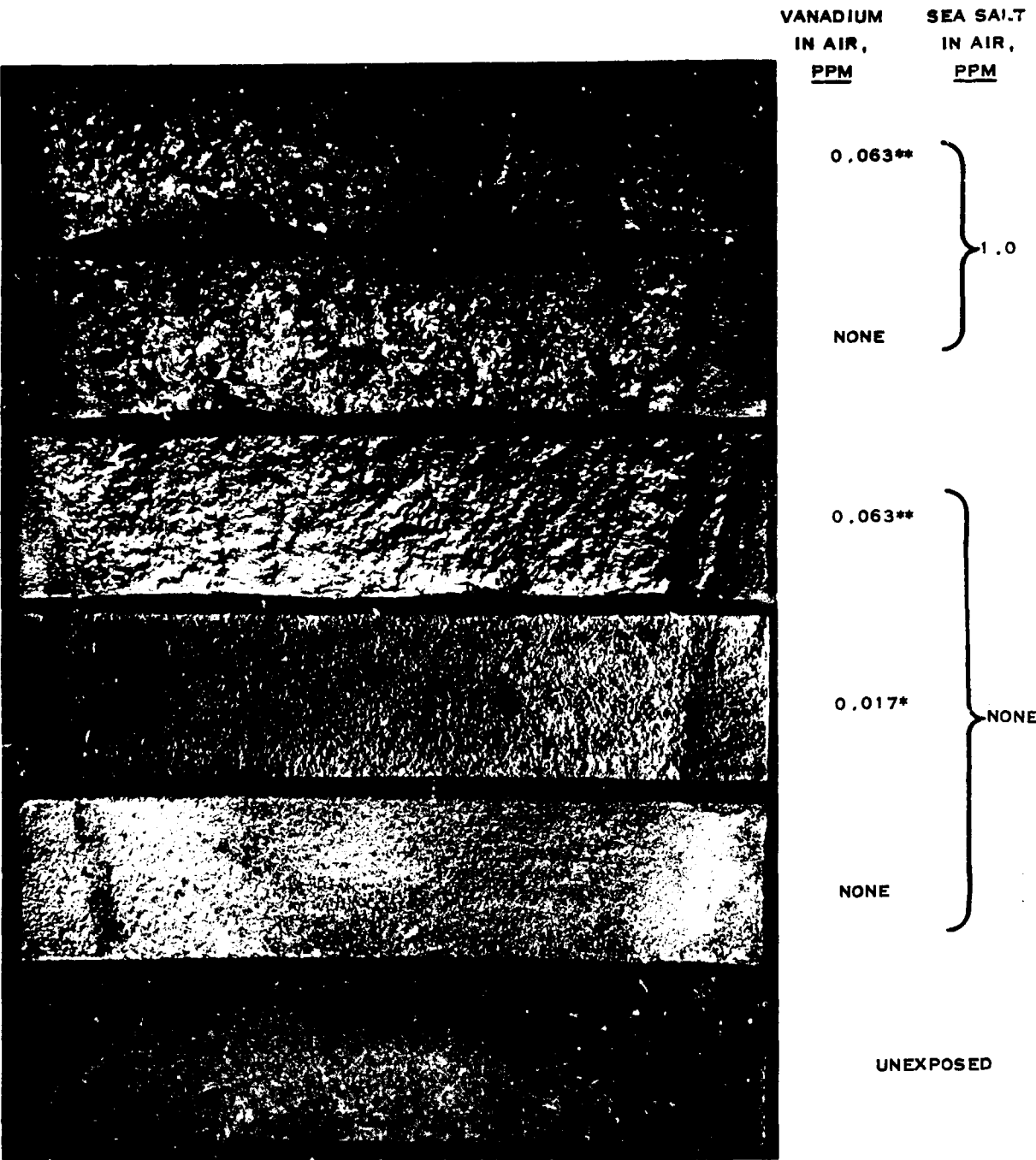
TABLE 6DEPOSIT WEIGHT DATA FOR INCONEL 713C

Vanadium in Air, ppm	Sea Salt in Air, ppm	Test Specimen Number	Exposure Time, hours	Deposit Weight(a)	
				mg	mg/cm ²
0.017 (b)	0.0	2	5	146.1	7.34
0.017	0.0	3	10	192.8	9.69
0.017	0.0	6	15	231.2	11.62
0.017	0.0	1	20	170.2	8.55
0.017	0.0	4	25	225.3	11.32
0.017	0.0	4A	30	188.0	9.45
0.017	0.0	1A	35	159.6	8.02
0.017	0.0	6A	40	149.6	7.52
0.017	0.0	3A	45	164.8	8.28
0.017	0.0	2A	50	181.4	9.12
0.017	0.0	5	55	182.3	9.16
0.063 (c)	0.0	2	5	128.1	6.44
0.063	0.0	3	10	142.6	7.17
0.063	0.0	6	15	164.3	8.26
0.063	0.0	1	20	111.7	5.61
0.063	0.0	4	25	142.3	7.15
0.063	0.0	4A	30	140.5	6.93
0.063	0.0	1A	35	152.6	7.53
0.063	0.0	6A	40	128.2	6.32
0.063	0.0	3A	45	147.5	7.28
0.063	0.0	2A	50	131.1	6.47
0.063	0.0	5	55	149.5	7.38
0.063 (c)	1.0	2	5	280.4	14.09
0.063	1.0	6A	5	297.0	14.65
0.063	1.0	3	10	542.6	27.27
0.063	1.0	3A	10	442.6	21.83
0.063	1.0	6	15	496.4	24.94
0.063	1.0	2A	15	420.6	20.75
0.063	1.0	1	20	389.0	19.55
0.063	1.0	4	20	516.9	25.97
0.063	1.0	5	20	505.5	24.94

(a) Obtained by difference between weight of specimen after test and weight of specimen after cleaning.

(b) Equivalent to 1.0 ppm vanadium in fuel at 2000 F conditions.

(c) Equivalent to 3.8 ppm vanadium in fuel at 2000 F conditions.



2X MAGNIFICATION AFTER ELECTRO-CLEANING

*CONCENTRATION EQUIVALENT TO 1.0 PPM VANADIUM IN FUEL AT 2000F TEST CONDITION.
**CONCENTRATION EQUIVALENT TO 3.8 PPM VANADIUM IN FUEL AT 2000F TEST CONDITION.

FIGURE 29
INCONEL 713C SPECIMENS AFTER 20 HOURS AT 2000F TEST CONDITION

4.2.2. Metallography

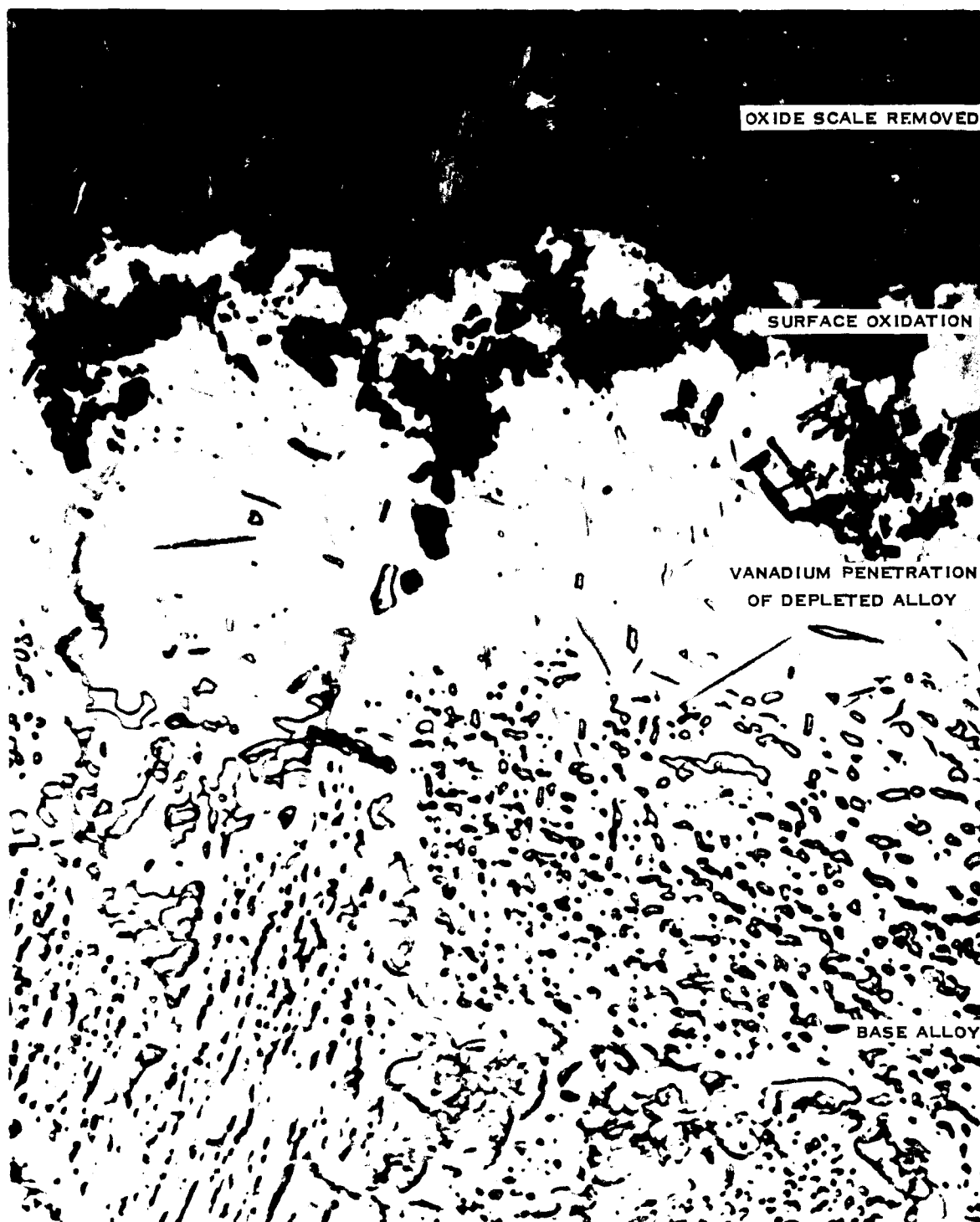
Information concerning the mode and the intensity of corrosive attack, sustained by the superalloy with the various combinations of sea salt and vanadium is pertinent to this investigation. High levels of specimen weight loss are obviously objectionable; but, low values do not establish a lack of metal damage. Attack may penetrate the metal matrix by deep intercrystalline corrosion without significant metal loss. Metallographic examination of selected test specimens was made (Appendix 3, Section 10.3.) to establish the depth of attack at the corrosion interface and determine whether the use of weight-loss data is a valid measure of metal damage for this investigation.

Typical Inconel 713C specimens as received from Misco (Figure 9) were free from any unusual surface scale or impurities and the surface composition of the alloy was not altered by depletion.

Exposure of Inconel 713C in an environment free of vanadium and sea salt at the 2000 F condition produced only slight, uniform surface oxidation. However, the surface composition of the alloy was altered by diffusion, and the depth of this zone of alloy depletion increased with exposure time to reach approximately four mils after 55 hours. A metallographic cross-section of such a specimen is shown in Figure 10. The usual surface scale is not evident in this, and subsequent, photomicrographs because these specimens have been electro-cleaned.

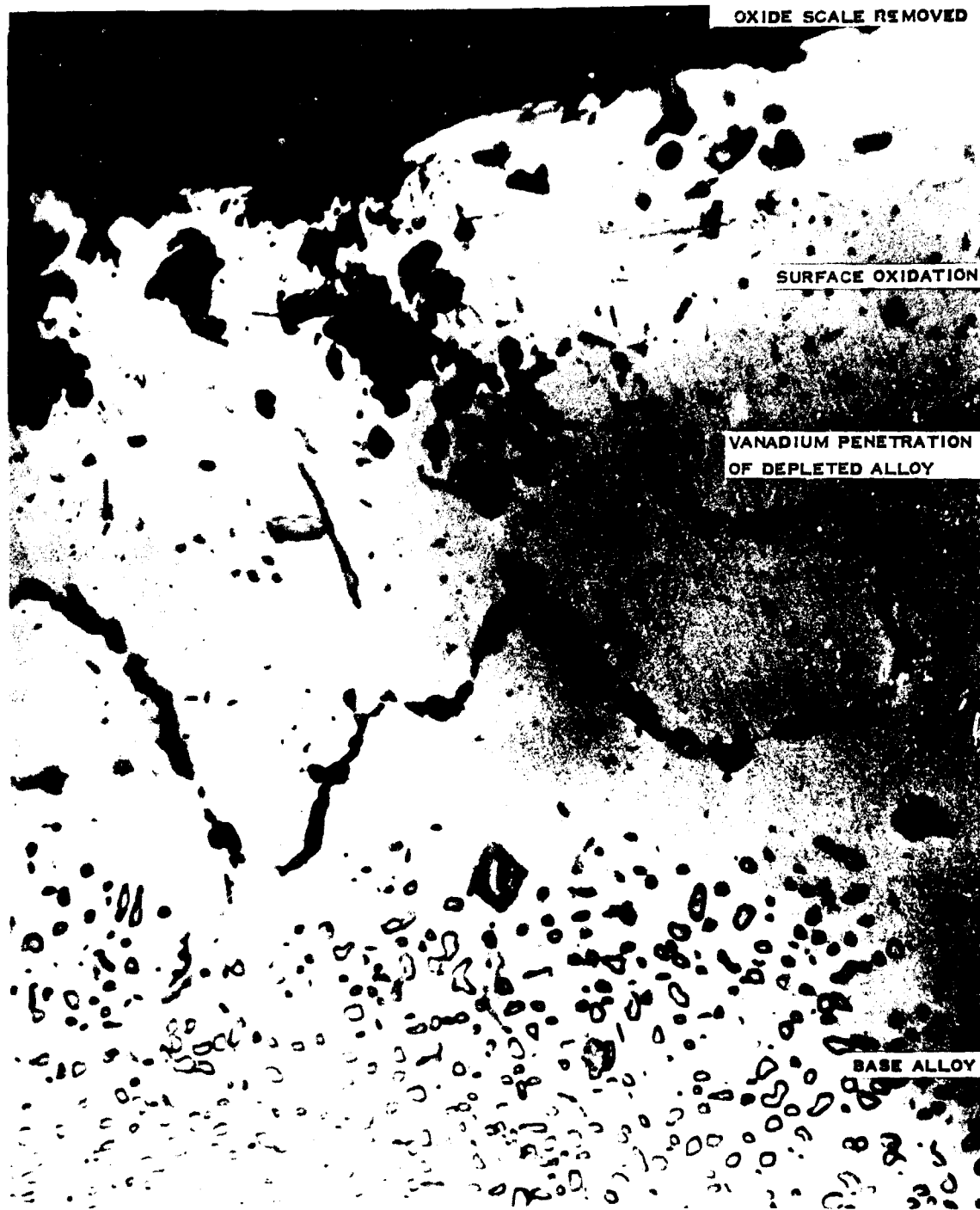
With the addition of vanadium to this sea-salt-free environment, the corrosion interface was modified by oxide penetration into the alloy-depleted zone. It is assumed that this is vanadium pentaoxide which is progressing along grain boundaries; as described in the literature on fire-side corrosion of boilers, etc. However, this penetration was limited to the weakened layer of the alloy at the surface, as shown in Figures 30 and 31. These photomicrographs show an increase in depth of vanadium penetration with an increase in concentration of vanadium in air. With 0.063 ppm vanadium in air the attack appeared to progress by the undermining and subsequent loss of whole grains of alloy from the surface of the specimen, without deep intercrystalline penetration into the base matrix of the alloy.

Exposure of Inconel 713C with sea salt in the environment, but free of vanadium, at the 2000 F condition produced an accelerated-oxidation attack. In general, the attack advanced on a broad front without deep-intercrystalline penetration of the corrosive agents and was led by penetration of randomly-dispersed, light-grey globules of metallic sulfides. The formation of these sulfides was associated with changes in the surface composition of the alloy, which is characterized by chromium depletion. Rapid oxidation of the weakened alloy follows (Figure 11). The depth of the zone of alloy depletion was less than 0.4 mil, which is in sharp contrast to the order of magnitude greater depth shown in Figure 10 with simple surface oxidation.



METALLOGRAPHIC CROSS-SECTION OF INCONEL 713C SPECIMEN AFTER 55 HOURS AT 2000F TEST CONDITION WITH 0.017 PPM VANADIUM IN AIR. ELECTRO-CLEANED. MARBLE'S REAGENT ETCHED. 1000X MAGNIFICATION.

FIGURE 30
SURFACE OXIDATION WITH VANADIUM PENETRATION



METALLOGRAPHIC CROSS-SECTION OF INCONEL 713C SPECIMEN AFTER 55 HOURS AT 2000F TEST CONDITION WITH 0.063 PPM VANADIUM IN AIR, ELECTRO-CLEANED, MARBLE'S REAGENT ETCHED, 1000X MAGNIFICATION.

FIGURE 31
SURFACE OXIDATION WITH VANADIUM PENETRATION

With the addition of vanadium to this sea-salt environment, the corrosion interface was modified by oxide penetration in the alloy-depleted zone, as shown in Figure 32. Thus, vanadium attack in the presence of sea salt appears to be similar to that previously shown in Figure 31 without sea salt. However, the depth of the layer of depleted alloy has been greatly reduced by the accelerated-oxidation attack with sea salt, previously shown in Figure 11. The corrosive effects of vanadium and sea salt appear to be additive, and evidence of both mechanisms can be seen more clearly at higher magnification in Figure 33.

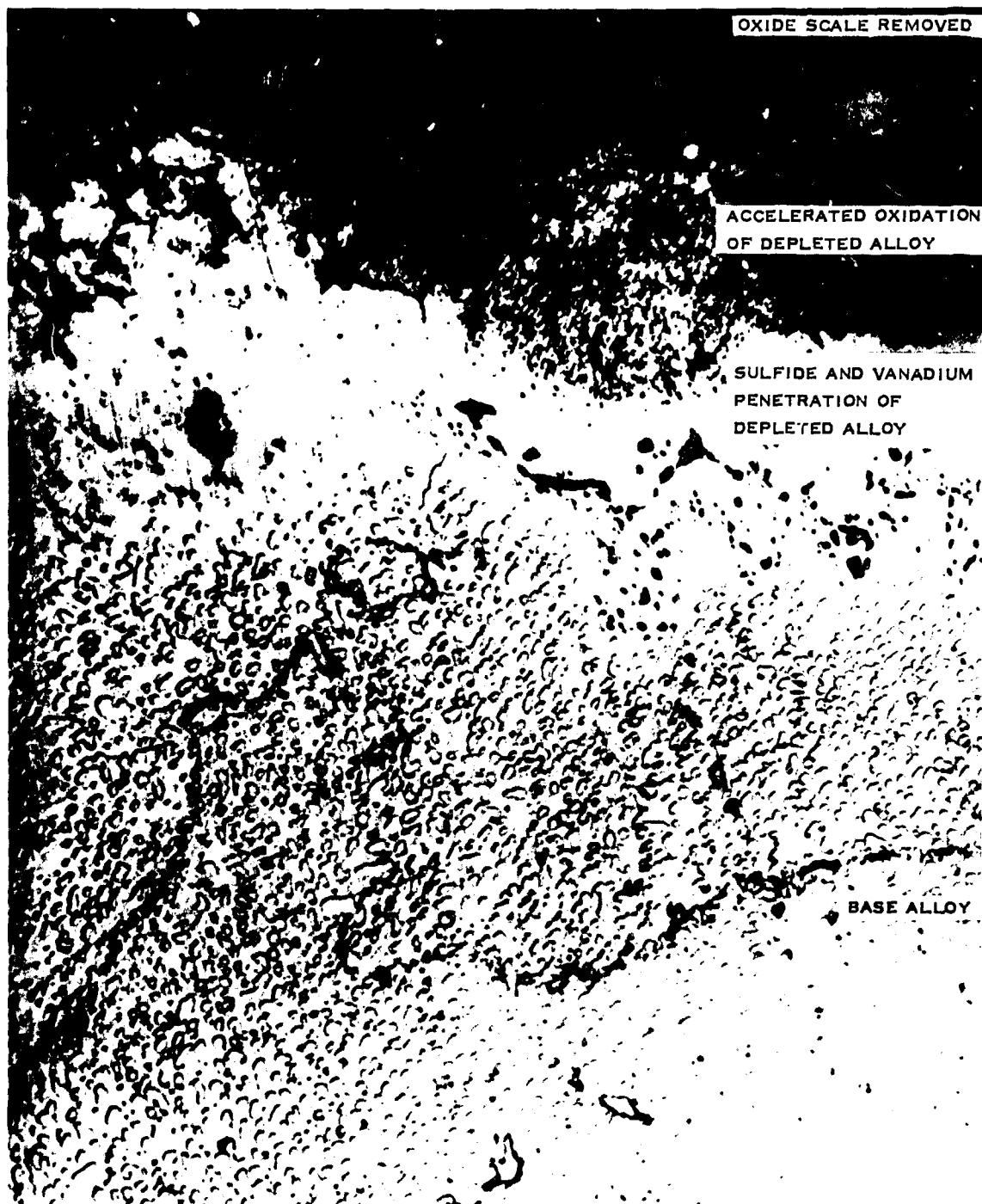
From this examination it is concluded that vanadium attack was limited to penetration of the surface layer of depleted alloy. This attack was in addition to the usual surface oxidation normally encountered and the accelerated surface oxidation encountered in a marine environment. The use of weight loss by the specimens is suitable for estimating the extent of corrosive attack on Inconel 713C over the range of conditions investigated, although this measurement may tend to underestimate the attack on specimens exposed in the presence of vanadium.

4.2.3. Weight-Loss

An estimate of variance of weight loss has been found to be a percentage of the value and, as in previous investigations, weight loss per unit area will be used in analyses to provide uniform variance.

Weight-loss data for Inconel 713C specimens exposed with 0.40 weight per cent sulfur in fuel and zero or 1.0 ppm sea salt in air are shown in Table 1. Data obtained in the current investigation for specimens of Inconel 713C exposed with 0.40 weight per cent sulfur in fuel, zero or 1.0 ppm sea salt in air, and 0.017 or 0.063 ppm vanadium in air are shown in Table 7. In the tests with vanadium in air, two heats of Inconel 713C were used because sufficient specimens from the heat used in the previous program were not available. There are indications that resistance to attack varied for the two heats; however, the effect of vanadium on hot corrosion of the specimens was larger than the difference between heats of specimens and the two heats did not interfere with the evaluation.

For the tests in the absence of sea salt in air, one specimen was exposed at each of the eleven time periods with and without vanadium in air. This provides a balanced experiment and an Analysis of Variance (AOV) of logarithms of weight-loss of the specimens was made (Table 8). This AOV shows a significant effect of vanadium on hot corrosion; however, to determine which levels of vanadium are significantly different an additional evaluation of the data must be made. The Least Significant Difference (LSD), at the 95 per cent confidence level, for testing differences in the means of logarithms of weight loss per unit area for the three concentrations of vanadium was calculated, from the error term of the AOV, to be 0.116023. The antilogarithm of the LSD (1.31) is the minimum ratio of two means, in terms of actual weight-loss, that could be declared statistically different at the 95 per cent confidence level. This analysis shows that the presence of 0.063 ppm vanadium in air, in the absence of sea salt in air, significantly increased hot corrosion over that with either zero



METALLOGRAPHIC CROSS-SECTION OF INCONEL 713C SPECIMEN AFTER 20 HOURS AT 2000F TEST CONDITION WITH 1.0 PPM SEA SALT AND 0.063 PPM VANADIUM IN AIR. ELECTRO-CLEANED. MARBLE'S REAGENT ETCHED. 1000X MAGNIFICATION.

FIGURE 32
ACCELERATED SURFACE OXIDATION WITH VANADIUM PENETRATION



METALLOGRAPHIC CROSS-SECTION OF INCONEL 713C SPECIMEN AFTER 20 HOURS AT 2000F TEST CONDITION WITH 1.0 PPM SEA SALT AND 0.063 PPM VANADIUM IN AIR. ELECTRO-CLEANED. CARPELLA'S REAGENT ETCHED. 3000X MAGNIFICATION.

FIGURE 33
ACCELERATED SURFACE OXIDATION WITH VANADIUM PENETRATION

TABLE 7

WEIGHT LOSS DATA FOR INCONEL 713C

Vanadium in Air, ppm	Sea Salt in Air, ppm	Test Specimen Number	Exposure Time, hours	Total Specimen Weight Loss	
				mg	mg/cm ²
0.017 (c)	0.0	2 (b)	5	133.1	6.69
0.017	0.0	3 (b)	10	334.2	16.79
0.017	0.0	6 (b)	15	540.4	27.16
0.017	0.0	1 (b)	20	641.3	32.23
0.017	0.0	4 (b)	25	470.0	23.62
0.017	0.0	4A (b)	30	670.6	33.70
0.017	0.0	1A (b)	35	459.5	23.09
0.017	0.0	6A (b)	40	727.7	36.57
0.017	0.0	3A (b)	45	2577.5	129.52
0.017	0.0	2A (b)	50	1166.7	58.63
0.017	0.0	5 (b)	55	1875.8	94.26
0.063 (d)	0.0	2 (b)	5	620.4	31.18
0.063	0.0	3 (b)	10	1356.6	68.17
0.063	0.0	6 (b)	15	1612.4	81.02
0.063	0.0	1 (b)	20	2170.5	109.07
0.063	0.0	4 (b)	25	2837.1	192.82
0.063	0.0	4A (a)	30	1907.9	94.12
0.063	0.0	1A (a)	35	2246.2	110.81
0.063	0.0	6A (a)	40	3274.8	161.56
0.063	0.0	3A (a)	45	4039.6	199.29
0.063	0.0	2A (a)	50	4322.7	213.25
0.063	0.0	5 (a)	55	5065.5	249.90
0.063 (d)	1.0	2 (b)	5	691.1	34.73
0.063	1.0	6A (a)	5	2265.9	111.78
0.063	1.0	3 (b)	10	4364.9	219.34
0.063	1.0	3A (a)	10	3706.9	182.87
0.063	1.0	6 (b)	15	6525.2	327.90
0.063	1.0	2A (a)	15	7183.2	354.37
0.063	1.0	1 (b)	20	7642.3	384.03
0.063	1.0	4 (b)	20	10251.8	515.16
0.063	1.0	5 (a)	20	8900.0	439.06

(a) Specimens from Misco Heat No. RW072.

(b) Specimens from Misco Heat No. RW382.

(c) Equivalent to 1.0 ppm vanadium in fuel at 2000 F conditions.

(d) Equivalent to 3.8 ppm vanadium in fuel at 2000 F conditions.

TABLE 8

ANALYSIS OF VARIANCE OF WEIGHT-LOSS OF INCONEL 713C SPECIMENS

<u>Source of Variation</u>	<u>Degrees of Freedom</u>	<u>Sum of Squares</u>	<u>Mean Square</u>
Total	32	5.630109	
Hours (H)	10	2.931348	0.293135 *
Vanadium (V)	2	2.358448	1.179224 *
H x V	20	0.340313	0.017015

$\sigma = 0.130442$ (Coefficient of Variation = 35.0%).

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

LSD for comparison of means of 11 values = 0.116023.

Table of Means

<u>Concentration of Vanadium in Air, ppm</u>	<u>Weight Loss</u>	
	<u>log mg/cm²</u>	<u>Geometric Mean, mg/cm²</u>
0.063	2.073792	118.5
0.017	1.515894	32.8
0.000	1.497910	31.5

<u>Comparison</u>	<u>Difference, log mg/cm²</u>	<u>Ratio</u>
0.063 ppm minus 0.017 ppm Vanadium	0.557898 *	3.61
0.063 ppm minus 0.000 ppm Vanadium	0.575882 *	3.77
0.017 ppm minus 0.000 ppm Vanadium	0.017984	1.04

Asterisk (*) indicates a significant difference at 95% confidence level.

or 0.017 ppm vanadium in air and shows no significant difference in hot corrosion between zero and 0.017 ppm vanadium in air. Hot corrosion with 0.063 ppm vanadium in air was 3.77 times as large as with zero vanadium in air and 3.61 times as large as with 0.017 ppm vanadium in air and with 0.017 ppm vanadium in air was 1.04 times as large as with zero vanadium in air.

In making an Analysis of Variance of the data in the presence of 1.0 ppm sea salt in air, some data were omitted to balance the analysis. In the presence of 1.0 ppm sea salt in air and 0.063 ppm vanadium in air, two specimens were exposed at 5, 10 and 15 hours while three specimens were exposed for 20 hours. To balance this test the logarithms of weight-loss for specimens 1 and 4 were averaged, and thus at each time period data were available with specimens of Inconel 713C from each of the two heats. In the absence of vanadium in air, data from the first four time periods for the first test were used. An Analysis of Variance of logarithms of weight-loss for these data are summarized in Table 9. This analysis shows that the presence of 0.063 ppm vanadium in air, in the presence of 1.0 ppm sea salt in air, significantly increased hot corrosion of Inconel 713C specimens, with the hot corrosion being 2.24 times as large with vanadium as without vanadium.

To relate hot corrosion to time of exposure for each combination of sea salt and vanadium in air, regressions were calculated using logarithm of weight-loss per unit area and hours of exposure or logarithm of hours of exposure. The regressions using logarithms of both weight loss per unit area and hours provided the best fit of the data. The calculated regression equations are shown in Table 10 and the data are shown graphically in Figure 34. This figure shows the detrimental effect of 0.063 ppm vanadium in air with both zero and 1.0 ppm sea salt in air and shows no appreciable effect of 0.017 ppm vanadium in air with zero sea salt in air.

The following equation was developed relating the hot corrosion data obtained with the three levels of vanadium in air in the absence of sea salt in air.

$$Y = 4.28876196X_1 - 3.26853405X_2 - 351.7179310X_3 + 177.1192658X_4 + 174.67897594$$

where Y = logarithm of weight loss, mg/cm²,

X₁ = logarithm of hours of exposure,

X₂ = (logarithm of hours) (ppm vanadium in air + 1.0)

X₃ = (ppm vanadium + 1.0), and

X₄ = (ppm vanadium + 1.0)².

The Standard Error of Estimate (S.E.E.) for this overall equation is 0.133670, which is in good agreement with the individual regressions calculated with vanadium in air at fixed concentrations (Table 10). The weight-loss data for the three concentrations of vanadium in air in the absence of sea salt in air and the calculated regression lines from the above equation are shown in Figure 35.

TABLE 9

ANALYSIS OF VARIANCE OF WEIGHT-LOSS OF INCONEL 713C SPECIMENS
(1.0 ppm Sea Salt in Air)

<u>Source of Variation</u>	<u>Degrees of Freedom</u>	<u>Sum of Squares</u>	<u>Mean Square</u>
Total	15	2.314175	
Vanadium (V)	1	0.492010	0.492010 *
Hours (H)	3	1.651741	0.550580 *
V x H	3	0.003104	0.001034
Error	8	0.167320	0.020915

$\sigma = 0.144620$ (Coefficient of Variation = 39.5%)

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

Table of Means

	<u>Weight Loss</u>	
	<u>log mg/cm²</u>	<u>Geometric Mean, mg/cm²</u>
0.063 ppm Vanadium in Air	2.318544	208.2
Zero Vanadium in Air	1.967827	92.8
Difference	0.350717 (a)	

(a) Weight loss in the presence of vanadium is 2.24 times as great as in the absence of vanadium in air.

TABLE 10REGRESSION EQUATIONS FOR WEIGHT-LOSS VS TIME

<u>Vanadium in Air, ppm</u>	<u>Sea Salt in Air, ppm</u>	<u>Regression Equations (a)</u>	<u>S.E.E. (b)</u>
0.0	0.0	$Y = -0.013745 + 1.087534X$	0.083982
0.017	0.0	$Y = 0.200830 + 0.946100X$	0.184448
0.063	0.0	$Y = 1.009669 + 0.765565X$	0.107210
0.0	1.0	$Y = 0.708920 + 1.252846X$	0.122590
0.063	1.0	$Y = 0.834905 + 1.417389X$	0.147662

(a) $Y = a + bX$, where Y = logarithm of weight loss per unit area (mg/cm^2) and X = logarithm of test duration, hours.

(b) Standard Error of Estimate in terms of logarithms.

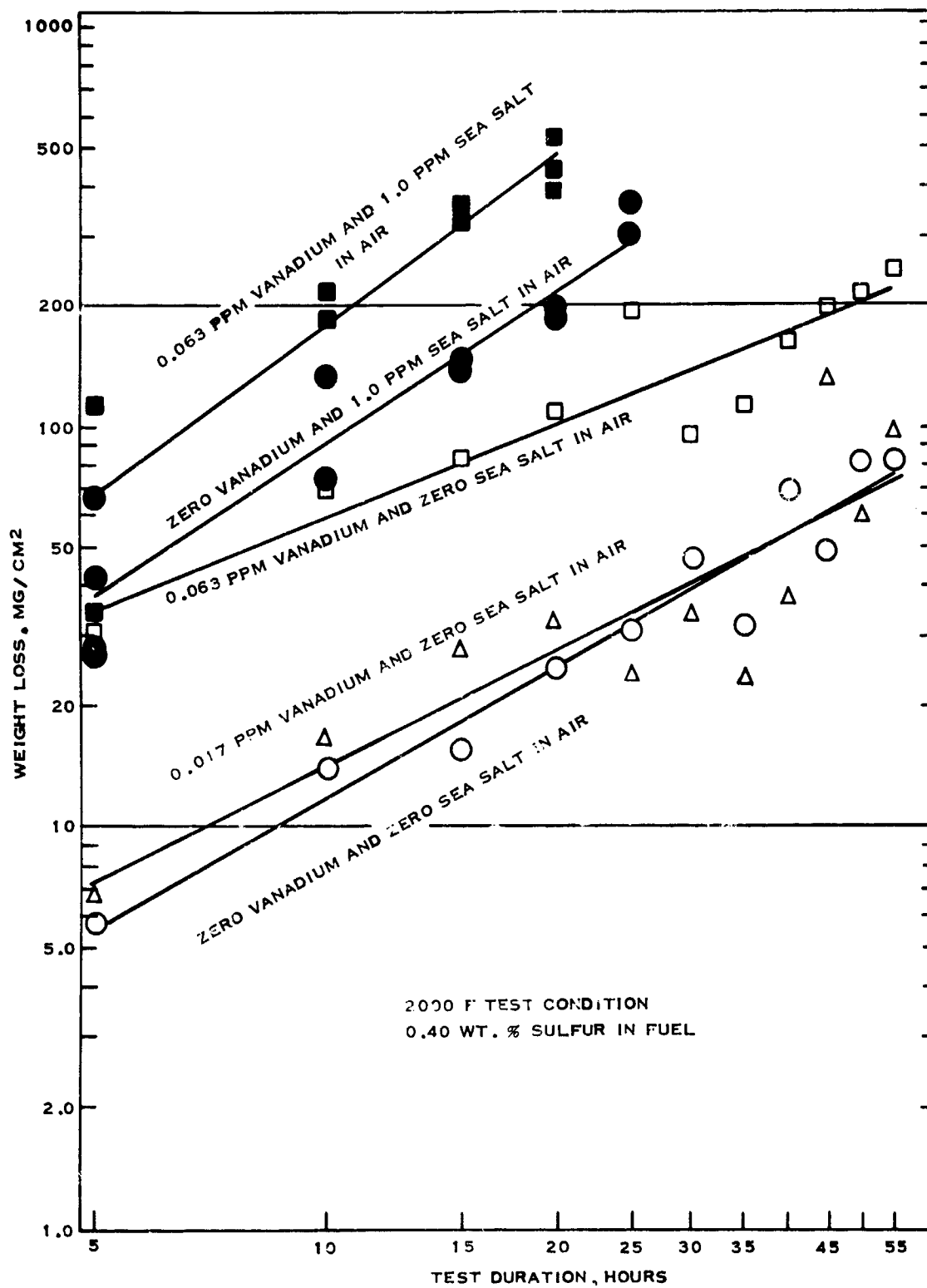


FIGURE 34
HOT CORROSION OF INCONEL 713C

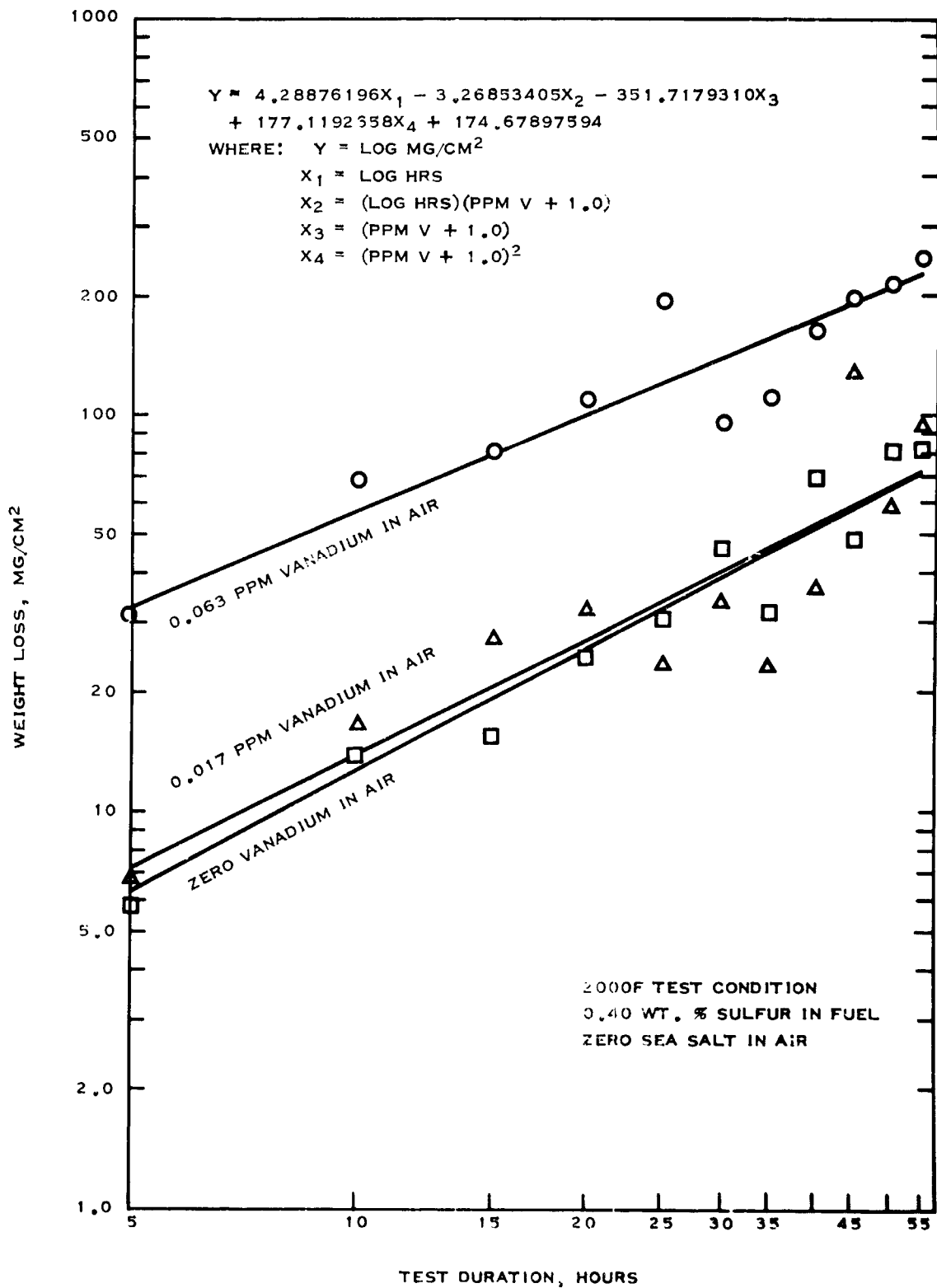


FIGURE 35
HOT CORROSION OF INCONEL 713C

This investigation has shown that the presence of 0.063 ppm vanadium in air (equivalent to 3.8 ppm vanadium in fuel at the 2000 F test conditions) more than doubles hot corrosion of Inconel 713C specimens with either zero or 1.0 ppm sea salt in air. The presence of 0.017 ppm vanadium in air (equivalent to 1.0 ppm vanadium in fuel at the 2000 F test conditions) did not significantly increase hot corrosion of Inconel 713C in the absence of sea salt in air. Additional data are needed before a firm conclusion can be drawn as to the effect of vanadium on hot corrosion of superalloys used in the hot section of gas turbine engines. The effect of 0.017 ppm vanadium in air in the presence of 1.0 ppm sea salt in air on hot corrosion of Inconel 713C and the effect of 0.017 ppm vanadium in air with zero and 1.0 ppm sea salt in air on hot corrosion of several coating-alloy systems should be obtained to complete the evaluation.

4.3. Effect of Temperature on Hot Corrosion

The purpose of this screening investigation was to examine 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) to aid in designing a program to evaluate the effect of sulfur, particularly at very low concentrations, in fuel on hot corrosion of coated superalloys.

Previous studies (1) showed that a coated superalloy was immune to attack under the conditions of exposure in the 5-hour test and thus did not provide an evaluation of the effect of sulfur in fuel on hot corrosion of the coated superalloy in a marine environment. In further studies (Section 4.1.) of hot corrosion of coated superalloys at the 2000 F condition it was found that coating failure could be induced by extending exposure time to cover a range of from 5 up to 55 hours and thus provide a measure of the effect of sulfur in fuel on hot corrosion of coated superalloys. As demonstrated in Section 4.1., sea salt is a primary cause of hot corrosion; however, studies with a coated superalloy (5) have shown a large experimental error when test severity was accelerated by increasing the concentration of sea salt in air from a realistic level of 1.0 ppm to 10.0 ppm and it has been concluded that testing should be confined to sea salt concentrations of 1.0 ppm sea salt in air or less. Thus at a given temperature, the length of exposure is the one route available for increasing test severity.

Specimens of Misco MDC-9 coated Inconel 713C were available for this screening program from the same batch of material used in the program reported in Section 4.1. and one of the combinations of sulfur in fuel and sea salt in air was selected to permit the use of data from Section 4.1. for one temperature in this investigation.

To simulate the environment in the turbine section of an aircraft engine, a cascade holder supporting six test specimens was mounted in the exhaust section of the Phillips 2-inch combustor. The facility was operated to obtain exposure of the test specimens at high pressure (15 atmospheres), over a range of temperatures from 1400 to 2000 F, with velocities from 565 to 745 feet per second at the test specimens with air fuel ratios of from 60 to 120.

Further details concerning the test equipment, test materials, and test program are presented in Appendices 1, 2, and 3 respectively, which are Sections 8., 9., and 10. of this report.

Visual and metallographic examination and weight loss of specimens will be used to evaluate the effect of temperature on hot corrosion and thus aid in designing a future program to evaluate the effect of sulfur in fuel on hot corrosion of coated superalloys exposed in a marine environment over a range of temperatures.

4.3.1. Visual

The condition of representative Misco MDC-9 coated Inconel 713C specimens following exposure at the four temperature conditions is shown by photomicrographs (2X magnification) of specimens mounted in the holder after 45 hours of exposure (Figure 36). Deposits decrease and hot corrosion increases with an increase in exposure temperature.

The appearance of cleaned specimens following exposure for 45 hours at the four temperature conditions is shown by photomicrographs (2X magnification) in Figure 37. After 45 hours of exposure specimens from tests at 1400 and 1600 F show little or no evidence of attack. At the 1800 F condition two small areas of attack are shown and at the 2000 F condition attack is general covering the entire surface of the specimen. This figure also shows that the cleaning technique removes a substantial portion of surface deposit or scale and provides specimens that are visibly clean. The one exception was the presence on some specimens of material on the portion of the specimen in contact with the holder and this has been identified in Section 10.2 (Appendix 3) as pick up of material from the holder.

4.3.2. Deposits

Many investigators feel that deposition of sodium sulfate on the metal surface is a normal precursor to hot corrosion. The range of temperature covered in the current investigation permitted an evaluation of the amount and composition of deposits on test specimens and their effect on hot corrosion.

The weights of deposits on the specimens were obtained as the difference between the weight of a specimen upon removal from the holder and the weight of the specimen after cleaning. By oversight, some of the specimens were not weighed upon removal from the holders; however, the deposit weights on the specimens weighed are shown in Table 11. The weight of deposits on the specimens decreased with an increase in exposure temperature.

From thermodynamic equilibrium data we have calculated the phase relationship for sodium sulfate over a range of temperatures and pressures. These data are shown in Figure 38. In these calculations it was assumed that all of the sodium in the sea salt was converted to sodium sulfate, with sulfur oxides furnished by combustion of the fuel. In this figure, for a given combination of temperature and sea salt in air, if the point of intersection falls to the left of the appropriate pressure line sodium

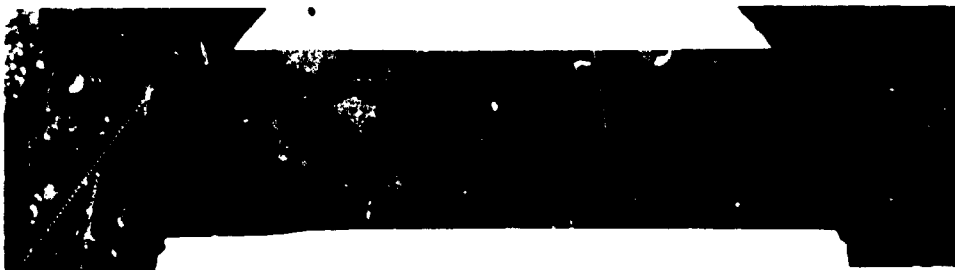
TEST
CONDITION,
F



2000



1800



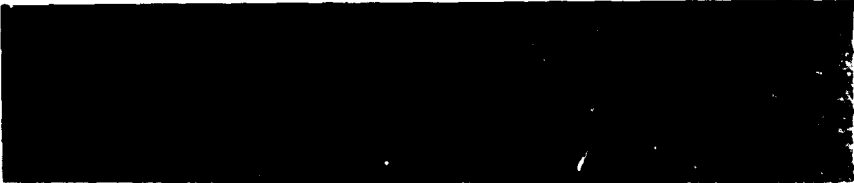
1600



1400

2X MAGNIFICATION OF MISCO MDC-9 COATED INCONEL 713C SPECIMENS
MOUNTED IN HOLDER AFTER EXPOSURE FOR 45 HOURS
WITH 1 PPM SEA SALT IN AIR AND 0.4 WT % SULFUR IN FUEL

FIGURE 36
EFFECT OF EXPOSURE TEMPERATURE ON ACCUMULATION OF
DEPOSITS ON SPECIMENS

	TEST	SPECIMEN
	CONDITION, F	WEIGHT LOSS MG/CM ²
	2000	114.7
	1800	2.4
	1600	0.3 GAIN
	1400	0.5 GAIN

2X MAGNIFICATION AFTER SONICALLY-CLEANING SPECIMENS
EXPOSED FOR 45 HOURS WITH 1 PPM SEA SALT IN AIR AND
0.40 WT % SULFUR IN FUEL

FIGURE 37
EFFECT OF EXPOSURE TEMPERATURE ON HOT CORROSION
OF MISCO MDC-9 COATED INCONEL 713C SPECIMENS

TABLE 11

DEPOSIT WEIGHT DATA FOR MISCO MDC-9 COATED INCONEL 713C
(1.0 ppm Sea Salt in Air and 0.4 wt. % Sulfur in Fuel)

<u>Temperature,</u> <u>F</u>	<u>Test (a)</u> <u>Specimen</u> <u>Number</u>	<u>Total</u> <u>Exposure</u> <u>Time,</u> <u>hours</u>	<u>Deposit Weight (b)</u>	
			<u>mg</u>	<u>mg/cm²</u>
1400	2	15	215.0	10.61
1400	6	15	160.7	7.93
1400	3	30	330.1	16.28
1600	2	15	113.4	5.59
1600	6	15	86.2	4.25
1600	3	30	139.3	6.87
1800	2	15	17.2	0.85
1800	6	15	14.7	0.72
1800	3A	15	29.8	1.47
1800	5	30	24.1	1.19
1800	2A	30	39.6	1.95
1800	6A	30	33.7	1.66
1800	1	45	57.2	2.82
1800	4	45	67.6	3.34
1800	5	45	39.3	1.94

- (a) Test specimen number indicates position in cascade holder.
 A letter following the position number indicates a replacement test specimen, following removal of the initial test specimen.
- (b) Obtained by difference between weight of specimen after test and weight of specimen after cleaning.

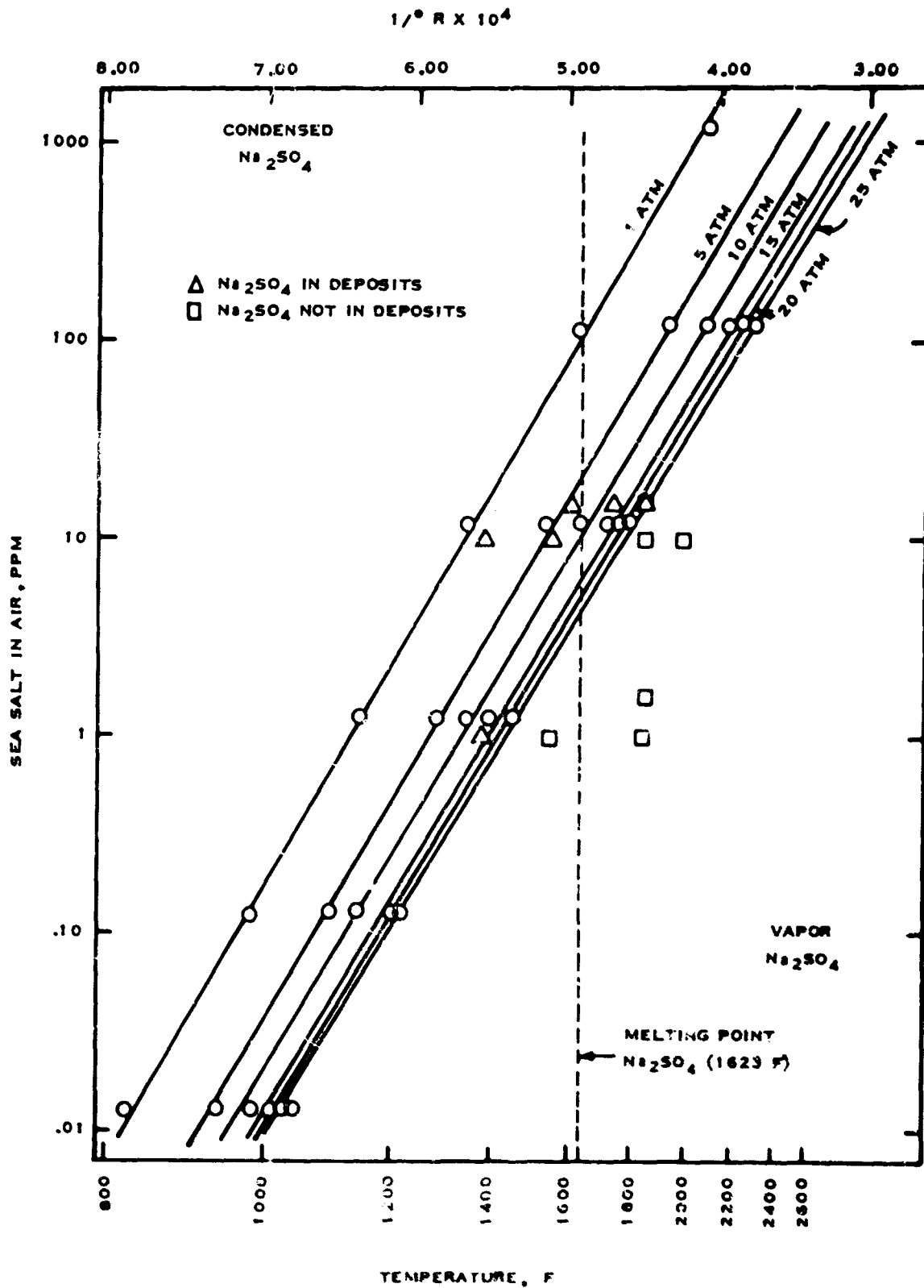


FIGURE 38
PHASE RELATIONSHIP FOR SODIUM SULFATE

sulfate would be present in the condensed state and could deposit on test specimens. If the point of intersection is to the right of the pressure line sodium sulfate would exist only in the vapor state and would not deposit on test specimens. For the current screening program with specimens exposed at 15 atmospheres pressure with 1.0 ppm sea salt in air, sodium sulfate would be expected to condense as a solid on specimens exposed only at 1400 F test condition (1375 F calculated specimen temperature).

Analysis of deposits on the Misco MDC-9 coated Inconel 713C specimens exposed for 45 hours at the 1400, 1600, and 1800 F conditions were obtained and the data are shown in Table 12. An analysis of the surface of the deposits was obtained by x-ray diffraction with the deposit in place, and analyses of total deposits were obtained on a sample of powdered deposits removed by scraping. Four forms of sodium sulfate were identified both on the surface and in the total deposit from the specimen exposed at the 1400 F condition; but sodium sulfate was not detected in the deposits on specimens exposed at higher temperatures. These data are shown in Figure 38 where a triangle indicates the presence and a square the absence of sodium sulfate in the deposits from specimens exposed at the combination of specimen temperature and sea salt in air. In previous studies (6,7) similar analyses were obtained on deposits from specimens exposed at 1400 to 2000 F test conditions with 1.5, 10.0, or 15.0 ppm sea salt in air and these data are also shown in Figure 38. With one exception the presence or absence of sodium sulfate found, by analyses, in the deposits agree with the prediction of the figure. Sulfur was found in the sample of total deposits for each test condition in the current investigation. Its concentration at the 1400 F condition was that expected for sodium sulfate, but was much smaller at the 1600 and 1800 F conditions. At the two higher temperatures, where sodium sulfate was not detected, sulfur is present in either amorphous material or in the complex, unidentified, salts.

Under the conditions where sodium sulfate was found in the deposits, metal oxides were not found; while at the higher temperatures, where sodium sulfate was not present, metal oxides were found. This indicates that the condensation of sodium sulfate is not a requisite for hot corrosion.

4.3.3. Metallography

Cross-section coupons from selected specimens of Misco MDC-9 coated Inconel 713C were examined; as detailed in Appendix 3, Section 10.3., of this report. The mode and intensity of corrosive attack was evaluated following exposure over the range of conditions of this investigation. In particular, evidence was sought concerning the absence of deep intercrystalline corrosion; which must be established for our use of loss-in-weight by the specimen to be a valid measure of metal damage.

A metallographic cross-section of a typical MDC-9 coated Inconel 713C specimen as received from Misco is shown in Figure 15, and the coating is described in Section 4.1.2.

TABLE 12

DEPOSIT ANALYSIS DATA FOR MISCO MDC-9 COATED INCONEL 713C
 (1.0 ppm Sea Salt in Air and 0.40 wt. % Sulfur in Fuel)
 (45 hour Exposure Specimens)

<u>Test Condition. F</u>	<u>1400</u>		<u>1600</u>		<u>1800</u>	
<u>Deposit Location</u>	<u>Surface</u>	<u>Total</u>	<u>Surface</u>	<u>Total</u>	<u>Surface</u>	<u>Total</u>
Sulfur, wt % (a)		22.2		9.1		3.7

Unidentified (b)	X	X (c)	X	X	X	X
Corundum Al_2O_3	-	-	-	-	X	-
Nickel II Chromite $NiCr_2O_4$	-	-	-	X	X	X
Aluminum Nickel $AlNi_3$	-	-	X	-	X	-
Bunsenite NiO	-	-	-	X	-	X
Chromium	-	-	X	-	-	-
Thenardite Na_2SO_4	X	X	-	-	-	-
Metathenardite Na_2SO_4	X	X	-	-	-	-
Sodium Sulfate, Form III Na_2SO_4	X	X	-	-	-	-
alpha Sodium Sulfate I Na_2SO_4	X	X	-	-	-	-

Notes:

(a) ASTM D-1552

(b) X-ray Diffraction (X = pattern present)

(c) Same as surface deposit unidentified

Exposure of Misco MDC-9 coated Inconel 713C specimens for test durations of up to 45 hours in a sea-salt environment at the 1400, 1600, and 1800 F test conditions showed them to be very resistant to hot corrosion. Metallographic cross-sections of such specimens are shown in Figures 39, 40, and 41 respectively. In general, the coatings remained intact; however, with increasing exposure temperature there was evidence of increasing separation of the coating remnant by corrosion products between its layers. This effect may have resulted in the cracks which were found in specimens exposed at the 2000 F test condition (Section 4.1.2.). Also, there is evidence of gradual diffusion of the aluminum and chromium into the base alloy, which depletes the diffused-layer; and this increased with exposure temperature to become quite prominent at the 2000 F test condition.

No evidence of corrosive attack on the base alloy was found in specimens exposed at the 1400 and 1600 F test conditions. In those areas where the MDC-9 coating was penetrated at the 1800 F test condition, the mode and intensity of attack was similar to that observed with bare Inconel 713C (Section 4.1.2.). The accelerated-oxidation attack that is typical of hot corrosion is shown in Figure 42. It should be noted that, characteristically, the attack advances on a broad front without deep-intercrystalline penetration of sulfides or oxides.

Normally, the corrosive attack experienced by the Inconel 713C specimens during this investigation did not penetrate the Misco MDC-9 coating; but when it did, the depth of surface penetration was usually less than one mil. This serves to justify the use of the metal-loss data as a valid measurement of the extent of corrosive attack on this coating-alloy system over the range of conditions investigated.

4.3.4. Weight Loss

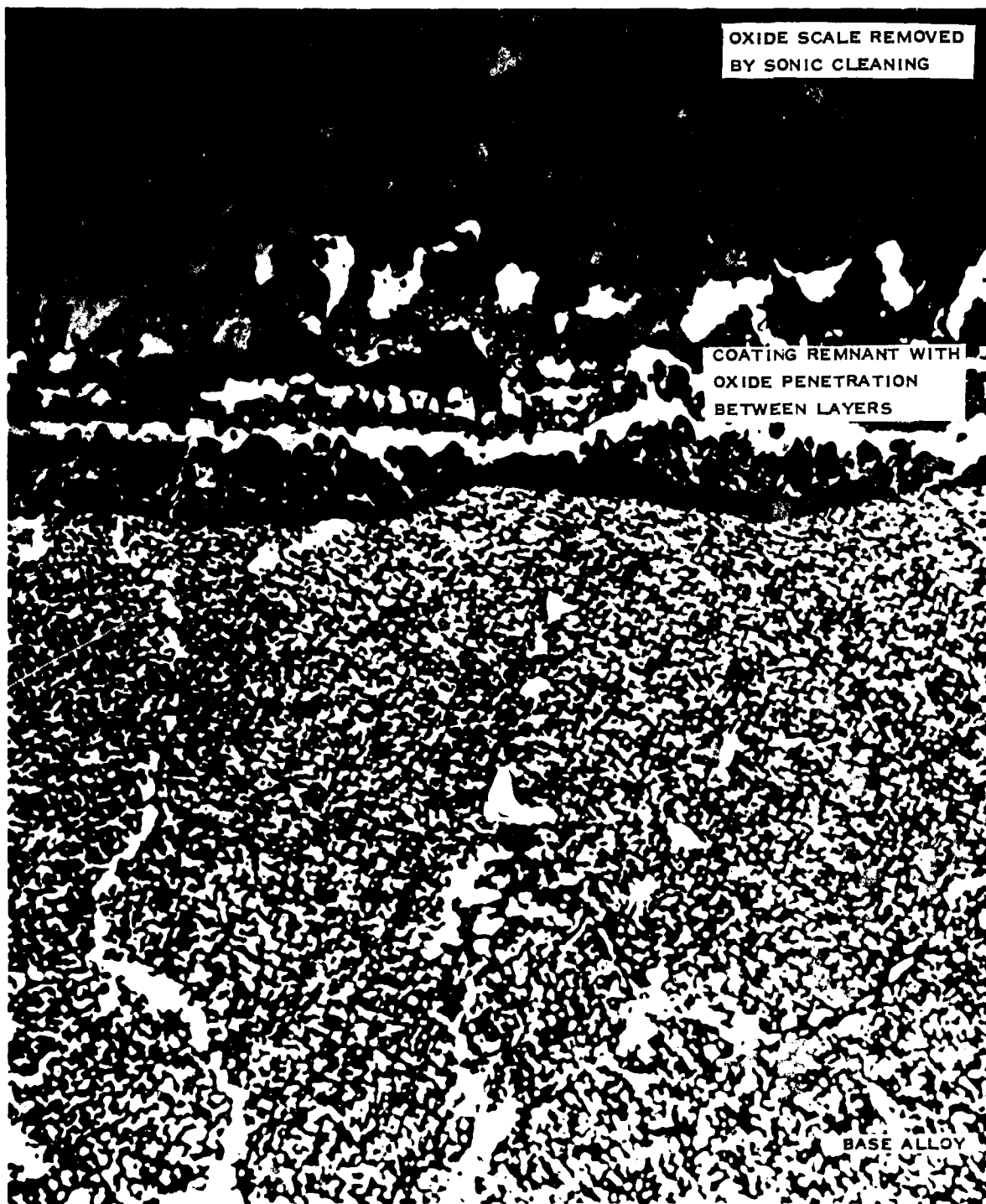
As discussed in Section 4.1.3., the use of logarithms have been shown to provide a basis of uniform variance for weight-loss data and an exponential growth curve has expressed the relationship of weight-loss with time of exposure. The data in the current investigation will be treated in the same manner.

The weight-loss per unit area for specimens exposed at 1400, 1600, and 1800 F for various periods of time in this program are shown in Table 13. Data for specimens exposed at 2000 F condition in a previous program are also included in the table. As discussed in Section 10.2, Appendix 3, the method of cleaning exposed specimens was modified for this investigation to include scrubbing the specimens with a stainless-steel-wire brush. This cleaning technique removed an average of 6.8 mg of deposits not removed by the fiber brush in the previous investigation. To place all of the data on a uniform basis, the weight losses for specimens exposed at the 2000 F condition were adjusted by the addition of 6.8 mg before converting to weight loss per unit area. To eliminate negative numbers, and permit the use of logarithms, all of the weight loss per unit area data were adjusted by the addition of 2.0 mg/cm².



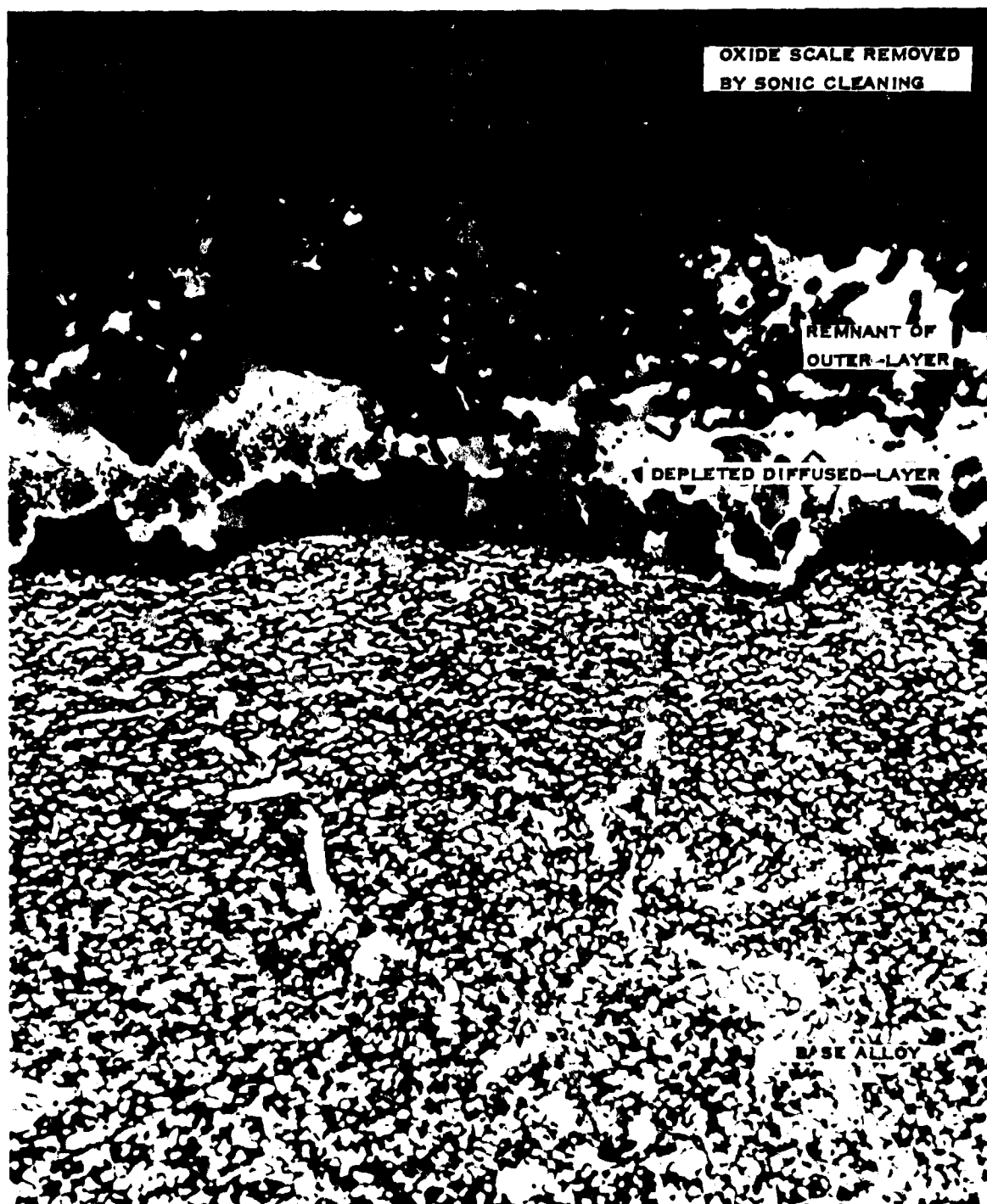
METALLOGRAPHIC CROSS-SECTION OF SPECIMEN AFTER 45 HOURS AT 1400 F TEST
CONDITION WITH 1.0 PPM SEA SALT IN AIR AND 0.40 WT % SULFUR IN FUEL.
3% SULFURIC ACID-ELECTROLYTIC ETCHED. 1000X MAGNIFICATION.

FIGURE 39
OXIDATION OF MISCO MDC-9 COATING ON INCONEL 713C SPECIMEN AT 1400 F



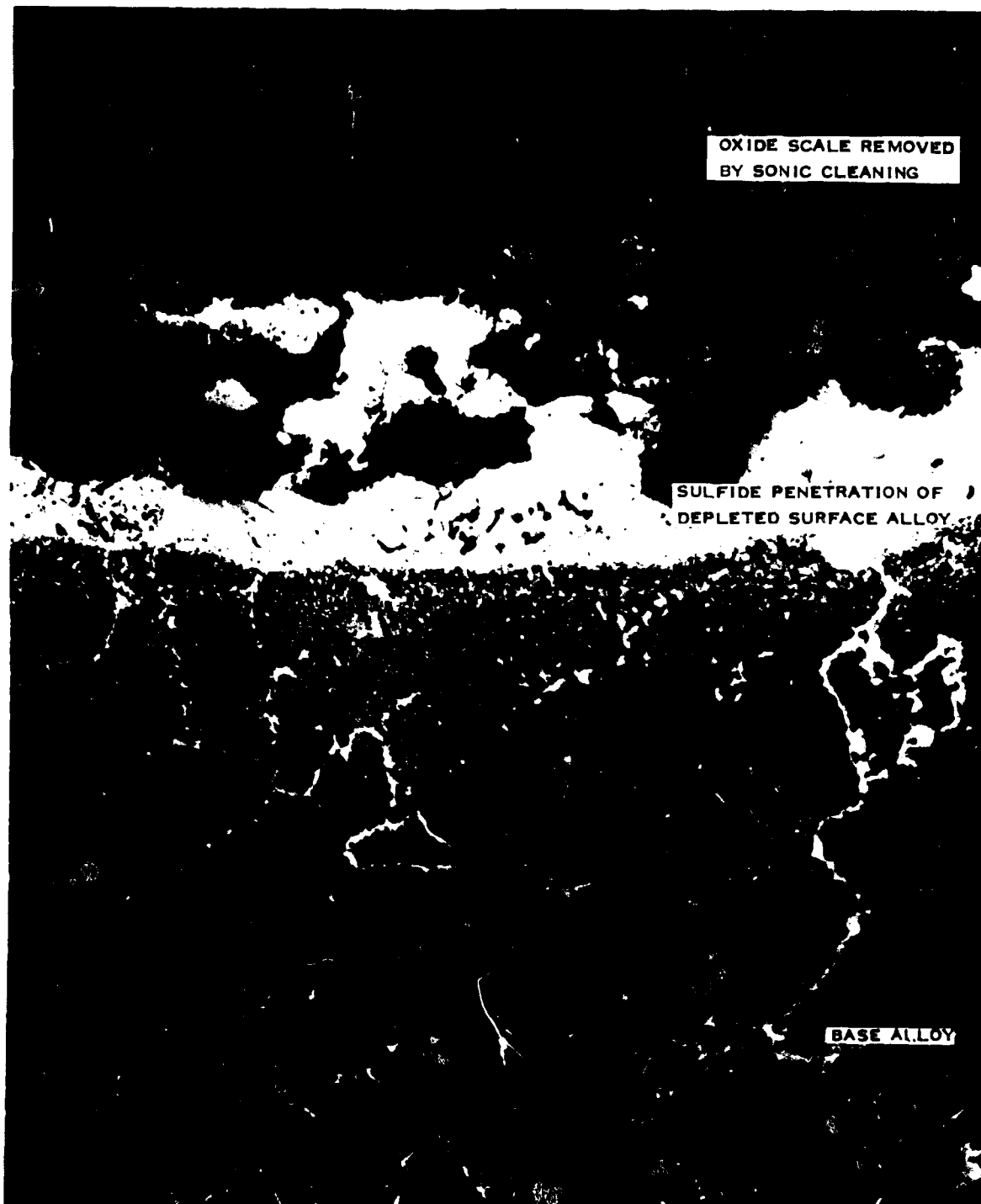
METALLOGRAPHIC CROSS-SECTION OF SPECIMEN AFTER 45 HOURS AT 1600 F TEST
CONDITION WITH 1.0 PPM SEA SALT IN AIR AND 0.40 WT % SULFUR IN FUEL.
3% SULFURIC ACID-ELECTROLYTIC ETCHED. 1000X MAGNIFICATION.

FIGURE 40
OXIDATION OF MISCO MDC-9 COATING ON INCONEL 713C SPECIMEN AT 1600 F



METALLOGRAPHIC CROSS-SECTION OF SPECIMEN AFTER 45 HOURS AT 1800 F TEST
CONDITION WITH 1.0 PPM SEA SALT IN AIR AND 0.40 WT % SULFUR IN FUEL,
3% SULFURIC ACID-ELECTROLYTIC ETCHED. 1000X MAGNIFICATION.

FIGURE 41
OXIDATION OF MISCO MDC-9 COATING ON INCONEL 713C SPECIMEN AT 1800 F



METALLOGRAPHIC CROSS-SECTION OF SPECIMEN AFTER 45 HOURS AT 1800 F TEST
CONDITION WITH 1.0 PPM SEA SALT IN AIR AND 0.40 WT % SULFUR IN FUEL.
2% SULFURIC ACID-ELECTROLYTIC ETCHED. 400X MAGNIFICATION.

FIGURE 42
ACCELERATED OXIDATION OF MISCO MDC-9 COATED INCONEL 713C SPECIMEN

TABLE 13

WEIGHT-LOSS DATA FOR MISCO MDC-9 COATED INCONEL 713C SPECIMENS
 (1.0 ppm Sea Salt in Air and 0.4 wt % Sulfur in Fuel)

Total Time, hours	Test Specimen Weight Loss, mg/cm ² (a)			
	<u>1400 F</u>	<u>1600 F</u>	<u>1800 F</u>	<u>2000 F (b)</u>
5	-	-	-	2.09
10	-	-	-	2.58
15	1.51	2.10	0.52	4.38
15	1.75	2.07	1.39	-
15	1.66	1.76	1.46	-
20	-	-	-	1.98
20	-	-	-	13.13
25	-	-	-	5.15
25	-	-	-	67.83
30	1.56	1.80	0.59	91.48
30	1.31	1.64	1.84	-
30	1.67	1.64	1.39	-
35	-	-	-	47.48
40	-	-	-	68.27
45	1.52	1.74	4.44	117.00
45	1.30	2.44	4.71	-
45	1.67	2.36	2.11	-

(a) Weight loss adjusted by adding 2.0 mg/cm².

(b) Initial weight loss increased by 6.8 mg to correct for the additional deposits removed by wire brushing specimens from tests at lower temperatures.

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The adjusted weight loss data for each temperature were fit to equations of the form of Equation 3 (Section 4.1.3.). The constants, b, for the equations at each of the four temperatures were tested for statistical significance (95 per cent confidence level) by the use of a statistical "t" test. It was found that, b, was not significantly different from zero for exposure temperatures of 1400 and 1600 F; however, the values were significantly different from zero at 1800 and 2000 F. Since the relative rates of corrosion were not significantly different from zero at 1400 and 1600 F the best estimate of weight loss at each temperature is a mean of the nine determinations. The equations for the two temperature conditions (1800 and 2000 F) showing a significant relative rate of corrosion are shown in Table 14.

TABLE 14

REGRESSION EQUATIONS FOR WEIGHT-LOSS VS TIME

Exposure Temp., F	Regression Equations (a)	S.E.E. (b)
1800	$Y = -0.335227 + 0.018014 X$	0.248247
2000	$Y = -0.042543 + 0.049406 X$	0.394859

(a) $Y = a + bX$, where Y = logarithm of weight loss per unit area and X = test duration, hours.

(b) Standard Error of Estimate in terms of logarithms.

Using the statistical "t" test it was determined that the relative rate of corrosion at 2000 F exposure temperature was significantly greater than at 1800 F. From an Analysis of Variance (95 per cent confidence level) of the data at 1400 and 1600 F it was determined that the weight loss for specimens exposed at 1600 F was greater than for specimens exposed at 1400 F. A comparison of the effect of temperature on weight loss can be made from the following calculated adjusted weight loss values for 45 hours of exposure.

Temperature, F	Adjusted Weight Loss, mg/cm ²
1400	1.47
1600	2.02
1800	2.99
2000	151.6

These data show an accelerating attack with increasing temperature. There is no indication of an increase in attack in the presence of sodium sulfate (1400 F) as has been indicated by some investigators.

The purpose of the current investigation was to obtain data to aid in designing a program to evaluate the effect of sulfur, particularly at very low concentrations, in fuel on hot corrosion of coated superalloys in a marine environment. In such a program the severity of the test at each condition should be sufficient to establish the relative rate of hot corrosion and it would be desirable to penetrate the coating and thus provide a measure of coating life without extrapolation of regression equations.

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In Section 4.1.4. a criterion of 5.0 mg/cm^2 was selected as the point of coating failure. Since the data in this screening program have been adjusted by the addition of 2.0 mg/cm^2 the criterion for coating failure should be adjusted to 7.0 mg/cm^2 . Direct estimates of the exposure time required for coating failure with Misco MDC-9 coated Inconel 713C specimens exposed in the presence of 0.40 weight per cent sulfur in fuel and 1.0 ppm sea salt in air can be obtained from the regression equations for 1800 and 2000 F exposure conditions. The calculated time to failure at the 2000 F condition is 18 hours and from an extrapolation of the regression equation the calculated time to failure at 1800 F is 66 hours. No direct estimate of exposure time for coating failure at the 1400 and 1600 F conditions is available since no significant change in weight loss with time was found in the 45 hours of exposure in this screening program. Rough estimates of the minimum time of exposure for coating failure, ignoring any possible interaction with temperature, can be obtained by linear extrapolations of time to failure vs temperature and such extrapolations indicate a time to coating failure of 110 hours at the 1600 F condition and 160 hours at the 1400 F condition.

In Section 4.1. it is shown that sea salt in air is a primary cause of hot corrosion. In Table 4 it is shown that a reduction of sea salt in air at the 2000 F condition with 0.40 weight per cent sulfur in fuel increased time to failure for each of the coating-alloy systems by a factor of four. While no data are available as to the effect of sea salt in air on coating life at temperatures below 2000 F, it would be expected that similar increases in coating life would be found at the lower temperature conditions.

While it has not been firmly established by this investigation it is apparent that exposure times of several hundred hours, or more, will be required to obtain coating failure for some coating-alloy systems at some conditions.

A program to evaluate the effect of sulfur in fuel (including very low levels) on hot corrosion of coated superalloys in a marine environment over a range of temperatures should include at least three levels of sulfur in fuel, two levels of sea salt in air and four levels of temperature for each of several coating-alloy systems. To establish the life of a coating-alloy system at each combination of sulfur in fuel, sea salt in air and exposure temperature, specimens with varied exposure times are required. With the current test section in the Phillips Hot Corrosion Test Rig, which holds six test specimens, a program requiring specimen exposures of several hundred hours or more becomes prohibitive with respect to manpower and materials. The obvious method of reducing the program to a manageable size is to increase the number of specimens that can be mounted in the test section to the point that sufficient specimens of each coating-alloy system of interest can be included in each test and thus require operation at each sea salt in air, sulfur in fuel and temperature only one time. The design of such a test section will be discussed in Section 4.4.

4.4. Revised Test Section

It has been stated by Champion (8) that corrosion of metals, and particularly local corrosion, is dependent on the breakdown of a protective film on the metal or the distribution of nuclei on the metal, and these phenomena must be expected to obey the normal laws of chance to a large extent. Since corrosion is a natural process, there is a limit to the control which can be exercised to increase reproducibility without seriously interfering with the natural course of the process. General statistical methods are, therefore, a considerable aid in the design of corrosion tests and in the interpretation and expression of the results.

Statistical methods for analysis and interpretation of data depend upon obtaining a random sample of a population and upon obtaining an estimate of experimental error or a measure of repeatability. A sample must contain two or more observations to provide a measure of the experimental error and the larger the experimental error the larger the sample that is required to detect differences of a given size between two populations. Thus, in hot corrosion testing, multiple specimens should be used for each exposure condition. While corrosion is a random process and a large experimental error is expected from the process, the conditions for exposure should be maintained as uniformly as possible to minimize experimental error.

The screening program reported in Section 4.3. indicated that tests of several hundred hours duration or more would be required under some conditions to evaluate the effect of sulfur in fuel on coated superalloys exposed over a range of temperatures in both a marine and nonmarine environment. To complete the proposed study of the effect of sulfur in fuel on hot corrosion of superalloys over a range of temperatures a number of coating-alloy systems must be included to provide a satisfactory evaluation. The present test section for the Phillips Hot Corrosion Test Rig mounts only six test specimens and with this equipment the expenditure of time and materials for such an evaluation would be prohibitive. The obvious method for increasing productivity would be to revise the test section to provide simultaneous exposure of a larger number of test specimens.

In the design of a new test section the desirable features of the current section should be retained and the less desirable features improved. Primary features of the current test section that should be retained in a new design are capabilities for exposing specimens at high pressure, high temperature, and high velocity.

In the current test section the six specimens are mounted in three stages in a cascade with each successive stage rotated 120 degrees to prevent channeling of hot gases. While an effect of position of specimens in the cascade could not be detected in hot corrosion tests it is believed that a minor effect on hot corrosion could be present from small temperature differences among the three stages and from specimens in the second and third stages being in the shadow of prior specimens. These effects, if present, should be eliminated in a new design. With flat strips used in the current test section it is difficult to measure the amount of attack by visual means. The use of round pins as test specimens would permit an evaluation, by metallographic means, of the depth of penetration to supplement the use of weight-loss in evaluating the extent of attack on test specimens.

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A cross section of a new test section, designed to meet the above objectives, is shown in Figure 43. In this design round pins are used as test specimens (Figure 44). Forty eight specimens are mounted in a retainer (Figure 45) and are rotated in the hot gas stream from the Phillips 2-inch combustor. A view of this test section mounted in the hot-corrosion test-way is shown in Figure 46. 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 worm-gear drive is provided to rotate the specimen retainer 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 $7\frac{1}{2}$ degrees from the prior row and thus no specimen is in the shadow of another specimen.

The Phillips 2-inch combustor will be operated in the same manner with the new test section as with the cascade test section and the pressure and temperature capabilities of the test facility will be maintained. The unblocked area at the test specimens is greater with the new test section than with the previous section (5.04 vs. 1.59 square inches) and the gas velocity with the new test section will be less. While the gas velocity at the test specimens, at the 2000 F condition, will be reduced from 745 to 235 feet per second the new test section can still be considered to operate at a high gas velocity.

The 48 positions for test specimens in the new test section will permit simultaneous exposure of multiple specimens of a number of superalloys and coating-alloy systems in a program to evaluate the effect of sulfur in fuel on hot corrosion in a marine environment over a range of temperatures. The ability to simultaneously expose the superalloys and coating-alloy systems of interest to each combination of sulfur in fuel, sea salt in air, and exhaust gas temperature will reduce the time and materials required for the program to a realistic level. Multiple specimens of each alloy will also permit a better estimate of experimental error for use in the evaluation.

Fabrication of a test section of the new design has been completed and a program initiated to determine, and correct, any mechanical problems. The following test schedule was set up for the preliminary evaluation of the new test section.

- (1) Operate the test section in place, cold, for a short period.
- (2) Operate the test section in place, with hot gas only, for a short period.
- (3) Operate the test section with fuel up to 2000 F condition for a short period of time.
- (4) Operate the test section with fuel and sea water up to 2000 F condition for a short period of time.

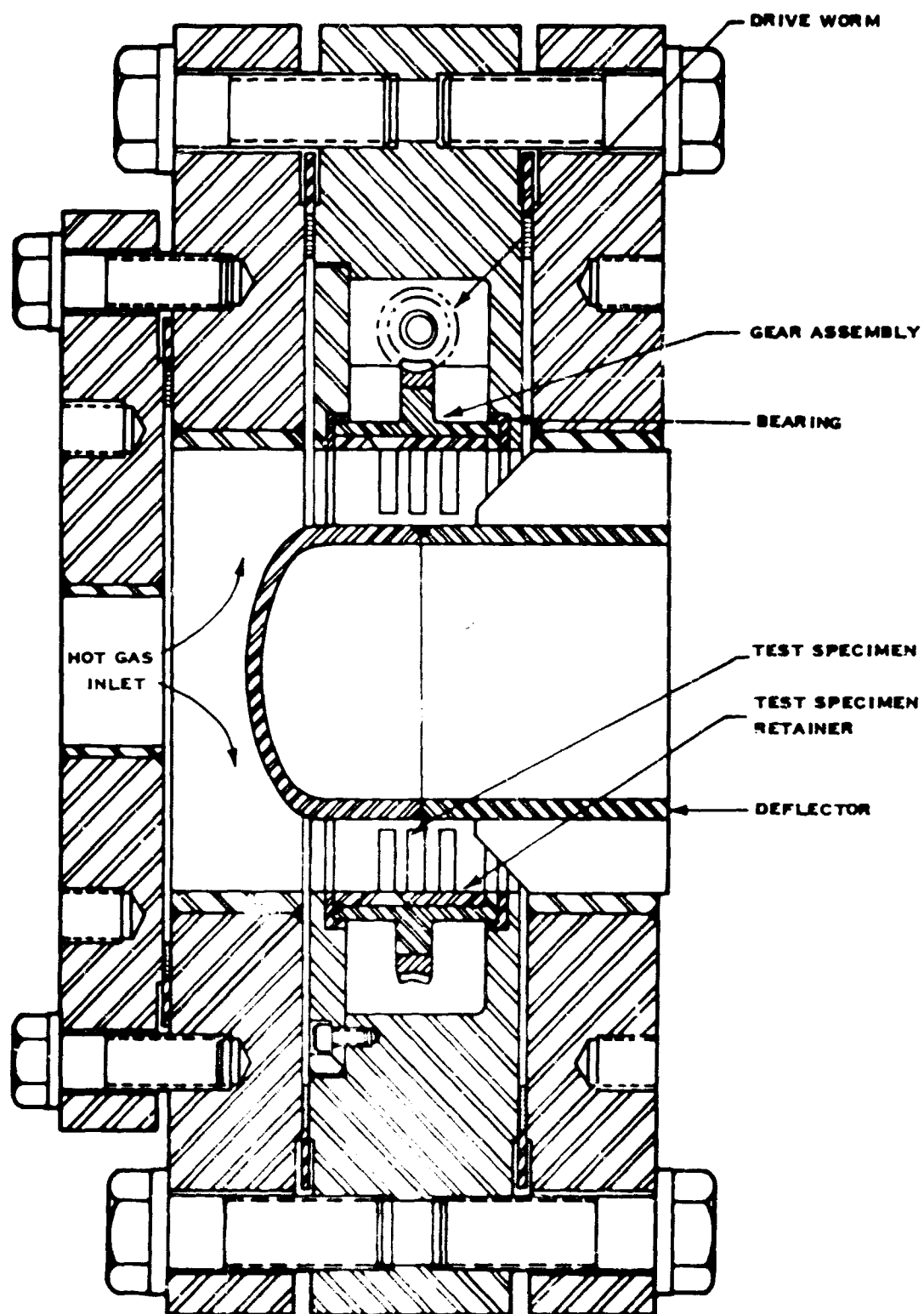


FIGURE 43
ROTATING SPECIMEN TEST SECTION FOR HOT CORROSION STUDIES

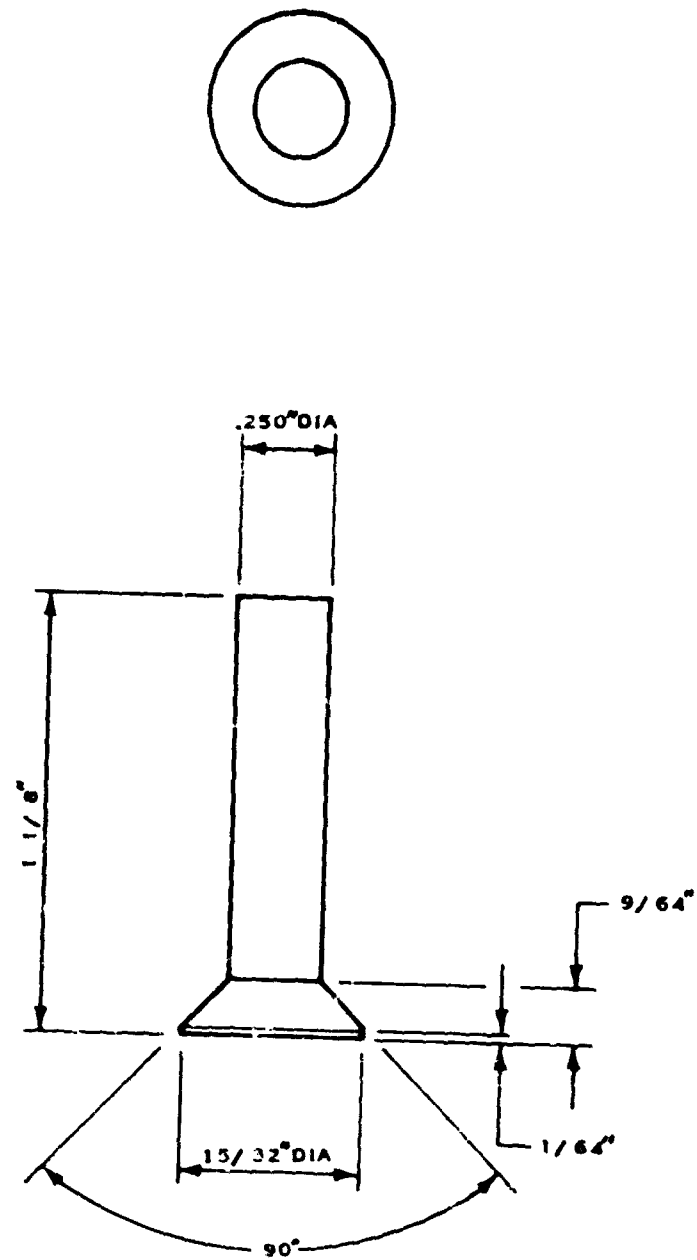


FIGURE 44
TEST SPECIMEN

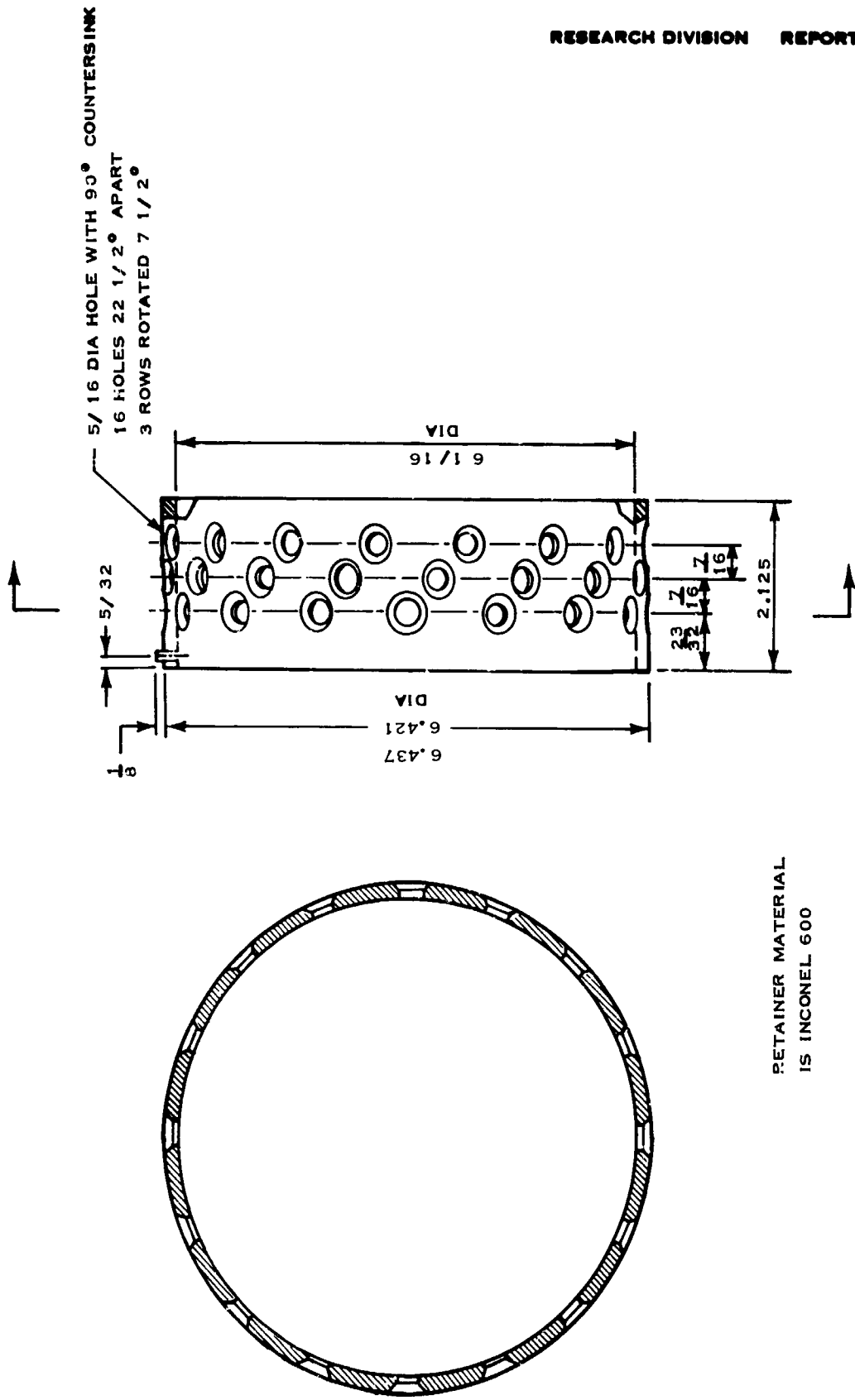


FIGURE 45
TEST SPECIMEN RETAINER

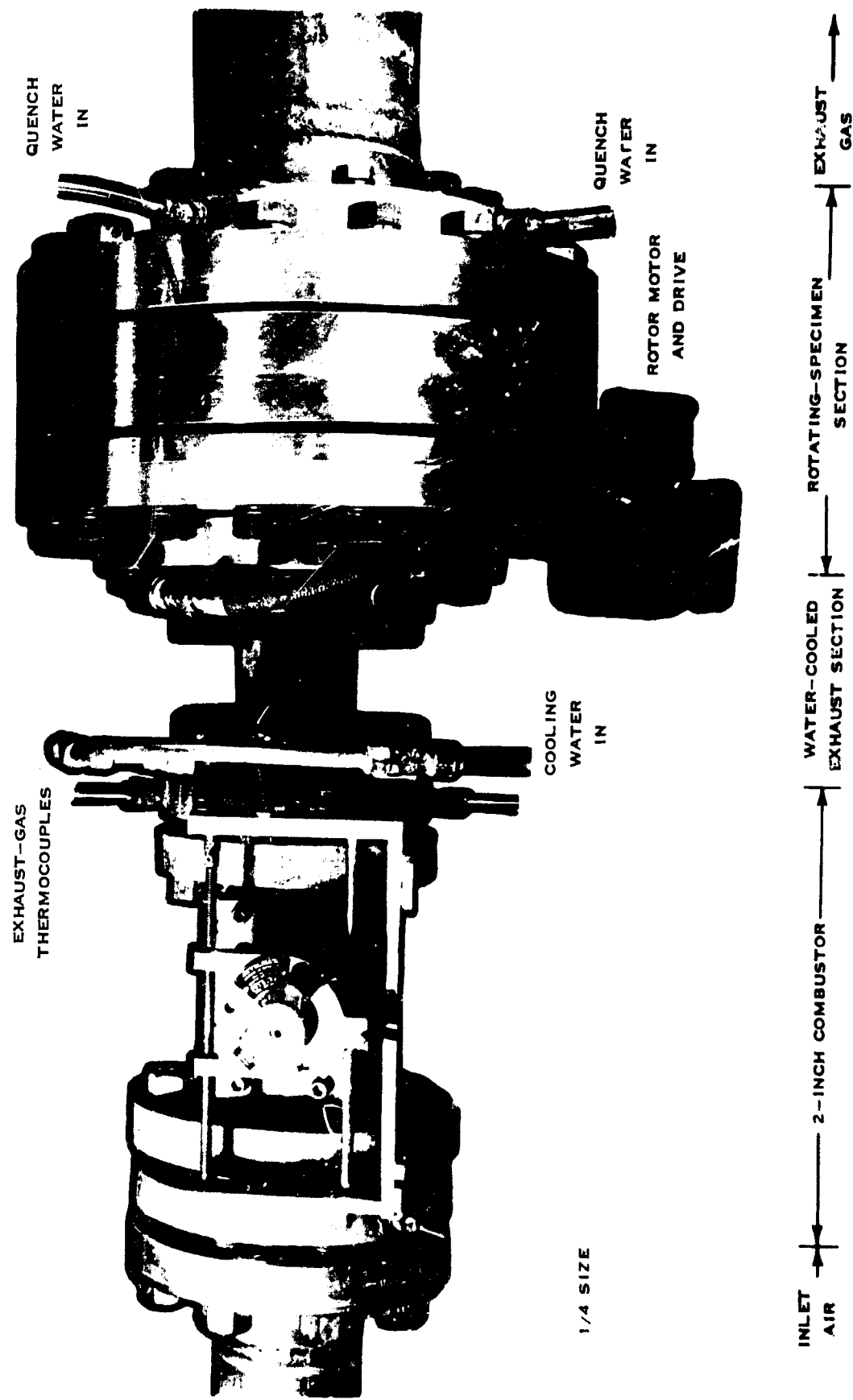


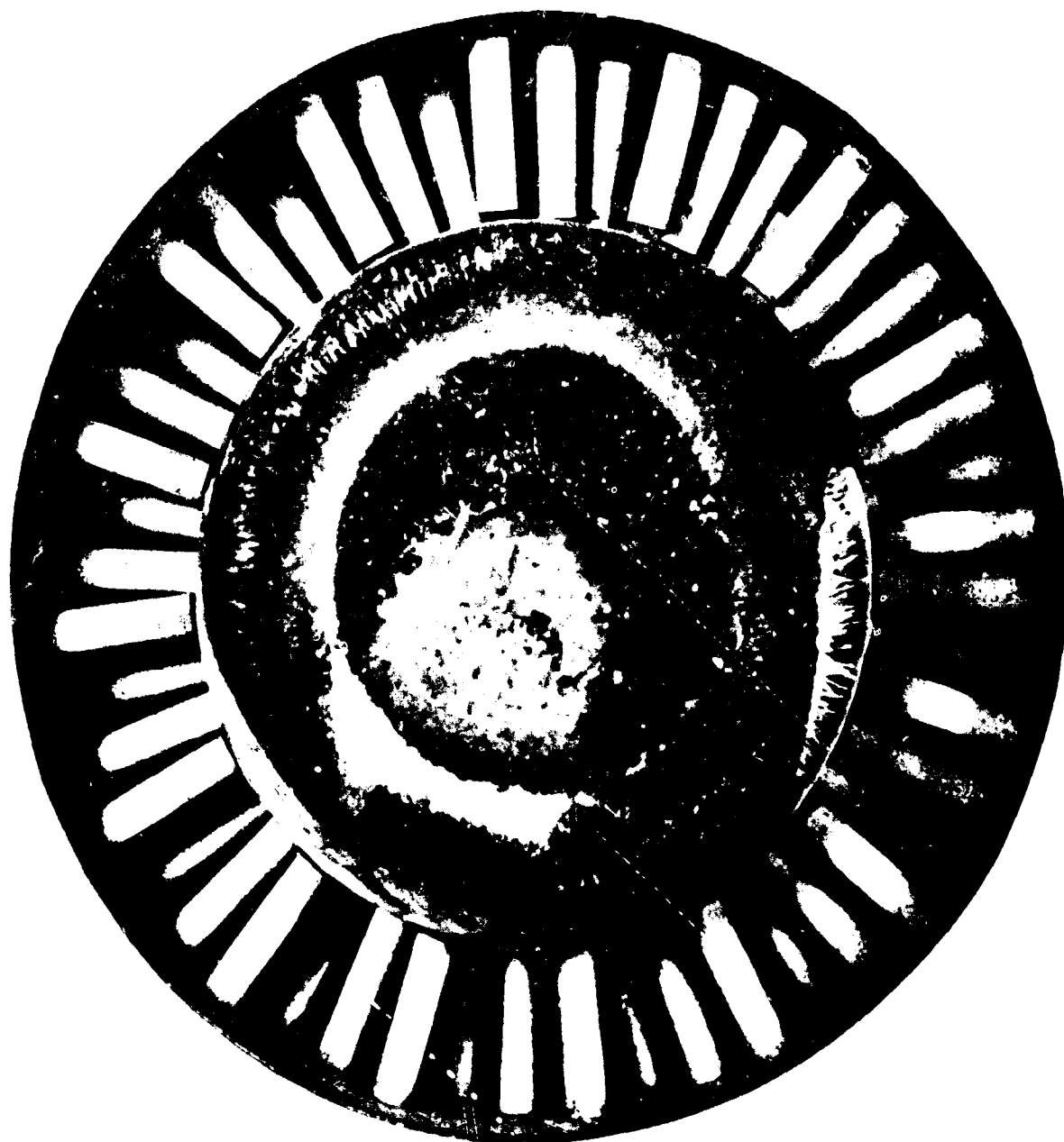
FIGURE 46
PHILLIPS ROTATING-SPECIMEN HOT-CORROSION TEST RIG

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- (5) Conduct a hot corrosion test, without sea salt, at 2000 F condition using Inconel 713C and Howmet MDC-1 coated Inconel 713C specimens with exposures of up to 55 hours.
- (6) Conduct a hot corrosion test, with 1.0 ppm sea salt in air, at 2000 F condition using Inconel 713C and Howmet MDC-1 coated Inconel 713C specimens with exposures of up to 55 hours.

After some modifications the test section was operated satisfactorily at 1200 F (Phase 2) with hot gas only. Mechanical problems developed when the test section was operated up to 2030 F with fuel and heated inlet air. These problems were attributed primarily to clearances although some changes in materials of construction were also indicated. Before removing the test section for major modifications a short test was conducted with 0.40 weight per cent sulfur in fuel and 10.0 ppm sea salt in air at 2000 F gas temperature without specimen rotation to determine whether the appearance of the specimens indicated any unusual distribution of the gas stream. The appearance of the specimens was uniform as indicated in Figure 47.

The mechanical and material problems are being corrected and upon completion the above program will be started again. With the successful completion of the program the test rig should be ready to conduct the planned program to evaluate the effect of sulfur in fuel on hot corrosion, in a marine environment, of a number of bare and coated superalloys over a range of temperatures.



FULL SIZE

FIGURE 47
DISTRIBUTION OF SEA-SALT RESIDUE IN ROTATING-SPECIMEN SECTION OF
PHILLIPS HOT-CORROSION TEST RIG AFTER 50 MINUTES AT 2000F TEST
CONDITION WITH 10 PPM SEA SALT IN AIR

5. FUTURE WORK

The effect of sulfur in fuel was found to vary among the various bare superalloys used in previous programs (1); i.e., a reduction in fuel sulfur had no effect with some superalloys, and increased hot corrosion with two superalloys under some conditions. With three coating-alloy systems 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) decreased 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 below the melting point of sodium sulfate (1523 F) with superalloys that were unaffected by changes in sulfur content at higher temperatures.

To complete the investigation of the effect of sulfur in fuel on hot corrosion of bare and coated superalloys in a marine environment a number of coating-alloy systems should be evaluated at one or more temperatures below and above the melting point of sodium sulfate. This investigation should include one or more low levels of fuel sulfur. From the current investigation it is estimated that exposure times of up to several hundred hours or more will be required at some conditions to measure the life of coatings. A new test section has been designed for the Phillips Hot Corrosion Test Rig that will permit the simultaneous exposure of 48 test specimens to the various corrosive environments. The increased number of test specimens that can be mounted in the test section should permit conducting the proposed program with a realistic expenditure of time and materials.

Preliminary evaluation of the new test section has indicated some mechanical and material problems which are being corrected. As soon as satisfactory performance of the new test section can be demonstrated a test program will be initiated to complete the evaluation of the effect of sulfur in fuel on hot corrosion of coated superalloys in a marine environment over a range of temperatures.

6. ACKNOWLEDGEMENTS

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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.

DEPOSIT ANALYSIS BY L. V. WILSON

THERMODYNAMIC EQUILIBRIUM STUDIES BY DR. N. W. RYAN AND LUCIEN BAGNETTO.

DESIGN OF TEST SECTION BY L. T. JOHNSON.



VELMA GOOCH

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2. Quigg, H. T. and Schirmer, R. M., "Effect of Sulfur in JP-5 Fuel on Hot Corrosion of Coated Superalloys in Marine Environment." Summary Report for Naval Air Systems Command Contract NOW 66-0263-d. Phillips Petroleum Company Research Division Report 4792-67R. Bartlesville, Oklahoma, June 1967, 71 pp.
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8. APPENDIX 1
(Test Equipment)

8.1. Test Facility

A view of Phillips Petroleum Company's research facility for study of the combustion characteristics of JP fuels is shown in Figure 48.

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 49.

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

8.2. Test Rig

A scale diagram of the Phillips test rig used in these studies of hot corrosion is shown in Figure 50.

The combustor, which embodies the principal features used in modern aircraft-turbine engines, is 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 internal-deflector skirts for film cooling of surfaces exposed to flame. The design of the test rig permits easy access to the fuel nozzle, combustor liner, test specimens, etc. The combustor installation was disassembled, inspected, and reconditioned after every test period, which was at 5-hour intervals during these investigations.

8.3. Sea-Water Injection

Sea water was injected in the quench zone of the combustor, as indicated in Figure 50, 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 concentration. The sea water was divided into two metered portions and introduced through opposing jets to obtain uniform distribution of sea salt in air by impingement of the jet streams.

8.4. Specimen Location

The general location of the specimen holders in the exhaust gas from the 2-inch combustor is shown in Figure 50. They are separated from the combustor by a 6-inch, water-cooled spool; and are followed by another 12-inch, water-cooled, spool prior to quench-water injection. Each holder accommodates two specimens for test (metal strips 1/8 by 1/2 by 2-3/8 inches).

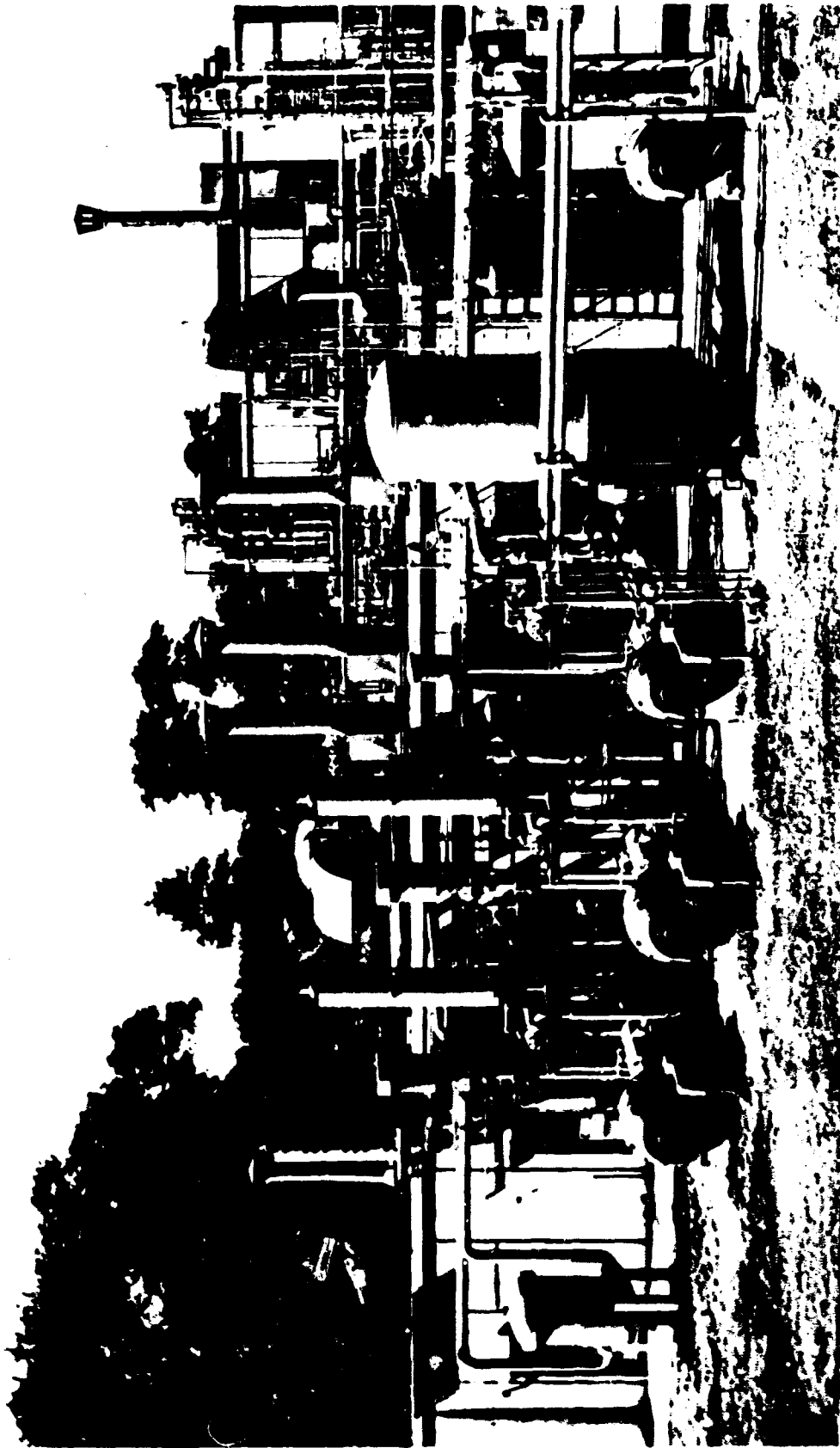


FIGURE 48
PHILLIPS RESEARCH FACILITY FOR JP FUELS

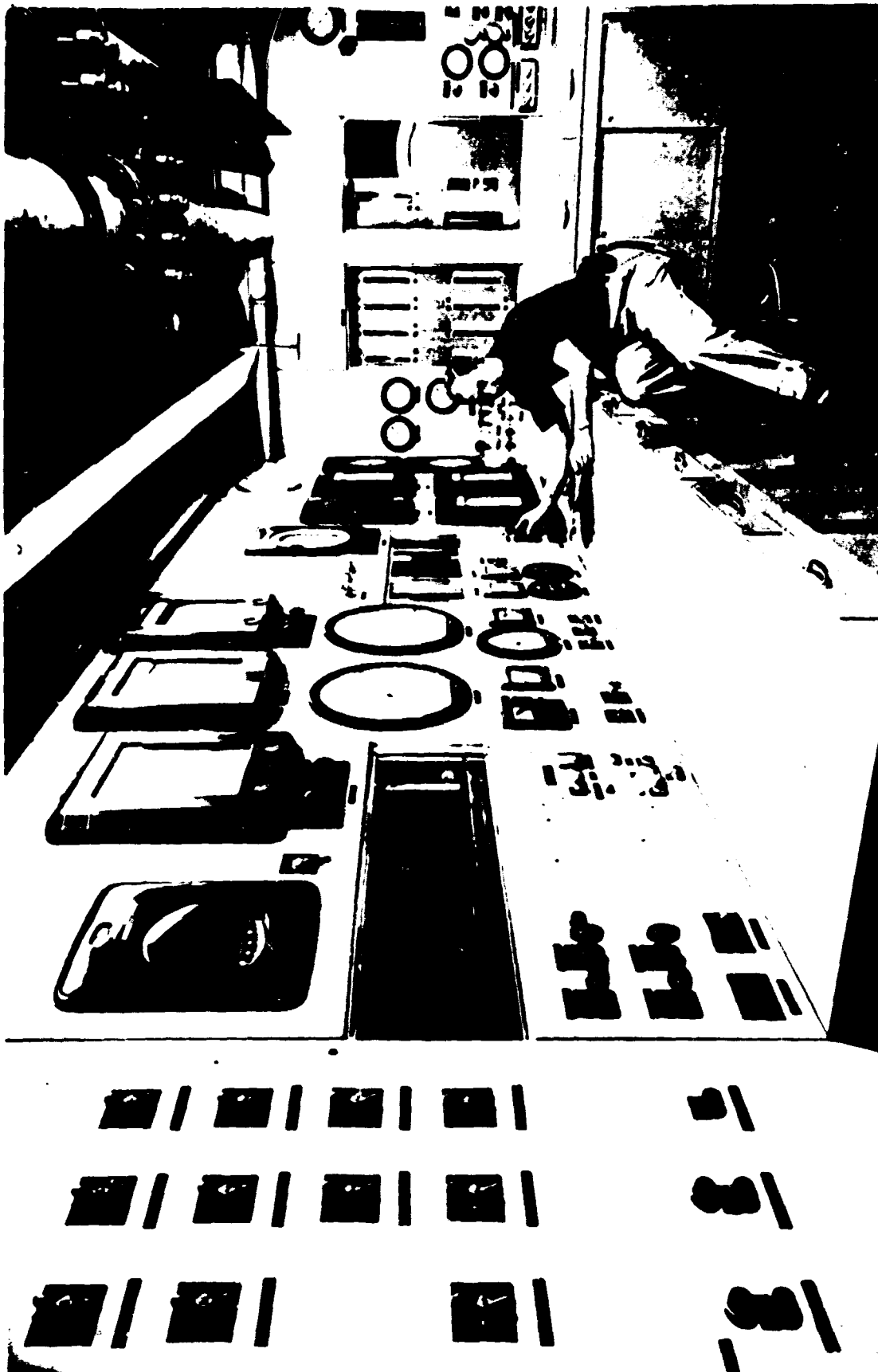


FIGURE 49
CONTROL ROOM FOR HIGH-PRESSURE COMBUSTOR

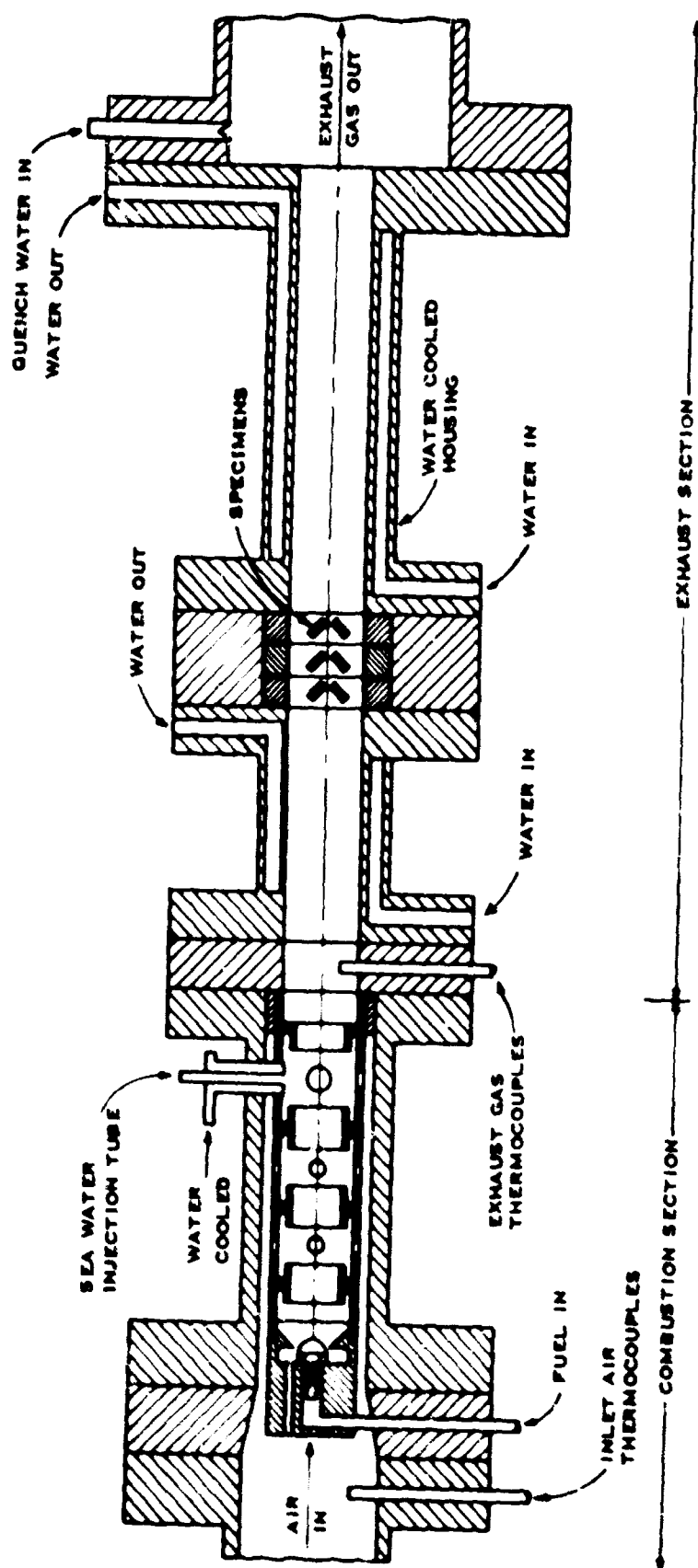


FIGURE 50
TEST-WAY FOR HOT-CORROSION STUDY

Three holders were stacked to form a cascade, with each successive holder rotated 120 degrees to prevent channeling of the hot-gas flow. A view of the cascade as assembled for mounting in the test rig is shown in Figure 51.

The cross-sectional area of the 2-inch pipe in which the specimen holders are located is 3.36 square inches; however, the unblocked area in the specimen holder is only 1.59 square inches. The holders maintain the specimens at an angle of 45 degrees to the axis of the pipe in which they are located. This provides for acceleration of the gas flow over the surface of the specimens, much as over the turbine blading in an actual engine. The specimens are subjected to appreciable gas-pressure loading while the test is in progress. It is sufficient to slightly bow the Inconel 713C specimens at the 2000 F test condition, and the effect increases with exposure time.



2X MAGNIFICATION

FIGURE 51
SPECIMENS MOUNTED IN CASCADE

9. APPENDIX 2
(Materials)

9.1. Test Fuels

The base fuels selected for use in these investigations were segregated samples of production ASTM Type A aviation-turbine fuel. The physical and chemical properties of interest to these investigations are presented in Table 15. The average values of pertinent properties from the Bureau of Mines Product Survey (9) over the 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 fuels contain less than 0.0040 per cent by weight of sulfur. Two fuels of higher sulfur contents were produced by blending to 0.040 and 0.40 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 (10). The components and their concentrations are shown in Table 16. The abundance of various elements in the synthetic formula compares very favorably with the average sea water composition (1).

TABLE 15

PHYSICAL AND CHEMICAL PROPERTIES OF TEST FUEL

	<u>Test Fuel</u> <u>Base (a)</u>	<u>Average</u> <u>JP-5 (b)</u>
Distillation Temperature, F		
Initial Boiling Point	326	-
5 Volume per cent evaporated	352	-
10 Volume per cent evaporated	357	-
20 Volume per cent evaporated	358	382
30 Volume per cent evaporated	377	-
40 Volume per cent evaporated	388	-
50 Volume per cent evaporated	398	413
60 Volume per cent evaporated	410	-
70 Volume per cent evaporated	423	-
80 Volume per cent evaporated	438	-
90 Volume per cent evaporated	457	455
95 Volume per cent evaporated	473	-
End Point	493	-
Gravity, degrees API	45.9	41.9
Gum, milligrams per 100 mls.	0.5	1.1
Smoke Point, millimeters	26.2	22.6
Hydrogen Content, weight per cent	14.0	13.6
Composition, parts per million		
Sulfur	< 40 (c)	1030
Metals		
Iron	< 0.1	-
Vanadium	< 0.1	-
Nickel	< 0.1	-
Copper (d)	0.037	-
Hydrocarbon Types, volume per cent		
Normal Paraffins	27	-
Isoparaffins	23	-
Cycloparaffins	36	-
Olefins	0.33	1.6
Aromatics	13.37	15.1

Notes:

- (a) Typical values for segregated samples of production ASTM Type-A aviation-turbine fuel, processed from West Texas crude and finished by hydrotreating.
- (b) U. S. Bureau of Mines Petroleum Product Survey, 1957 - 1966 (9).
- (c) Higher sulfur content test fuel obtained by blending to desired sulfur level using ditertiary butyl disulfide.
- (d) Spectro-photometric analysis.

TABLE 16
COMPOSITION OF SYNTHETIC SEA WATER (a)

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

- (a) ASTM D665 (10).
(b) Use cp chemicals.
(c) Use distilled water.

9.3. Vanadium

Vanadium salt solutions were blended with deionized water or dilute sea water for injection through the sea water jets to provide concentrations of 0.017 or 0.063 ppm vanadium in air. The vanadium salt solution was prepared from Fisher Laboratory Chemical, Vanadyl Chloride, VOCl_2 F. W. 137.864, Lot No. 762489. An analysis of this material showed it to be 76.07 per cent vanadyl chloride. Stock solutions of the vanadyl chloride was prepared using 4.7376 grams of the vanadyl salt, 100 ml of concentrated nitric acid and deionized water to make 3 liters. The nitric acid was added to solubilize the vanadium salt.

9.4. Test Specimens

Two base alloys were used in these investigations (Inconel 713C and Mar M-200), which are nickel-base alloys used in previous studies (1). Investment-cast specimens of these superalloys were obtained from Misco Division, Howmet Corporation. The chemical analyses for the two heats of Inconel 713C and one heat of Mar M-200, furnished by the supplier, are shown in Table 17.

The investment castings were finished by the supplier by grinding to provide specimens having a smooth uniform surface with a thickness of 0.125 ± 0.005 inches, a width of 0.500 ± 0.030 inches and a length of 2.375 ± 0.030 inches. The specimens were inspected by fluorescent penetrant (Zyglo) and X-ray to insure freedom from cracks, porosity and inclusions.

Specimens of Inconel 713C with two different coatings and Mar M-200 with one coating were obtained from Misco. Both bare and coated Inconel 713C specimens for a program were cast from the same heat. The coatings are characterized by the following descriptions.

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.

MDC-9 is a composite coating, rich in aluminum and chromium, which was applied by a pack-diffusion process to obtain a total thickness of approximately 2 mils. The inner diffused zone is approximately 45 per cent of the total coating thickness.

From physical measurements of random samples of specimens the average surface area was calculated to be 20.27 square centimeters for Inconel 713C (Heat Number RW072) and for MDC-1 coated Mar M-200 and 19.90 square centimeters for Inconel 713C (Heat Number RW382). The average weight from a random sample of each material is shown in Table 18.

TABLE 17

COMPOSITION OF INVESTMENT CAST TEST SPECIMENS

<u>Alloying Elements</u>	<u>Chemical Analysis, Per Cent</u>		
	<u>Inconel 713C</u>		<u>Mar M-200</u>
Nickel	Balance (a) (72.1)	Balance (b) (72.3)	Balance (c) (60.1)
Cobalt	40.1	0.25	9.9
Chromium	13.10	13.45	9.07
Molybdenum	4.51	4.61	-
Tungsten	-	-	12.2
Aluminum	5.73	5.87	4.92
Titanium	0.73	0.84	2.05
Manganese	<0.1	<0.1	0.1
Iron	0.93	0.11	0.19
Zirconium	0.095	0.086	0.042
Vanadium	-	-	-
Silicon	0.12	<0.1	0.05
Boron	0.008	0.006	0.016
Sulfur	0.004	0.003	0.005
Carbon	0.12	0.11	0.15
Phosphorus	-	-	-
Copper	<0.1	<0.1	0.1
Cb + Ta	2.31	2.07	-
Cb	-	-	1.10

(a) Misco Heat Number RW072.

(b) Misco Heat Number RW382.

(c) Misco Heat Number RLO88.

TABLE 18

INITIAL WEIGHT OF TEST SPECIMENS

<u>Specimens</u>	<u>Average Initial Weight, mg</u>
Inconel 713C (uncoated) (Heat No. RW072)	19,605
Inconel 713C (uncoated) (Heat No. RW382)	18,969
MDC-1 coated Inconel 713C	19,528
MDC-9 coated Inconel 713C	19,780
MDC-1 coated Mar M-200	20,915

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 52. 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. Specimens of bare and MDC-1 coated Inconel 713C and MDC-1 coated Mar M-200 were immersed in molten sodium hydroxide at 750 to 790 F with 1/3 amp/sq cm passing through the specimens for a period of 10 min. 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 53.

From visual and metallographic examination it was found that electro-cleaning removed a portion of the coating and modified the interface between the diffused layer of the coating and the base metal with MDC-9 coated Inconel 713C. A water-wash (sonic cleaning) technique was adopted for cleaning the MDC-9 coated Inconel 713C specimens in this program. This technique consisted of immersing the specimens in deionized water in an ultrasonic bath for 90 minutes. Specimens were then brushed with a fiber brush, rinsed in acetone, dried, and weighed. For the program conducted at 1400, 1600, and 1800 F this method of cleaning was modified by the use of a fine stainless-steel wire brush and a detergent rather than the fiber brush. With the stainless-steel wire brush, from 3.6 to 13.2 milligrams (6.8 milligrams average for all specimens) of deposits were removed that had not been removed with the fiber brush. The use of the stainless-steel brush on unexposed specimens of MDC-9 coated Inconel 713C removed less than one milligram from a specimen and it was concluded that the brushing was not disturbing the coating.

After this cleaning procedure the specimens appeared visibly clean with the exception a small amount of material on some of the specimens that appeared to be material transferred from the specimen holder at the point of contact. One specimen was examined metallographically (Figure 54) to determine if this was metal transfer to the specimen from the holder or a modification of the specimen. Figure 54 confirms that the material left on the specimen at the point of contact with the holder is metal transferred from the holder.

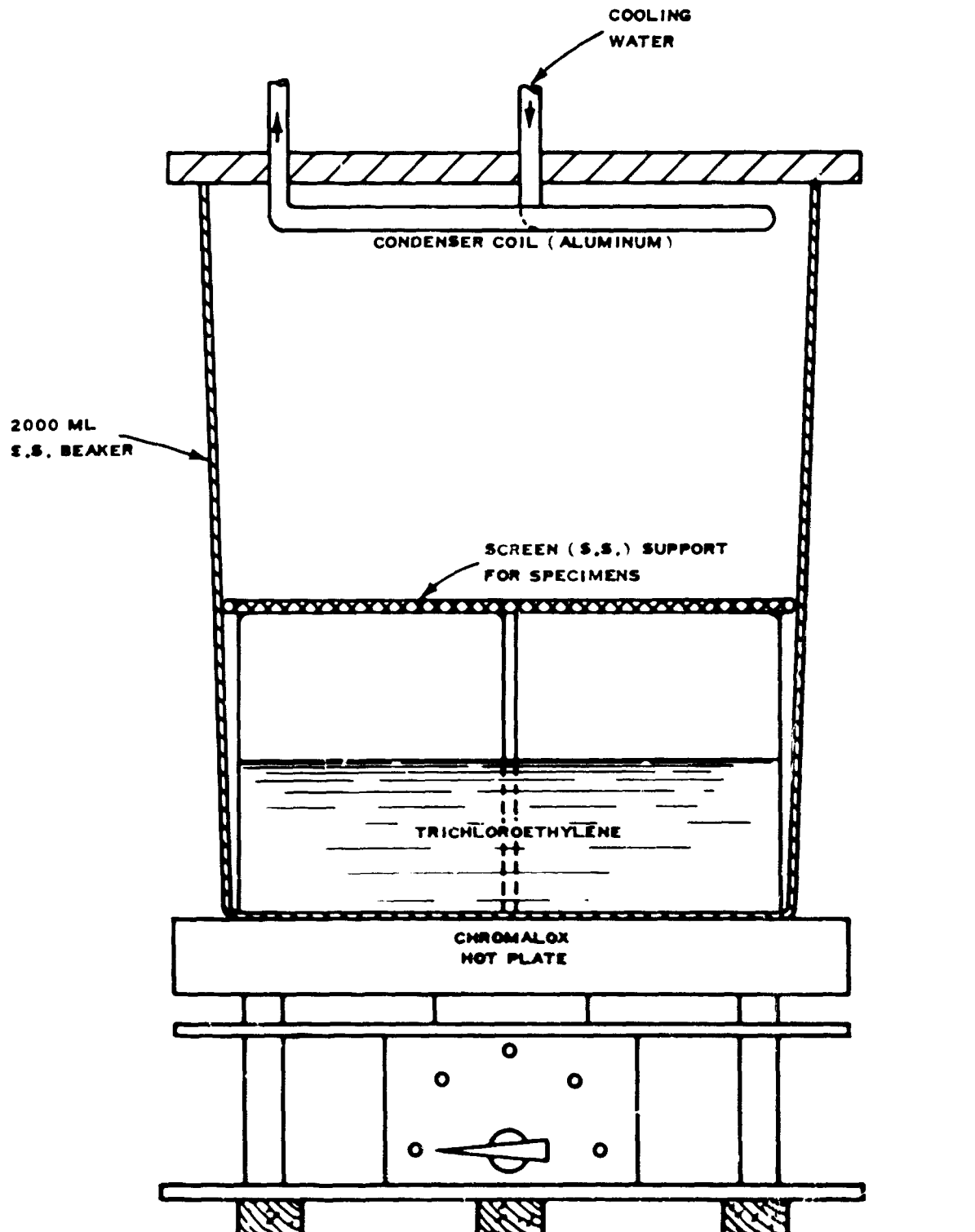


FIGURE 52
VAPOR-DEGREASING APPARATUS FOR PRE-TEST CLEANING OF SPECIMENS

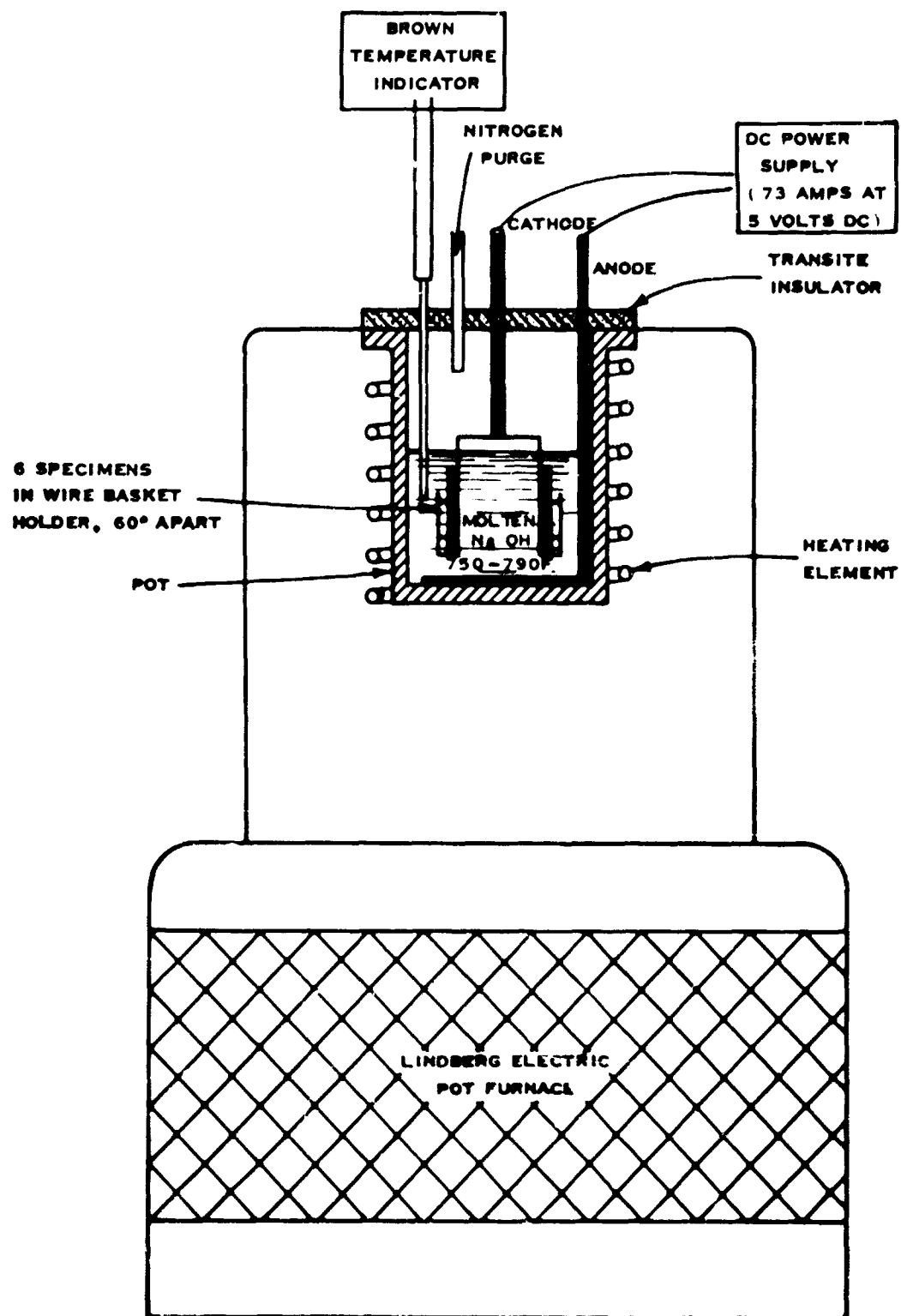
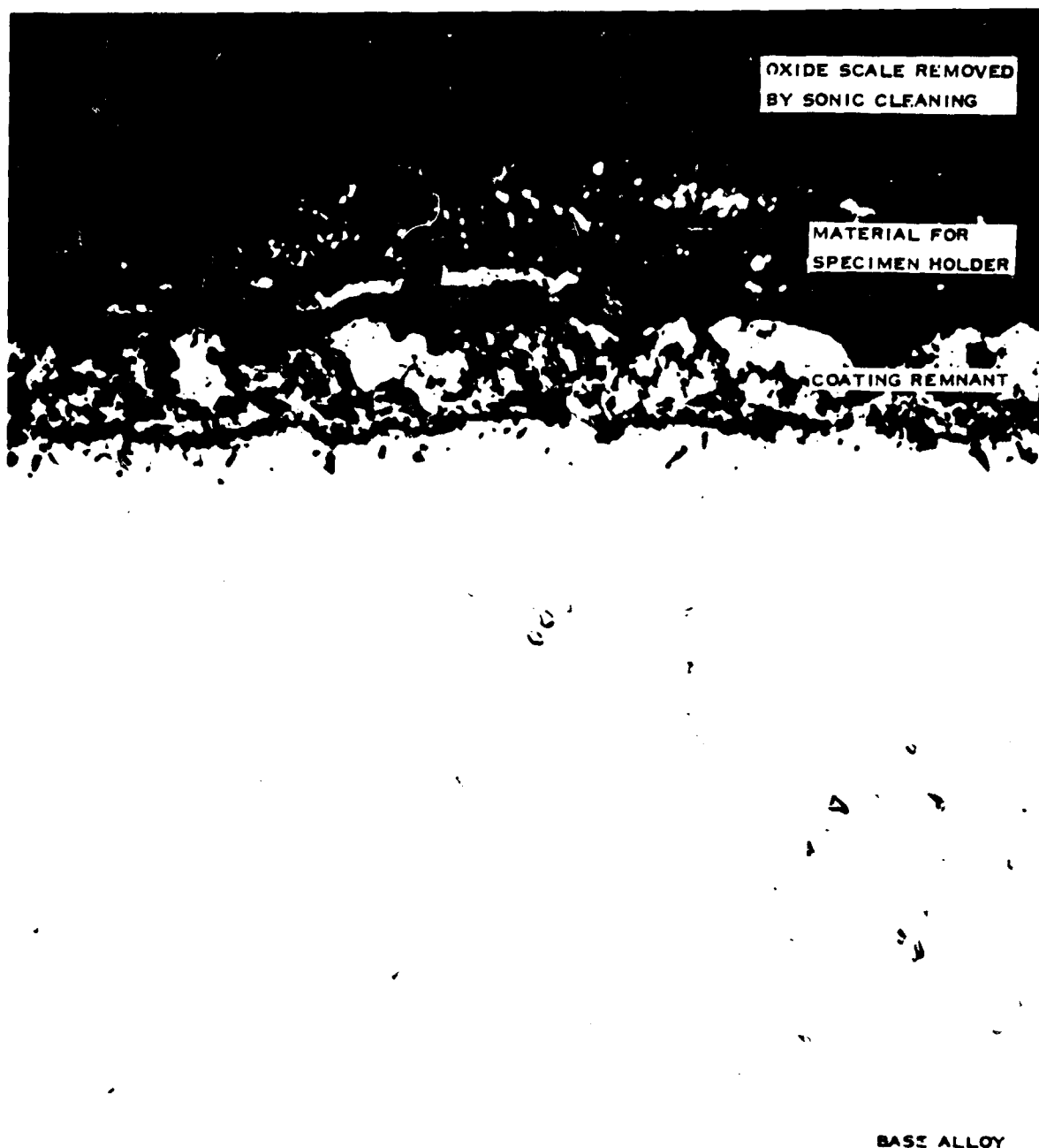


FIGURE 53
ELECTRO-DESCALING APPARATUS
FOR POST-TEST CLEANING OF SPECIMENS



METALLOGRAPHIC CROSS-SECTION OF SPECIMEN AFTER 45 HOURS AT 1800 F TEST
CONDITION WITH 1.0 PPM SEA SALT IN AIR AND 0.40 WT % SULFUR IN FUEL.
UNETCHED. 400X MAGNIFICATION.

FIGURE 54
MATERIAL PICK-UP FROM SPECIMEN HOLDER
BY MISCO MDC-9 COATED INCONEL 713C SPECIMEN

10.3. Metallographic Examination

Following exposure, specimens were cleaned for determination of their loss in weight, and subsequently specimens were chosen from each test for metallographic examination. The selection was made to allow study of the effect of long-time exposure of the coating-alloy systems at the 2000 F test condition, as well as changes in the concentration of corrosive agents which characterized the different tests.

Each specimen was sectioned in three places, with one section at the point of maximum visible attack. The three pieces were placed in a 1-inch red Bakelite mold with the coupon of most interest (maximum attack) in the center. This arrangement aided in obtaining a flat surface 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.

Polishing was done in one direction. The edge of most interest was kept on the back side while polishing, so the polishing motion is across the coupons toward the edge to be kept flat.

In general, the attack was most evident on the impact surface of the specimen, as expected, because it had maximum exposure to the corrosive agents. Photomicrographs showing typical attack on the specimens were taken there at 200X or 500X and 1500X or 2000X, and in some cases at other magnifications. Also, the coupons were frequently etched to show areas of alloy depletion and grain structure. Typical photomicrographs, from the several hundred taken in our investigations, are presented to illustrate the nature of the attack experienced by the superalloy and coating over the range of conditions investigated.

10.4. Test Rig Operation

The Phillips Test Facility and Test Rig used for these investigations are described in Sections 8.1 and 8.2 of Appendix 1. The operating conditions used for the various programs during these investigations are shown in Table 19.

In all tests the flow through the sea water injection jets (Figure 50) is maintained constant at 1.75 pounds per hour per jet or 3.5 pounds per hour total. In the absence of sea salt, deionized water is used. For tests with sea salt, the sea water is diluted to give the desired concentration of sea salt in combustor air.

TABLE 19

OPERATING CONDITIONS FOR PHILLIPS 2-INCH COMBUSTOR

<u>Test Variables</u>	<u>Test Conditions</u>			
Temperature, deg. F				
Nominal Gas	1400	1600	1800	2000
Exhaust Gas (a)	1358	1544	1861	2039
Test Specimen, metal (b)	1375	1537	1814	1970
Test Specimen, surface (c)	-	1656	1924	2074
Combustor Inlet Air	800	1000	800	1000
Pressure, atmospheres				
Combustor Inlet Air	15	15	15	15
Mass Flow Rate, pounds per hour				
Air	7200	7200	7200	7200
Fuel	60	60	120	120
Air-Fuel Ratio	120	120	60	60
Flow Velocity, feet per second at Test Specimen (d)	565	625	685	745
Test Duration, hours (e)	Varied	Varied	Varied	Varied

Notes:

- (a) Values calculated using mean specific heats (11) for 100 per cent combustion efficiency. These values are confirmed by linear regression with measured temperatures in Appendix 1 of Reference 1.
- (b) Calculated mean value from thermocouple measurement of strip temperature vs calculated gas temperature in Appendix 1 of Reference 1.
- (c) Calculated mean value from optical pyrometer readings vs calculated gas temperature shown in Appendix 1 of Reference 1.
- (d) Calculated value based on unblocked area in specimen holder of 1.59 square inches.
- (e) Operating cycle was 55 minutes at test condition followed by 5 minutes with fuel off.

In all tests the three stages of the cascade (Figure 51) were loaded with six new specimens prior to the start of a test. The cyclic test was operated in periods of 5-hours with the fuel nozzle, combustor dome and liner, sea water injection tubes and test specimens examined at the end of each period. The basic plan for the experiments consisted of removal of one test specimen, with replacement, at each 5-hour period for the first five periods and then continuing the test to 55 hours, or until visual inspection of the specimens indicated heavy attack and the possible loss of a specimen with further exposure to the hot gasses. To minimize any effect of position in the cascade, a system of rotation of the position of specimens was used where the specimen holder in the first stage of the cascade was moved to the third stage and the other two holders advanced one position at each five hour interval. The order of specimen removal is shown in Table 20. For test conducted at the 1400, 1600 and 1800 F conditions the order of specimen removal was modified, as shown in Table 21, with each specimen exposed in each of the three stages of the cascade. Thus total exposure on each specimen was 15, 30 or 45 hours.

TABLE 20
ORDER OF TEST SPECIMEN REMOVAL
(2000 F Test Conditions)

<u>Test Period</u>	<u>Test Time, Hours</u>	<u>Test Specimen Location</u>					
		<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>	<u>6</u>
1	5	1 ₅	(2 ₅)	3 ₅	4 ₅	5 ₅	6 ₅
2	10	(3 ₁₀)	4 ₁₀	5 ₁₀	6 ₁₀	1 ₁₀	2 ₅
3	15	5 ₁₅	(6 ₁₅)	1 ₁₅	2 ₁₀	3 ₅	4 ₁₅
4	20	(1 ₂₀)	2 ₁₅	3 ₁₀	4 ₂₀	5 ₂₀	6 ₅
5	25	3 ₁₅	(4 ₂₅)	5 ₂₅	6 ₁₀	1 ₅	2 ₂₀
6	30	5 ₃₀	6 ₁₅	1 ₁₀	2 ₂₅	3 ₂₀	4 ₅
7	35	1 ₁₅	2 ₃₀	3 ₂₅	4 ₁₀	5 ₃₅	6 ₂₀
8	40	3 ₃₀	4 ₁₅	5 ₄₀	6 ₂₅	1 ₂₀	2 ₃₅
9	45	5 ₄₅	6 ₃₀	1 ₂₅	2 ₄₀	3 ₃₅	4 ₂₀
10	50	1 ₃₀	2 ₄₅	3 ₄₀	4 ₂₅	5 ₅₀	6 ₃₅
11	55	3 ₄₅	4 ₃₀	5 ₅₅	6 ₄₀	1 ₃₅	2 ₅₀

Notes:

- () Remove test specimen and replace with new test specimen.
Terminate test when failure of a specimen appears imminent.

TABLE 21

ORDER OF TEST SPECIMEN REMOVAL
(1400, 1600, and 1800 F Test Conditions)

<u>Test Period</u>	<u>Test Time, hours</u>	<u>Test Specimen Location</u>					
		<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>	<u>6</u>
1	5	1 ₅	2 ₅	3 ₅	4 ₅	5 ₅	6 ₅
2	10	3 ₁₀	4 ₁₀	5 ₁₀	6 ₁₀	1 ₁₀	2 ₁₀
3	15	5 ₁₅	(6 ₁₅)	1 ₁₅	(2 ₁₅)	3 ₁₅	4 ₁₅
4	20	1 ₂₀	2 ₅	3 ₂₀	4 ₂₀	5 ₂₀	6 ₅
5	25	3 ₂₅	4 ₂₅	5 ₂₅	6 ₁₀	1 ₂₅	2 ₁₀
6	30	5 ₃₀	6 ₁₅	1 ₃₀	2 ₁₅	(3 ₃₀)	4 ₃₀
7	35	1 ₃₅	2 ₂₀	3 ₅	4 ₃₅	5 ₃₅	6 ₂₀
8	40	3 ₁₀	4 ₄₀	5 ₄₀	6 ₂₅	1 ₄₀	2 ₂₅
9	45	5 ₄₅	6 ₃₀	1 ₄₅	2 ₃₀	3 ₁₅	4 ₄₅

() Remove specimen and replace with new specimen.

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13. ABSTRACT An experimental investigation is in progress to determine whether the 0.4 per cent by weight of sulfur which is currently allowed in JP-5 fuel is a safe level for protection of <u>coated</u> turbine blades in high-performance engines when operated in a marine environment. Comparisons are made which show the effect of three different levels of sulfur in fuel, both with and without 1.0 ppm sea salt in air. A significant decrease in the relative rate of corrosion when sea salt was removed from the air shows it to be the primary cause of hot corrosion. These data indicate that little or no benefit would result from a reduction in the sulfur limit for JP-5 fuel. A screening program conducted at 2000 F conditions with 0.4 weight per cent sulfur in JP-5 fuel shows the presence of 0.063 ppm vanadium in air more than doubles hot corrosion attack on Inconel 713C specimens with either zero or 1.0 ppm sea salt in air and 0.017 ppm vanadium in air had no significant effect on hot corrosion of Inconel 713C specimens in the absence of sea salt in air. A screening program indicated that exposure times of several hundred hours or more would be required at some conditions to provide a meaningful evaluation of the effect of sulfur in fuel on hot corrosion of coated superalloys over a range of temperatures from 1400 - 2000 F. A new test section for the Phillips Hot Corrosion Test Rig has been designed and fabricated which should increase the productivity by allowing simultaneous exposure of 48 test specimens rather than six specimens as in the current rig.		

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14. KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
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