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EFFECT OF A CHROMIUM-CONTAINING FUEL ADDITIVE ON HOT CORROSION

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Cleveland, Ohio 44135

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16. Abstract

Four superalloys were tested at 900° C in high velocity combustion gases containing synthetic sea salt and, in some cases, a chromium-containing fuel additive. While the additive reduced hot corrosion of the alloys over the 100 hour test period, the attack was not eliminated nor was the mode of attack changed. Reduction of the number of thermal cycles had as large a beneficial effect as the Cr additive. Intermittent washing during testing had either small beneficial or adverse effects depending on the alloy.

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ABSTRACT

Four cast superalloys (one cobalt-base and three nickel-base) were tested to 900° C for one hundred hours in Mach 0.3 combustion gases. Five parts per million of synthetic sea salt were added to the gases in the combustion chamber. Several types of thermal cycle and washing procedures were employed. Similar tests were made with the addition of 300 parts per million of a chromium-containing fuel additive. In both sets of tests the extent of hot corrosion was evaluated by specific weight change and metal recession. In general, the chromium additive in the fuel reduced the extent of hot (salt) corrosion but did not eliminate it. The percent reduction of hot corrosion attack was similar for all four alloys. As great a reduction of hot corrosion was achieved by reducing the number of thermal cycles during the test from 100 to five or six. The effect of washing the alloys every ten cycles as opposed to the end of the test was erratic; some alloys were attacked slightly more, others somewhat less. A NiCrAlY coating was found to be more effective in reducing hot corrosion than either the fuel additive or the washing schedule.

INTRODUCTION

Hot corrosion has been identified as a major cause of failure of blades and vanes in many types of gas turbines: aircraft, marine, and

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ground power. The literature of hot corrosion has been reviewed by Stringer[1] and expanded recently[2]. The mechanisms of hot corrosion attack are complex. Briefly, attack involves fluxing of the protective oxide scale primarily by Na_2SO_4 which is formed from a reaction of ingested sodium salts and sulfur in the fuel. The fluxing of the oxide scale results in a greatly enhanced attack of the metal by oxidation and sulfidation. The attack is so greatly enhanced that blade or vane failure can take place in less than one hundred hours in extreme cases.

While the application of surface coatings is the usual method used to protect alloys from hot corrosion, some recent work of Bornstein and DeCrescente[3,4] seems to indicate that a fuel additive (a chromium (Cr) compound was their choice) might accomplish the same goal. Additives such as MgO or other Mg compounds have been used for many years in oil burning turbines to minimize vanadium corrosion. Such additives would be an attractive solution to hot corrosion. Unlike coatings, one probably does not have to worry about effects on the turbine component's mechanical properties and such additives could be a less expensive solution to corrosion attack. One unknown in the use of such an additive is the effect of repetitive thermal cycling on the rate of hot corrosion attack. A commercial, Cr-containing, fuel additive has not been produced.[5] The purpose of this study was to evaluate this additive under controlled laboratory test conditions. The approach used in this study was to add synthetic sea salt to the combustion gases and a Cr additive to the fuel of a Mach 0.3 burner rig. Four commercial superalloys and one commercial coating were tested under a variety of conditions for 100 hours at 900° C. The extent of hot corrosion was judged by net specific weight change and metal recession.

MATERIALS AND PREPARATION

The alloys used in this study and their nominal compositions are shown in table I. The three nickel-base alloys are commercial aircraft gas turbine blade alloys while the cobalt-base alloy has found use as a turbine vane material. The compositions of the alloys vary substantially -- especially in chromium content (10 to 23 percent Cr). In general, high chromium content alloys, especially cobalt-base, are associated with superior hot corrosion resistance and the alloys examined in this study were chosen, in part, for their variation in chromium level. In addition to the base alloys listed, a few tests were run on MM-509 which had been commercially overlay coated with NiCrAlY by physical vapor deposition.

All of the samples were cast to the size and shape shown in Fig. 1. After removal of the mold, the surfaces of the castings were glass bead blasted and cleaned in alcohol. After cleaning, the samples were weighed to ±0.2 mg and their wedge diameters (at A-A in Fig. 1) were measured to ±2 μM.

PROCEDURES

The burner rig used for these tests is shown in Fig. 2 and has been described in Ref. 6. The test (an accelerated hot corrosion test) conditions are defined in table II. In brief, eight samples are rotated rapidly in front of the nozzle of a Mach 0.3 burner rig. After a predetermined exposure interval, the burner pivots away from the samples bringing a forced air cooling nozzle into place. The samples are cooled for a short time and the heating cycle begins again. At approximately 10 to 20 hour exposure intervals the samples were weighed. In specified runs the samples were periodically weighed, then washed, and finally reweighed. Washing consisted of immersion of each blade in 300 cc of water at 80° C for two hours. This was followed by soft brushing in running water, an alcohol rinse, and air drying. The specific test conditions are summarized in table III. These conditions were chosen to allow an evaluation of the effects of the fuel additive as well as of cycle frequency and of the washing schedule.

Before testing started a few runs were made with an axially rotating platinum collector to make sure that the test conditions would allow salt and additive deposition. With no salt or additive only a negligible weight pickup was noted. When the additive was injected at a concentration of 300 ppm (recommended in Ref. 5), ESCA analysis indicated that the deposit was rich in Cr. When both the additive and 5 ppm synthetic sea salt were used, ESCA analysis indicated that the deposit was rich in Cr, Na, and S. The deposit was too small for phase identification. The collector was preheated to 900° C before salt injection to prevent condensation prior to reaching test conditions.

At the conclusion of the tests all the samples were given a final water wash as above and the wash water was filtered. Both the filtrate and the water insoluble residue were spectroscopically analyzed. The samples were given a final weighing and then sectioned at A-A , Fig. 1. The cross section was examined metallographically and then the diameter measured with a traveling microscope along the same line as the original measurement (t_0). To determine the depth of scale formation the distance from scale-metal interface to scale-metal interface across the diameter was measured and subtracted from t_0. The maximum depth of attack was determined in a similar fashion by measuring from either the edge of the depletion zone or from the deepest point of attack, whichever was most severe. Finally, the degree of attack was judged on the combined basis of the measurements just described and on weight loss.

RESULTS AND DISCUSSION

Specific Weight Change

The specific weight changes of the washed specimens determined at the conclusion of each of the runs (100 hrs) are presented in table IV. Figure 3 contains weight change versus time data for the runs where weights were determined after washing. Under all conditions, uncoated IN-100 was the most susceptible to hot corrosion while uncoated MM-509 was the least. IN-792 and IN-738 were of intermediate susceptibility. While the alloys' hot corrosion resistance can be ranked by their increasing Cr content, the function is not simply linear. The effect of the Cr fuel additive was beneficial for all alloys in all cases but one: IN-792 tested for 100 cycles with washing only at the conclusion of test. The amount of improvement varied considerably but averaged about a factor of two. However, as can be seen from table IV and Fig. 3, under no conditions was the additive really as effective as the overlay coating (a factor of 100 improvement). The effect of the intermittent washing, which was done to simulate the recommendations of Ref. 5, seems to have been slightly deleterious. On the other hand the runs with fewer cycles seem to have resulted in slightly less attack. In fact, lessening the cycle frequency was almost as beneficial as the additive, although both together gave the best results except for coating. In effect the additive appeared to have delayed or reduced hot corrosion, but not eliminated it.

Metal Recession Measurements

These measurements are subject to many visual interpretation problems and inaccuracies, but they are the only direct measurement of metal degradation.[7] The recession data obtained in this study are presented in table V. Because of difficulties in measurement of depth of scale values and the conservative nature of the maximum depth of attack, the latter measurements should be given the most credence. Since these measurements are made in the hot zone they are not affected by what occurs in other parts of the sample as the weight change data might be. However, a comparison of tables IV and V shows that both types of measurement give substantially the same rankings including the anomaly with the IN-792.

Structural Changes

The effects of hot corrosion are often quite apparent visually as can be seen in Fig. 4. This figure shows the appearance of all samples exposed for 100 one-hour cycles at 900° C with 5 ppm synthetic sea salt and with and without the Cr fuel additive. These specimens were washed every ten cycles, but they are typical of all the runs. The attack on IN-100 and to a lesser extent IN-738 and IN-792 is obvious. MM509 shows little evidence of attack with or without a coating. However, a closer (10×) look at the coated surface run without the fuel additive shows a small amount of coating spallation in the hot zone. That this is only minor damage will be seen below. The most important point of Fig. 4 is that the additive does not eliminate corrosive surface attack.

The mode of microstructural attack is shown in Figs. 5 to 8. Typical of the three nickel base alloys is IN-738 which is shown in Fig. 5. It is a γ/γ' alloy. These phases can be seen in the unaffected metal beneath the depletion zone. The results of hot corrosion are the same for all salt conditions with a heavy external scale layer over a zone, depleted in γ', which is filled with voids. The nature of the corrosive attack can easily be seen by comparison with the oxidative-only attack shown in Fig. 5(d).

In addition to light microscopy extensive Scanning Electron Microscopy (SEM) was performed, including x-ray mode analysis. Fig. 6 is typical of this work. The extensive voids result from a dissolving away of corrosion products (e.g., sodium chromate) during the polishing operation which is done in the presence of water. Apparently, the salt corrosion attack occurs to some extent down the grain boundaries and this step is followed by rapid oxidation of the depleted metal. Volumes of metal can be clearly seen surrounded by the reaction zone. These metal regions will either subsequently oxidize or spall off with the oxide during thermal cycling. No evidence of sulfides could be found on any sample. However, due to interference between Mo and S in the nondispersive analysis used, no conclusion can be drawn as to the presence of sulfides in the corrosion zone. Even though MM 509 was attacked much less, see above, the mode of attack appears to be the same (see Fig. 7). Grain boundary corrosion was followed by rapid oxidation with only the depth of attack varying with these conditions. Again no sulfides were found in the x-ray microanalysis. Clearly the hot corrosion attack, as was the case for the nickel alloys, is qualitatively different from the case where only oxidation attack took place (Fig. 7(d)).

The microstructure of coated MM 509 (Fig. 8) showed no attack at all. Not even the spalled areas of the coating which were evident by surface examination (Fig. 4) could be seen in any part of the microstructure. It appears that the overlay coating is very resistant to corrosive attack.

To summarize the structural evaluation, it appears that the corrosive attack occurs without and with the Cr additive. The additive and test conditions affect the amount of attack but do not alter the manner of that attack.

Wash Water Analysis

Analyses of the wash water for the two long cycle runs are shown in table VI. These runs were chosen because they contained the maximum buildup of salt and corrosion products due to minimum thermal cycling spallation. The composition of the wash water is expressed in weight percent of the total sample in the case of the residue. The filtrate values are in terms of weight percent of the dried sample. Obviously this filtrate consisted largely of unreacted sodium sulfate so the percentage levels of the other constituents must be viewed with care. In addition, the total residue samples were generally only a few milligrams. The results indicated are therefore difficult to interpret but have several interesting points. One is that the Cr additive has little or no effect on the composition of either the residue or the water soluble deposits. Secondly, Al is not combined into a soluble corrosion product. Finally, Mo and V are very rapidly corroded by the salt attack and appear in the water soluble corrosion products. This is especially true of IN-100 and may account for its being the least resistant alloy to hot corrosion.

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CONCLUSIONS

As a result of testing the effect of 300 ppm of a Cr fuel additive on the hot corrosion attack of four cast superalloys exposed for 100 hours at 900° C in Mach 0.3 combustion gases containing 5 ppm synthetic sea salt (an accelerated test), the following conclusions may be drawn:

1. The chromium additive reduces the hot corrosion attack by as much as a factor of two. However, hot corrosion is not eliminated nor is the mode of attack altered by the chromium additive at the 300 ppm level.

2. The effect of the additive was similar for all four alloys including those of high chromium content (16 to 23 percent) as well as those with lower levels (10 to 12.7 percent).

3. Thermal cycling was found to influence hot corrosion attack. A decrease in cycling generally led to less attack: for the more resistant alloys this reduction ranged from a factor of from 2 to 3. However, as in the case of the additive, the mode of attack was not altered.

4. Intermittent water washing of test specimens to remove soluble salt deposits had only a small and variable effect on hot corrosion.

5. An overlay coating of NiCrAlY was an effective barrier to hot corrosion.

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4. N. S. Bornstein, M. A. Decrescente, and H. A. Roth, Metall. Trans. 4, 1799 (1973).

5. R. C. Farmer, Gas Turbine Internl. 15, 32 (1974).

6. G. Santoro, NASA TM X-71734 (1975).

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TABLE I. - ALLOY COMPOSITION (W/O)

Element	IN-100	IN-792	IN-738	MM509
Cr	10	12.7	16	23
Ni	Bal.	Bal.	Bal.	10
Co	15	9.0	8.5	Bal.
Al	5.5	3.2	3.4	---
Ti	4.7	4.2	3.4	0.2
Mo	3	2.0	1.75	----
W	----	3.9	2.6	7
Ta	----	3.9	1.75	3.5
Nb	----	0.9	-----	----
V	1.0	----	-----	----
Mn	----	----	0.2	----
Fe	----	----	0.5	----
Si	----	----	0.3	----
Zr	0.06	0.10	0.10	0.5
B	0.014	0.02	0.01	----
C	0.18	0.21	0.17	0.6

TABLE II. - BURNER RIG TEST PARAMETERS (REF. 6)

Rig: 0.3 Mach Pratt & Whitney type

Fuel: Type A-1, ASTM D-1655. Dissolved sulfur content over one year period varied from 0.02 to 0.05 weight percent

Air/fuel ratio: Approximately 25/1 at one atmosphere pressure

Salt: 5 ppm synthetic sea salt, ASTM D-1141

Temperature: Maximum metal temperature, 900° C. Temperature profile given below

Cycle: Variable time at temperature. Three minutes forced air cooling at 3.4×10^{4} N/m^{2} (5 psi)

Temperature Profile

Distance from top of specimen		Temperature, °C
cm	in.	
0.64	0.25	860
1.27	.5	882
1.91	.75	896
2.54	1.0	896
3.18	1.25	885
3.81	1.5	870
4.45	1.75	837

TABLE III. - TEST CONDITION SUMMARY

[All tests of 100-hour duration]

Run	5 ppm salt	300 ppm additive	Wash schedule	Cycles
O	No	No	None	100
N	Yes	No	End of test	100
S	Yes	No	Every 10 cycles	100
Z	Yes	No	Every 10 cycles	100
K	Yes	No	End of test	6
W	Yes	Yes	End of test	100
X	Yes	Yes	Every 10 cycles	100
Y	Yes	Yes	Every 10 cycles	100
I	Yes	Yes	End of test	5

TABLE IV. - NET SPECIFIC WEIGHT CHANGE AFTER 100 HOURS AT 900° C (MG,CM^{-2})

		Washed every 10 cycles		Washed only at end of test			
	100 cycles			100 cycles		5 cycles	6 cycles
Alloy	No salt (a)	Salt + additive (b)	Salt only (b)	Salt + additive (a)	Salt only (a)	Salt only (a)	Salt + additive (a)
IN-100	0.88	-436	-879	-830	-814	-701	-660
IN-792	0.94	-10.6	-22.3	-30.7	-21.2	-16.2	-6.7
IN-738	0.52	-19.0	-34.4	-19.6	-24.2	-15.8	-9.6
MM509 bare	-1.28	-9.6	-20.7	-4.5	-9.9	-6.2	-4.6
MM509 coated	0.51	0.25 (c)	-0.18 (c)	-----	-----	-----	-----

[a] Average of two samples, 1 run.

[b] Average of three samples, 2 runs.

[c] One sample only.

TABLE V. - METAL RECESSION AFTER 100 HOURS AT 900° C (MICROMETERS)

		Washed every 10 cycles		Washed only at end of test			
	100 cycles			100 cycles		5 cycles	5 cycles
Alloy	No salt (a)	Salt + additive (b)	Salt only (b)	Salt + additive (c)	Salt only (c)	Salt only (c)	Salt + additive (c)
IN-100	20/40	1730/1780	2770/2810	2090/2090	2830/2920	2810/2840	3110/3190
IN-792	30/50	210/410	290/600	320/640	240/600	200/400	90/250
IN-738	10/50	200/350	240/460	190/450	160/450	230/340	80/210
MM509	20/70	80/160	80/280	[a]50/[a]170	50/170	60/140	40/100

[a] One sample.

[b] Three samples, two runs.

[c] Two samples, one run.

A/B

A - Depth of scale formation

B - Maximum depth of attack

TABLE VI. - ANALYSIS OF DEPOSITS REMOVED BY WASHING

[K: Six cycles at 900° C, 100 hours, salt only]
[I: Five cycles at 900° C, 100 hours, salt plus additive]
[Values in weight percent]

Element	Analysis of	IN-100		IN-792		IN-738		MM-509	
		K	I	K	I	K	I	K	I
Mo	Filtrate	12.5	13.0	0.4	0.1	0.3	0.1	0.1	<0.1
	Alloy	3	3	2	2	1.7	1.7	None	None
	Residue	0.4	----[a]	0.3	0.7	0.3	0.9	0.2	----
V	Filtrate	2.7	2.0	----	----	----	----	----	----
	Alloy	1	1	None	None	None	None	None	None
	Residue	----	1.0	----	0.5	----	0.5	----	1.0
W	Filtrate	0.7	0.3	0.9	0.4	1.0	0.5	0.9	0.4
	Alloy	None	None	3.9	3.9	2.6	2.6	7	7
	Residue	----	----	0.6	0.9	0.4	0.9	0.8	0.4
Cr	Filtrate	0.2	0.2	0.4	0.4	0.6	0.5	0.3	0.6
	Alloy	10	10	12.7	12.7	16	16	23	23
	Residue	6.8	6.5	7.3	5.9	11.4	7.9	9.8	10.9
Al	Filtrate	----	0.1	----	<0.1	----	<0.1	----	<0.1
	Alloy	5.5	5.5	3.2	3.2	3.4	3.4	None	None
	Residue	4.0	4.1	1.7	1.7	2.8	1.8	0.9	0.9
Ni	Filtrate	0.3	0.4	<0.1	----	<0.1	----	----	----
	Alloy	Bal.	Bal.	Bal.	Bal.	Bal.	Bal.	Bal.	Bal.
	Residue	47	48.7	27.8	29.5	12.8	33.6	21.2	24.7
Co	Filtrate	0.1	0.2	<0.1	----	----	<0.1	<0.1	<0.1
	Alloy	15	15	9	9	8.5	8.5	Bal.	Bal.
	Residue	10.4	9.8	6.8	7.0	9.8	5.8	34	25
Ti	Filtrate	----	<0.1	----	<0.1	----	<0.1	----	<0.1
	Alloy	4.7	4.7	4.2	4.2	3.4	3.4	0.2	0.2
	Residue	4.1	3.7	3.4	3.1	4.8	2.2	0.9	1.0
Na	Filtrate	29.7	34.0	29.2	30.4	29.2	38.4	35.0	38.8
	Alloy	None	None	None	None	None	None	None	None
	Residue	----	----	----	----	----	----	----	----
Mg	Filtrate	0.1	0.1	0.2	0.3	0.2	0.4	0.2	0.3
	Alloy	None	None	None	None	None	None	None	None
	Residue	<0.1	<0.1	<0.1	2.7	2.2	1.6	4.8	12.0

[a] Not detected.

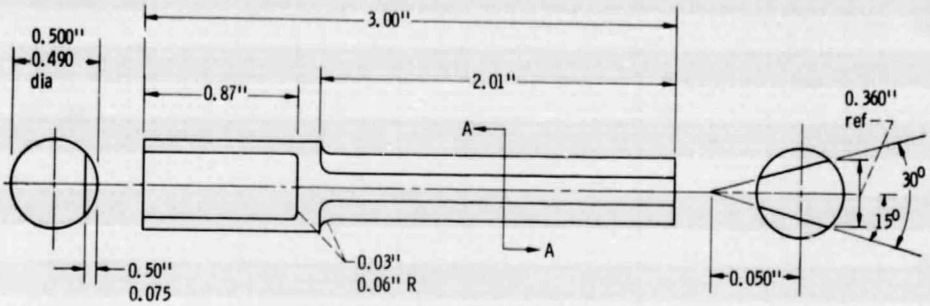

Figure 1. - Burner rig sample geometry.

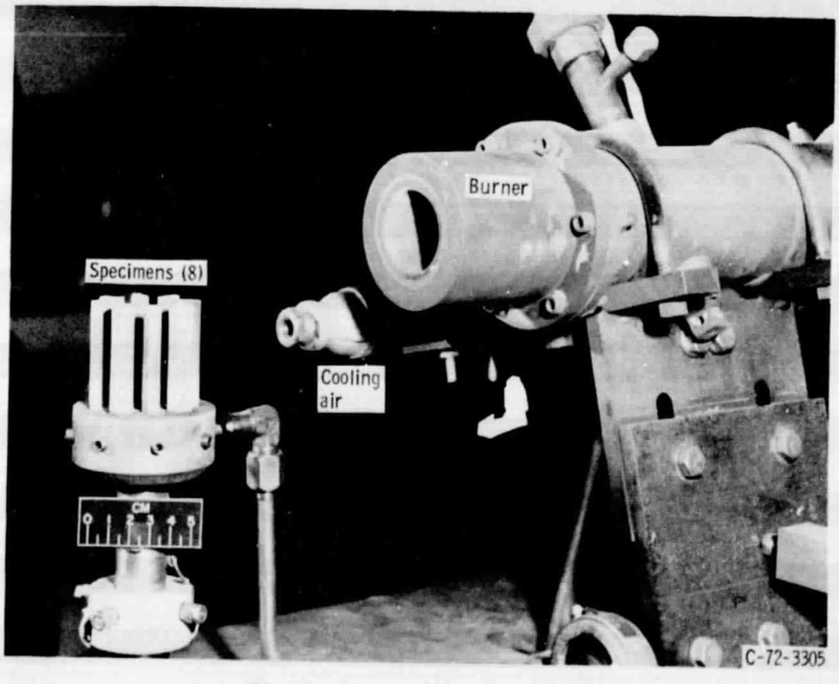

Figure 2. - Mach 0.3 oxidation apparatus.

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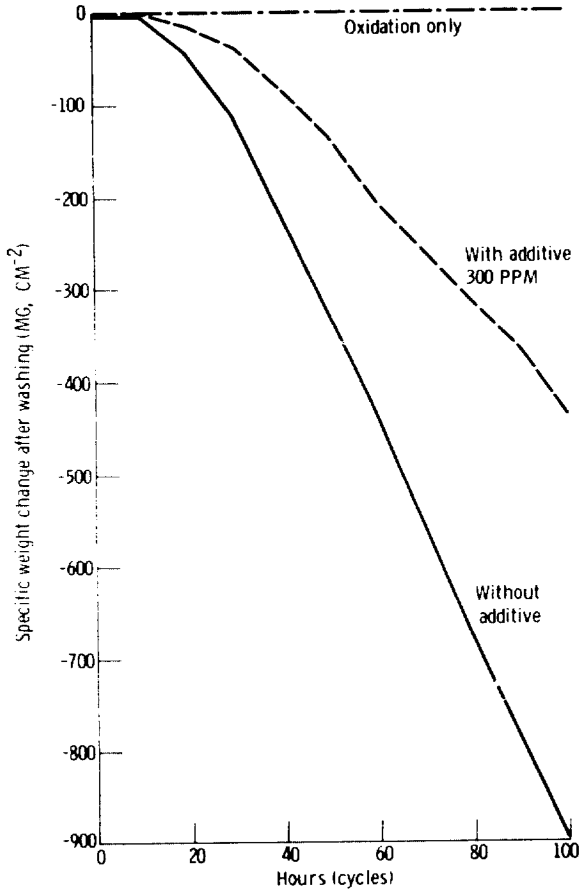
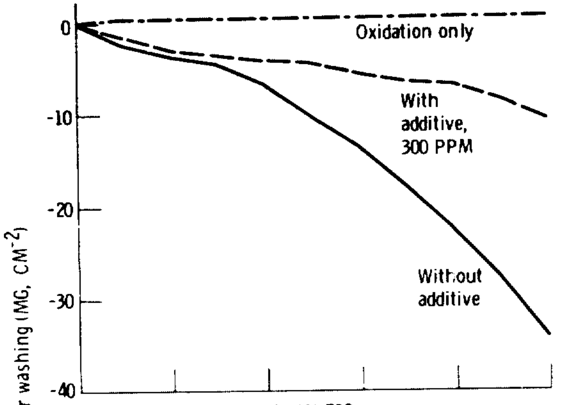
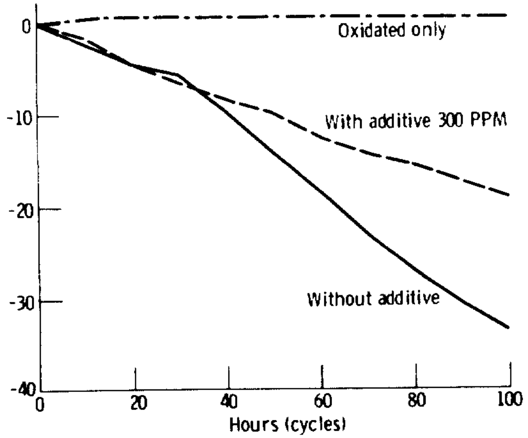


(a) IN-100.

Figure 3. - Effect of Cr additive on hot corrosion at 900° C. One hour cycles, washed every 10 cycles, 5 PPM salt.

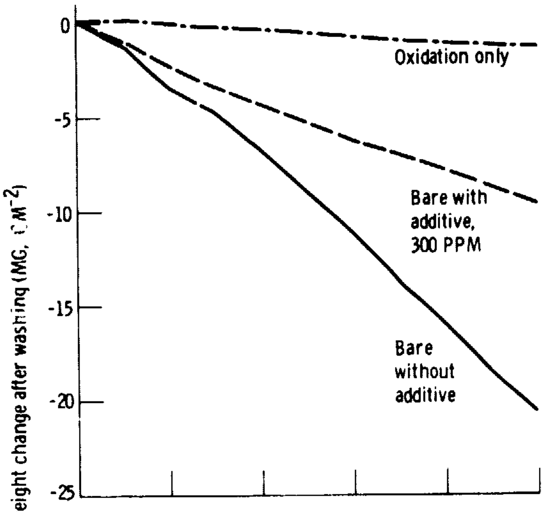


(b) IN-792.

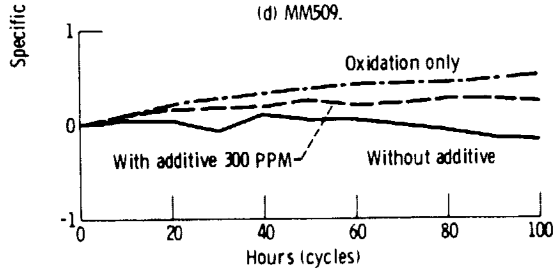


(c) IN-738.

Figure 3. - Continued.



(d) MM509.



(e) Coated MM509.

Figure 3. - Concluded.

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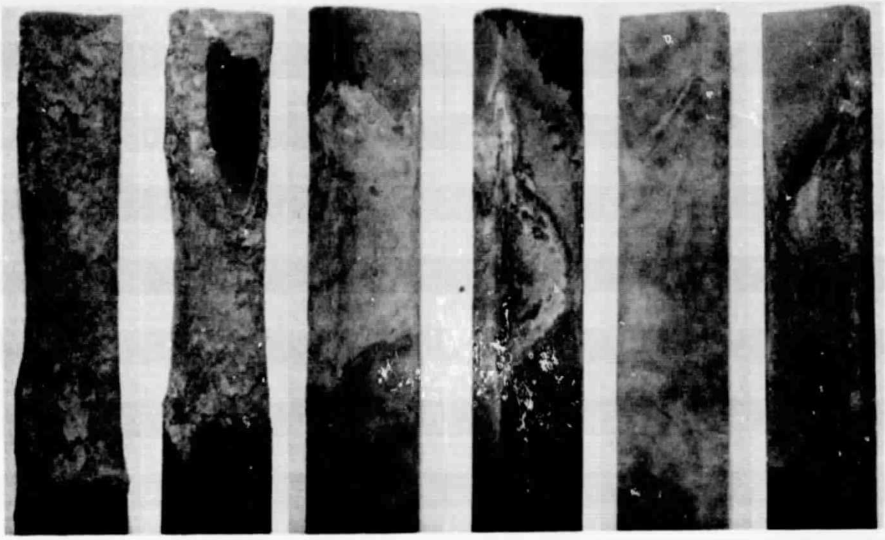

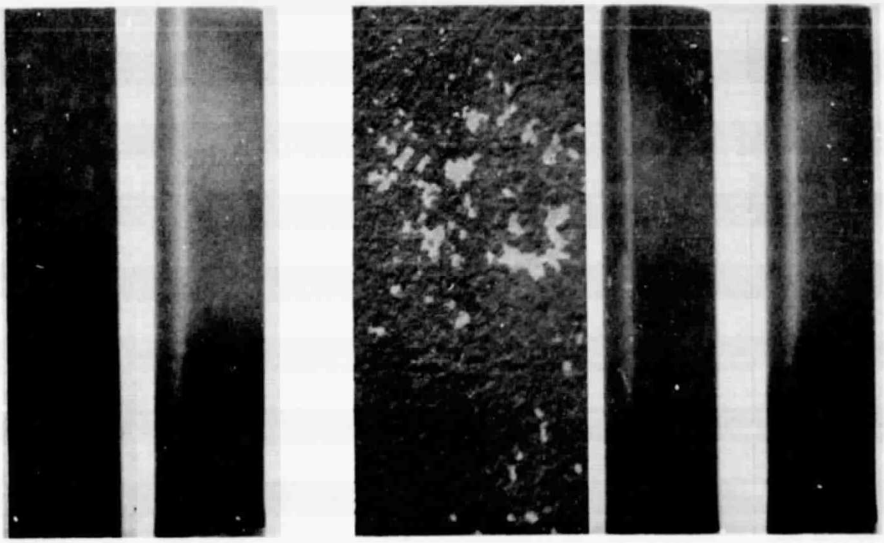

Figure 4. - Surface attack after 100, one hour cycles at 900° C, M0.3. Samples washed every ten cycles. Salt 5PPM, additive 300 PPM. 1.5 X except as noted.

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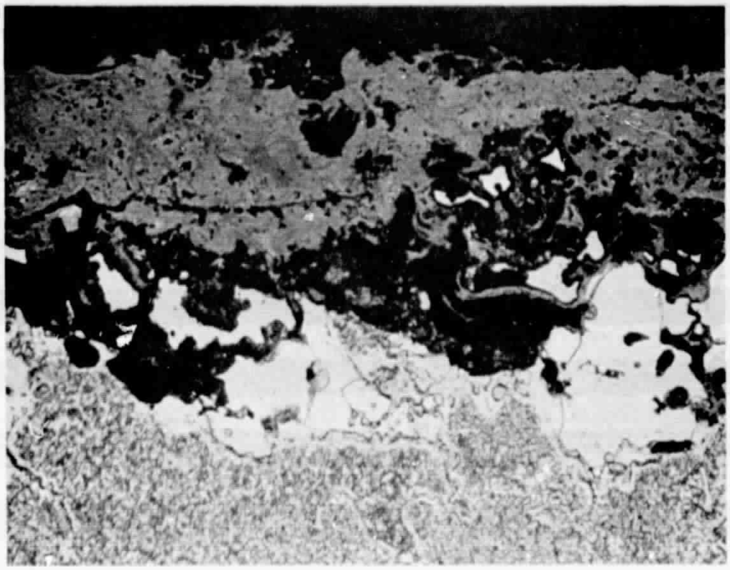
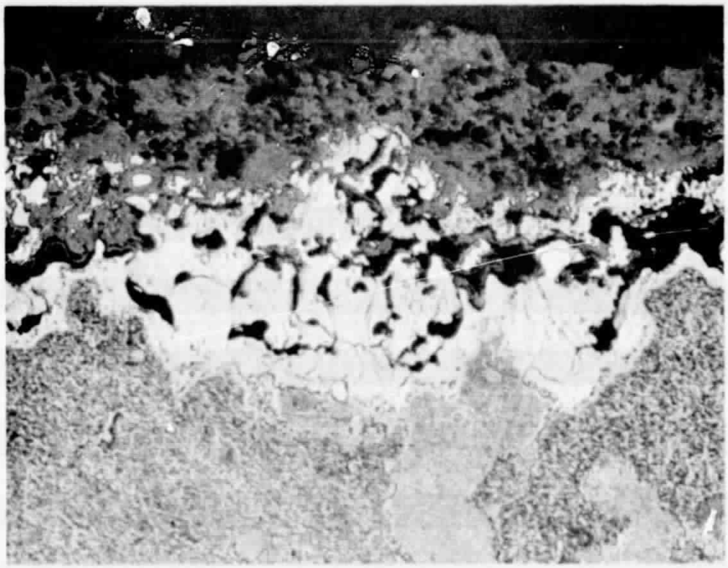


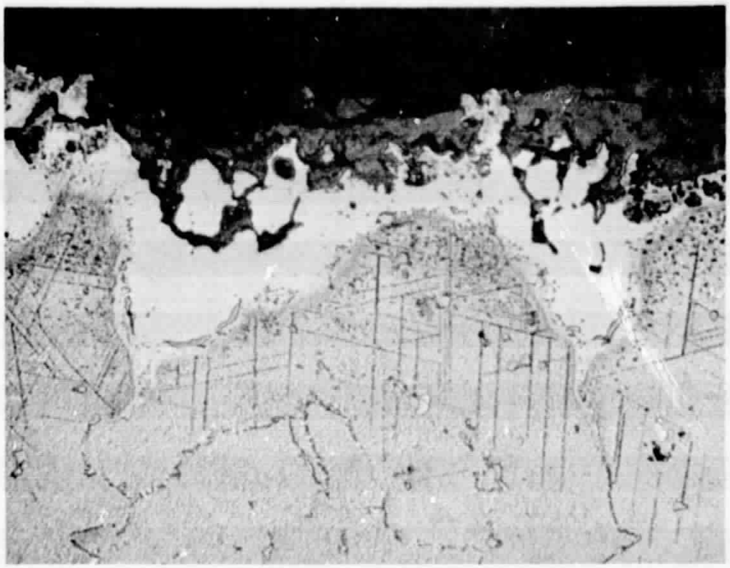
Salt only



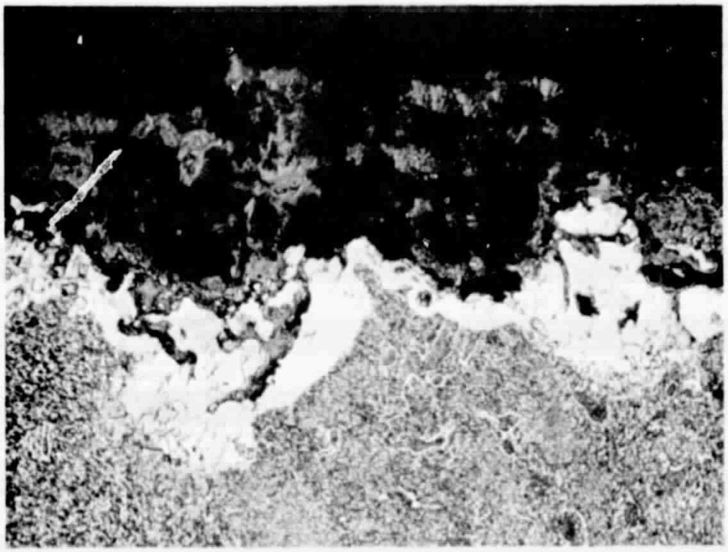
Salt + additive

(a) 100-One hour cycles washed every 10 cycles.

Figure 5. - Effect of MO.3 hot corrosion at 900° C on the microstructure of IN-738. Salt-5PPM, additive-300PPM; etched. X250.



Salt only

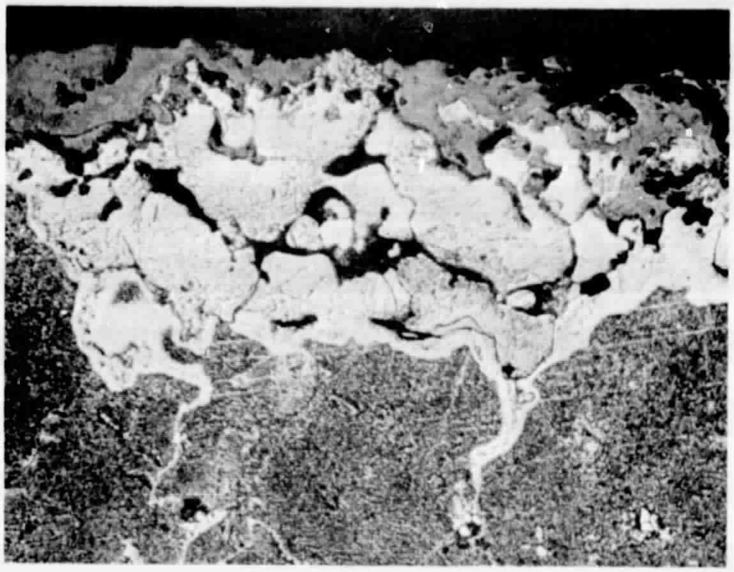


Salt + additive

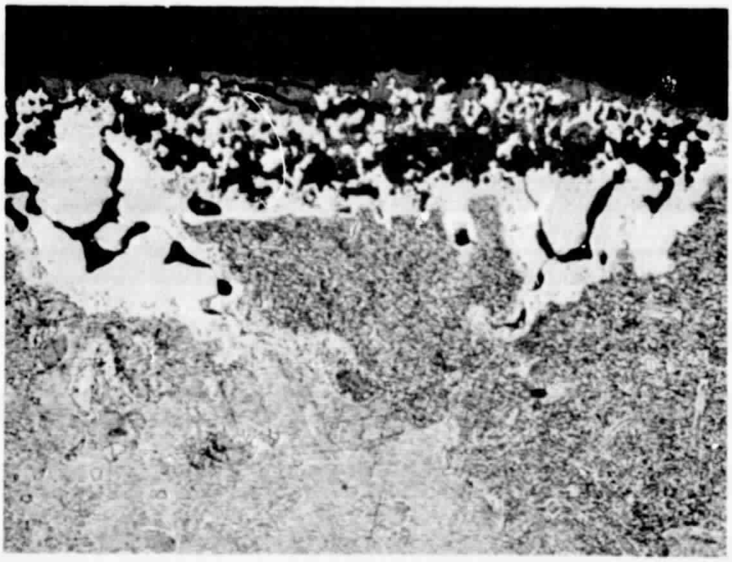
(b) 100-One hour cycles washed only at end of test.

Figure 5. - Continued.

E-9931



6 cycles, salt only



5 cycles, salt + additive

(c) Washed only at end of test.

Figure 5. - Continued.

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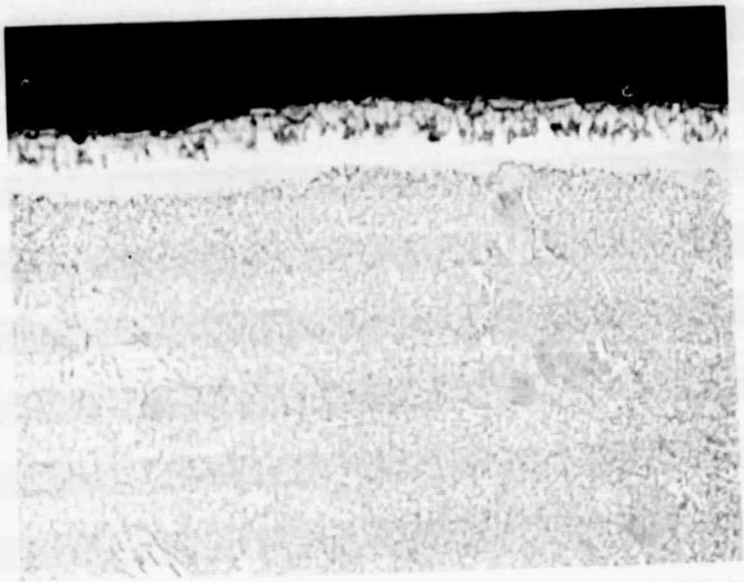

(d) No salt, no additive.

Figure 5. - Concluded.

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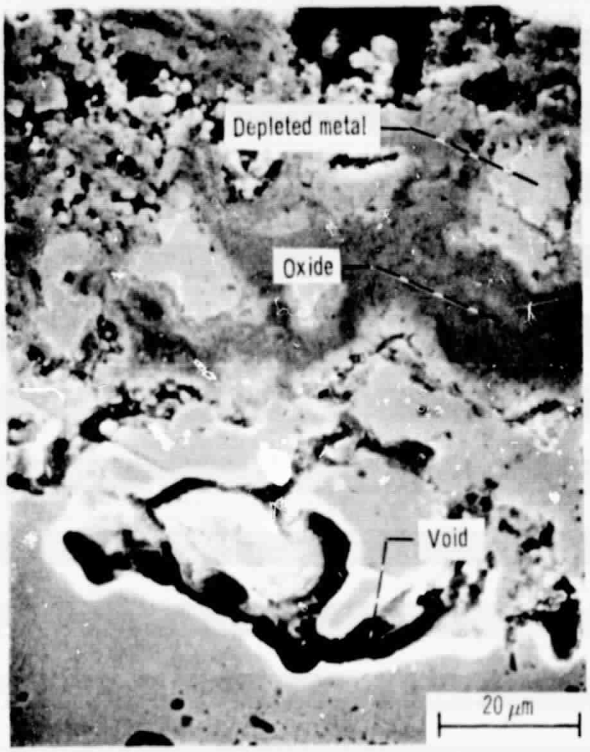
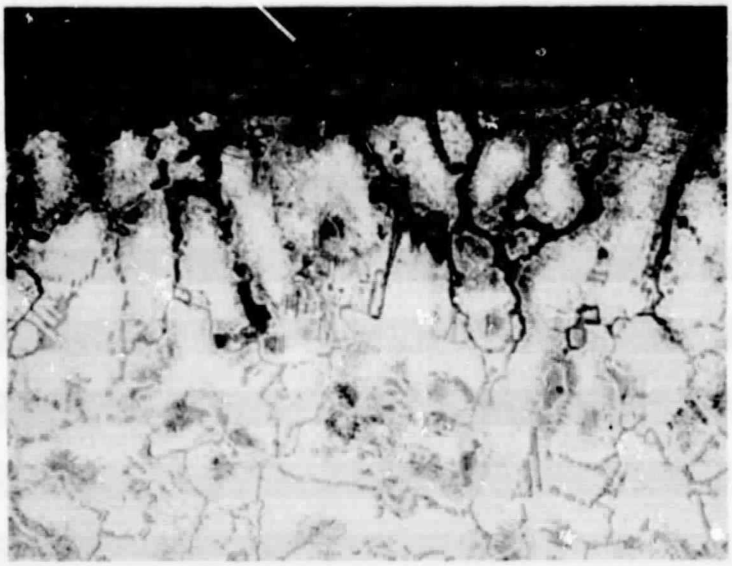
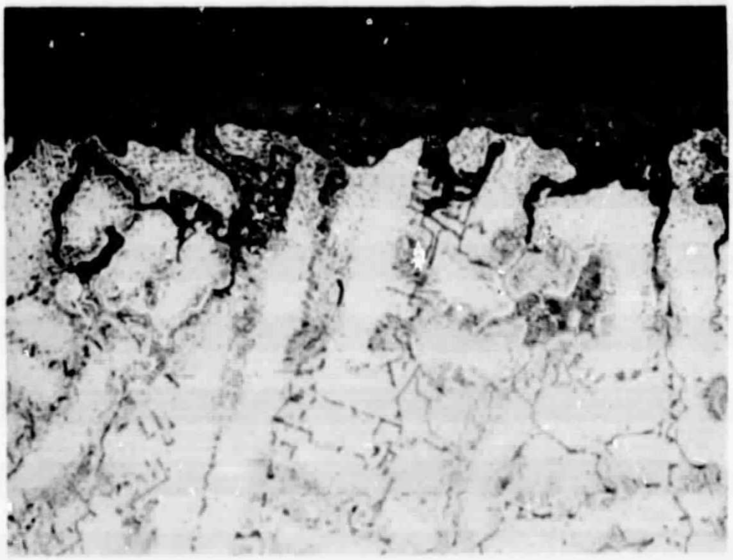


Figure 6. - Scanning electron microscopy of IN-738 after 100 hours at 900° C. MO.3, 5PPM salt, 6 cycles; unetched. X1000.

E-8831



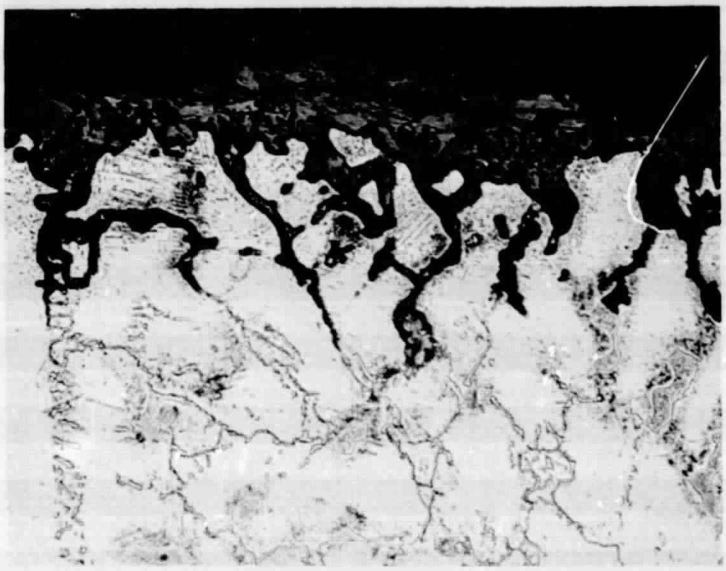
Salt only



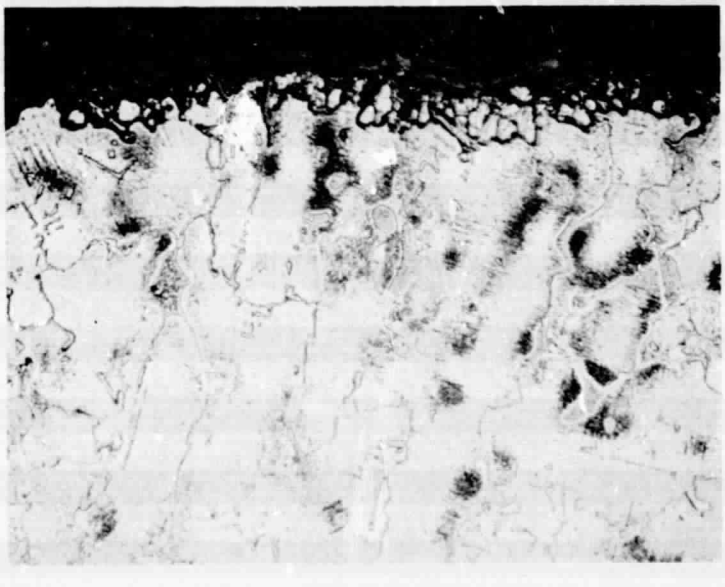
Salt + additive

(a) 100-One hour cycles washed every 10 cycles.

Figure 7. - Effect of MO.3 hot corrosion at 900° C on the microstructure of MM 509. Salt-5PPM, additive-300PPM; etched. X250.



Salt only



Salt + additive

(b) 100-One hour cycles washed only at end of test.

Figure 7. - Continued.

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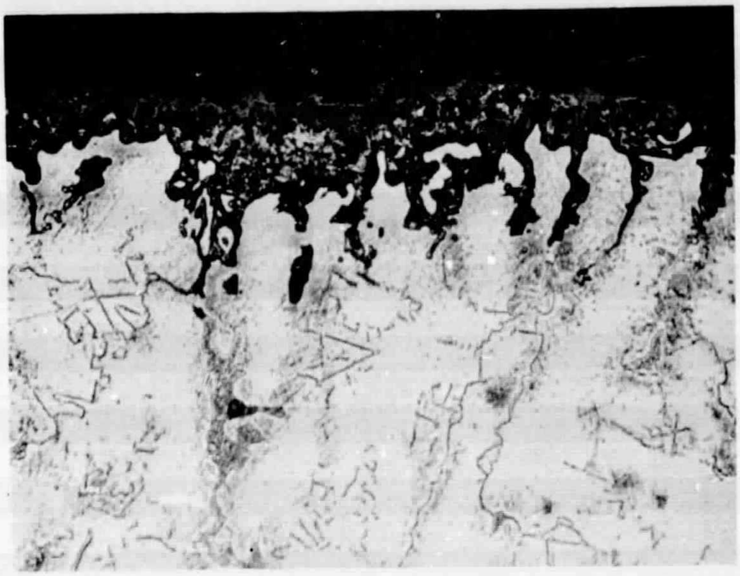
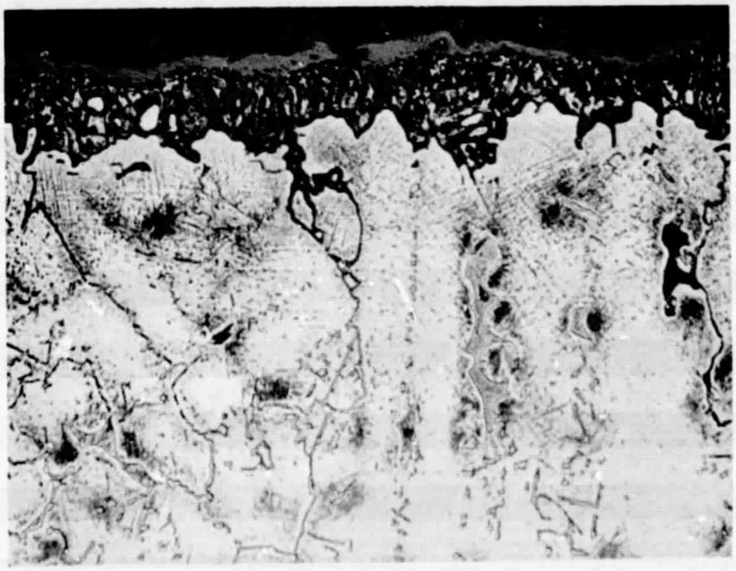


6 cycles, salt only

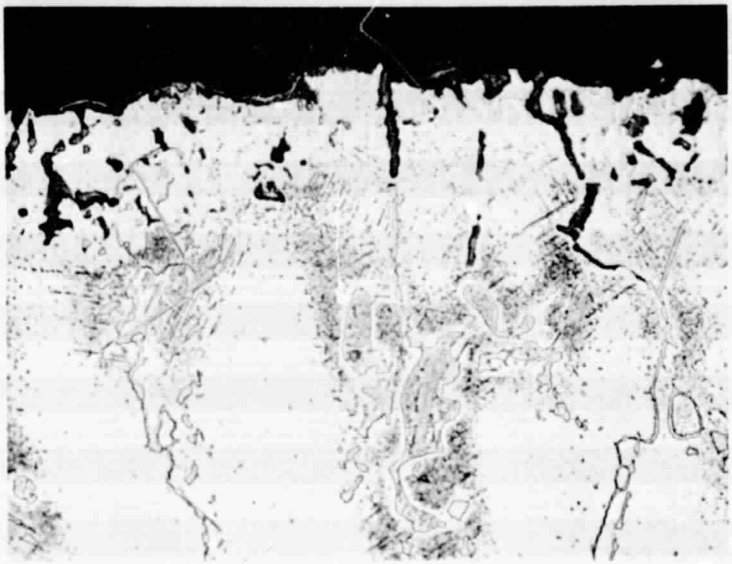


5 cycles, salt + additive

(c) Washed only at end of test.

Figure 7. - Continued.

E-8831



(d) No salt, no additive.

Figure 7. - Concluded.

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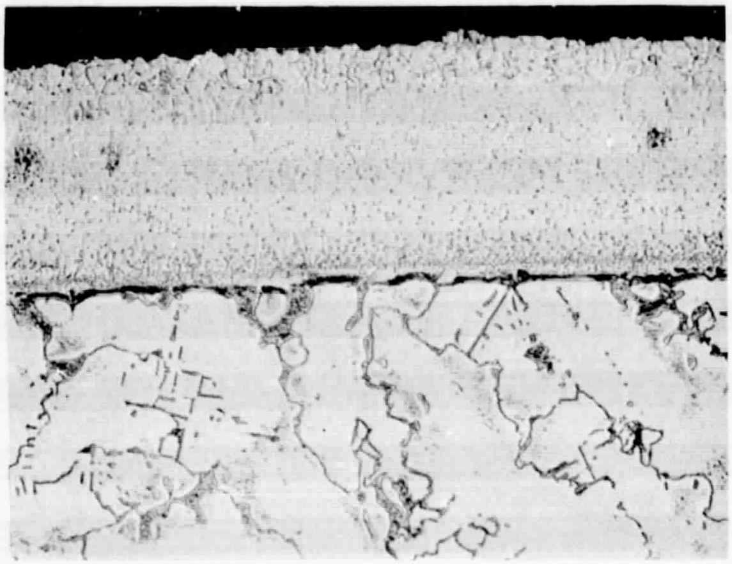

Salt only

Salt + additive

Figure 8. - Effect of MO.3 hot corrosion at 900° C on the microstructure of coated MM 509. Salt-5PPM, additive-300PPM, 100-one hour cycles, washed every 10 cycles; etched. X250.