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Technical Note N-1071

PROTECTIVE PROPERTIES OF COATINGS AS MEASURED BY DEW-CYCLE ACCELERATED WEATHERING

By

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January 1970

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ABSTRACT

With a new set of test conditions in an accelerated weathering machine, it was possible to determine protective properties of coatings under conditions of cycling light, heat, and humidity and in a salty environment. This salt-dew-cycle test combines the environment of the dew-cycle Weather-Ometer and of the underrust test. The scribed panels are periodically dipped in salt water, and increased rusting, blistering, and undercutting at the scribe is thereby obtained.

INTRODUCTION

The Naval Civil Engineering Laboratory investigates and evaluates new coatings for the Naval Facilities Engineering Command, and conducts research related thereto. The evaluation of the performance of such coatings under conditions similar to those encountered in actual service requires considerable time, especially if the coatings have long service lives. There is thus, a strong need for reliable accelerated test methods.

Many accelerated tests have been described in the literature;[1] however, all these tests appear to be of limited usefulness. Some tests show limited correlation for very specific applications; others were found suitable for comparing two or three coatings, without much being known about their general applicability.

Accelerated tests have generally been more useful in the prediction of surface changes in the coatings than in the prediction of the long term protective ability of the coatings. Thus, accelerated weathering machines are claimed by some to give useful information about surface changes caused by aging, such as chalking, fading, or loss of gloss.[2] No accelerated tests are known that are useful in reliably assessing the long term protection, or resistance to corrosion, provided by coatings.

The salt-spray test[3] is often used for the comparison of coatings, but this method is very inaccurate.[4] In the salt-spray test, scribed panels are continuously exposed to a high humidity produced from the atomization of salt water. There are no cyclic changes in the environment, and there is therefore no wetting and drying and the effect of radiant energy from sunlight is completely ommitted.

It would thus be desirable to combine in one test the effects of heat and light and the effects of salt and high humidity. The simplest method would be the cyclic-spraying with salt water of panels in a Weather-Ometer. However, the corrosive conditions so produced could not be tolerated long by conventional weathering machines. One attempt to provide such a combination resulted in very rapid corrosion of the weathering machine.[5]

Another approach to this problem was investigated at NCEL. This was essentially a combination of the environments of two other test methods. One of these was the environment of a relatively new method of using a dew cycle in a Weather-Ometer; the other was an environment similar to that encountered in the underrust test. The dew cycle subjects the panel alternately to light and to dew formation without runoff.[6] The underrust test subjects the scribed area of the panel to salt and high humidity.[7] The results of experiments with this method of testing coatings are described below.

EXPERIMENTAL METHODS AND RESULTS

The accelerated weathering machine which was used in these experiments was a xenon-arc Weather-Ometer with humidity control (Atlas Electric Devices Co., Model 60-WR). The rack of this Weather-Ometer was modified to hold the 2-3/4-by-5-7/8-inch panels. Two horizontal scribes, 1-3/4 inches long, were placed 1-1/4 inches from the top and from the bottom of each panel, and these were each intersected by two vertical scribes, 1/2 inch long. The panel arrangement in the Weather-Ometer is shown in Figure 1. Duplicate sets of panels were used; one set was placed in the top rack and one in the bottom rack.

During the dark cycle, the panels were cooled by water spray from two nozzles which replaced the normal rack spray arrangement. The spray nozzles were selected to produce maximum cooling with a minimum amount of demineralized water. These nozzles required approximately three gallons of water per hour each, during the operation of the spray. A cloth was held behind the panels of each quadrant to use the cooling water efficiently, and behind the cloth, stainless steel protective devices were installed between the panels, as shown in Figure 2, to prevent penetration of the water spray through the cloth to the front of the panels. A deflector was mounted inside the panel rack, as shown in Figure 3, to prevent the spray from hitting the xenon arc lamp or the panels on the opposite side when empty quadrants of the rack passed in front of the spray.

The water used to spray the rack was passed through a refrigerated cooling unit. A timing device made it possible to spray the rack during the first portion of the dark cycle and to stop the spray during the remainder of the dark cycle. Light cycles and dark cycles of 30 minutes each were employed.

At one-week intervals, the panels were removed from the Weather-Ometer and the lower scribed areas were dipped in one-percent sodium chloride solution. Thorough wetting of the scribe by the salt solution was insured by careful stroking with a soft brush. After one minute of immersion, the panels were removed, and excess salt solution was blotted from the edges and surfaces of the panel.

Each time the panels were replaced in the Weather-Ometer, the panels from the bottom rack were placed in the top rack, and vice-versa, to provide for even distribution of light on all panels. Periodically, the panels were rated and/or photographed. To highlight surface changes, the ilumination was placed at a low angle (a tangent of 0.167) and the photographs of the scribed areas were magnified five times.

Exposure of Thin Coating Systems

The initial experiments were conducted with five coating systems approximately 3 mils thick, consisting of oil, alkyd, vinyl-alkyd, phenolic, and Saran systems. These coatings are listed in Table 1. In this initial experiment the cooling water was not refrigerated and the spray was applied continuously during the total half hour of the dark cycle. The conditions were such that at the end of the light cycle the dry bulb temperature was 42.6°C with a relative humidity of 58 percent. At the end of the dark cycle the dry bulb temperature was 35.0°C and the relative humidity was 88 percent. The test was continued for a total of 1254 cycles, or 1,254 hours (about 52 days), of exposure.

The results of this experiment are shown in Figures 4-8, which show the conditions of portions of the upper and lower scribes. The rusting at the scribe, the development of filiform corrosion, and the blister formation are listed in Table 2.

Four of the above five systems had previously been exposed in an underrust test.[7] For this test the panels were hung in a glass humidity chamber in the laboratory at 40°C and at a relative humidity of 85%. An upper and a lower horizontal scribe were made on each panel with a 60-degree scribing tool. The lower scribe was dipped in one-percent sodium chloride solution at one-week intervals. The amounts of filiform corrosion obtained after 49 days, or approximately 1176 hours, are listed in Table 3. The filiform corrosion was confined to the lower scribe. The total area of all scribes had rusted, but there was essentially no blistering.

Exposure of Thick Coating Systems

Another experiment was performed with the thirteen thicker systems listed in Table 4. The thicknesses of these systems varied from about 6 mils to about 36 mils.

In this experiment, the spray water was cooled to approximately 12°C and the spray was applied during the first 20 minutes of the half-hour dark cycle. The dry bulb temperature at the end of the light cycle was 41.5°C and the relative humidity was 74 percent. At the end of the dark cycle the temperature was 29.8°C and the relative humidity was 96 percent. The experiment was continued for a total of 1,200 hours (50 days).

The panels were rated periodically, and at the end of the experiment, for blistering, undercutting, rusting, and general protection. The blistering rating was performed as nearly as possible according to ASTM method D 714-56, except that the blistering was along the scribe and that the blister size was recorded as seen through a two-power magnifying glass. The undercutting and rusting were rated as none (10), light (L), medium (M), or heavy (H). The general protection rating was an overall rating taking the previous factors into account and assigning a value of 10 for complete protection and a value of 7 for estimated failure. The ratings after 1200 hours of Weather-Ometer exposure are listed in Table 5. Photographs of the scribed portions of the coated panels that showed noticeable deterioration are shown in Figures 9 to 15.

The changes in gloss of the coating surfaces between the two scribes were also determined. The original gloss, the final gloss, the loss in gloss and the percentage loss are listed in Table 6.

The thirteen thick coating systems had all been subjected to exposure tests under marine atmospheric environments at Kwajalein, Marshall Islands, at Kaneohe, Hawaiian Islands, and at Port Hueneme, California.[8] In Table 7, the quality ratings for the scribed panels are given for each coating at each location. The quality rating assigned was approximately ten times the number of years required for failure of the coated panels under those particular conditions. For one of the coating systems at Kaneohe and for a majority of the coating systems exposed at Port Hueneme, failure had not taken place at the last rating, and the quality rating was therefore only a minimum value. (Quality ratings preceeded by ">" are based on a final protection rating of "8", those preceeded by ">>" are based on a final rating of "9", and those preceeded by ">>>" are based on a final rating of "10".) For the Kwajalein exposure tests, there are listed in Table 7 not only the quality ratings, but also the performance ratings after three years of exposure. The latter ratings include general protection, blistering, and undercutting, as described above, but no magnification was used in assigning the blistering rating.

DISCUSSION

In accelerated weathering machines run in the normal manner, very long exposures are generally needed to produce rusting and blistering, and thus evaluate the protective performance of coatings. With a dew cycle and with panels containing scribes maintained in a salty condition, rusting, blistering, and undercutting were obtained in a relatively short exposure period. This procedure combines the effects of light changes, heat changes, humidity changes, and saline conditions in one test. For simplicity, it will be referred to as the salt-dew-cycle test.

Some precautions must be taken when setting up the salt-dew-cycle test conditions. The dew formation must be sufficiently great to thoroughly wet all the panels, but it must not be so great as to produce run-off on the panels. The dew formation is affected by the color and texture of the coating surface. It is also affected by the heat transmission from the back of the panel, which is being cooled, to the front of the panel, on which the dew will form. By using cooled water and a spray of shorter duration it was possible to produce dew formation more rapidly and then maintain the dew for a longer period of time, while the spray was off and the light was not yet on.

In the first experiment with the five thinner coating systems, the differences in the performances of the coatings were quite strong.

The oil system performed best in the salt-dew-cycle test, followed by the Saran system, the alkyd and the phenolic systems, and lastly by the vinyl-alkyd system. This experiment was performed as a preliminary trial and no atmospheric exposure results were available for the same five systems.

The results with the salt-dew-cycle test were different from those obtained in the underrust test. In the underrust test, the scribed panels were continuously exposed at a high relative humidity (85% at 40°C). Four of the systems (all but the Saran) had been so exposed. The chief deterioration was filiform corrosion from the scribe that had been dipped in salt water. No filiform corrosion was obtained from scribes that were not treated with salt.

Filiform corrosion is a type of corrosion which occurs on coated panels under high humidity. It starts at exposed steel and proceeds under the coating in thin, long channels. These may progress in varying directions, but they never intersect.

In the salt-dew-cycle test, less filiform corrosion was produced and more blistering was obtained than in the underrust test. This is a more natural type of deterioration. The upper scribes which had not been treated with salt were not immune from deterioration (as in the underrust test), but the deterioration was considerably reduced. Not only the type of deterioration, but also the order of performance differed in the salt-dew-cycle test and in the underrust test.

Different performances would, of course, be expected with different accelerated tests, otherwise all tests would give the same correlation with field performance. It is of interest that the performance of the five thin coating systems in laboratory immersion in seawater at 25°C was in the reverse order from that given above for the salt-dew-cycle test.[9]

The thirteen systems for which atmospheric exposure data were available were all much thicker coating systems. None of these produced the filiform corrosion or the extensive blistering near the scribe that was produced with the thin coating systems. There also was a less pronounced difference between the effects of the scribe that was dipped in the salt solution and the scribe that was not dipped. But half the panels definitely had greater deterioration at the lower scribe mark that had been kept salty, and none had greater deterioration at the upper scribe.

No definite correlation could be established between the results obtained with the Weather-Ometer and the results of atmospheric exposures. The two systems which had the poorest performance at Kwajalein and at Kaneohe, Systems 113 and 114, (as well as the two systems with the poorest performance at Port Hueneme, Systems 113 and 115) did produce blistering and undercutting in the Weather-Ometer but so did five other systems of better performance.

The performances at Kwajalein and Kaneohe were so similar that there was no significant difference in correlation with the salt-dew-cycle test results. Correlation with field exposure results at Port Hueneme may be slightly higher, but so few of the coating systems had reached the point of failure at Port Hueneme, that no conclusions could be drawn.

It was expected that changes in gloss obtained in the Weather-Ometer would not be directly related to the protection provided to scribed panels, but that they should rather be related to the surface changes during atmospheric exposure. No accurate ratings of coating surface changes in field exposure were available for correlation, because of the emphasis on rating of the protective performance, and because the coating surface conditions were dependent on rainfall prior to inspection. However, the gloss changes should be related to the general stability of the coatings and might therefore be useful in the development of a prediction equation for the overall performance of the coatings.

One of the difficulties in making comparisons between the results of the atmospheric exposure tests, as shown in Table 7, and the results with the salt-dew-cycle test, as shown in Table 5, is the fact that the ratings of both methods are somewhat subjective and are difficult to record in quantitative form. Another difficulty is the fact that in some cases different results were obtained with duplicate panels. Thus the panel with the poorest performance in the Weather-Ometer was one panel of System 117, which had very heavy undercutting. However, the other panel of the same system showed no undercutting whatsoever. The former of these panels could have had a defect in the metal or a defect in application. Three other systems also had one panel which showed no undercutting or blistering and one panel which did show undercutting and/or blistering.

The salt-dew-cycle test produces definitely greater and more rapid changes on panels coated with thin systems than on panels coated with thick systems. The testing of thick systems could therefore be accelerated by thinner application of the coating on the test panels. But because the filiform corrosion appears to proceed by a somewhat different process from that of the undercutting and blistering, a reduction of the coating thickness may change the validity of the results. It is possible that this test is more applicable to the testing of thin coatings, such as appliance coatings or automotive coatings, than it is to the testing of protective coatings for marine structures.

In the salt-dew-cycle test as described above, only one scribe mark was periodically immersed in salt water. The chief reason for not immersing the other scribe was to verify the assumption that this small amount of salt would appreciably increase the deterioration produced at the scribe. It is recommended that in future tests both scribes of each panel be periodically treated with salt solution so as to essentially double the number of sites to be rated and thereby increase the validity of the data.

CONCLUSIONS

1. With the salt-dew-cycle test (using a dew cycle in an accelerated weathering machine and scribed coated panels periodically dipped in salt water) it is possible to determine protective properties of coatings under conditions of cycling light, heat, and humidity and in a salty environment.

2. Strong effects at the scribe, including blistering near the scribe and filiform corrosion, are obtained with thin coating systems.

3. Lesser effects at the scribe, but including blistering at the scribe and undercutting, are obtained with thicker coating systems.

4. Results to date do not give conclusive information about the correlation of this method with performance in atmospheric exposure tests.

ACKNOWLEDGMENTS

The furnishing of coating evaluation results, by Dr. R. L. Alumbaugh and Messrs. C.V. Brouillette and A. F. Curry, both published[8] and unpublished, is gratefully acknowledged.

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Table 1. Thin Coating Systems

System	Specification	Description	Thickness (mils)
1	TT-P-86a	Oil, red lead, primer	1.1
	TT-P-102a	Oil topcoat (gray)	1.1
			Total 2.2
2	MIL-P-15328B	Formula 117, wash primer	0.5
	MIL-P-17545B	Formula 116, alkyd, red lead, primer	1.2
	MIL-E-15130C	Alkyd enamel (gray)	1.2
			Total 2.9
3	MIL-P-15328B	Formula 117, wash primer	0.3
	MIL-P-15929B	Formula 119, vinyl, red lead primer	2.2
	MIL-E-15936B	Formula 122/27, vinyl-alkyd enamel (gray)	1.2
			Total 3.7
4	MIL-P-15328B	Formula 117, wash primer	0.4
	MIL-P-12742B	Phenolic, water-immersible, primer	1.4
	MIL-E-12507A	Phenolic enamel (white)	1.3
			Total 3.1
5	MIL-L-18389	Formula 113/54, vinylidene resin, orange	1.7
	MIL-L-18389	Formula 113/54, vinylidene resin (white)	1.7
			Total 3.4

Table 2. Salt-Dew-Cycle Test Results with Thin Coating Systems

System	Comparative Performance	Scribe	Portion Rusted %	Filiforms: Approximate Number	Filiforms: Maximum Length (mm)	Blister Density *
1	A	upper	10	--	--	--
		lower	70	--	--	--
2	C	upper	90	--	--	--
		lower	100	50	16	MD
3	E	upper	100	30	6	F
		lower	100	30	10	D
4	D	upper	100	20**	7	--
		lower	100	35	7	MD
5	B	upper	100	--	--	F
		lower	100	--	--	M

*F = few, M = moderate, MD = moderately dense, D = dense

**faint filiforms

Table 3. Underrust Test Results with Thin Coating Systems

System	Comparative Performance	Filiforms		
		Approximate Number	Maximum Length (mm)	Thickness
1	B	40	10	faint
2	D	60	24	faint
3	C	30	11	strong
4	A	20	6	moderate

Table 4. Thick Coating Systems

System	Description	Thickness (mils)
111	Epoxy metal primer	4.3
	Epoxy-phenolic finish (medium gray)	10.2
	Total	14.5
112	Polyurethane zinc chromate primer	1.3
	Polyurethane finish (gray)	6.3
	Total	7.6
113	Epoxy zinc chromate primer	3.4
	Epoxy finish (white)	9.4
	Total	12.8
114	Modified phenolic primer	7.9
	Modified phenolic finish (gray)	8.0
	Total	15.9
115	Epoxy primer	0.9
	Epoxy intermediate	1.4
	Epoxy finish (gray)	5.3
	Total	7.6
116	Vinyl red lead iron oxide primer	1.6
	Chlorosulfonated polyethylene finish (gray)	6.5
	Total	8.1
117	Mica-filled phenolic mastic primer	13.0
	Phenolic mastic finish (gray)	4.8
	Total	17.8
118	Vinyl-phenolic strontium chromate iron oxide primer	1.5
	Vinyl finish (gray)	5.0
	Total	6.5
119	Epoxy primer	1.1
	Epoxy intermediate	2.6
	Epoxy finish (tan)	2.5
	Total	6.2
120	Vinyl-phenolic strontium chromate iron oxide primer	1.5
	Vinyl mastic finish (black)	35.0
	Total	36.5
121	Coal-tar urethane (black)	8.0
	Total	8.0
122	Pretreatment primer	0.5
	Vinyl red lead primer	3.4
	Aluminum-pigmented vinyl finish (aluminum)	3.9
	Total	7.8
123	Saran, formula 113/54, alternate coats of orange and white (white)	7.0
	Total	7.0

Table 5. Salt-Dew-Cycle Test Results with Thick Coating Systems

System and Panel**	Rating at Upper Scribe*				Rating at Lower Scribe*			
	Protection	Blistering	Undercutting	Rusting	Protection	Blistering	Undercutting	Rusting
111-A	10	10	10	L	10	10	10	L
-B	10	10	10	L	10	10	10	L
112-A	10	10	10	L	10	10	10	L
-B	10	10	10	L	10	10	10	L
113-A	10	10	10	L	9	8 M	L	L
-B	10	10	10	L	9	2 M	L	L
114-A	10	10	10	L	8	2 M	L	L
-B	10	10	10	L	10	10	10	L
115-A	10	10	10	L	10	10	10	L
-B	9	8 F	10	L	9	8 F	10	L
116-A	10	10	10	L	10	10	10	L
-B	10	10	10	L	10	10	10	L
117-A	10	10	10	L	9	10	10	M
-B	10	10	10	L	2	10	H	L
118-A	9	4 F	L	L	8	4 MD	L	L
-B	10	10	10	L	10	10	10	L
119-A	9	4 MD	L	L	9	4 M	L	L
-B	9	6 F	10	L	9	4 F	10	M
120-A	10	10	10	L	9	9 F	10	L
-B	10	10	10	L	9	9 F	10	L
121-A	10	10	10	L	10	10	10	L
-B	10	10	10	L	10	10	10	L
122-A	10	10	10	L	10	10	10	L
-B	10	10	10	L	10	10	10	L
123-A	10	10	10	L	10	10	10	L
-B	10	10	10	L	10	10	10	L

*As described in the text, under "Exposure of Thick Coating Systems."

**A and B denote duplicate panels.

Table 6. Gloss Changes in Accelerated Weathering

System	Gloss Values			
	Original	Final	Loss	% Loss
111	60	9	51	85
112	72.5	4.9	67.6	93
113	89	21	68	76
114	44.8	7.3	37.5	84
115	94	47	47	50
116	6.9	6.0	0.9	13
117	14.7	2.6	12.1	82
118	7	7.6	-0.6	-9
119	16.5	7.5	9	52
120	1.5	2.5	-1.0	-67
121	40.6	4.2	36.4	90
122	9	6	3	33
123	6.1	7.0	-0.9	-15

Table 7. Field Performance of Scribed Panels
with Thick Coating Systems

System	At Kwajalein				At Kaneohe	At Port Hueneme
		Rating After 3 Years*				
	Quality*	Protection	Blistering	Undercutting	Quality*	Quality*
111	28	7	2 D	M	>60	>>> 50
112	17	<6	2 D	H	27	>50
113	14	<<8	2 D	M	9	25
114	9	<<7	2 D	H	25	>80
115	40	8	2 D	L	35	60
116	65	9	4 MD	L	>>95	>>>85
117	49	9	2 F	10	82	>>115
118	36	8	2 M	M	84	>>115
119	45	8	2 D	L	58	>>65
120	37	8	2 M	10	85	>>65
121	18	<6	2 D	H	33	80
122	39	9	2 MD	10	60	>115
123	27	6	2 D	10	70	>>>115

*As described in the text, under "Exposure of Thick Coating Systems."

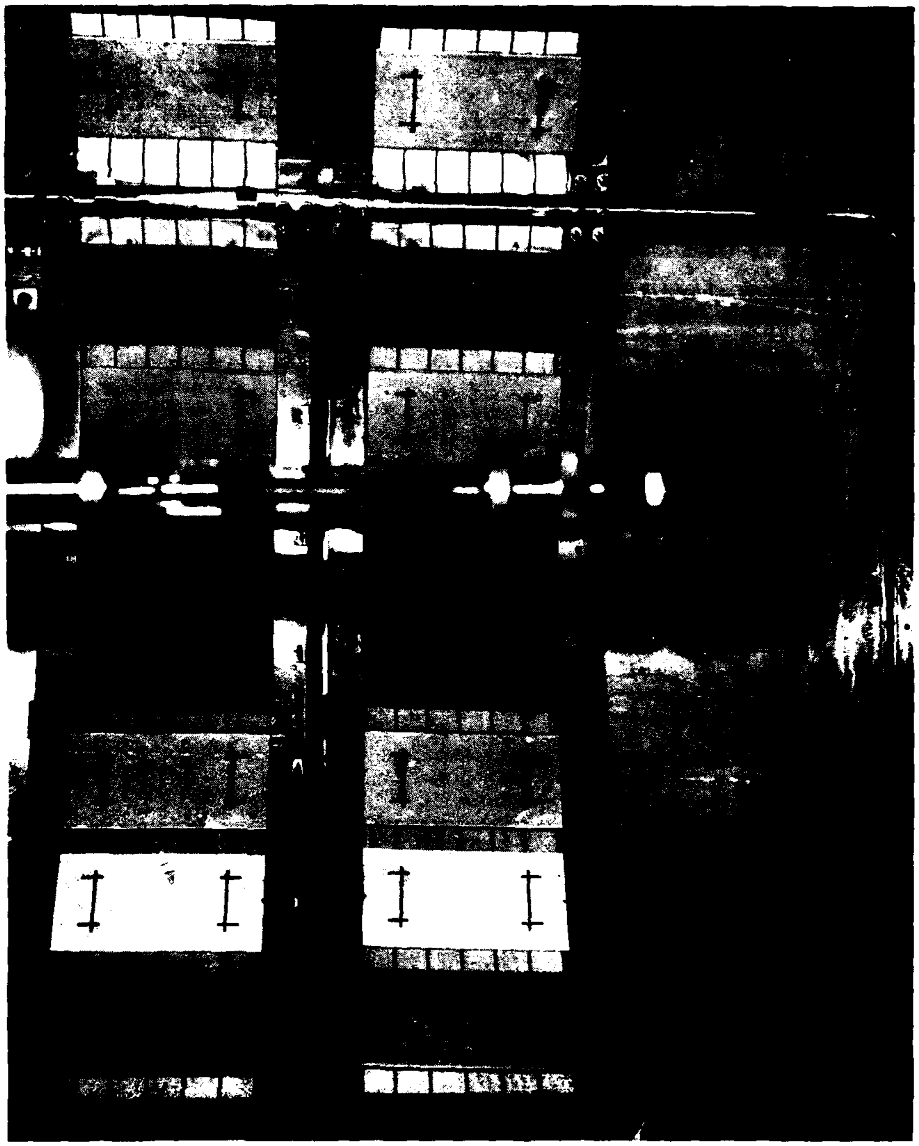


Figure 1. Panel arrangement in Weather-Ometer

Figure 2. Cooling of panels in Weather-Ometer

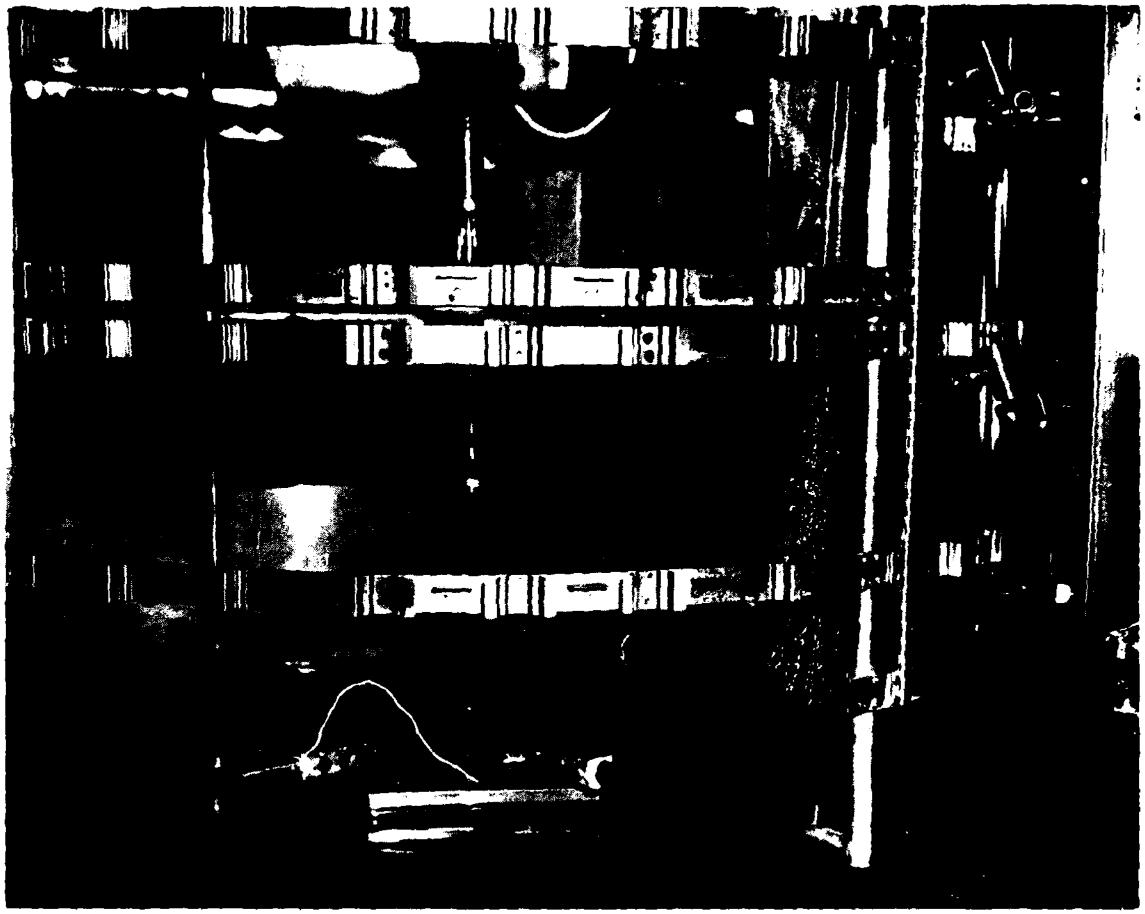
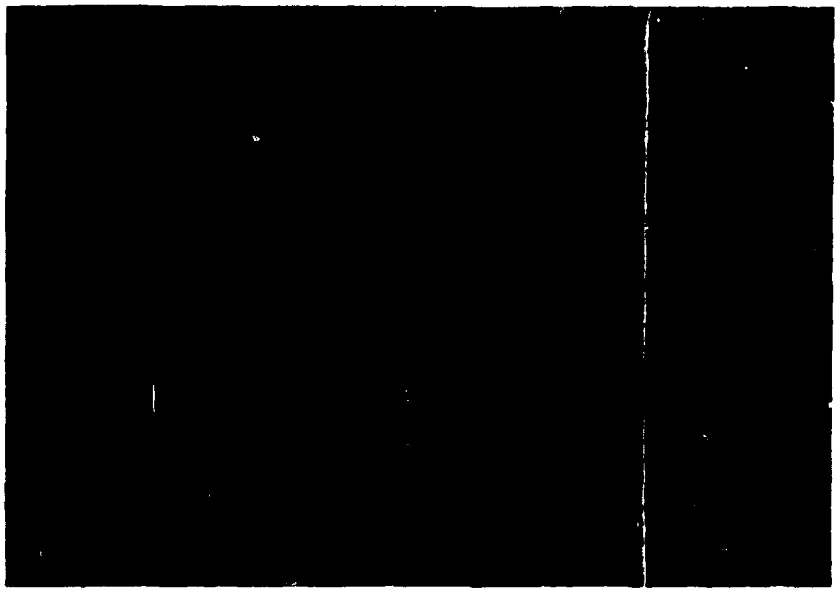


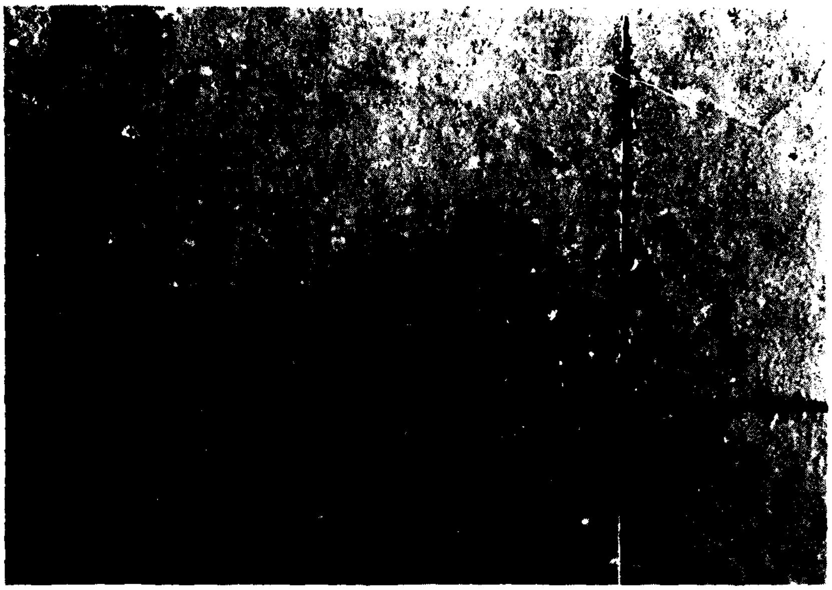
Figure 3. Spray nozzles and deflector in Weather-Ometer



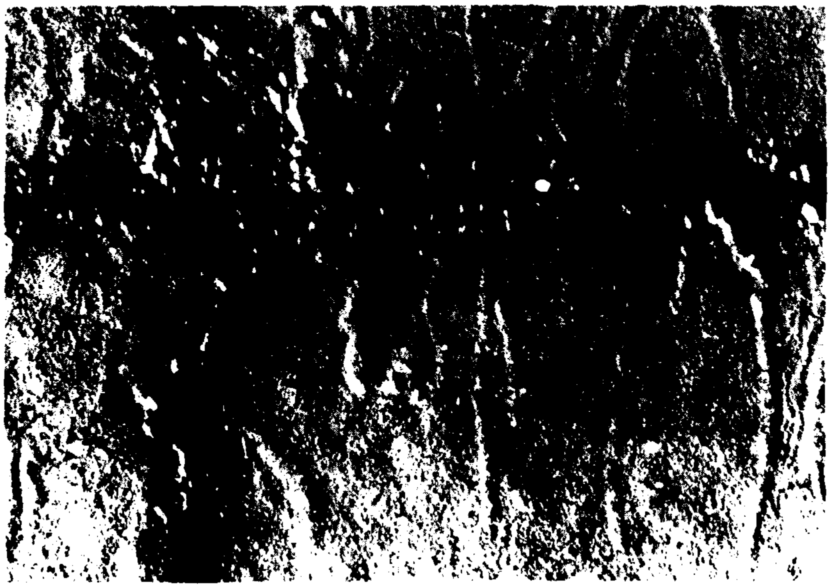
upper scribe

lower scribe

Figure 4. Exposed panels of System 1

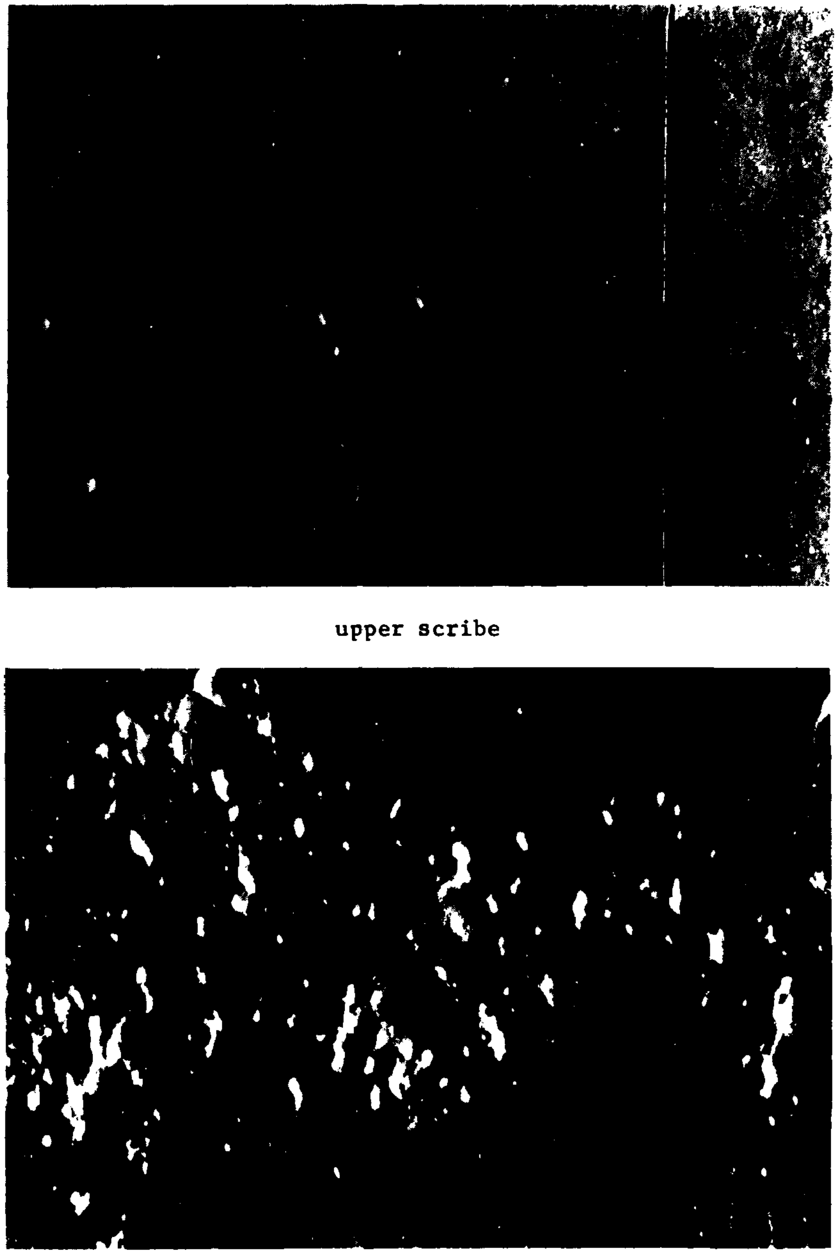


upper scribe



lower scribe

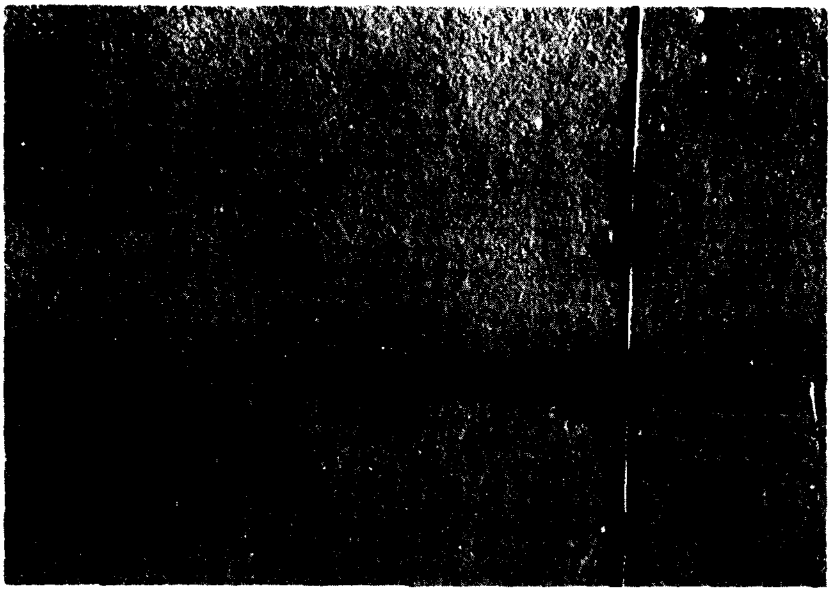
Figure 5. Exposed panels of System 2



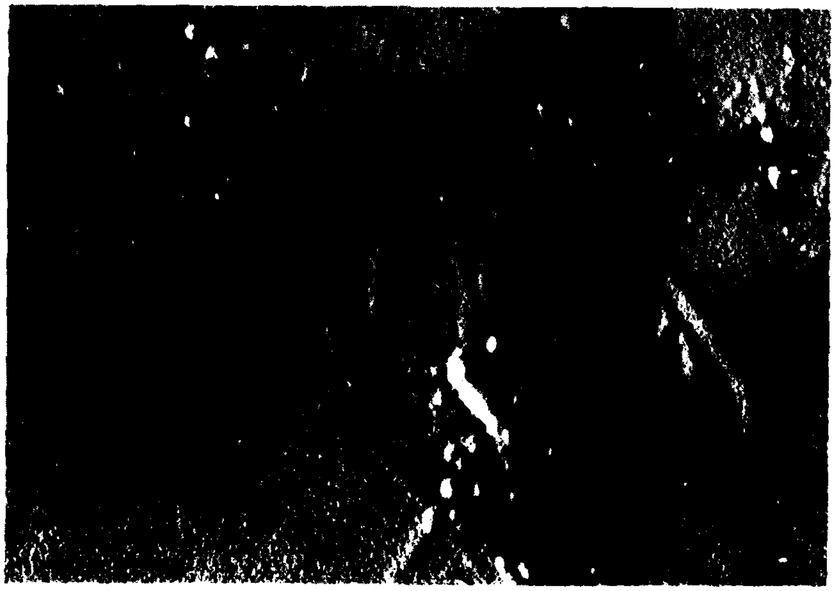
upper scribe

lower scribe

Figure 6. Exposed panels of System 3



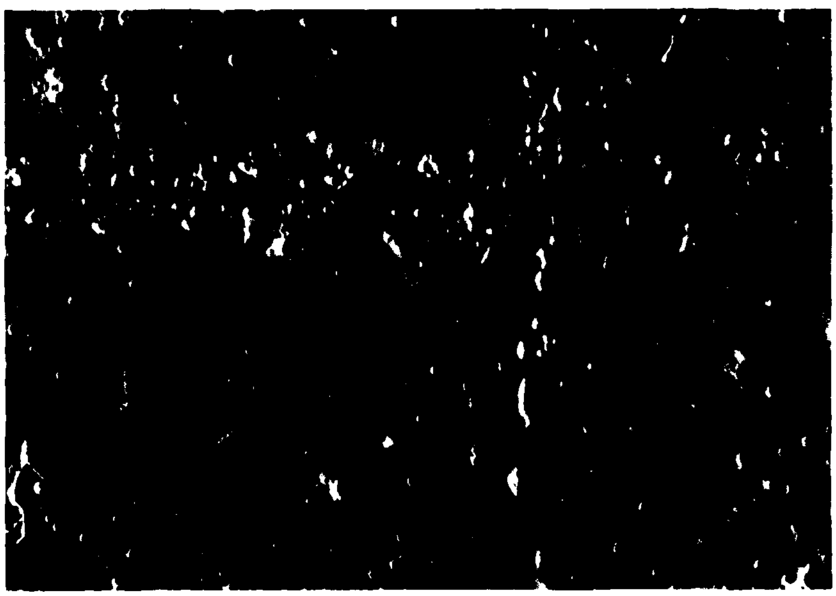
upper scribe



lower scribe

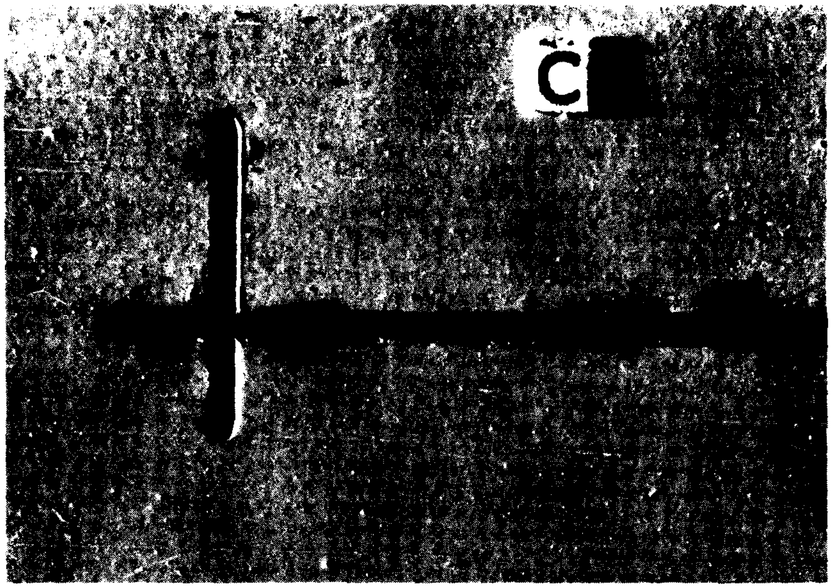
Figure 7. Exposed panels of System 4

upper scribe

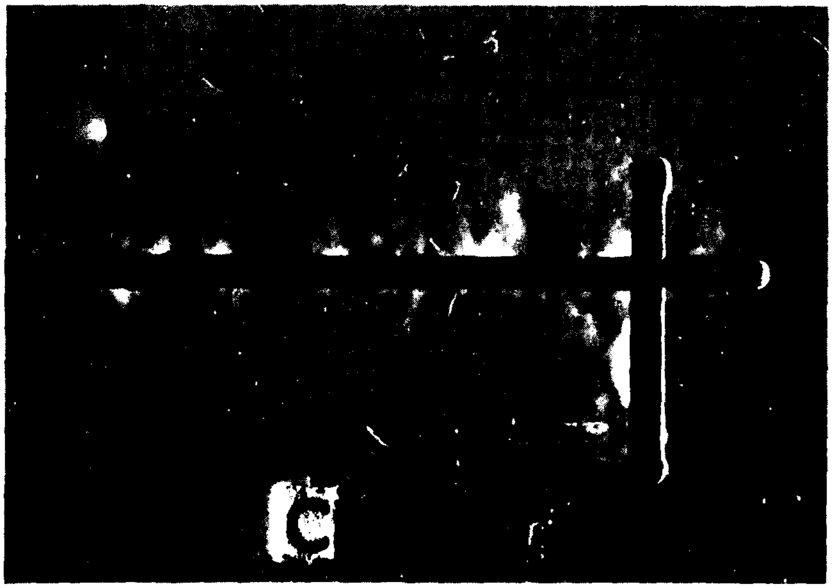


lower scribe

Figure 8. Exposed panels of System 5

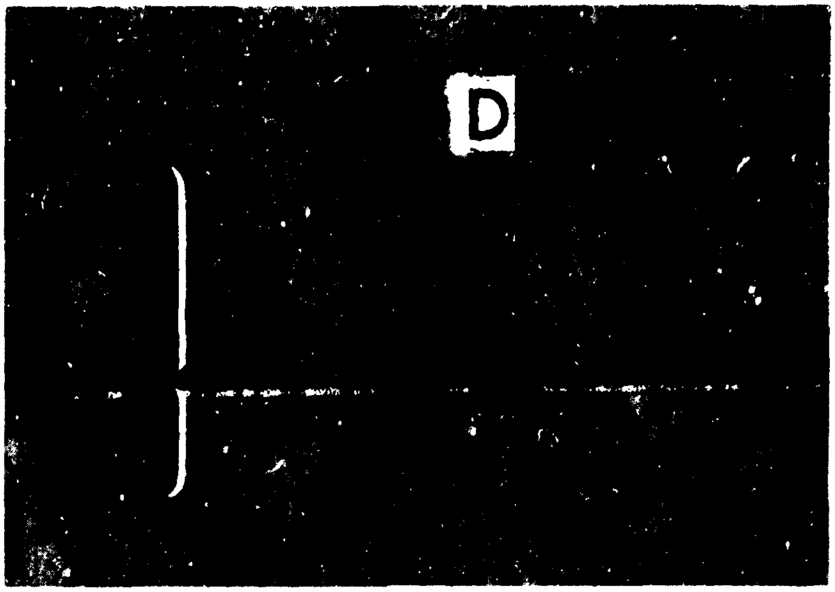


upper scribe



lower scribe

Figure 9. Exposed panels of System 113



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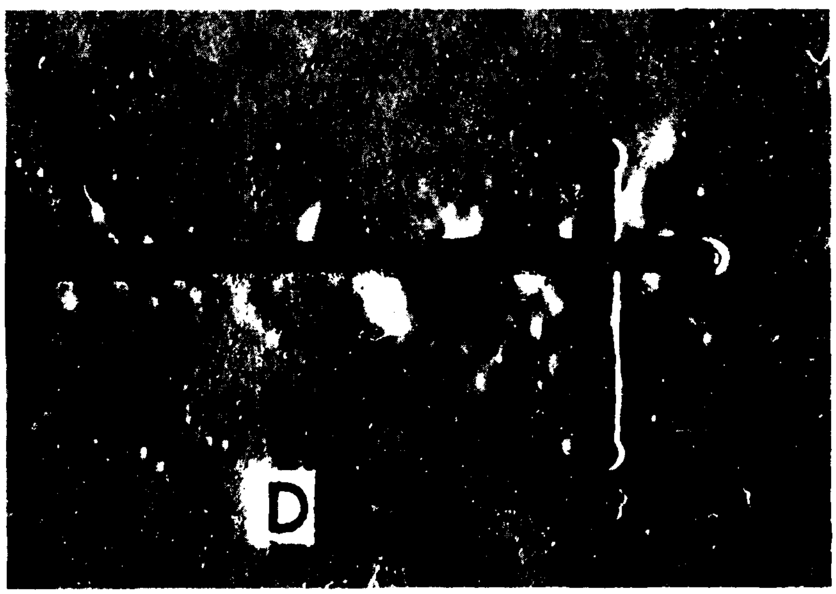
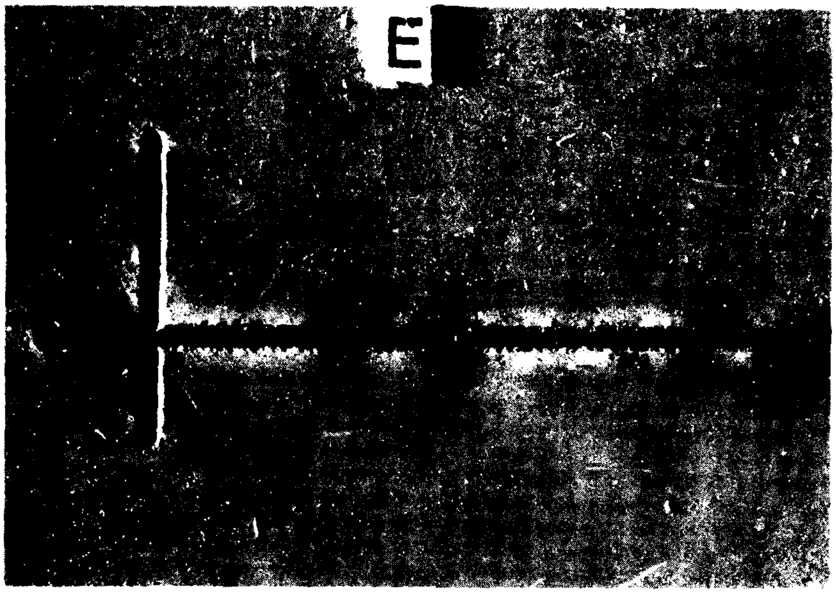
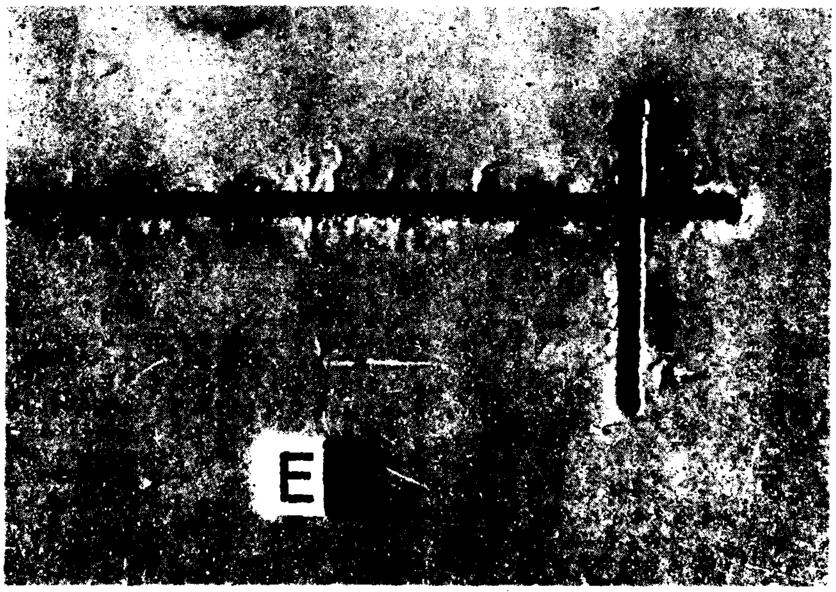


lower scribe

Figure 10. Exposed panels of System 114

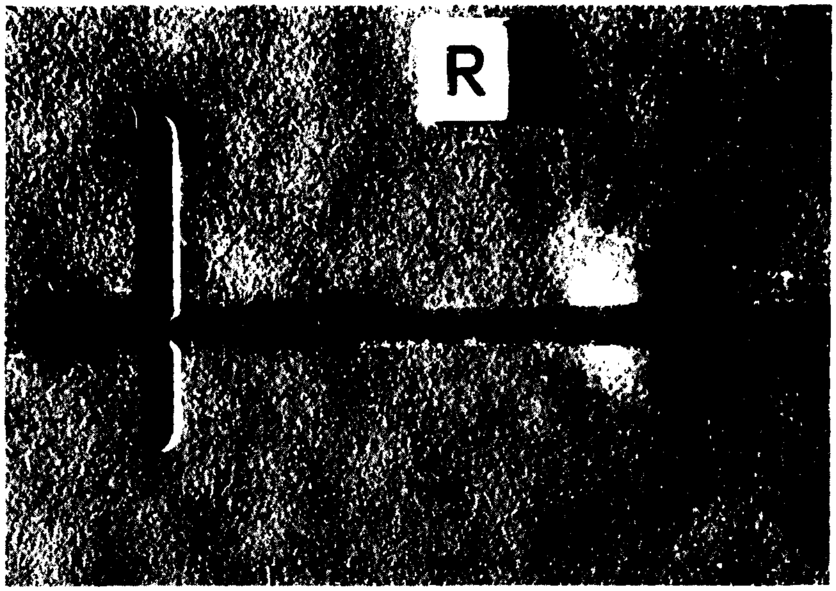


upper scribe

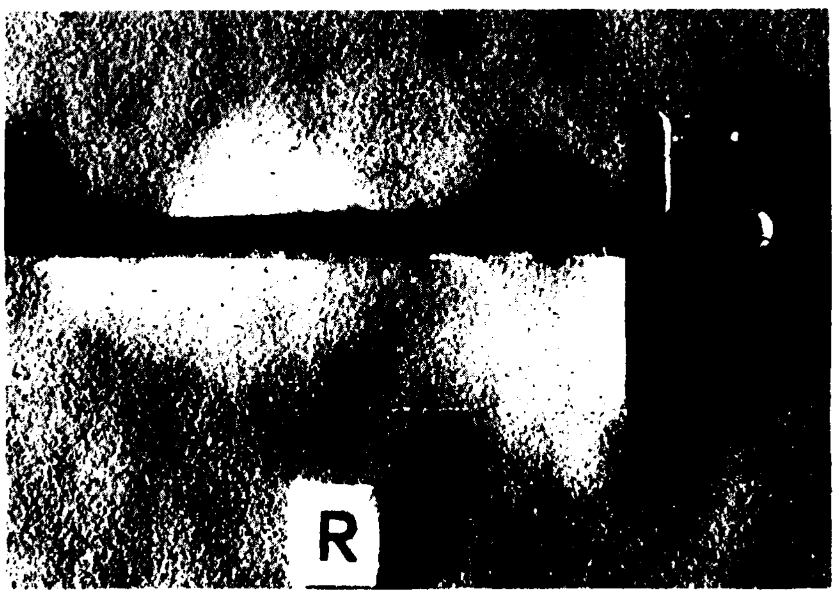


lower scribe

Figure 11. Exposed panels of System 115

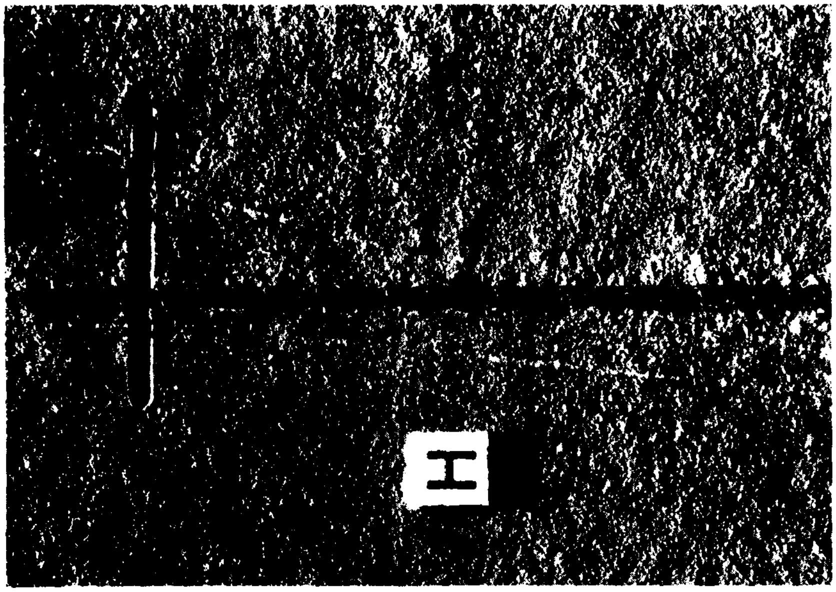


upper scribe



lower scribe

Figure 12. Exposed panels of System 117



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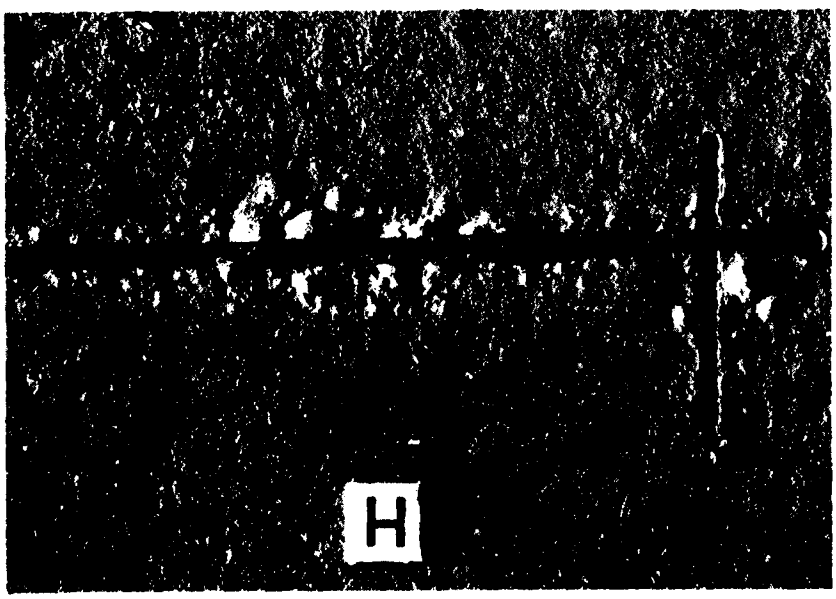
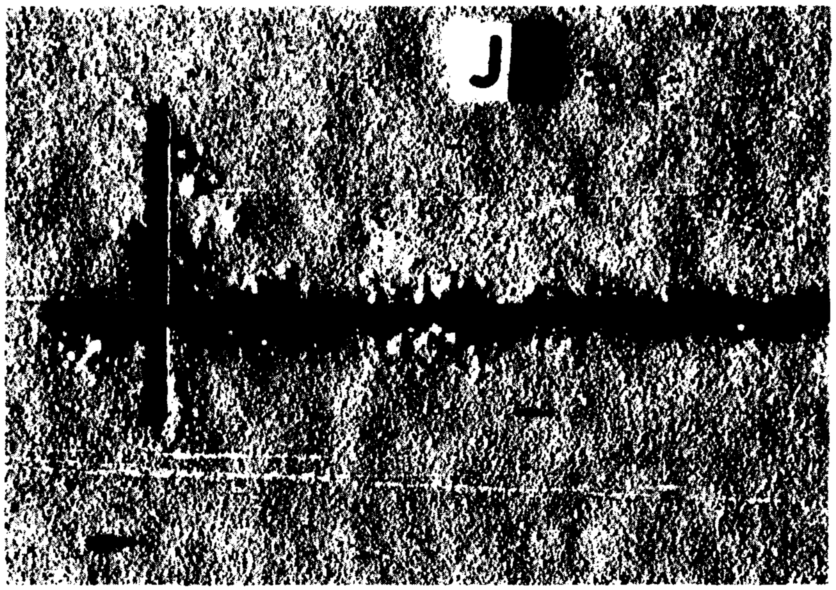


lower scribe

Figure 13. Exposed panels of System 118



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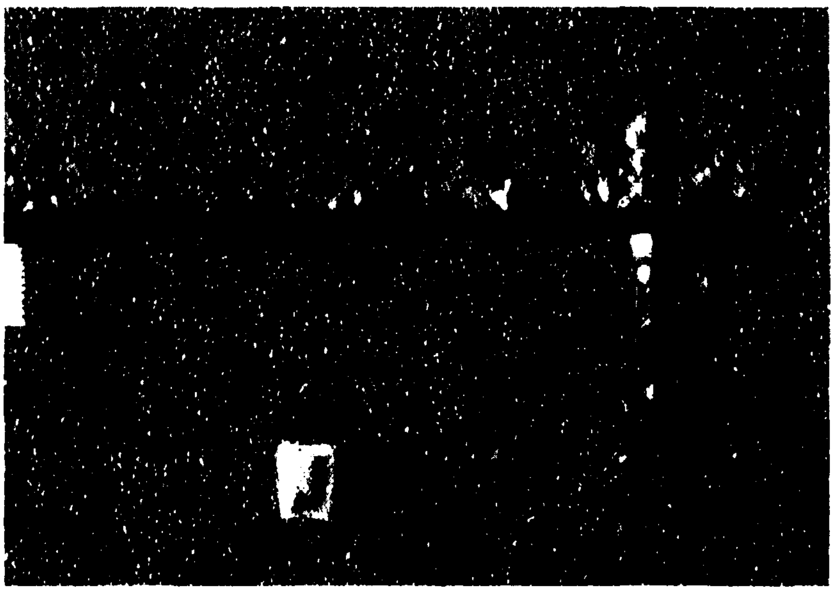
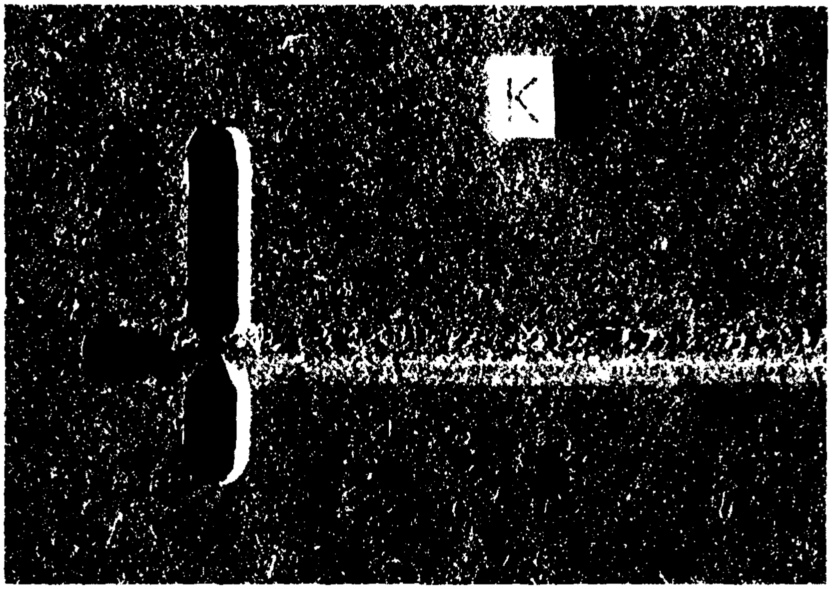
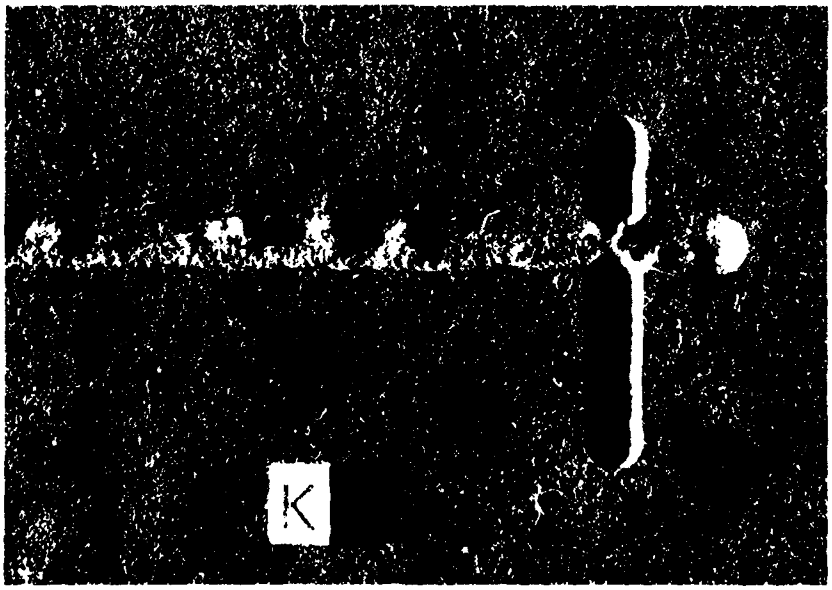


lower scribe

Figure 14. Exposed panels of System 119



upper scribe



lower scribe

Figure 15. Exposed panels of System 120

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Security Classification

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	Naval Facilities Engineering Command

13. ABSTRACT

With a new set of test conditions in an accelerated weathering machine, it was possible to determine protective properties of coatings under conditions of cycling light, heat, and humidity and in a salty environment. This salt-dew-cycle test combines the environment of the dew-cycle Weather-Ometer and of the underrust test. The scribed panels are periodically dipped in salt water, and increased rusting, blistering, and undercutting at the scribe is thereby obtained.

Unclassified
Security Classification

14. KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Protective coatings						
Coatings						
Protection						
Physical properties						
Weathering						
Corrosion environments						
Marine atmospheres						
Light (visible radiation)						
Artificial weathering tests						
Sea water corrosion						
Atmospheric corrosion tests						
Heat						
Humidity						
Rusting						
Blistering						
Corrosion						

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