Rock Island Arsenal Laboratory

TECHNICAL REPORT

EFFECT OF CURE CONDITIONS ON WEAR LIFE AND CORROSION PROTECTION OF A RESIN-BONDED SOLID FILM LUBRICANT

By

F. S. Meade

G. P. Murphy

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G. P. Murphy

Approved by:

A. C. HANSON
Laboratory Director

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Rock Island Arsenal
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ABSTRACT

A resin-bonded solid film lubricant was applied to grit blasted steel, zinc phosphatized steel, and preheated zinc phosphatized steel. The coating was then cured at temperatures from 200 to 500°F for times ranging from 10 to 180 minutes. The effect of these cure conditions on wear life and corrosion protection were determined.

The following information was obtained from this investigation: (1) Grit blasted steel is inferior to the other two substrates. (2) At cure temperatures above 300°F, the resin-bonded solid film lubricant does not prevent the loss of water of hydration from the zinc phosphate coating and the resultant loss in corrosion protection. (3) No one set of cure conditions gives optimum wear life and corrosion protection. (4) Wear life increase with increasing cure time and temperature. (5) Corrosion protection increases with decreasing cure time and temperature. (6) Cure conditions depend on the application for which the resin-bonded solid film lubricant is to be used.

RECOMMENDATIONS

For zinc phosphatized steel, the following cure conditions should be used: (1) If maximum wear life is the most important factor, cure for 30 minutes at 400°F. (2) If maximum corrosion protection is the most important factor, cure for 60 minutes at 325°F. (3) For applications requiring simultaneously good wear life and good corrosion protection, cure for two hours at 325°F. This recommendation is made with the understanding that both the wear life and corrosion protection obtained will be less than that obtained under the conditions stated in (1) and (2) above.

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OBJECT

To determine the optimum cure conditions from the point of view of both wear life and corrosion protection when a resin-bonded solid film lubricant (RIA Compound 9A) is applied over zinc phosphatized steel.

To determine the effect of RIA Compound 9A on the loss of water of hydration from a zinc phosphate substrate.

INTRODUCTION

Studies made at Rock Island Arsenal[1,2,3,4] have shown that zinc phosphatized steel is heated, the coating loses its water of hydration. When zinc phosphatized steel is heated in air, the following loss of water of hydration occurs: 200°F, 50%; 300°F, 80%; 400°F, 95%; 500°F, 95+%. It has also been shown that as the temperature of a zinc phosphatized coating is raised above 300°F, the corrosion protection provided by the coating is reduced.

It has been customary to cure resin-bonded solid film lubricants such that maximum wear life was obtained. Recently, it has become apparent that such coatings can be so formulated so as to provide simultaneous corrosion protection. In order to improve this corrosion protection, additional information concerning curing time and temperatures became desirable in view of the fact that the effect of heating phosphatized coatings was known.

PROCEDURE

The type of test specimens used in this study were as follows: (1) Falex pins and V-blocks made from AISI 3135 steel and AISI 1137 steel respectively, and (2) panels 2 x 3 inches made from AISI 1020 steel. The specimens were treated as follows: One batch was grit blasted to a surface finish of 145-155 microinches rms. The remaining specimens were grit blasted to the above surface finish and then zinc phosphatized according to Specification MIL-P-16232B, Type 2, Class 3[5]. Part of the above zinc phosphatized specimens were preheated before application of the resin-bonded solid film lubricant. After the solid film lubricant had been applied the coating was cured for various times and temperatures. These cure conditions were as follows: 3 hours at 200°F; 2 hours at 300°F; 1 hour at 400°F, and 1/2 hour at 500°F.

The same time and temperatures used to cure the solid lubricant film were used to preheat the zinc phosphatized test specimens. For example, if the RIA Compound 9A coating was to be cured for one hour at 400°F, the zinc phosphatized specimens were also heated one hour at 400°F prior to the application of the solid film lubricant.

The solid film lubricant formulation used was slightly different from the RIA PD-651[6] formulation. The formulation was adjusted so as to provide a corrosion protection period of 172 hours and a wear life of 520 minutes. This contrasts with the usual 72 hour corrosion protection period and 580 minutes wear life. These figures, of course, are for nonpreheated zinc phosphatized steel with a one hour at 400°F cure.

The modified formulation was used as it was thought that the longer corrosion protection provided by this formulation would aid in distinguishing the effect of cure conditions and loss of water of hydration on the solid film coating.

RESULTS AND DISCUSSION

(A) 20% Salt Spray Corrosion Tests

With the exception of the tests on grit blasted steel, the salt spray tests were run in triplicate. Only one test was run on grit blasted steel because the failure time was less than average deviation of twenty-four hours for the salt spray test. For each of the three salt spray tests, the sets of three panels were placed in different positions in the salt spray cabinet. This was done in order to eliminate as much as possible any variation in results due to cabinet position.

The salt spray cabinet was operated at a temperature of 95°F. The air pressure was adjusted so as to provide a solution collection rate of 1.0 to 1.4 ml. per hour. Failure time was taken as the number of hours necessary for three rust dots to appear on at least two of the three test panels.

The results of the salt spray test are given in Table I. For each cure condition and substrate, the results for each test and the average results are given.

The results for the grit blasted substrate showed that the cure conditions had no effect on the test results.

In order to determine the effect of the cure conditions and substrate on the salt spray life as given in Table I, an analysis of variance was run on the data. Since the salt spray life for the grit blasted steel was significantly less than for the two other substrates, this data was not used in the analysis. The analysis of variance table is given in Table II.

TABLE I

20% SALT SPRAY TEST
(Hours to Failure)

	CURE CONDITIONS			
SUBSTRATE	3 HOURS @200°F	2 HOURS @300°F	1 HOUR @400°F	1/2 HOUR @500°F
Grit blasted steel	3	5	5	4
Average	3	5	5	4
Preheated zinc phosphatized steel	70	221	70	70
	66	228	156	66
	72	310	96	48
Average	69	253	107	61
Zinc phosphatized steel	121	245	169	97
	132	276	156	66
	96	310	192	72
Average	116	277	172	78

TABLE II

ANALYSIS OF VARIANCE OF SALT SPRAY RESULTS

SOURCE	SUM OF SQUARES	DEGREES OF FREEDOM	MEAN SQUARE
Substrate	8,778.38	1	8,778.38
Cure Condition	136,554.13	3	45,518.04
Interaction	2,170.12	3	723.37
Subtotal	147,502.63	7	
Within Groups	23,072.00	16	1,442.00
Total	160,574.63	23	

The F ratio for interaction, that is, the mean square for interaction divided by the mean square for within groups, is 0.5. The F table[7] for 3 and 16 degrees of freedom at a 95% confidence level is 3.24. Since the value 0.5 is less than 3.24, we can say that the interaction is not significant. Since the interaction is not significant, we may pool the two estimates of variance, namely interaction and within group to form a new analysis of variance table. This table is given in Table III.

From the mean square values in Table III, the "F" ratio was calculated for substrate and cure conditions. These values were 6.62 and 34.40 respectively. Their "F" table values at 95% confidence level for their respective degrees of freedom were 4.38 and 3.13 respectively. Therefore, since the "F" ratio for both substrate and cure conditions are greater than their respective "F" table value, it may be concluded that both substrate and cure conditions have a significant effect on the salt spray life of resin bonded solid film lubricants.

In order to further study the substrate effect, Student's "t" tests were run comparing the salt spray wear life for preheated and nonpreheated panels. The "t" value and the corresponding "t" table value are given in Table IV for each temperature level.

TABLE III

ANALYSIS OF VARIANCE AFTER POOLING ESTIMATES OF VARIANCE

SOURCE	SUM OF SQUARES	DEGREES OF FREEDOM	MEAN SQUARE
Substrate	8,778.38	1	8,778.38
Cure Condition	136,554.13	3	45,518.04
Residual	25,424.12	19	1,328.53
Total	170,574.63	23	

TABLE IV

STUDENT "t" VALUES FOR EFFECT OF PREHEATING ZINC PHOSPHATIZED PANELS ON SALT SPRAY LIFE OF RESIN BONDED SOLID FILM LUBRICANTS

TEMPERATURE (°F)	CALCULATED "t" VALUE	TABLE "t" VALUE[7]
200	4.24	2.78
300	.70	2.78
400	.50	2.78
500	1.50	2.78

This data shows that preheating has a significant effect only at the 200°F cure temperature level. Apparently at 200°F cure temperature the solid film coating does prevent loss of water of hydration, thus increasing the corrosion protection of the zinc phosphate coating in comparison to the corrosion protection given by the preheated zinc phosphate coating. This may account for the longer corrosion protection for the nonpreheated panels coated with solid film lubricant as given in Table I. At all other temperature levels tested the solid film lubricant coating does not prevent loss of water of hydration. Thus any corrosion protection the zinc phosphate coating itself would afford would be greatly reduced for cure temperature above 300°F. However, this doesn't mean that the overall corrosion protection afforded by the solid film lubricant applied to zinc phosphatized steel is necessarily decreased by these higher cure temperatures.

To determine the effect of cure conditions on salt spray corrosion protection for RIA 9A applied to zinc phosphatized steel, Student's "t" tests were run comparing the various cure conditions. These "t" values and their corresponding "t" table values are given in Table V.

TABLE V

EFFECT OF CURE TEMPERATURE ON SALT SPRAY LIFE

TEMPERATURE (°F)	CALCULATED "t" VALUE	TABLE "t" VALUE[7]
300 vs. 200	7.44	2.78
300 vs. 400	4.80	2.78
300 vs. 500	9.48	2.78
400 vs. 200	3.82	2.78
400 vs. 500	6.86	2.78

As seen in this table, all cure temperature have a significant effect on the salt spray life of the resin bonded solid film lubricant. Combining this fact with the data in Table I, it appears that 300°F for 2 hours is the best cure temperature with 1 hour at 400°F next best. Both 200°F and 500°F temperature are inferior to the above two.

The poor salts spray life for the 500°F cure is probably due to degradation of the resin at the 500°F cure temperature. In the case of the 200°F cure, it's low salt spray life is probably due to the fact that the crosslinkage between the epoxy and phenolic resin is not nearly as complete as for the 300 and 400°F cure. Neville and Lee[8] recommend a 300°F to 400°F cure for the epoxy phenolic system.

(B) Falex Wear Life

Falex wear tests were run for the various substrates and cure conditions. The test procedure used was that given in Specification MIL-L-22273(WEP)[9], with the exception, of course, that various substrates were used instead of the specified manganese phosphate. The results of the test are given in Table VI. Four tests were run for each substrate and cure condition; the average of the 4 tests being considered as the wear life. Both the individual test values and the average value are given in this table. It will be noted that 8 tests were run for the 3 hours at 200°F cure on zinc phosphatized steel. This was due to the wide variation in individual test results for the first four runs.

Since there was such a wide deviation in the test results for this 3 hours at 200°F cure condition, the 200°F cure condition was omitted from the following analysis of variance because the variances were not all from the same population and the analysis of variance would be meaningless.

The analysis of variance of the wear life data, neglecting that for the 200°F 3 hour cure, is given in Table VII.

The "F" ratio for interaction is 12.4. This value is significantly greater than the "F" table value of 2.73 for 4 and 27 degrees of freedom and a 95% confidence level. Therefore, there is significant interaction. Examination of the data in Table VI shows that the interaction is probably due to the substrate effect. The wear life for grit blasted steel shows a sharp increase between the 400 and 500°F cures. This increase is not evident for the other two substrates. The table also shows that wear life of dry lubricants applied to grit blasted steel is significantly less than for the other substrates.

TABLE VI

FALEX WEAR LIFE
(Minutes)

SUBSTRATE	CURE CONDITIONS: 3 HOURS @200°F	2 HOURS @300°F	1 HOUR @400°F	1/2 HOUR @500°F
Grit blasted steel	0	0	7	130
	0	0	3	175
	0	0	17	225
	0	0	49	171
Average	0	0	19	175
Preheated zinc phosphatized steel	109	103	542	494
	49	120	420	479
	140	130	511	400
	202	130	460	385
Average	125	121	483	440
Zinc phos-phatized steel	9 477	290	571	530
	223 65	319	414	500
	299 6	208	536	409
	3 4	180	547	360
Average	91	250	517	450

TABLE VII

ANALYSIS OF VARIANCE OF WEAR LIFE

SOURCE	SUM OF SQUARES	DEGREES OF FREEDOM	MEAN SQUARE
Substrate	22,530	2	11,265
Cure Condition	10,815	2	5,407
Interaction	3,913	4	978
Subtotal	37,258	8	
Within Group	2,128	27	79
Total	39,386	36	

If a Student's "t" test is run comparing the wear life on grit blasted steel for the 400°F and 500°F cures, a value of 7.43 is obtained. This value is significantly greater than the table value of 2.45. Therefore, for the grit blasted steel substrate, the wear life for the 500°F cure is significantly greater than the wear life for any other cure temperature.

Since the grit blasted steel caused interaction and since we have treated it's results separately, an analysis of variance was run for the remaining two substrates and 3 cure temperatures. These results are given in Table VIII.

The "F" value for interaction is 2.34; when compared to the "F" table value of 3.55 for 2 and 18 degrees of freedom at the 95% confidence level, it is evident that there is no significant interaction.

The estimates of variance, interaction and within group, were pooled and the "F" values for substrate and cure condition were recalculated. These "F" values were 4.15 and 40.6, respectively. These corresponding "F" table values at 95% confidence level for the appropriate degrees of freedom were 4.35 and 3.49, respectively.

TABLE VIII

REVISED ANALYSIS OF VARIANCE OF WEAR LIFE

SOURCE	SUM OF SQUARES	DEGREES OF FREEDOM	MEAN SQUARE
Substrate	620	1	620
Cure Condition	12,143	2	6,071
Interaction	514	2	257
Subtotal	13,277	5	
Within Group	1,977	18	110
Total	15,254	23	

The substrate had no effect on wear life for the 300°, 400°, and 500°F cure conditions, again showing that at these temperatures loss of water of hydration from the phosphate coating was probably equivalent to that of the preheated phosphate coating. Due to the wide variation in test results at the 200°F cure temperature, no definite statement can be made as to the preheating effect.

The cure temperature over the 300°F to 500°F range, however, does significantly effect the wear life of RIA Compound 9A.

Student's "t" was run for the wear life results on zinc phosphatized steel for the various temperatures. The "t" values are given in Table IX. The table shows both 400°F and 500°F cures are significantly different from the 300°F cure. Also, there is no advantage of curing beyond 400°F. Since data in Table VI shows the wear life for the 400°F cure is double than that for the 300°F cure, the 400°F cure would be the best for long wear life.

Although the loss of water of hydration may have a deleterious effect on wear life, it is overcome by using the 400°F cure, as the wear life for this cure temperature is much greater than it is for the lower cure temperatures necessary for little loss of water of hydration.

TABLE IX

EFFECT OF CURE CONDITIONS ON WEAR LIFE OF RIA COMPOUND 9A APPLIED TO ZINC PHOSPHATIZED STEEL

TEMPERATURE (°F)	CALCULATED "t" VALUE	TABLE "t" VALUE[7]
300 vs. 400	4.90	2.45
300 vs. 500	3.50	2.45
400 vs. 500	1.43	2.45

Comparing the results for the salt spray and wear life tests, it is readily apparent that the same cure conditions do not give optimum corrosion protection and at the same time optimum wear life. Therefore, it was necessary to investigate the possibility that another set of cure conditions could be used to obtain a suitable compromise between the two.

A partial factorial design experiment was set up using the 300°F to 400°F cure temperature range and cure times of 10 to 120 minutes. The results for both salt spray and wear life tests are given in Tables X and XI.

An examination of the two tables shows that the trend is for the salt spray life to increase with decreasing cure temperature and also with decreasing cure time. While the wear life,on the other hand,is exactly opposite, increasing with increasing cure temperature and time.

A few additional tests were run using these trends in the hope that a compromise might be obtained between the trends. The cure time at 400°F was reduced to 30 minutes and the following results were obtained: 525 minutes wear life and 192 hours salt spray life. This was not significantly different than the 60 minute cure.

Next, the cure temperature was decreased in the hope that a too drastic decrease in wear would not occur. Tests at 325°F and 120 minutes gave a salt spray life of 277 hours and a wear life of 336 minutes. This is a 60% increase in corrosion protection over the 400°F and 60 minute cure figure, however, it is a 55% decrease in wear life. Test results at

TABLE X

HOURS OF SALT SPRAY LIFE

TIME (Minutes)	TEMPERATURE (°F) 300	325	350	375	400
10			316		239
30		412		192	
45	240		192		
60		388			172
120	277			144	

TABLE XI

MINUTES OF WEAR LIFE

TIME (Minutes)	TEMPERATURE (°F) 300	325	350	375	400
10			152		120
30		50		41	
45	8		45		
60		154			517
120	250			491	

350°F for two hours gave 186 hours salt spray life and an estimated 410 minute wear life. There was no significant increase in corrosion protection and a 20% decrease in wear life.

Tests run on RIA PD-651 material cured at 325°F for two hours showed a salt spray life of 188 hours and a wear life of 355 minutes. This is an increase of 160% in corrosion protection and a decrease of 40% in wear life when compared to the results for the 400°F for one hour cure.

Although the results for the 325°F for two hours cure for RIA PD-651 materials meet the corrosion protection and wear life requirements given in the purchase description, it is probably best to select cure conditions based on the application for which the solid film lubricant is to be used.

If long wear life and some corrosion protection is desired, a cure of 400°F for 30 minutes would be best. If a long period of corrosion protection with some lubrication is desired, 325°F for one hour is probably the best.

The 325°F for two hours cure would be best if relatively long wear life and corrosion protection are required.

In conclusion, the following points are emphasized:

1. Above 300°F resin bonded solid film lubricants do not prevent the loss of water of hydration for the phosphate coating.

2. There is no one set of optimum cure conditions.

3. Corrosion protection provided by the solid film lubricant increases with decrease cure time and temperature.

4. Wear life of solid film lubricants increases with increasing cure time and temperature.

5. The best cure conditions to use will depend on the application to which the solid film lubricant is to be utilized.

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58-1134	5-14-58	Evaluation of Bonded Dry Lubricants
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U. S. ARMY
ROCK ISLAND ARSENAL
ROCK ISLAND, ILLINOIS

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9 May 1963

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1. As indicated on the inside of the cover of the inclosed Rock Island Arsenal Laboratory Report 63-959, this report has been sent to the Office of Technical Services. It is, therefore, desired that this report not be listed in the buff section of TAB.

FOR THE COMMANDER:

Arthur C. Hanson

1 Incl
as (20)

A. C. HANSON
Laboratory Director