

AD-754 568

SOLID LUBRICANT COATINGS CURABLE AT
225 F-300 F

George Murphy, Jr.

Army Weapons Command
Rock Island, Illinois

July 1972

DISTRIBUTED BY:

NTIS

National Technical Information Service
U. S. DEPARTMENT OF COMMERCE
5285 Port Royal Road, Springfield Va. 22151

AD754568

AD

SWERR-TR-72-48

**SOLID LUBRICANT COATINGS
CURABLE AT 225°F-300°F**



TECHNICAL REPORT

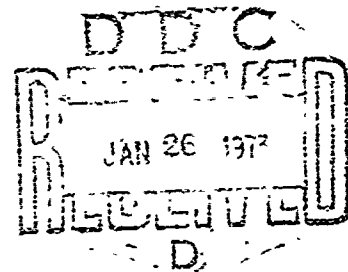
George Murphy Jr.

July 1972

Reproduced by
NATIONAL TECHNICAL
INFORMATION SERVICE

U.S. Department of Commerce
Springfield VA 22151

RESEARCH DIRECTORATE



WEAPONS LABORATORY AT ROCK ISLAND

RESEARCH, DEVELOPMENT AND ENGINEERING DIRECTORATE

U. S. ARMY WEAPONS COMMAND

DISPOSITION INSTRUCTIONS:

A Destroy this report when it is no longer needed. Do not return it to the originator.

DISCLAIMER:

The findings of this report are not to be construed as an official Department of the Army position unless so designated by other authorized documents.

AD

RESEARCH DIRECTORATE
WEAPONS LABORATORY AT ROCK ISLAND
RESEARCH, DEVELOPMENT AND ENGINEERING DIRECTORATE

U S. ARMY WEAPONS COMMAND

TECHNICAL REPORT

SWERR-TR-72-48

SOLID LUBRICANT COATINGS
CURABLE AT 225°F-300°F

George Murphy Jr

July 1972

DA 16062105A107

AMS Code 502E 11 80100 01

Approved for public release, distribution unlimited.

ABSTRACT

Experimental solid-film lubricant coatings based on urea-formaldehyde, epoxy-polyamide, epoxy-silane, alkyd-urea, melamine-acrylic, and epoxy-urea resins were formulated by personnel of the Research Directorate, Weapons Laboratory at Rock Island. None of these coatings when cured at temperatures of 225-300°F had antiwear or corrosion preventing properties comparable to the fully cured MIL-L-46010A type of solid lubricant coating. Of the experimental formulations tested, those based on the urea-formaldehyde and epoxy-silane resins gave the best results. However, with the addition of a curing agent, boron trifluoride monoethylamine complex, to a qualified MIL-L-46010A product, the product could be cured at 275°F, and all test requirements of Military Specification MIL-L-46010A could be satisfied.

FOREWORD

This investigation was carried out under DA Project 1G062105A107, AMS Code 502E.11.80100.01, "Lubricants, Friction and Wear." The unit under which the study was made is entitled "Wear and Corrosion Preventive Resin-Bonded Solid Film Lubricant Curable at 225°F."

CONTENTS

	<u>Page</u>
Title Page	i
Abstract	ii
Foreword	iii
Table of Contents	iv
Objective	1
Background	1
Approach	2
Procedure	2
Results and Discussion	4
Conclusions	12
Recommendations	12
Literature Cited	13
Distribution	14
DD Form 1473 (Document Control Data - R&D)	17

Unclassified

Security Classification

DOCUMENT CONTROL DATA - R & D		
<i>(Security classification of title, body of abstract and indexing annotation must be entered when the overall report is classified)</i>		
1. ORIGINATING ACTIVITY (Corporate author) U. S. Army Weapons Command Research, Dev. and Eng. Directorate Rock Island, Illinois 61201		2a. REPORT SECURITY CLASSIFICATION Unclassified
		2b. GROUP
3. REPORT TITLE SGLID LUBRICANT COATINGS CURABLE AT 225°F-300°F		
4. DESCRIPTIVE NOTES (Type of report and inclusive dates)		
5. AUTHOR(S) (First name, middle initial, last name) George Murphy, Jr.		
6. REPORT DATE July 1972	7a. TOTAL NO. OF PAGES 18	7b. NO. OF REFS 8
8a. CONTRACT OR GRANT NO.	8b. ORIGINATOR'S REPORT NUMBER(S) SWERR-Tr-72-48	
d. PROJECT NO DA 1G062105A107		
c. AMS Code 502E.11.80100.01	9a. OTHER REPORT NUMBER(S) (Any other numbers that may be assigned this report)	
10. DISTRIBUTION STATEMENT Approved for public release, distribution unlimited.		
11. SUPPLEMENTARY NOTES	12. SPONSORING MILITARY ACTIVITY U. S. Army Weapons Command	
13. ABSTRACT Experimental solid-film lubricant coatings based on urea-formaldehyde, epoxy-polyamide, epoxy-silane, alkyd-urea, melamine-acrylic, and epoxy-urea resins were formulated by personnel of the Research Directorate, Weapons Laboratory at Rock Island. None of these coatings when cured at temperatures of 225-300°F had antiwear or corrosion preventing properties comparable to the fully cured MIL-L-46010A type or solid lubricant coating. Of the experimental formulations tested, those based on the urea-formaldehyde and epoxy-silane resins gave the best results. However, with the addition of a curing agent, boron trifluoride monoethylamine complex, to a qualified MIL-L-46010A product, the product could be cured at 275°F, and all test requirements of Military Specification MIL-L-46010A could be satisfied. (U) (Murphy, George Jr.)		

DD FORM 1473
1 NOV 65

REPLACES DD FORM 1473, 1 JAN 64, WHICH IS OBSOLETE FOR ARMY USE.

Unclassified

Security Classification

1a

14 KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
1. Lubrication						
2. Corrosion Protection						
3. Solid Film Lubricants						
4. Resins						
5. Cure Temperature						
6. Curing Agents						

it

OBJECTIVE

The objective of this study was to develop a solid-film lubricant coating with properties equivalent to those of the MIL-L-46010A type of solid lubricant coating, but curable at 225°F to 300°F.

BACKGROUND

Solid-film lubricants are currently used extensively on weapons such as the M16A1 rifle. From the point of view of wear prevention, corrosion protection, adhesion, and fluid resistance, the MIL-L-46010A¹ type of solid-film lubricant coating is adequate for many weapons applications. However, this coating has the disadvantage that its recommended cure temperature of 400°F causes deterioration of some of the alloys used in weapons such as the M16A1 rifle. For example, at temperatures above 300°F, the yield strength, tensile strength, elongation and impact resistance of 7075 and 6061 aluminum alloys are adversely affected.^{2,3,4}

Because of this detrimental temperature effect, the fully cured MIL-L-46010A coating cannot be used on these aluminum alloy parts. The solid-film lubricant coatings currently used on aluminum parts are either commercially available proprietary materials which are cured at 250°F - 300°F or the MIL-L-46010A coating cured at 300°F. None of these are as effective as the fully cured MIL-L-46010A coating.⁵ Therefore, a need exists for a solid-film lubricant curable at 225°F-300°F that has the properties of the fully cured MIL-L-46010A type coating.

The determining factor at which temperature a bonded solid-film lubricant coating can be cured is the temperature at which its resin binder is fully cured. A survey of the literature indicates which types of resins are fully cured at a temperature of 225°F-300°F. The literature shows that the following types of organic resin systems are feasible: (1) urea-formaldehyde, (2) epoxy-polyamide, (3) epoxy-silane, (4) alkyd-urea, (5) melamine-acrylic, and (6) epoxy-urea.

Payne⁶ states that the urea-formaldehyde-based finishes are normally baked at 250°F-300°F. However, acid catalyzed urea-formaldehyde-based finishes have been cured at 140°F-160°F. Epoxy-polyamide is basically an air-cure system in which a reaction between the amino group in the

polyamide and the epoxy resin forms a cross-linked copolymer. The epoxy-silane resin is a commercial experimental material curable at 250°F which is reported to give good corrosion protection. This resin was developed as a primer coating. Alkyd-urea resins are cured in the same temperature range as the urea-formaldehyde resins (250°F-300°F) and have similar properties; however, they are not as brittle as the urea-formaldehyde coatings. The melamine-acrylic resins are cured at 250°F to give a hard, resistant film. The urea-formaldehyde resins, which are amino in characteristics can also cross-link with epoxy resins when cured at 300°F-325°F.

APPROACH

Formulation Work

Based on information on properties of resin systems gained from the literature, a number of formulations were made and were compared with existing solid-film lubricants. The initial formulations that were considered best were varied in composition and cure conditions to find the optimum combination.

Modification of Existing Formulation

A curing agent, boron trifluoride monoethylamine complex, was added to an existing MIL-L-46010A formulation to lower the curing temperature of the solid-film lubricant. The modified coating was tested against the specification requirement of Military Specification MIL-L-46010A.

PROCEDURE

Formulation Work

Formulations that contained approximately 27 to 30 per cent by weight resin and 70 to 73 per cent by weight pigment system were prepared. Compositions of these formulations are given in Table I. These formulations were tested for storage stability by periodic examination for evidence of gelation or livering. The lubricant formulations were also subjected to the wear life test of MIL-L-45010A specification. In this test, the coating is applied to zinc phosphatized test specimens, the coated specimens are cured for 2 hours at 225°F, and then the endurance life of the coatings is determined on the Falex Wear Tester in accordance with Method 3807.1 of Federal Test Method Standard 791B.⁷

TABLE I
INITIAL SOLID LUBRICANT FORMULATIONS

Component Resin (Type)	Formulation (% by weight)									
	MA-1	FA-1	FA-2	EU-1	EP-1	EP-2	EP-3	U-1	ES-1	
Acrysol WS-24 (Acrylic)	24.0									
Uformite MM83 (Melamine)	5.0									
Duraplex A-29 (Alkyd)	21.0	21.0								
Uformite F200E (Urea)	9.0	9.0		9.0						
Epon 1007 (Epoxy)				21.0						
EKR 2002 (Epoxy)					18.0	16.0	18.0			
Marsamid 115 (Polyamide)					9.0	9.0	5.0			
Plaskon 3300 (Urea)								27.0		
XZ-8-5059 (Epoxy-Silane)									30.0	
Percent Resin	29.0	30.0	30.0	30.0	27.0	25.0	23.0	27.0	30.0	
Pigment										
Molybdenum Disulfide	38.0	37.0	37.0	37.0	39.0	40.0	41.0	38.0	37.0	
Antimony Trioxide	24.0	24.0	24.0	24.0	25.0	26.0	26.0	25.0	24.0	
Dibasic Lead Phosphite	9.0	9.0	8.5	8.7	9.0	9.0	10.0	9.0	9.0	
Percent Pigment	71.0	70.0	69.5	69.7	73.0	75.0	77.0	72.0	70.0	
Catalyst										
Phosphoric Acid								1.0		
Paratoluenesulphonic Acid			0.5	0.3						

When the formulations having the best storage stability and wear life were determined, variations of these materials were made to find the optimum combination of resin, pigment, catalyst, and cure conditions that would give the longest wear life.

Modification of Existing Formulation

A concentration of 0.6 per cent by weight boron trifluoride monoethylamine complex was added to a qualified MIL-L-46010A product. The formulation was applied to test specimens and cured under several conditions of time and temperature; it was then tested for solvent resistance to dioxane and wear life according to the procedures of specification MIL-L-46010A. When the optimum cure conditions were determined, the BFMEA modified MIL-L-46010A coating was subjected to all MIL-L-46010A specification tests.

RESULTS AND DISCUSSION

Formulation Work

The results of the Falex wear tests on the initial experimental formulations and some commercial formulations after a 2-hour cure at 225°F are given in Table II. The data show that only one formulation, ES-1, was comparable in wear life to the commercial formulations identified as 626, 99A, and RIAPD 703. However, all formulations were comparable to, or better than, the MIL-L-46010A coating in wear life when cured at 225°F instead of at the normal 400°F cure. The data also show that the wear life of none of the experimental lubricants was comparable to the 500 minute wear life obtainable for the fully cured MIL-L-46010A coating.

In addition to wear life, the gelation time of the experimental formulations was also determined. The number of months required for gelation of the formulation to occur is given in Table III. When gelation occurs, the formulation is no longer useful. If this gelation occurs in less than 6 months, the formulation is considered to have poor storage stability. The data in Table III show that, for the experimental formulations, only those based on the urea-formaldehyde and epoxy-silane binder had sufficient storage stability to be considered for further formulation work.

TABLE II
SOLID LUBRICANT TEST DATA,
INITIAL FORMULATIONS (CURED 2 HOURS AT 225°F)

<u>Solid Lubricant Formulation</u>	<u>Falex Wear Life (Minutes)</u>
MA-1	17
FA-1	3
FA-2	21
EP-1	13
EP-2	12
EP-3	22
EU-1	91
U-1	19
ES-1	117
MIL-L-46010	15
MIL-L-46147	160
626	92
99A	148

TABLE III
 GELATION TIME OF INITIAL FORMULATIONS

<u>Solid Lubricant Formulation</u>	<u>Gelation Time (Months)</u>
MA-1	2.0
FA-1	4.0
FA-2	4.0
EP-1	0.5
EP-2	0.5
EP-3	0.5
EU-1	3.0
U-1	14.0 ⁺
ES-1	14.0 ⁺
MIL-L-46010	6.0 ⁺
MIL-L-46147	8.0 ⁺
626	6.0 ⁺
99A	6.0 ⁺

The composition of additional formulations used in the optimization study is given in Table IV, and the wear life of these formulations when tested under various cure conditions is given in Table V.

Of the epoxy-silane formulations, ES-2 is optimum. This formulation, however, was only slightly better than ES-1, which is essentially the same as ES-2 except that it contained no catalyst. Increasing the cure temperature over the range 225°F-300°F had no effect on the wear life of the epoxy-silane solid lubricant coatings.

For the urea-formaldehyde type of formulations, U-2 is optimum because wear life decreased when the amount of resin was either greater or less than 32 per cent. The data also show that, for the urea-formaldehyde type of coatings, wear life is dependent upon the cure temperature.

Corrosion tests of formulations ES-2 and U-2 showed that both of these formulations protected the zinc-phosphated steel panels from rust for about 70 hours in the salt fog test when these panels were cured for 2 hours at 275°F. Although these formulations do not have properties equivalent to those of the fully cured MIL-L-46010A coating (100 hours minimum protection in the salt fog test), the results are encouraging and show that development of a low cure-temperature coating equivalent to standard MIL-L-46010A may be possible.

Modification of Existing Formulation

The MIL-L-46010A formulation with 0.6 per cent catalyst, boron trifluoride monoethylamine, added was found to be curable to the solvent resistant state in 45 minutes at 275°F. Data on wear life of the modified coating under various cure conditions are shown in Table VI.

These data show that, although the coating is solvent-resistant after a 45-minute cure at 275°F, a 120-minute cure at this temperature is necessary to obtain a Falex wear life by which the MIL-L-46010A wear life requirement is satisfied. Since the 120-minute cure at 275°F was the lowest cure conditions at which the modified MIL-L-46010A coating would meet the Falex wear life requirement, these conditions were used to fully evaluate the modified coating. The results of this evaluation are given in Table VII. These results

TABLE IV
OPTIMIZED SOLID FILM LUBRICANT FORMULATIONS

<u>Component</u>	<u>Formulation (% by weight)</u>						
	<u>ES-1</u>	<u>ES-2</u>	<u>ES-3</u>	<u>U-1</u>	<u>U-2</u>	<u>U-3</u>	<u>U-4</u>
Resin (Type)							
XZ-8-5059 (Epoxy-Silane)	30.0	30.0	35.0	27.0	32.0	35.0	37.0
Plaskon 3300 (Urea)							
Percent Resin	30.0	30.0	35.0	27.0	32.0	35.0	37.0
Pigment							
Molybdenum Disulphide	37.0	37.0	35.0	38.0	36.0	34.0	33.0
Antimony Trioxide	24.0	24.0	22.0	25.0	23.0	22.0	21.0
Dibasic Lead Phosphate	9.0	8.5	8.0	9.0	8.0	8.0	8.0
Percent Pigment	70.0	69.5	65.0	72.0	67.0	64.0	62.0
Catalyst							
Phosphoric Acid		0.5		1.0	1.0	1.0	1.0

TABLE V
 FALEX WEAR LIFE (MINUTES), OPTIMIZED FORMULATIONS

<u>Formulations</u> <u>Cure Conditions</u>	<u>ES-1</u>	<u>ES-2</u>	<u>ES-3</u>	<u>U-1</u>	<u>U-2</u>	<u>U-3</u>	<u>U-4</u>
2 hours at 225°F	117	133	98	19			
2 hours at 275°F	125			267	334	90	55
1 hour at 300°F	120			400			

TABLE VI

EFFECT OF CURE CONDITIONS ON FALEX WEAR LIFE
OF MODIFIED MIL-L-46010A COATING

<u>Cure Time (Minutes)</u>	<u>Cure Temperature (°F)</u>	<u>Falex Wear Life (Minutes)</u>
60	250	22
120	250	15
45	275	15
60	275	55
120	275	800
120	300	738

TABLE VII

SPECIFICATION TEST RESULTS FOR BFMEA MODIFIED MIL-L-46010A SOLID-FILM LUBRICANT

<u>Test</u>	<u>Test Requirement</u>	<u>Test Results</u>
Solids Content	Minimum 40%	40.6
Film Thickness	.0002-.0005 inches	.0005 inches
Wear Life	Minimum 450 minutes	800
Load-Carrying Capacity	Minimum 2000 pounds	3250
Corrosion Protection	Minimum 100 hours	500 ⁺
Film Adhesion	Coating not removed by tape	Pass
<u>Fluid Resistance</u>		
Trichloroethylene	Pass film adhesion	Pass
Carbon Removing Compound	Pass film adhesion	Pass
Standard Hydrocarbon Test Fluid	Pass film adhesion	Pass
Rifle Bore Cleaner	Pass film adhesion	Pass
Aviation Gasoline	Pass film adhesion	Pass
Petroleum Base Hydraulic Fluid	Pass film adhesion	Pass
Jet Fuel	Pass film adhesion	Pass
Aircraft Lubricating Oil	Pass film adhesion	Pass
Nonpetroleum Base Hydraulic Fluid	Pass film adhesion	Pass
Silicate Ester Type Lubricating Oil	Pass film adhesion	Pass
Diester Type Semifluid Lubricating Oil	Pass film adhesion	Pass
Silicone Damping Fluid	Pass film adhesion	Pass
Synthetic Aircraft Turbine Lubricating Oil	Pass film adhesion	Pass
Thermal Stability	Pass film adhesion	Pass
Graphite and Powdered Metals	None	None
Storage Stability (6 months)		
Wear Life	Minimum 450 minutes	608
Corrosion Protection	Minimum 100 hours	300
Workmanship	Completely dispersed by moderate shaking or stirring	Pass

show that the modified coating meets all the specification requirements, including the six months storage stability test. The Falx wear test was also run on the modified coating after 8 months, and a wear life of 20 minutes was obtained. This shows that the catalyst was no longer effective since 20 minutes is the wear life of the unmodified MIL-L-46010A coating when cured for 2 hours at 275°F.

CONCLUSIONS

1. Of the low temperature-cured experimental formulations tested, only those based on urea-formaldehyde or epoxy-silane binder have antiwear, corrosion preventive, and storage stability properties that approach those of the fully cured MIL-L-46010A coating. However none of the materials tested matched the performance of the fully cured MIL-L-46010A coating.

2. The addition of boron trifluoride monoethylamine to the MIL-L-46010A solid-lubricant formulation decreased the cure temperature from 400°F to 275°F without an unfavorable effect on the properties of the coating.

RECOMMENDATION

Since the work with the boron fluoride curing agent was limited to one qualified MIL-L-46010A lubricant, further investigation should be carried out to determine its effect on other qualified MIL-L-46010A lubricants before a final recommendation for the use of the curing agent can be made.

LITERATURE CITED

- 1 Military Specification MIL-L-46010A, "Lubricant Solid Film, Heat Cured, Corrosion Inhibiting," December 1969.
2. Gonser, T. and Goettsch, P., "Mechanical Property Evaluations of 7075 Aluminum Alloy Forgings for the M16A1 Lower Receiver," August 1969, AWC Science and Technology Laboratory Report 69-2305.
- 3 Mantell, C. L., Engineering Materials Handbook, 1958, McGraw-Hill Book Company.
- 4 Alcoa Structural Handbook, 1960, Aluminum Company of America.
- 5 Meade, F. S., "Comparison of Solid Film Lubricants Everlube 626, Electrofilm 66C, and a Specification MIL-L-46010(MR) Product," September 1969, AWC Science and Technology Laboratory Report 69-2434.
6. Payne, H. F., Organic Coatings, 1961, John Wiley and Sons Inc., Vol I.
- 7 "Wear Life of Dry, Solid-Film Lubricants," Method 3807 1, Federal Test Method Standard 791B, January 1969
- 8 Rock Island Arsenal Purchase Description 703, "Lubricant, Solid Film: Air Drying, Corrosion Inhibiting (In Pressurized Containers)," December 1967.