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	REPORT NO. NAEC AML 2454 DATE 16 May 1966	
	THE CORROSION PROTECTION AFFORDED BY VARIOUS COATING SYSTEMS IN AIRCRAFT FASTENER AREAS	1
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AERONAUTICAL MATERIALS LABORATORY

REPORT NO. NAEC AML 2454 DATE 16 May 1966

THE CORROSION PROTECTION AFFORDED BY VARIOUS COATING SYSTEMS IN AIRCRAFT FASTENER AREAS

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ABSTRACT

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Decision

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A stress-cycling test (at -103°F and room temperature) designed to simulate the spectrum of stresses which could occur during 500 aircraft flying hours (roughly 1 PAR interval), indicates that current specification and experimental aircraft coatings cannot meet these conditions without cracking around fastener heads.

The coatings used were the MIL-C-22750 epoxy, the MIL-L-81352(WEP) all-acrylic, and two polyurethanes.

The implication of this test is that, at the present state of the coatings art, paint films of the non-rubbery type cannot be expected to provide adequate corrosion protection by themselves in the fastener areaa persistent trouble area on naval aircraft.

I. INTRODUCTION

A. References (a) and (b) established this problem assignment 'o develop improved corrosion resistant primers for protection of aircraft, with specific emphasis on the protection of aluminum countersinks fitted with cadmium plated steel fasteners.

B. Reference (c) was a report which indicated that aircraft fastener areas (cadmium plated steel fasteners in aluminum countersinks) could be effectively protected against corrosion by treatment with a MIL-S-8802 sealant, Products Research Company's PR1422 (B2). The treatment recommended therein was, for maximum protection, PR1422 (B2) in the countersink prior to fastener installation and a PR1422 (B2) overlay after installation. A somewhat less effective technique was the application of the PR1422 (B2) overlay alone for those aircraft where it is impractical to apply the PR 1422 (B2) in the countersinks.

C. The conclusions of the reference (c) investigation were based on stress-cycling tests of MIL-C-22750 epoxy and polyurethane coated aluminum specimens with and without PR1422 (B2) treatment, followed by salt spray tests and examination of the disassembled stripped specimen components.

D. With the exception of one very elastomeric polyurethane system (two were used), all the specimens exhibited noticeable paint cracking around fasteners, including those specimens which had been treated with PR1422 (B2) prior to painting. The explanation for the corrosion protection qualities of the PR1422 (B2) appears to lie in its ability to remain soft and flexible enough to withstand severe stressing, and impermeable enough to withstand moisture and electrolyte penetration even though the paint system is cracked and flaked.

E. The stress tests employed in reference (c), while severe, were not designed to simulate the actual spectrum of stresses encountered by military aircraft, or to correlate with length of service life. It was of interest, therefore, to know if current specification and experimental aircraft coating systems exhibit fastener head cracking after a reasonable length of service life, e.g., one PAR. If, say, the MIL-C-22750 epoxy system cracked after one simulated PAR whereas the MIL-L-81352 all-acrylic or polyurethane did not, then the use of the latter systems is indicated and further protection methods such as PR1422 (B2) may be superfluous. In this connection, it was noted above that the elastomeric polyurethane did not crack during stressing. Even so, corrosion did occur in the fastener area. However, reference (c) has explained that even though the coating was intact, it was nevertheless broken down by exhibiting severe stretching (beyond its elastic limit) around the fastener heads. The stretched coating was probably highly permeable to moisture and electrolytes.

F. Accordingly, the objective of this investigation was to determine which of the current specification and experimental coating systems is most desirable for aircraft use by virtue of its crack resistance around fastener

heads after one simulated PAR tour of duty. Towards this end, the Aeronautical Structures Laboratory of the Naval Air Engineering Center provided the stress-cycling test parameters for simulating the spectrum of stresses encountered by military aircraft during 500 flying hours, which is approximately equivalent to 15 months of duty or 1 PAR. The coating systems tested to these conditions were MIL-C-22750 epoxy, MIL-L-81352 all-acrylic, and two polyurethanes.

II. EXPERIMENTAL PROCEDURES

A. Specimen Preparation:

(1) The Figure 1 specimens were fabricated from unclad 7075 aluminum alloy. The fasteners were cadmium plated steel, AN 509-10R-12.

(2) The unassembled three sections of the specimens were treated with Iridite 14-2 (including countersinks). The faying surfaces were then shopprimed with two coats of MIL-P-23377 epoxy primer, dried overnight, and the specimens assembled.

(3) Just prior to fastener installation, the countersinks were treated with primers, sealant, or left untreated. When treated, the primers or sealant were wet-applied by stirring rod and the fasteners immediately installed at a torque of 25 in.-lb. The excess primer or sealant was wiped from around the fastener heads with a clean cloth. (These test areas will be utilized in future corrosion work not included in this report.)

(4) The installed fastener heads were treated for $1\frac{1}{2}$ minutes with MIL-C-5410 (1 x 1 water) and then flushed off with water. The heads were then brush treated for about 5 minutes with Iridite 14-2 containing ARP #2 detergent for better wetting.

(5) At this stage, a band of Products Research Company's PR1422 (B2) sealant was applied over the fastener head areas of some of the specimens. The band was about 6-8 mil thick and $\frac{1}{2}$ inch wide for each fastener row and was applied across the entire width of the specimen.

(6) A 1/32 inch thick $1\frac{1}{2}$ inch cross was made at the top and bottom of each specimen with masking tape so that a bare test area could be had after painting. (These test areas will be utilized in future corrosion work not included in this report).

(7) The specimens were then primed and topcoated with the epoxy, acrylic, or polyurethane systems according to the coating schedules of Tables 1 and 2. The taped crosses were now removed exposing an unpainted, Iridited test cross. Note that topcoat formulations are listed in Table 3.

(8) After air drying for 1 week, the specimens were exposed on the roof of the Aeronautical Materials Laboratory from late July 1965 to January 1966. The specimens were removed from exposure for a short period (1 week)

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so that $1\frac{1}{2} \times \frac{1}{2}$ inch unpainted test areas could be machined with a lathe on the top and bottom of the specimens. These areas differed from the above cross area in that the former were machined through the Iridite treatment. (These areas will be utilized in future corrosion work not included in this report).

(9) After exposure, some of the specimens were baked to simulate aerodynamic heating: 90 hours at 260° F, followed by 10 hours at 300° F, followed by 2½ hours at 350° F followed by ½ hour at 400° F. Tables 1 and 2 indicate those specimens which were baked.

(10) A layer of pressure sensitive tape (3M Polyvinyl Fluoride-Y9057) was applied over the frstener heads of one epoxy painted specimen, #218 of Table 1.

B. The Cyclic Loading Test (See Plate 8):

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(1) It has been calculated that if a specimen such as described in Figure 1 were subjected to a pulling force of 11250 lb. along its longitudinal axis and then released to a minimum pull of 1535 lb. so that the specimen is under tension for the complete cycle, then 1000 of such cycles would approximate, on the specimen, the spectrum of stresses which would occur on military aircraft during 500 flight hours (which is roughly equivalent to one PAR cycle or 15 months). It is noted that the 1535 lb. limit represents the minimum stresses to which an aircraft is subjected in flight, corresponding to steady state flying conditions without acceleration or deceleration. The 11250 lb. limit represents a very high stress level corresponding to a 7.33 g maneuver condition which would occur during sharp changes in the aircraft's acceleration or change in direction. Calculation indicates that the actual stress on the narrow width of the specimen when pulled to 11250 lb. is 30,000 lb./in.2 (11250 lb. force + cross sectional area, 0.125 x 3 inches). The actual stress on the fastener head at 11250 lb. is difficult to calculate and must be determined empirically for each fastener, but it is estimated that the stress should be about three times the stress on the specimen width, or 90,000 lb./in.².

(2) Painted stress specimens such as described in Figure 1 and elsewhere in this section were installed vertically in the jaws of a Krouse Fatigue machine which had been programmed to provide the maximum and minimum stresses described above, and also lesser stresses. The ratio of maximum to minimum stress was always 10/1 since this was most practical with the machine. Hence, the max./min. stress for the max 11,250 pull was 11250/1125 instead of the idealized 11250/1535. This decreases the minimum stress level from 4000 lb./in.² to 3000 lb./in.² which is considered a minor error for this test.

(3) Upon installation, some specimens were packed with dry ice by enclosing an insulated metal box about each specimen and filling the box with small dry ice chips. Twenty minutes were permitted for temperature equilibrium prior to the introduction of stresses. After this time, the surface temperature of the specimens was measured by thermocouple to be about -103°F (the dry ice temperature is -109°F.) At this point, the apparatus was adjusted to produce the min. and max. loads and the specimen was cycled. The cycles were read by meter.

Note that not all specimens were stress-cycled at cold temperature conditions-some were cycled at room temperature.

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(4) The temperature conditions, number of cycles, stress configuration, and other test details for each specimen are listed in Tables 1 and 2.

III. RESULTS

The below listed results may be placed in clearer perspective by consideration of the stress cycling test parameters which were employed on the Figure 1 specimens to simulate 500 flying hours on 1 PAR: maximum stress, 30,000 lb./in.²; minimum stress, 3000 lb./in.²; number of cycles, 1000; temperature, -103°F.

A. The MIL-C-22750 epoxy, the MIL-L-81352 lacquer, and two polyurethane topcoats exhibited cracking around fastener heads when tested to simulate 1 PAR. (One of the polyurethanes, P114-1, was satisfactory when tested at -60° F as per reference (d), the conical mandrel flexibility test).

B. In many cases, the above coatings exhibited fastener head cracking when tested at conditions notably less severe than the simulated 1 PAR test.

(1) <u>Example</u>: Most specimens, regardless of topcoat, exhibited fastener head cracking when the maximum stress was reduced from 30,000 lb./in.² to 17,500 lb./in.².

(2) <u>Example</u>: All specimens, regardless of topcoat, exhibited faster head cracking when the simulated 1 PAR test was conducted at room temperature.

C. The coatings in paragraph A. above which were baked to simulate aerodynamic heating offered markedly less resistance to cracking around fastener heads than the same type unbaked coatings.

D. The coatings in paragraph A. which were applied over the PR1422 (B2) sealant strip generally exhibited cracking around fastener heads after the simulated 1 PAR test.

E. The pressure sensitive polyvinyl fluoride tape applied over the fastener heads of one epoxy coated specimen exhibited cracking around fastener heads.

F. High speed motion pictures (400 frames per second) taken of the coated fastener areas during cycling and projected on a screen clearly shows movement between the fastener and surrounding structure. This movement is also apparent in Plates 1-7 which are single frames from the motion pictures and show the paint crack widen and narrow with increased and decreased load during one cycle.

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IV. CONCLUSIONS

A. The implication of the stress-cycling test is that it is improbable that any current specification or experimental non-rubbery type coating system, per se, can provide reasonable long-term corrosion protection to aircraft fastener areas.

B. Even the Pl14-1 polyurethane coating which has unusually good flexibility for a rigid, non-rubbery type suitable for aircraft use (satisfactory to the conical mandrel flexibility test at -60° F) exhibited cracking around fastener heads when evaluated in the simulated 1 PAR test.

V. RECOMMENDATIONS

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A. No recommendations are made at this stage of the subject investigation.

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REFERENCES

- (a) BUWEPS 1tr RRMA-52:SK/108 of 3 May 1961
- (b) BUWEPS 1tr kRMA-52:SK/22 of 23 Feb 1965
- (c) Report No. NAEC AML 2346 of 2 Feb 1966
- (d) Federal Test Method Standard No. 141, Method 6222, Flexibility, Conical Mandrel Test

ACKNOWLEDGEMENTS

Sincere appreciation is given to Mr. Ralph Vining of the Aeronautical Structures Laboratory for providing the parameters for the stress-cycling test and the design of the specimens, and for his patient instruction in some of the more fundamental aspects of this test; to Mr. Michael Stellabotte of the Metallurgical Division of the Aeronautical Materials Laboratory for his instruction in the use of the Krouse Flexure Apparatus; and to Messrs. John Stewart and Holt Smith for their assistance in operating the K-F apparatus.



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Coating System-Bé <u>Prior to Stressi</u> <u>Yes</u>	gu											
Condition of Coating Around Most Fastener Heads	Fine cracking Slightly more cracki	Cracking	Cracking	Satisfactory Cracking	Cracking	Satisfactory Cracking	Cracking	Cracking	Satisfactory Satisfactory Cracking	Cracking	Satisfactory Satisfactory Cracking	Satisfactory
No. of Cycles	1000	1000	1000	1000 1000	1000	1000 1	1000	1000	1000 1000 1000	1000	1000 1000 1000	1000
en en Min.	1560 3000	1560	15600	1560 1880	1560	1560 3000	1880	1560	1560 1880 3000	1560	1560 1880 3000	1560
Specim Specim (1bs./i Max.	15600 30,000	15600	15600	15600 18800	15600	15600 30,000	18800	15600	15600 18800 30,000	15600	15600 18800 30,000	15600 30,000
s on m-1bs. Min.	575 1125	575	575	575 700	575	575 1125	004	575	575 700 1125	575	575 700 1125	575
Force Specime Max.	5800 11250	5800	5800	5800 7000	5800	5800 11250	7000	5800	5800 7000 11250	5800	5800 7000 11250	5800 11250
Coating System ⁽¹⁾	WPI + P46 + P79 Epoxy Same specimen	WPI + P46 + P79 Epoxy	WPI + P46 + P79 Epoxy	P46 ↔ 95K Acrylic Same specimen	P46 + 95K Acrylic	P46 + 95K Acrylic Sam¢ specimen	P46 + 95K Acrylic	P46 4 43MS Polyurethane	P46 + 43MS Polyurethane Same specimen Same specimen	P46 + 43MS Polyurethane	P46 + 43MS Polyurethane Same specimen Same specimen	P46 + 43MS Polyurethane Same snerimen
Specimen Designation	14	9	11	16	N 6 TARI	00 	89	75		- 18 - 18	6 2	18

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Coating System Prior to Stree Yes		××	×	×	×			~	~
Condition of Coating Around <u>Most Fastener Heads</u> Crackine	Cthe Read	Cracking	Cracking	Cracking	Cracking	Cracking of tape around fastener heads	Satisfactory-No observable crack-	ıng of paint system Fine cracking	fine cracking
No. of Cycles 1000	1000	0001	1000	1000	1000	1000	1000	1000	1000 1
thes on the set of the	1560	1560	1560	1560	3000	3000	3000	3000	3000
Stress Speci Speci Speci Max. 18800	15600	15600	15600	15600	30,000	30,000	30,000	000,000	0,000
tes on ten-1bs. Min. 700	<u> </u> 275	575	575	575	1125	1125	1125	1125 3	1125 3
Ford Specim Max	5800	5800	5800	5800	11250	11250	11250	11250	11250
Coating System ⁽¹⁾ P46 + P114-1 Polvurethane	P46 + 95K Acrylic	P46 + P43MS Polyurethane	P46 + P43MS Polyurethane	P46 + P43MS Polyurethane	?46 + P114-1 Polyurethane	246 + P79 Epoxy + 3M Polyvinyl ^m luoride Tape (Y-9057)	PI + P46 + P79 over PRI422 ribbon	PI + P46 + P79 over PK1422 ribbon	PI + P46 + P79 cver PR1422 ribbon
Designation B	615	166	173	- 91 748LE 1	A	8 11 10 10	9 0E 0	141 I 141	142 WE F

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NOTES TO TABLE 1

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- (1)WPI = Wash primer, MIL-C-8514
 - P46 = Epoxy primer, MIL-P-23377
 - P79 = Epoxy topcoat, MIL-C-22750 (Insignia White)
 - PR1422 = Products Research Company's PR1422, Class B filleting compound. Prepared by mixing 75 parts by weight of Part I to 10 parts by weight of Part II.
 - P43MS = AML formulated white polyurethane topcoat
 - P114-1 = AML formulated white polyurethane topcoat

95K = White all-acrylic topcoat, MIL-L-81352(WEP)

PAGE 3 OF 3 PAGES

CYCLIC LOADING TESTS AT ROOM TEMPERATURE

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		E C L C L C L C L		Stresse	s on			
Specim	en	Specime	en-lbs.	(1bs./1	n.2)	No. of	condition of Coatine Around	Coating System-Baked Prior to Stressing
Designa	tion Coating System	Max	पम्प्र	Мах.	Min	Cycles	Most Fastener Heads	Yes v v v v v v v v v v v v v v v v v v v
72	P46 + P43MS Polyurethane	11250	1125	30,000	3000	1000	Cracking	×
73	P46 + P43MS Polyurethane	11250	1125	30,000	3000	1000	Cracking	×
168	P46 + P43MS Polyurethane	11250	1125	30,000	3000	1	Cracking	×
169	P46 + P43MS Polyurethane	11250	1125	30,000	3000	-	Cracking	×
66	P46 + 95K Acrylic	11250	1125	30,000	3000	1000	Cracking	×
101	P46 + 95K Acrylic	11250	1125	30,000	3000	1000	Cracking	×
174	P46 + 95K Acrylic	11250	1125	30,000	3000	1	Cracking	R
175	P46 + 95K Acrylic	11250	i 125	30,000	3000	-1	Cracking	× ×
300	P46 + P114-1 Polyurethane	11250	1125	30,000	3000	1	Cracking	xt no. ×
301	P46 + P114-1 Polyurethane	11250	1125	30,000	3000	-1	Cracking	NAEC A
302	P46 + P114-1 Polyurethane	11250	1125	30,000	3000	1000	Cracking	ML 245 ×
NOTES:	<pre>(1) P46 = Epoxy primer, (3) P95K = White all-acr</pre>	AIL-P-233 ylic topo	77; (2) oat; (4	P43MS =	= AML fo = AML	ormulated formulat	l white polyurethane ed white polyurethane:	5. topcoat

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TABLE 2

Parts by

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FORMULATION DATA FOR TOPCOATS

A. Polyurethane, 114-1

Ingredients	Weight
TiO ₂ (R610)	360
Castor Oil #1066, Spencer-Kellog Company	288
Cellosolve Acetate (Urethane Grade)	138
Xylene	138

Grind in pebble mill, then mix 29.8 parts by weight to:

Aliphatic Isocyanate XP1699, Spencer-Kellog Company 343

B. Polyurethane, P43MS

	Parts by
Ingredients	Weight
Desmophen 650, Motay Chemical Company	321
TiO ₂ (R610)	267
Dri-Film, General Electric Company	0.8
Zinc Naphthenate (8%)	1.2
Cellosolve Acetate (Urethane Grade)	223
Xylene	40
Cyclohexanone	50

Gind in pebble mill, then to 90.3 parts by sought of above, add:

Desmoden N,	Mobay Chemical Company	45

C. Acrylic, MIL-L-81352(WEP)

Ingredients	Parts by <u>Weight</u>
710 ₂ (R610)	720
Acryloid A21 (30% solution) Rohm & Haas Co.	1720
Acryloid B44 (40% solution) Rohm & Haas Co.	1284
Santicizer 160, Monsanto Co.	54
Cellosolve Acetate	175

Grind in pebble mill

D. Epoxy, MIL-C-22750

See Specification



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1st PHOTO IN PICTURE SEQUENCE OF FASTENER HEAD AREA DURING 1 CYCLE OF TEST PHOTO NO: CAN-375450(L)-5-66 PLATE 1



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3rd PHOTO IN PICTURE SEQUENCE OF FASTENER HEAD AREA DURING 1 CYCLE OF TESTPHOTO NO:CAN-375452(L)-5-66PLATE 3

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4th PHOTO IN PICTURE SEQUENCE OF FASTENER HEAD AREA DURING 1 CYCLE OF TEST PHOTO NO: CAN-375453(L)-5-66



5th PHOTO IN PICTURE SEQUENCE OF FASTENER HEAD AREA DURING 1 CYCLE OF TEST PHOTO NO: CAN-375454(L)-5-66

PLATE 5

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6th PHOTO IN PICTURE SEQUENCE OF FASTENER HEAD AREA DURING 1 CYCLE OF TEST PHOTO NO: CAN-375455(L)-5-66



7th PHOTO IN FICTUFE SEQUENCE OF FASTENER HEAD AREA DURING 1 CYCLE OF TEST PHOTO NO: CAN-375456(L)-5-66



KROUSE DIRECT STRESS TESTING MACHINE WITH SPECIMEN MOUNTED

PHOTO NO: CAN-375245(L)-4-66

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CLOSE-UP OF MOUNTED SPECIMEN

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FIGURE 1 - THE STRESS SPECIMEN USED IN THE CYCLIC LOADING APPARATUS

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