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DiEGME Resistant Materials for Fuel Tank Protection

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ABSTRACT

Di-ethylene glycol monomethyl ether (DiEGME) is a fuel system icing inhibitor (FSII) added to both JP-5 and JP-8 jet fuel to prevent water from freezing in integral fuel tanks and also functions as a biostat. The use of DiEGME is currently causing fuel tank coatings to peel, especially in the headspace where the condensate is mostly a DiEGME/water mixture. Fuel tank coatings are used to protect the fuel tank from corrosion due to water contamination of the fuel. Additionally, the coatings protect cadmium plated fasteners from reacting with sulphur compounds in the fuel. Peeling topcoat paint chips and flakes can collect in fuel system filters and screens blocking fuel to the engines, thereby potentially causing engine flame out and possible loss of personnel and aircraft. DiEGME is primarily used on military aircraft, as commercial aircraft use inline fuel heaters in lieu of a FSII. Fuel tank topcoat peeling has been observed in the P-3 and it is anticipated that the currently available fuel tank coatings will continue to be problematic in the future naval asset, the P-8 Poseidon. In addition, the Air Force has experienced fuel tank topcoat peeling in the B-52, KC-135, and C-17. The focus of this effort is to develop a standard accelerated test method for the evaluation of fuel tank coatings for DiEGME resistance. The resulting test protocol will be used for determining acceptable DiEGME resistant materials and further the development of DiEGME resistant coatings. This effort is being performed in conjunction with the USAF and the SAE(1) Aerospace Materials Specification (AMS) G8 Aerospace Organic Coatings Committee.

Key Words: DiEGME, fuel tank coatings, corrosion

INTRODUCTION

In the 1950's, it was observed that many military fuel systems were prone to icing. In 1958, a B-52 accident brought the problem into the spotlight. In previous crashes, icing of the fuel system had been suspected as a likely cause, but the ice had melted in ensuing fires leaving no concrete evidence. In this instance, the USAF determined that icing of the fuel system strut filters and fuel pump screens had caused the engine to flame out and lose thrust. By the early 1960's, a fuel system icing inhibitor (FSII) in the form of ethylene glycol monomethyl ether (EGME) was being added to all jet fuel at military installations. FSII also served a dual purpose as a biostat.

To address environmental concerns in the 1990's, a switch from EGME to Di-ethylene glycol

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monomethyl ether (DiEGME) as a FSII was made. Additionally, a DoD/NATO-wide change was made in the early 1990's from the more flammable JP-4 to JP-8. The Navy uses the even less flammable JP-5 for carrier based assets. In 1995, about one year after these changes were initiated, coating failure was observed in the ullage of aircraft with integral fuel tanks. These failures were attributed to the greater presence of a DiEGME/water mixture than fuel in the vapor phase due to the decrease in fuel volatility from the fuel switch. In the liquid phase, DiEGME has a greater affinity for water than fuel, water being denser than fuel settles in the tank bottoms and causes additional coating failure. Fuel tank coatings are used to protect the tank from corrosion and to protect cadmium plated fasteners from reacting with sulfur compounds in the fuel. Fuel tank coatings are typically a single layer epoxy or polyurethane coating. There is some confusion in terminology as they are often described as an integral fuel tank primer, topcoat, or coating, all are considered synonymous in this effort.

Over time, DiEGME degrades fuel tank coatings and causes fuel tank topcoat peeling (FTTP). Paint chips and flakes can collect in fuel system filters and screens blocking fuel to the engines, potentially causing engine flame out and possible loss of personnel and aircraft. DiEGME is almost exclusively used on military aircraft, as commercial aircraft use inline fuel heaters in lieu of a FSII. FTTP has been observed in the P-3 and it is anticipated that the currently available fuel tank coatings will continue to be problematic in the future naval asset, the P-8 Poseidon. In addition, the Air Force has experienced fuel tank topcoat peeling in the B-52, KC-135, and C-17. The focus of this effort is to develop a standard accelerated test method for the evaluation of fuel tank coatings for DiEGME resistance and to modify the AMS-27725 to include the new test method and a new type for DiEGME resistant coatings. This effort is being performed in conjunction with the USAF, OEMs (i.e. Boeing, Lockheed-Martin), coatings manufacturers, and the SAE AMS G8 Aerospace Organic Coatings Committee.

Field Observations

The USAF first observed FTTP in the ullage of the B-52 aircraft in 1995, approximately 1 year after the conversion from JP-4 to JP-8 (Figure 1). The conversion to JP-8 is significant because DiEGME is less soluble in both JP-5 and JP-8 than in JP-4. This decrease in solubility has led to an increased concentration of DiEGME in the ullage and tank bottoms where water resides.

Figure 1: FTTP, B-52 aircraft, stopping at fuel line (Tinker-AFB)

Figure 2: Fuel tank topcoat blistering in a P-3 aircraft (FRC-SE)

The Navy began seeing FTTP in the P-3 aircraft in the early 2000's (Figures 2, 3, 4). The P-3 FTTP is less severe than that of the B-52 because the B-52 is routinely operated with less than a full fuel load for takeoff/weight considerations. Therefore, the B-52 has an increased ullage area when the aircraft is not flying. Also, the topcoat used on the exterior of the B-52 aircraft is less UV reflective than the P-3 topcoat leading to the possibility of increased fuel tank temperatures. FTTP in integral fuel tanks inevitably will occur regardless of the aircraft, but the rate will depend upon fuel tank conditions (i.e., temperature, humidity, etc.).

Figure 3: Fuel tank topcoat blistering in a P-3 aircraft (FRC-SE)

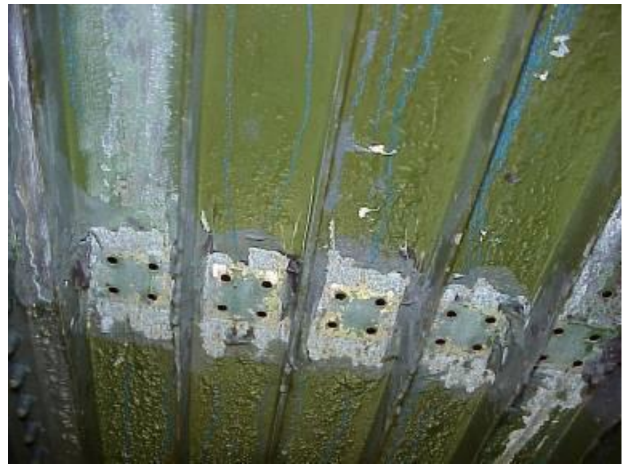

Figure 4: Fuel tank topcoat peeling/blistering in a P-3 aircraft (FRC-SE)

The accumulation of paint chips in fuel filters (Figure 5) and pump screens (Figure 6) can lead to potential fuel starvation[1] to engines, a decrease in mission readiness, and increased man-hours required to keep fuel circuits clean.

Figure 5: Paint chip contaminated fuel filter removed from a B-52 aircraft (Tinker-AFB)

Figure 6: Main tank pump screen from a B-52 aircraft (Tinker-AFB)

In addition to FTTP, there is evidence of microbial induced corrosion (MIC) as *bacillus cereus* microbes (Figure 7) were observed in corrosion pits of a fuel tank, though the vast majority of the coating failure exposes non-corroded substrate. The *bacillus cereus* microbe is commonly found in aviation fuel systems[2].

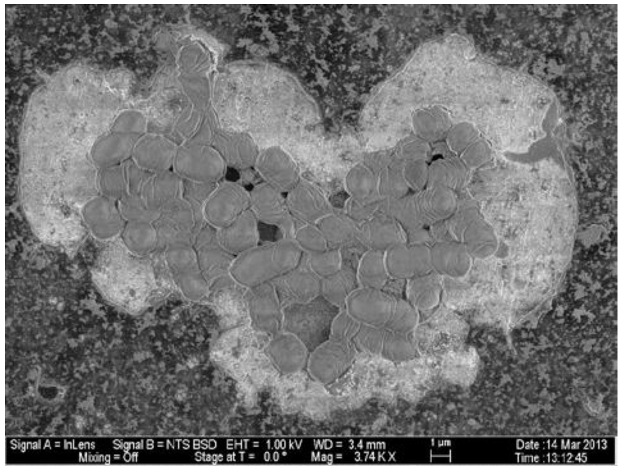

Figure 7: AA2024-T3 with bacillus cereus in a corrosion pit (AFRL-WPAFB)

EXPERIMENTAL

In order to determine optimal conditions to reproduce the coating failures observed in the in-service military integral fuel tanks. DiEGME coating degradation testing was performed at Fleet Readiness Center-Jacksonville (FRC-JAX), Naval Air Station-Patuxent River (NAS-PAX) and Wright-Patterson Air Force Base (WPAFB). This testing determined that DiEGME with a presence of water is detrimental to fuel tank coatings and conversion coating integrity.

In addition to experimental coatings, many of the coatings that were evaluated are currently qualified to industry and OEM materials specifications. Coatings were tested solely for DiEGME resistance. Coatings evaluated are listed in Table 1.

Table 1 – Coatings evaluated for DiEGME resistance

Manufacturer	Product Number
PPG	823-707/910-702/020-707
PPG	825X309/910-702/020-707
PPG	833K086/930KO88
Deft	69W004/69W004Cat
Deft	69W006/69W006Cat
Deft	69W006A/69W006Cat
Deft	69W013/69W004Cat
AXON/Hentzen	FT-9-Y4/EH-46/SR61
Sherwin Williams	CM560564/CM0120888
ANAC	454-4-1/CA-109
ANAC	20P1-21/PC-235
Hentzen	53091AEP
Hentzen	53093GEP
ACT Phoenix	Exokote 8003FTC
NIC Industries	Cera-Kote C556

Aluminum AA7075-T6 test panels were pretreated with Alodine 1200S in accordance with MIL-DTL-81706, Type I. Panels were allowed to dry in ambient laboratory conditions for 48 hours prior to being coated with the prospective DiEGME resistant fuel tank coatings. The coatings were allowed to cure for 21 days at ambient laboratory conditions prior to testing.

RESULTS AND DISCUSSION

FRC-SE

Initial NAVAIR testing was performed at FRC-SE, Jacksonville, FL and consisted of AA7075-T6 panels in enclosed Mason jars with a DiEGME/water mixture with concentrations ranging from 0 to 100% (0%, 25%, 50%, 60%, 70%, 80%, 90%, and 100%). Panels were exposed to both liquid (l) and vapor (v) conditions. The jars were placed into a cyclic environmental chamber and cycled between temperatures of 150°F for 20 hours and 10°F for 4 hours over a 30 day period. This testing confirmed that the presence of both water and DiEGME in a condensing environment is needed to replicate the conditions for fuel tank coating degradation. Coating degradation was assessed by blistering (Figure 8), adhesion (Table 2), and pencil hardness data (Table 3). This testing also showed that increasing water concentration increases conversion coating depletion, as observed by color change of the Alodine 1200S conversion coating (Figure 9). A depletion of conversion coating can contribute to corrosion of the fuel tank.

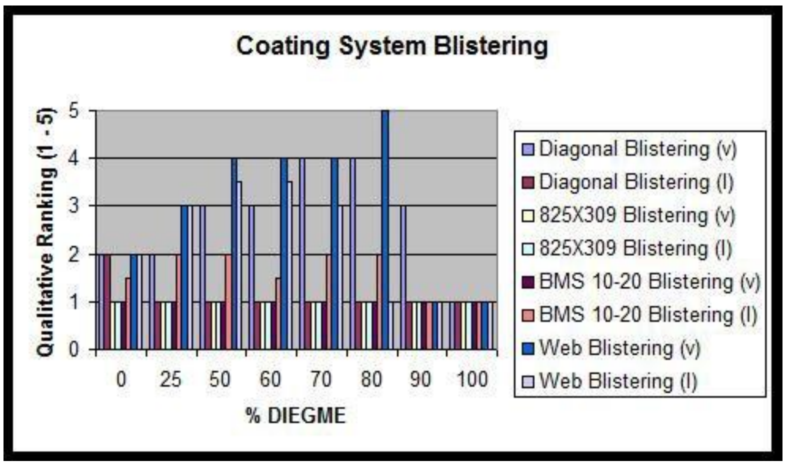

Figure 8: Blistering Data
(Qualitative Ranking 1 = no blisters, 5 = most blisters)

Table 2 – Adhesion Data
(S = No Adhesion Failure, F = Adhesion Failure, NA = Unable To Test)

% DIEGME	Panel	Diagonal Adhesion	825X309 Adhesion	BMS 10-20 Adhesion	Web Adhesion
0	A	S	S	S	F
0	B	S	S	F	F
25	A	F	S	F	F
25	B	S	S	F	F
50	A	F	S	F	F
50	B	F	S	F	F
60	A	F	S	F	F
60	B	F	S	S	S
70	A	F	S	F	F
70	B	F	S	F	F
80	A	F	S	F	F
80	B	F	S	F	F
90	A	F	S	S	S
90	B	F	S	NA	S
100	A	S	S	F	F
100	B	NA	NA	NA	NA

Table 3 - Pencil Hardness Testing per ASTM D3363

% DIEGME	Panel	Diagonal Hardness	825X309 Hardness	BMS 10-20 Hardness	Web Hardness
0	A	4H	4H	5H	NA
0	B	4H	4H	5H	NA
25	A	5H	4H	5H	NA
25	B	5H	4H	5H	NA
50	A	5H	4H	5H	NA
50	B	5H	4H	5H	NA
60	A	4H	3H	4H	NA
60	B	4H	3H	4H	NA
70	A	4H	3H	4H	NA
70	B	4H	3H	4H	NA
80	A	4H	3H	B	NA
80	B	4H	3H	B	NA
90	A	2H	H	5B	NA
90	B	2H	H	5B	NA
100	A	2H	H	5B	NA
100	B	2H	HB	5B	NA

Figure 9: Panels showing depletion of chromate conversion coating with increasing water concentration left to right is decreasing water concentration.

The coating degradation/adhesion loss is the result of the swelling of the integral fuel tank coating and the ingress of water. The evidence of chromate conversion coating depletion[3] may contribute to osmotic blistering due to the presence of the inorganic salts at the coating/substrate interface creating an osmotic gradient[4]. Inorganic salts in the form of chromic acid-chromic salts are present in conversion coatings utilized in integral fuel tanks. In addition, inorganic salts within the coating itself also can contribute to osmotic blistering. Most protective coatings will impede water intrusion to the coating-substrate interface, but the swelling of the coating by DiEGME uptake[5,6] makes for ease of water ingress.

Testing performed at FRC-SE concluded that DiEGME concentrations between 70-90% provide the highest detrimental coating effects from blistering, color change, and conversion coating depletion collectively. Some coatings are more sensitive to proper conversion coating application than others as is seen by the adhesion loss (Figure 10) coinciding with the conversion coating “dry line” (Figure 11).

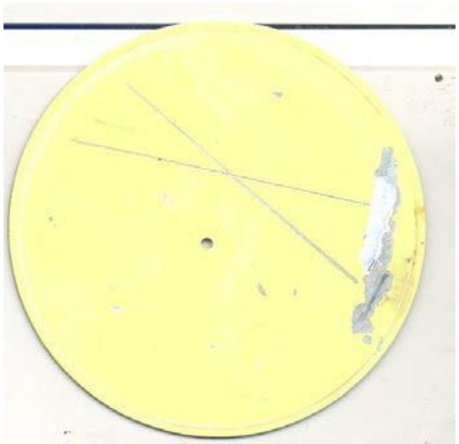

Figure 10: Conversion coating adhesion loss

Figure 11: Conversion coating dry line

NAS-PAX

The number of candidate coatings was increased during follow-on testing at NAS-PAX and an alternative condensation testing environment was further examined. Test panels were exposed to the vapor phase of the Mason jar test apparatus by vertical suspension and horizontally resting the panels across the jar opening. The jars were heated to 170°F with the tops of the jars being subjected to ambient laboratory conditions to induce condensation (Figure 12). An 80% DiEGME/20% water mixture was selected for use due to previous testing indicating that greatest coating degradation occurred between 70-90% DiEGME. Additionally, water is considerably more volatile than DiEGME and it was determined that a < 90% DiEGME concentration can easily be achieved when considering loss due to specimen "drag out" or evaporation. To maintain a DiEGME solution between 70-90% it was determined that the entire solution should be replaced after a 10% solution loss. Test panels were exposed for up to 35 days and evaluated periodically for blistering, adhesion, and pencil hardness.

Concurrently, the Coatings Technology Integration Office (CTIO-WPAFB) collaborated with NAVAIR PAX and performed similar condensation testing in an effort to achieve an agreed upon test protocol for addition to SAE AMS-27725 as well as a new coating type for DiEGME resistance. These collaborative efforts confirmed evidence of different modes of failure as is evident by the blistering on the horizontal panel (Figure 13) and the pencil hardness data (Figure 14).

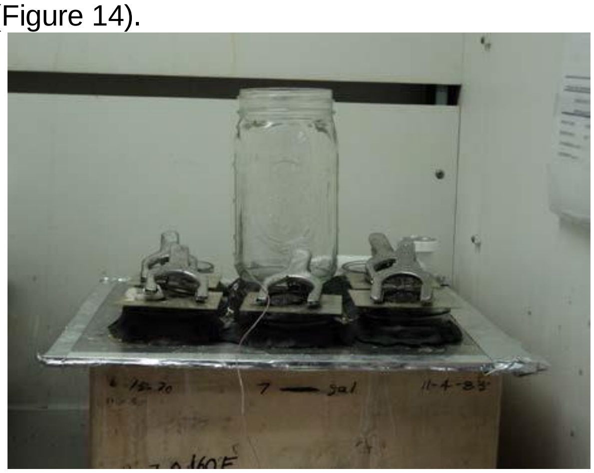

Figure 12: Test apparatus with the tops of the jars subjected to ambient conditions

Figure 13: Vertical panel intact and the horizontal panel blistering

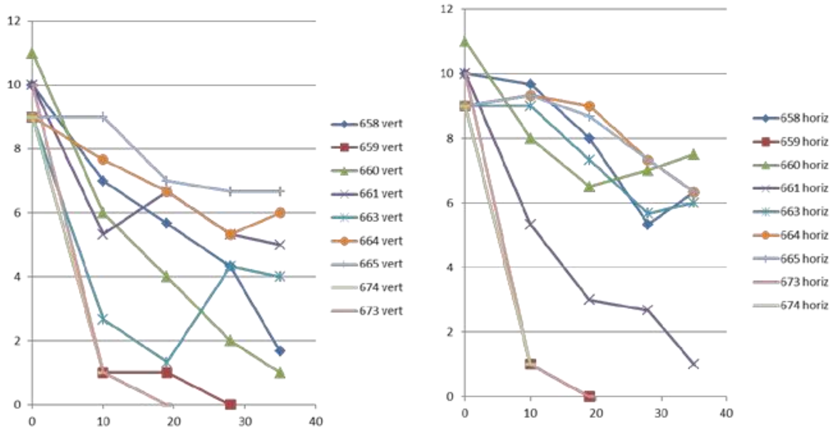

Figure 14: Pencil Hardness Equivalents (will not gouge); 4H-13, 3H-12, 2H-11, H-10, F-9, HB-8, B-7, 2B-6, 3B-5, 4B-4, 5B-3, 6B-2, <6B-0, complete delamination-0, 0 to 35 days.

Subsequent testing was performed with inserts of AA7075-T6 that were manufactured to fit inside the screw tops of the Mason jars to minimize evaporation (Figure 15). The panels were examined for visual defects and pencil hardness at ~30 day intervals to reduce solution drag out. In addition, the heating (165°F) of the Mason jars was confined to only that of the solution filled volume (~350 ml). This was done to further increase the condensation on the vertical hanging and horizontal panels (Figure 16). The temperature and testing protocol change was a collaborative decision made by CTIO-WPAFB and NAS-PAX for consistency of testing.

Figure 15: Test specimen jar and panels

Figure 16: Test apparatus, only solution heated.

The majority of vertical hanging panels in this round of testing exhibited lower pencil hardness test results than the horizontal panels (Figures 17, 18). Generally the vertical panels showed the greatest coating degradation, but in some instances the horizontal panels had increased coating degradation. This is attributed to the vertical panels having less condensation and a greater temperature exposure and the horizontal panels having greater condensation and a lower temperature exposure.

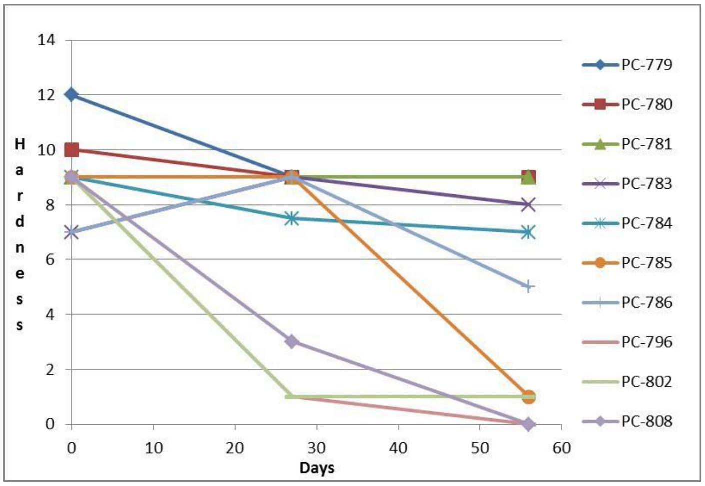

Figure 17: Vertical Hanging Panels

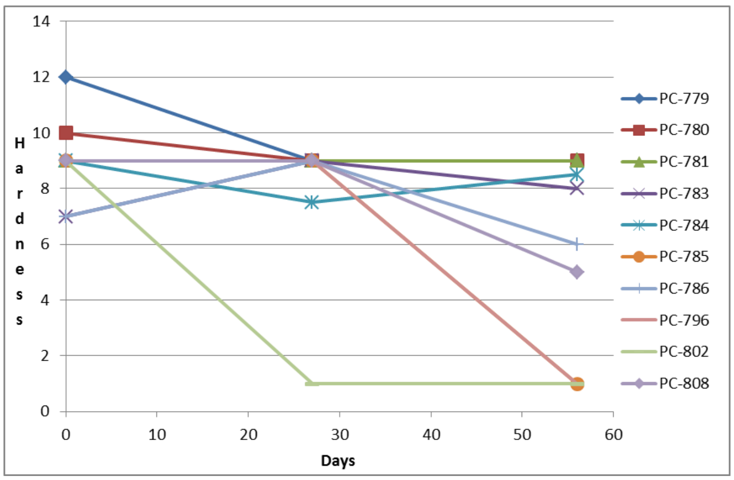

Figure 18: Horizontal Panels

Pencil Hardness Equivalents (will not gouge); 4H-13, 3H-12, 2H-11, H-10, F- 9, HB-8, B-7, 2B-6, 3B-5, 4B-4, 5B-3, 6B-2, <6B-0, complete delamination-0

Based on previous test results, a down-selection was performed on the coatings. Coatings that exhibited a decrease in pencil hardness of greater than two units or the adhesion loss/blistering of the coating were removed from further testing. Further testing was performed on down-selected coatings to verify reproducibility. The vertical and horizontal panels exhibited equivalent behavior in regards to pencil hardness (Figures 19, 20). Representative 60 day exposure panels (Appendix A) show wet tape adhesion results. Two PPG products and one Deft product exhibited blistering and adhesion failure attributed to a conversion coating deficiency. Averaged adhesion results disregarding conversion coating deficiency show that there are failures on both the horizontal and vertical panels (Table 4). One of the Deft coatings leached into the DiEGME/water solution as was seen by the “milky” solution color (Appendix A).

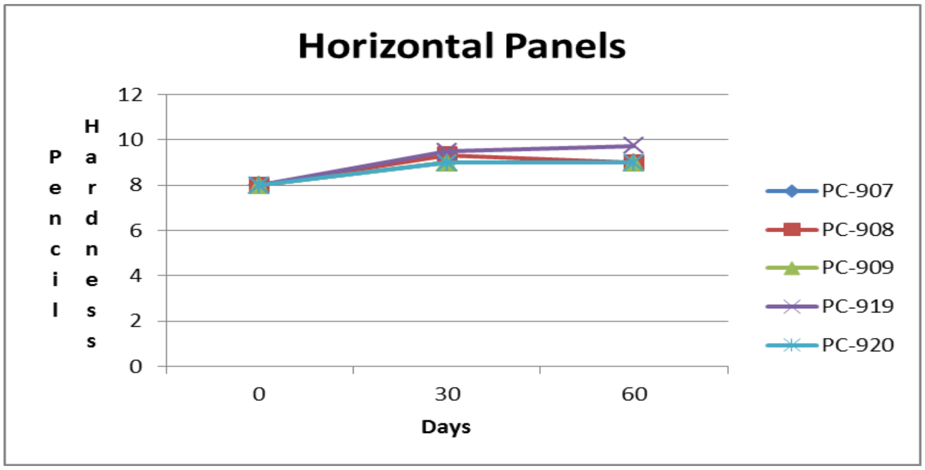

Figure 19: Pencil Hardness Results for Down-Select Horizontal Panels

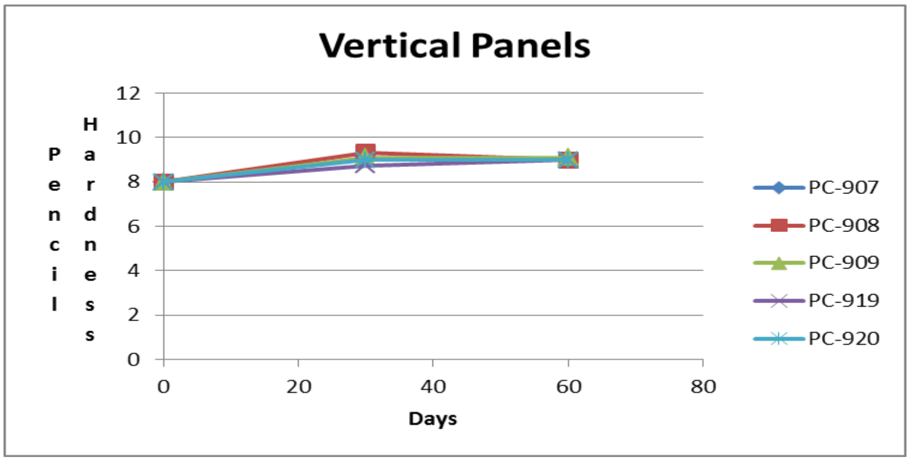

Figure 20: Pencil Hardness Results for Down-Select Vertical Panels

Table 4 - Adhesion, film thickness, and solution data

Product	Horizontal (adhesion)	Vertical (adhesion)	Thickness (mils)	Solution
PC-907	5A	5A	1.7	clear
PC-908	5A	5A	1.6	clear
PC-909	3.3A	5A	1.8	clear
PC-919	5A	3A	1.8	clear
PC-920	5A	5A	1.3	milky

Although there is concurrence by NAS-PAX and CTIO-WPAFB as to the environment of the coating exposure, there is divergence as to panel preparation (i.e., contamination, pretreatment, configuration etc.). NAVAIR PAX panels are intended to simulate an OEM applied coating and have also been scribed to simulate a damaged coating. CTIO-WPAFB is investigating lap-joint and fuel contaminated panels.

Synopsis of Testing Results

Coating	Sample Number	Pencil Hardness	Adhesion	Blistering	Notes	Relevant Figure
PPG 823-707/ 910-702/ 020-707	PC-664 PC-779 PC-907	Good	Good	None		19, 20 Appendix A
PPG 825X309/ 910-702/ 020-707	PC-663 PC-780 PC-908	Good	Good	None		19, 20, Appendix A
PPG 833K086/ 930KO88	PC-665 PC-781 PC-909	Good	Mixed results (horizontal)	None		19, 20, Appendix A
Deft 69W004/ 69W004Cat	PC-660 PC-783 PC-919	Good	Mixed results (vertical)	None		19, 20 Appendix A
Deft 69W006/ 69W006Cat	PC-661 PC-784 PC-920	Good	Good	None	Leaching	19,20 Appendix A
Deft 69W006A/ 69W006Cat	PC-785	Poor				17
Deft 69W013/ 69W004Cat	PC-786	Poor				17
AXON/Hentzen FT-9-Y4/EH-46/SR61	PC-796	Poor		Poor		17, 18, Appendix A
Sherwin-Williams CM560564/CM0120888	PC-802	Poor				17, 18
ANAC 454-4-1/CA-109	PC-808	Poor		Poor		17, 18, Appendix A
ANAC 20P1-21/PC-235	PC-658	Poor				14
Hentzen 53091AEP	PC-673	Poor				14
Hentzen 53093GEP	PC-674	Poor				14
ACT Phoenix Exokote 8003FTC	PC-659		Poor			Appendix A
NIC Industries: Cera-Kote C556					Leaching/ dissolution	Appendix A

Field Testing (B-52)

While laboratory testing was being performed, the AF simultaneously performed field testing of prospective fuel tank coatings on B-52 aircraft. Below are inspection results from coatings that were field tested.

Inspection Date: January 2013
Location: Barksdale AFB
Inspection Interval: 17 months
Product and Observation: PPG 833K086/930KO88 - No signs of coating degradation
Product and Observation: PPG 825X309/910-702/020-707 - No signs of coating degradation
Product and Observation: AXON/Hentzen FT-9-Y4/EH-46/SR61 - Peeling of the coating
Product and Observation: Deft 69W004/69W004Cat - No signs of coating degradation

Inspection Date: September 2012
Location: Tinker AFB
Inspection Interval: 36 months
Product and Observation: Sherwin-Williams CM560564/CM0120888 - Peeling of the coating
Product and Observation: ACT Phoenix Exokote 8003FTC - No signs of coating degradation
Product and Observation: PPG 833K086/930KO88 - No signs of coating degradation
Product and Observation: PPG 825X309/910-702/020-707 - No signs of coating degradation
Product and Observation: AXON/Hentzen FT-9-Y4/EH-46/SR61- Peeling of the coating
Product and Observation: NIC Industries: Cera-Kote C556 - Peeling of the coating

CONCLUSIONS

It was found that there are at least two different coating failure modes inherent to this testing. One mode is the combination of greater condensation and lower temperature and the other mode is the lesser condensation and higher temperature combination. Some coated panels in the horizontal position (lids) showed degradation sooner than their vertical counterparts (hanging) and vice-versa. Although most of the vertical hanging panels showed greater degradation than the horizontal panels for the equivalent amount of exposure, there are exceptions. Hence, there is value in testing in both orientations and coating degradation within the fuel tanks themselves is seen in both scenarios.

In addition, a DiEGME/water concentration of 80%/20% is recommended as long as total liquid loss does not exceed 10% volume, ensuring a concentration in the range of 80-90% DiEGME. When a solution loss of 10% is achieved the solution shall be replaced in its entirety, the rational is that the vapor pressure (volatilization) of water is much greater than that of DiEGME and that any solution loss will be almost entirely water. In agreement with the AF and laboratory testing, the solution temperature shall be maintained at 165° F ± 5°F and the top of the container shall be exposed to an ambient condition of 75° F ± 5°F, to induce condensation.The DiEGME resistance performance testing requirements shall be determined by pencil hardness testing in accordance with ASTM D3363, adhesion ASTM D3359, blistering ASTM D714, and visual inspection i.e. scribed panel undercutting, leaching, discoloration etc.

There is correlation of the field results to that of the laboratory data as the best performers are in the field tend to be the best performers in the laboratory. However, there is one coating that showed adhesion failure in the laboratory, but is performing satisfactorily in the field.

The continuation of this effort will involve corroboration with CTIO-WPAFB to determine the subsequent testing of DiEGME resistant coatings to additional AMS-27725 performance requirements, this testing would be performed after the coatings have been exposed to the DiEGME/water mixture.

Acknowledgements

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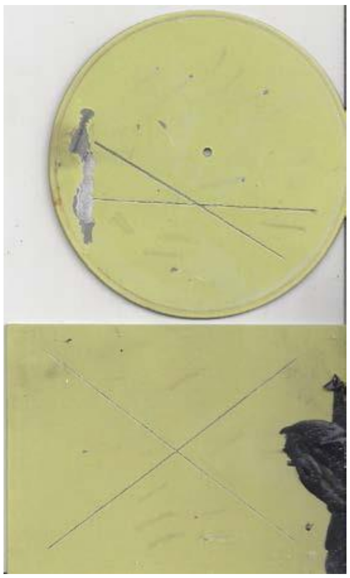
References

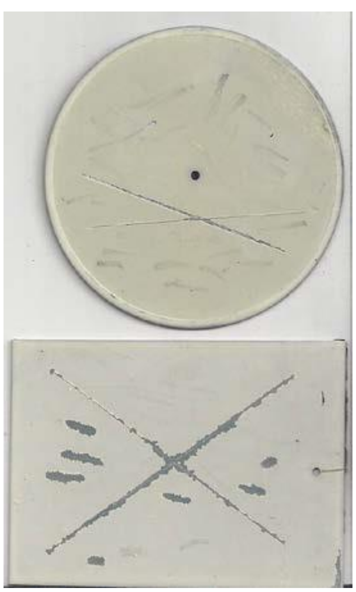
1) Deficiency Report (clogged fuel nozzles), Report Control Number FB6530-12-0114, AFSAS 845681, 2012
2) A.Rajasekar, Y. Ting, Ind. Eng. Chem.Res., 2010, 49, 6054-6061
3) P-3 Integral Fuel Tank (IFT) DIEGME Evaluation By J. E. Benfer (NAVAIR): Aug 2006
4) C. Hare, JPCL-PMC, February 1998
5) S. Zabarnick, R. Adams, Z. West, M. DeWitt, L. Shafer, R. Striebich, C. Delaney, D. Phelps, Energy Fuels, 2010, 24, 2614-2627
6) Exfoliation of Protective 1039 Polyamide – Epoxy Coating in B52 Fuel Cells Contract # EA 02-B52-027B Phase 1 report to Boeing Military and USAF April, 2004

Appendix A



PC-907 PC-908 PC-919, 60

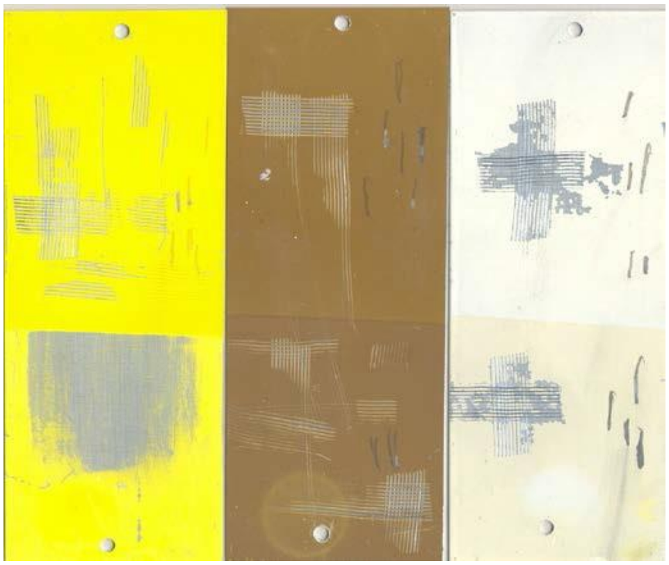
60 days@170F, 80/20



PC-909 PC-920

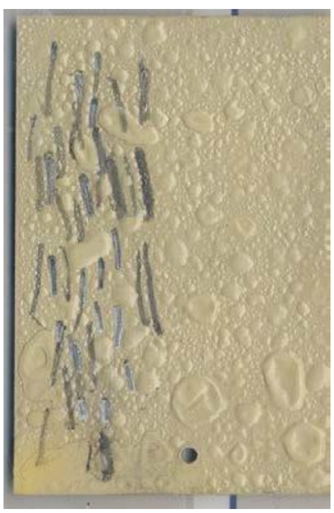
60 days@170F, 80/20

Appendix A



NIC C556 PC-796 PC-659

60 days, cycling of 20 hrs@ 200F and 4 hrs@10F, 80/20



PC-808, 47 days@170F, 80/20

PC-796, 26 days @170F, 80/20

Example of the leaching of a coating