

Advanced Thin Film Fluoropolymers for Paint Replacement Appliqué Technology

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ABSTRACT

This paper discusses the exciting progress made over the last several years in the ongoing development of thin film, peel and stick, fluoropolymer appliques. Final development and optimization of these paintless coatings systems are targeted for applications on various Navy and Marine Corps air, marine and land vehicles as alternatives to currently used paint based coating systems.

This presentation will focus on a new generation of multifunctional fluoropolymer based appliqué systems. Results to date have demonstrated:

1. The development of new pressure sensitive adhesives containing environmentally benign, organic based corrosion inhibitors.
2. Novel Peel and stick properties throughout a wide temperature range (-65°F to 350°F).
3. Resistance to a wide range of chemicals, solvents, UV radiation, and supersonic environmental conditions.

Key words: Applique, Fluoropolymer, Corrosion inhibitors, Pressure sensitive adhesives.

INTRODUCTION

Naval and Marine Corps aircraft suffer from pitting and crevice corrosion to a much higher degree than land based counterparts. Operation from air capable ships ensures that 7000 series aluminum alloys will be exposed to the chloride ion from salty spray and air. This aggressive corrosion environment increases: sailor maintenance demands, inspection requirements, and ultimately, total costs per flight hour.

Traditionally, paint is applied as a protective and decorative approach to mitigating this corrosion. Significant progress has been made enabling modern paint systems to comply with ever increasing air quality and human protection demands. Alternatively, paintless application of

films using pressure sensitive “peel and stick” adhesive technology is another corrosion mitigation approach gaining momentum. It is our contention that a logical solution to the corrosion problems on aircraft surfaces would result from application of a *thin fluoropolymer appliqué system with revolutionary corrosion inhibiting features embedded*. One of the present drawbacks of appliqué technology is the inability to prevent the chloride ion from reaching the substrate when the appliqué is compromised, torn away, or along seams and edges. This research seeks to address these problems by incorporating an environmentally acceptable inhibitor that retards pitting, corrosion fatigue and stress corrosion cracking of aluminum alloys, into the appliqué or adhesive system.

Due to the properties such as low surface energy, low dielectric constant, high thermal stability, inertness to most chemicals and excellent weatherability, fluoropolymer films offer an excellent alternative to anti-corrosive paints and coatings. In addition they are of considerable importance in microelectronic, sensor, biomedical, textile, filtration, and many other technologies¹. Fluoropolymers are inert to most chemical treatments which is an advantage for many applications, but this property also makes adhesion of other materials to fluoropolymer substrates difficult. Therefore, methods for adhering other materials to and within fluoropolymers receive continuing attention²⁻⁵.

Integument uses unique patented surface treatment technology that allows one to permanently bond virtually any material to fluoropolymer based films including: PTFE (Polytetrafluoroethylene), FEP (tetrafluoroethylene-co-hexafluoropropylene), PFA (Polyfluorinated alkoxy), MFA (Perfluorinated Methylalkoxy), ETFE (Ethylene Trifluoroethylene), ECTFE (Ethylenechloro-trifluoroethylene), and PVDF (Polyvinylidene Fluoride). All of these fluoropolymers have different properties including thermal and chemical resistance, abrasion resistance, flexibility, surface modulus, and elongation characteristics. By using unique surface treatment technology, Integument first developed a series of fluoropolymer films and sheets that were backed with various pressure sensitive adhesives for applications in the chemical and food processing and transportation industries.

Based on efforts to date, Integument has recently been awarded one of the top achievements in the chemical processing industry. Specifically, Integument received the 2001 Vaaler Award for “Innovative Solution to Corrosion Control” at the Chem Process show held in New York City.

More recently, over the last three years, Integument has been awarded various DoD, NIH, and DARPA grants for developing novel multifunctional fluoropolymer appliqué technology (i.e., paint replacement coating systems) for applications on various Navy and Marine air and sea craft.

For example, Integument has been awarded one Phase II Navy SBIR for developing a lightning strike (E³) protection appliqué for use on composite aircraft (V-22 Osprey) and, another Phase II award for the development of a high temperature paint replacement film containing environmentally benign corrosion inhibitors for applications on aircraft like the Joint Strike Fighter. This has led to two ongoing collaborative efforts with Bell Helicopter Textron, Inc. (BHTI) and Lockheed Martin Aerospace for continued development of multifunctional appliqué coating systems. Recent efforts funded by the DARPA are investigating new nanotechnology developed at Integument for incorporating various optical and electromagnetic properties into our appliqué systems. These efforts involve collaboration with the Center for Non-Destructive Evaluation at the Johns Hopkins University with the intention of creating paintless, peel and stick coating technology that provides:

1. Corrosion protection
2. CARC compatibility

3. Various E³ characteristics
4. Controlled optical reflectance properties
5. Real-time sensing capabilities
 - a. Corrosion
 - b. Chemical and Biological Agents
 - c. Lifecycle information

The key to successful development of the appliqué technology is related to the use of fluoropolymers which provide the base material for fabricating flexible composite film structures with devices, and components incorporated and protected from severe environments by an overlying fluoropolymer film. The key to successfully using fluoropolymers is then achieved via the use of Integument's novel surface and polymer nano-technology that allows fluoropolymers to be bonded to virtually any material including metals, metal oxides, ceramics, adhesives, and other polymers.

Given the limitations with paint systems previously discussed, reduction of in-port time and manpower to perform corrosion maintenance has taken on a whole new meaning. The ever increasing environmental challenges facing the Navy demand that a corrosion mitigation system be applied that is long life, trouble-free, and that possesses other unique war fighting advantages besides corrosion protection. Integument's fluoropolymer appliqué coating technology offers the promise to significantly reduce drudgery, life cycle costs and enhance aircraft operational capability.

There are several high temperature thermoplastic resins that are currently molded into thin films of 2 mils to 20 mils thickness with unique properties that make them attractive as corrosion barrier protection systems. The baseline for these polymer systems emanates from factory applied, field proven technology that is currently being used in the chemical processing industry where the environment is extremely corrosive and failures are intolerable. The research challenge is to develop methods of modifying these fluoropolymer films and associated adhesives to simultaneously permit bonding to aircraft components, and to possess system properties that will ensure corrosion inhibition under the mechanical and environmental rigors of naval aircraft operation.

Approaches for promoting adhesive bonding of various materials, including adhesives, to fluoropolymer surfaces typically require harsh chemical reagents (highly reducing alkalies, such as sodium naphthalide) or require complex sputtering or ion beam processes^{6,7}. Other methods include cross linking fluoropolymer surfaces with x-rays followed by chemical etching⁸. These methods are often difficult to use making them commercially non-viable, environmentally problematic, and can adversely affect the chemical and morphological characteristics of the surface that leads to short term degradation.

A unique surface modification using a H₂/MeOH or H₂/H₂O radio frequency glow discharge (RFGD) plasma treatment has been demonstrated as effective for defluorinating fluoropolymers to a precisely controlled extent and then activating these sites for the addition of alcohol functionality on a variety of substrates such as poly(tetrafluoroethylene-co- hexafluoropropylene) (FEP), poly(vinylidene fluoride) (PVDF), and poly(ethylenechlorotrifluoroethylene) (ECTFE)⁹⁻¹⁰. Modification occurs at the outermost 5.0 nm of these substrates without significantly affecting the material's morphology or the carbon backbone of these polymers. Because of this, the material's hydrophobic, anti-corrosive properties can be selectively maintained on specified sides or regions of these films. Furthermore, the remaining fluorine functionality at the surface and within the bulk of these materials has substantial electron withdrawing characteristics which affect the reactivity of the added alcohol functionality. The RFGD modified surfaces are consequently quite reactive and have been shown to be facile for

covalently bonding and adhering numerous materials such as, organosilane coupling agents¹¹, adhesives^{12,13}, metals¹⁴ and polyelectrolytes (including conducting polymers)¹⁵.

SAMPLE PREPARATION

Adhesion Testing:

1. Test coupon surface preparation: aluminum 2024 QQ-A-250/4 – T3, thickness T = 0.071”
 - Test coupons were 1” x 12” x 0.071” and were thoroughly cleaned using alkaline cleaner with scotch brite (per Mil-C-87937).
 - Test coupons were chem.-filmed according to application spec Mil-C-5541 (the material used to prepare chem.-film solution conformed to Mil-C 81760, class 3, form II) and air dried.
 - Test coupons were then primed with water reducible, low density, epoxy primer Mil-PRF-85582C or DEFT 44-GN-36 to a dry thickness of 0.8 to 1.2 mils. Primer was then cured at RT for at least 10 hours.

2. Application of candidate appliqué:
 - Test panels were scuff sand primed with sand paper grit 220 or finer to remove the gloss, then cleaned with cheesecloth moistened with isopropyl alcohol, follow by a clean with dry cheesecloth.
 - Appliqué films were then applied (*with NO adhesion promoter*) according to PSTC-1.

3. Adhesion testing: all tests were performed per PSTC-1, 3 coupons per temperature or condition at room temp, 250°F, 350°F and -65°F.

4. QUV Weatherometer Tests: all tests were performed for 30 days with cycling as follows exposure to UVA source for 17 minutes and exposure to 3 minutes water spray cycle at 60°C.

5. Fluid resistance: test coupons were totally immersed in test fluids. Coupons were adhesion tested after 7 days and also 14 days. Test values were recorded at not less than < 10% of room temperature requirement. Appliqué were inspected for any sign of degradation or softening. Test fluids included:
 - Hydraulic fluid Mil-H-23699
 - Synthetic hydraulic fluid Mil-H-83282
 - Engine lube oil Mil-I-7808
 - JP-5
 - Cleaning Solvents MIL-C-38738 Type II
 - Deicing Fluid UCAR “50/50” SAE/ISO Type 1
 - 5% Aqueous Cleaning Detergent

6. Temperature stability: Appliqué system was exposed to the following temperature range.
 - 180°F to 200°F
 - 200°F to 250°F
 - 250°F to 300°F
 - 300°F to 350°F

The appliqué was inspected for signs of curling, melting or degradation. Candidate samples (3” x 3”) were applied onto clean aluminum panel. After exposure, candidate samples were then shall be peeled (hand pressure). Test results are reported as: ease of removing sample from substrate, film tore upon removal, cohesive adhesive, estimate of the amount of adhesive left on substrate (for example:

100% cohesive adhesive, 75% adhesive left on test coupon, 25% adhesive left on film; or 100% adhesive transferred to test coupon).

7. Physical Properties

1. Tensile strength:
 - a. ECTFE – 8000 psi
 - b. MFA – 4000 psi
 - c.
2. Elongation: >200% for both films.

ADHESION STRENGTH AT TEMPERATURE

Tables 1-4 list 180° adhesion peel strengths for several appliquéés and adhesive systems as described. There were triplicate test coupons per test. Materials were brought up to temperature using a thermocouple. Once the samples were at temperature for 10 minutes, peel adhesion was performed (at the specified temperature). Adhesion strength was recorded in pounds per linear inch (PLI), the locus of failure, and how much adhesive is left behind on the panel is also listed. Three different adhesives were evaluated and labeled below as:

1. Integument
2. TRI 39-3
3. TRI 45-1

The Integument adhesive is a commercially available acrylic based pressure sensitive adhesive commonly used in military applications. Specifically it goes by the commercial trade name ADCHEM 747 and its formulation contains no corrosion inhibitors. TRI 39-3 and TRI 45-1 are proprietary formulations that are acrylic based and also contain an organic, environmentally, benign vapor corrosion inhibitor. The thickness of each adhesive layer is detailed in Tables 1-4 and were either 2 or 4 mil in thickness. The fluoropolymers investigated were a clear, 2 mil MFA without pigment and a 3 mil gray pigmented ECTFE material. Both of these fluoropolymers were backed with one or several of the adhesives described above and included in the Sample description in the Tables below.

QUV STABILITY

Table 5 lists the results stability of the applique materials to ultraviolet radiation exposure. All tests were performed for 30 days with cycling as follows: (1) exposure to UVA source for 17 minutes and (2) exposure to 3 minutes water spray cycle at 60°C.

FLUID RESISTANCE

Table 6 There were three triplicate test coupons per test. Test coupons were totally immersed in each fluid. Fluoropolymer films were then tested for adhesion strength after 14 days (as listed). Adhesion strength was recorded in pounds per linear inch (PLI), with a note towards any degradation and how much adhesive was left behind on the test coupon.

TEMPERATURE STABILITY

Each test coupon was exposed to the specified temperature for one hour. After exposure and cooling to room temperature the films were inspected and then hand peeled.

Report:

1. Ease of removing film from test coupon.

2. Note the locus and type of failure, i.e. (cohesive versus adhesive failure, film tearing, how much adhesive is left on the test coupon).

SUMMARY

One of the objectives of this ongoing work was to compliment both high temperature stability with adhesion that would allow easy application, removal and repair of the appliqué system. Clearly the current appliqué systems have demonstrated the thermal stability requirements, which were targeted including the development of two new adhesive systems that contain environmentally friendly corrosion inhibitors. The development of these adhesives also provided additional benefits due to:

1. Better adhesion with respect to easy application and removal (ca. 7 pli at room temperature as opposed to 15 pli for the original adhesive system).
2. Thermal stability throughout -65° to 350° range with adequate adhesion and removal properties. For example, the original adhesives were stable throughout the temp. range, however removal of the film resulted in almost 100% of the adhesive being left behind on the test panel at all temps except room temperature. The new adhesives demonstrated better bonding tenacity to the fluoropolymer appliqué material than to the test panels (i.e., > 7 pli to fluoropolymer; and $= 7$ pli to the 2024-T surface), thus effecting almost total removal of appliqué and adhesive from test panels even at temps throughout the investigated range. (Note: there is still a problem at -65° with respect to leaving adhesive behind on test panel).
3. Additionally, the new adhesives showed outstanding resistance to various fluids listed in this report even under total immersion for 14-day tests.
4. The new adhesives and appliqué showed outstanding resistance to QUVA weathering
5. Although not tested yet, the new adhesives have been formulated to contain an environmentally benign corrosion inhibitor.

In conclusion the results reported in this report demonstrate an appliqué system having characteristics that make it a viable alternative to currently used aircraft paint and coating systems.

FUTURE WORK

1. Further adhesive development in order to provide the best corrosion, temperature, and fluid resistance, coupled with the best characteristics for easy application and repair. Specifically, the optimal goal is to provide a multifunctional applique system that can be completely (i.e., with adhesive intact on fluoropolymer) removed and replaced.
2. Measurement of material lifecycles and factors that can be used to determine the exact point or age of the appliqué within its lifecycle at any given time. We are currently working on the incorporation of real time sensors that will allow one to monitor (among other things) appliqué lifecycle and corrosion onset to underlying substrates.
3. Development of non-destructive techniques for measuring exact age of appliqué within its lifecycle in-situ, (i.e., we will determine the lifetime of the appliqué system in conjunction with new non-destructive techniques for measuring the discreet stage of the appliqué lifecycle).

4. Development of non-destructive techniques for determining underlying disbonds or damage to underlying structures. For example, we would envision incorporating a BVID (Barely Visible Induced Damage) capability directly into the appliqué system that would correlate to underlying composite damage).

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TABLES

Table 1
Room Temperature Testing
(180° Peel Adhesion)

<u>Temperature</u>	<u>Sample</u> (3 sample avg.)	<u>Peel PLI/Failure</u>	<u>Failure Mode</u>	<u>% Adhesive Left</u>
Room Temperature	2 mil MFA 4 mil ADCHEM 747	9.4 pli (1 day)	Adhesive failure between appliqué and adhesive layer.	95% of adhesive left on primed 2024 substrate
Room Temperature	3 mil ECTFE 2 mil ADCHEM 747	14.8 pli (1 day)	Adhesive failure between appliqué and adhesive layer.	95% of adhesive left on primed 2024 substrate
Room Temperature	3 mil ECTFE 4 mil TRI 39-3	8.4 pli (1 day)	Adhesive failure between the substrate and the adhesive layer.	No adhesive left on primed 2024 substrate
Room Temperature	2 mil MFA 4 mil TRI 45-1	7.0 pli (1 day) 7.9 pli (4 day) 8.0 pli (7 day)	Adhesive failure between the substrate and the adhesive layer.	No adhesive left on primed 2024 substrate

Table 2
250° Temperature Testing
(180° Peel Adhesion)

<u>Temperature</u>	<u>Sample</u> (3 sample avg.)	<u>Peel PLI/Failure</u>	<u>Failure Mode</u>	<u>% Adhesive Left</u>
250°F Temperature	3 mil ECTFE 2 mil ADCHEM 747	1.9 pli	Cohesive failure within the adhesive layer.	50% of adhesive left on primed 2024 substrate
250°F Temperature	3 mil ECTFE 4 mils TRI 39-3	1.4 pli	Cohesive failure within the adhesive layer.	50% of adhesive left on primed 2024 substrate
250°F Temperature	2 mils MFA 4 mil TRI 39-3	1.5 pli	Cohesive failure within the adhesive layer.	50% of adhesive left on primed 2024 substrate

Table 3
350° Temperature Testing
(180° Peel Adhesion)

<u>Temperature</u>	<u>Sample</u> (3 sample avg.)	<u>Peel PLI/Failure</u>	<u>Failure Mode</u>	<u>% Adhesive Left</u>
350°F Temperature	2 mil MFA 4 mil ADCHEM 747	1.2 pli	Cohesive failure within the adhesive layer.	60% of adhesive left on primed 2024 substrate
350°F Temperature	3 mil ECTFE 2 mil ADCHEM 747	1.1 pli	Cohesive failure within the adhesive layer.	60% of adhesive left on primed 2024 substrate
350°F Temperature	3 mil ECTFE 4 mils TRI 39-3	0.7 pli	Cohesive failure within the adhesive layer.	60% of adhesive left on primed 2024 substrate
350°F Temperature	2 mil MFA 4 mil TRI 39-3	1.4 pli	Cohesive failure within the adhesive layer.	50% of adhesive left on primed 2024 substrate
350°F Temperature	2 mil MFA 4 mil TRI 45-1	0.4 pli	Cohesive adhesive failure. No film tearing.	50% of adhesive left on primed 2024 substrate

Table 4
-65° Temperature Testing
(180° Peel Adhesion)

<u>Temperature</u>	<u>Sample</u> (3 Sample Avg.)	<u>Peel PLI/Failure</u>	<u>Failure Mode</u>	<u>% Adhesive Left</u>
-65°F Temperature	2 mil MFA 4 mil ADCHEM 747	7.3 pli	Adhesive failure between the film and the adhesive layer.	95% of adhesive left on primed 2024 substrate
-65°F Temperature	3 mil ECTFE 2 mil ADCHEM 747	13.5 pli	Adhesive failure between the film and the adhesive layer.	95% of adhesive left on primed 2024 substrate
-65°F Temperature	3 mil ECTFE 4 mil TRI 39-3	7.0 pli	Adhesive failure between the film and the adhesive layer.	95% of adhesive left on primed 2024 substrate
-65°F Temperature	2 mil MFA 4 mils TRI 39-3	3.9 pli	Adhesive failure between the film and the adhesive layer.	95% of adhesive left on primed 2024 substrate
-65°F Temperature	2 mil MFA 4 mil TRI 45-1	11.24 pli	Adhesive failure between the film and the adhesive layer.	95% of adhesive left on primed 2024 substrate

Table 5
60° QUV Resistance Testing
(180° Peel Adhesion)

<u>Temperature</u>	<u>Sample</u> (3 sample avg.)	<u>Peel Failure</u>	<u>Failure Mode</u>	<u>% Adhesive Left</u>
60°C QUV Weatherometer	2 mil MFA 4 mil TRI 45-1	5.5 pli (30 day)	Failure between adhesive and primed substrate, film tear after 2 inches of peel	No adhesive left on primed 2024 substrate

Table 6
Fluid Resistance Testing
(180° Peel Adhesion)

<u>Fluid</u>	<u>Sample</u> (3 sample avg.)	<u>Peel Failure</u>	<u>Failure Observations</u>	<u>% Adhesive Left</u>
Hydraulic Fluid MIL-H-23699	3 mil ECTFE 4 mils TRI 39-3	4.7 pli (14 days)	Adhesive failure between the adhesive and the primed substrate, no film degradation	No adhesive left on primed 2024 substrate
Hydraulic Fluid MIL-H-23699	2 mil MFA 4 mils TRI 39-3	4.5 pli (14 days)	Adhesive failure between the adhesive and the primed substrate, no film degradation	No adhesive left on primed 2024 substrate
Hydraulic Fluid MIL-H-23699	2 mil MFA 4 mil TRI 45-1	5.1 pli (14 days)	Failure between the adhesive and the primed substrate, no film degradation	No adhesive left on primed 2024 substrate
Hydraulic Fluid MIL-H-23699	3 mil ECTFE 2 mil ADCHEM 747	13.3 pli (14 days)	Failure cohesive in the adhesive, no film degradation	70% of adhesive left on primed 2024 substrate
Hydraulic Fluid MIL-H-23699	2 mil MFA 4 mil ADCHEM 747	Film Broke adhesion higher than the cohesive strength of the film	NA	NA
Engine Lube Oil MIL-I-7808	3 mil ECTFE 4 mils TRI 39-3	5.0 pli (14 days)	Failure between the primed substrate and the adhesive, film no degradation	No adhesive left on primed 2024 substrate
Engine Lube Oil MIL-I-7808	2 mil MFA 4 mil TRI 39-3	4.3 pli (14 days)	Failure between the primed substrate and the adhesive, film no	No adhesive left on primed 2024 substrate

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Table 6 - Continued
 Fluid Resistance Testing
 (180° Peel Adhesion)

<u>Fluid</u>	<u>Sample</u> (3 sample avg.)	<u>Peel Failure</u>	<u>Failure Observations</u>	<u>% Adhesive Left</u>
Engine Lube Oil MIL-I-7808	2 mil MFA 4 mil TRI 45-1	4.1 pli (14 days)	Failure between substrate and the adhesive, film no degradation	No adhesive left on primed 2024 substrate
Engine Lube Oil MIL-I-7808	3 mil ECTFE 2 mil ADCHEM 747	13.0 pli (14 days)	Adhesive failure between the film and the adhesive layer, no film tearing.	85% of adhesive left on primed 2024 substrate
Engine Lube Oil MIL-I-7808	2 mil MFA 4 mil adhesive	Film failed during pull, adhesion greater than cohesive strength of the film	NA	NA
Synthetic Hydraulic Fluid MIL-H-83282	3 mil ECTFE 4 mils TRI 39-3	5.2 pli (14 days)	Failure between substrate and the adhesive, film no degradation	No adhesive left on primed 2024 substrate
Synthetic Hydraulic Fluid MIL-H-83282	2 mils MFA 4 mils TRI 39-3	3.2 pli (14 days)	Failure between substrate and the adhesive, film no degradation	No adhesive left on primed 2024 substrate
Synthetic Hydraulic Fluid MIL-H-83282	3 mil ECTFE 2 mil ADCHEM 747	13.0 pli (14 days)	Failure between the adhesive and the film, film no degradation	95% of adhesive left on primed 2024 substrate
Synthetic Hydraulic Fluid MIL-H-83282	2 mils MFA 4 mil ADCHEM 747	Film broke, adhesion greater than the cohesive strength of the film	NA	NA
JP-8	3 mils ECTFE 4 mils TRI 39-3	3.5 pli (14 days)	Adhesive failure between the adhesive and substrate, no film degradation	10% of adhesive left on primed 2024 substrate
JP-8	2 mil MFA 4 mil TRI 39-3	3.7 pli (14 days)	Cohesive failure in the adhesive, no film degradation	70% of adhesive left on primed 2024 substrate
JP-8	2 mil MFA 4 mil TRI 45-1	4.5 pli (14 days)	Adhesive failure between the adhesive and substrate, no film degradation	20% of adhesive left on primed 2024 substrate
JP-8	3 mil ECTFE 2 mil ADCHEM 747	5.0 pli (14 days)	Failure between the adhesive and the film, film no degradation	90% of adhesive left on primed 2024 substrate

Table 6 - Continued
 Fluid Resistance Testing
 (180° Peel Adhesion)

<u>Fluid</u>	<u>Sample</u> (3 sample avg.)	<u>Peel Failure</u>	<u>Failure Observations</u>	<u>% Adhesive Left</u>
JP-8	2 mil MFA 4 mil ADCHEM 747	Film broke during peel, adhesion greater than cohesive strength of the film	NA	NA
Cleaning Solvents MIL-C-38738 Type II	2 mil MFA 4 mil TRI 39-3	0.1 pli (14 days)	Failure between the film and the adhesive, film no degradation	100% of adhesive left on primed 2024 substrate
Cleaning Solvents MIL-C-38738 Type II	2 mil MFA 4 mil TRI 45-1	0.1 pli (14 days)	Failure between the film and the adhesive, film no degradation	100% of adhesive left on primed 2024 substrate
Deicing Fluid UCAR "50/50" SAE/ISO Type 1	2 mil MFA 4 mil 39-3	4.8 pli (14 days)	Failure between the adhesive and the substrate, film no degradation	10% of adhesive left on primed 2024 substrate
Deicing Fluid UCAR "50/50" SAE/ISO Type 1	2 mil MFA 4 mil TRI 45-1	5.1 pli (14 days)	Failure between the adhesive and appliqué, film no degradation	50% of adhesive left on primed 2024 substrate
5% Aqueous Cleaning Detergent	2 mil MFA 4 mil TRI 39-3	4.9 pli (14 days)	Failure between the adhesive and appliqué, film no degradation	75% of adhesive left on primed 2024 substrate
5% Aqueous Cleaning Detergent	2 mil MFA 4 mil TRI 45-1	5.5 pli (14 days)	Failure between the appliqué and adhesive, film no degradation	80% of adhesive left on primed 2024 substrate

Table 7
 1Hour Temperature Stability Testing
 (180° Peel Adhesion)

<u>Temperature</u>	<u>Sample</u> (3 samples avg.)	<u>Peel Failure</u>	<u>Failure Observations</u>	<u>% Adhesive Left</u>
180°F	3 mil ECTFE 4 mils TRI 39-3	Cohesive failure in the adhesive layer.	Difficult to peel, film looks fine no tearing of film	50% of adhesive left on primed 2024 substrate
180°F	2 mil MFA 4 mil TRI 39-3	Cohesive failure in the adhesive layer.	Difficult to peel, film looks fine no tearing of film	50% of adhesive left on primed 2024 substrate
180°F	2 mil MFA 4 mil TRI 45-1	Adhesive failure between the appliqué film and the adhesive layer	Difficult to peel, tearing of film	90% of adhesive left on primed 2024 substrate

Table 7 - Continued
 1Hour Temperature Stability Testing
 (180° Peel Adhesion)

180°F	3 mil ECTFE 2 mil ADCHEM 747	Adhesive failure between the appliqué film and the adhesive layer	Adhesive failure between the appliqué and adhesive layer, difficult removal	90% of adhesive left on primed surface
180°F	2 mil MFA 4 mil ADCHEM 747	Adhesive failure between the adhesive and the appliqué film	Film tore, adhesion exceeded the cohesive strength of the film, difficult removal	100% of adhesive left on primed aluminum
250°F	3 mil ECTFE 4 mils TRI 39-3	Cohesive failure within the adhesive layer.	No film tearing, hard to remove, no degradation of film evident	50% adhesive left on primed 2024 substrate
250°F	2 mil MFA 4 mil TRI 39-3	Cohesive failure within the adhesive layer.	Some film tearing, easy to remove, no degradation of film evident	60% adhesive left on primed 2024 substrate
250°F	2 mil MFA 4 mil TRI 45-1	Cohesive failure in the adhesive layer.	No film tearing, easy to remove	50% of adhesive left on surface of primed substrate
250°F	3 mil ECTFE 2 mil ADCHEM 747	Failure between the appliqué and the adhesive layer.	No film tearing, easily removed, no degradation of film evident	95% of adhesive left on primed 2024 substrate
250°F	2 mil MFA 4 mil ADCHEM 747	Cohesive failure in the adhesive layer	No film tearing, easily removed, no degradation of film evident	50% of adhesive left on primed 2024 substrate
350°F	3 mil ECTFE 4 mils TRI 39-3	Cohesive failure within the adhesive layer	No film tearing, easily removed, no degradation of film evident	40% of adhesive left on primed 2024 substrate
350°F	2 mil MFA 4 mil TRI 39-3	Cohesive failure within the adhesive layer	No film tearing, easily removed, no degradation of film evident	50% of adhesive left on primed 2024 substrate
350°F	3 mil ECTFE 2 mil ADCHEM 747	Failure between the appliqué and the adhesive layer.	Relatively easy removal, no film tearing or film degradation	100% of adhesive left on primed 2024 substrate
350°F	2 mil MFA 4 mil ADCHEM 747	Cohesive failure in the adhesive layer	Relatively easy removal, no film tearing or thermal degradation of film.	65% of adhesive left on primed 2024 substrate