FLUOROPOLYMER COATINGS FOR PREVENTION OF CORROSION

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ABSTRACT

Fluoropolymers have been used in coatings for more than forty years. These materials offer excellent weatherability, corrosion resistance, and stain resistance. However, fluoropolymers generally must be heated to form coatings. This makes field application extremely difficult usually limiting their use to coil coatings. A newer class of fluoropolymers, fluoroethylene vinyl ether (FEVE) resins was developed to overcome these problems. FEVE resins incorporate both fluoropolymer and reactive groups. This enables them to be used for either field or shop application, while still retaining many of the positive characteristics of fluoropolymers including weatherability. Initial applications of FEVE based coatings (mainly fluorourethanes) were in the architectural market, where their excellent gloss and color retention was desirable. FEVE resins are increasingly used to provide both weatherability and corrosion protection for metal industrial structures like bridges and offshore oil and gas platforms. Topcoat life exceeding 30 years is possible using FEVE resins. Because of this, fluorourethane topcoats are required on all bridges in Japan. FEVE coatings are used on vehicles and on military and civilian aircraft as well. FEVE resins are used in the Air Force's Advanced Performance Coating. The ultra-weatherability that makes FEVE coatings useful in architectural markets also contributes to their ability to protect structures, vehicles, and aircraft from corrosion.

Keywords: Fluoropolymer, FEVE, resin, weatherability

INTRODUCTION

Fluoropolymers have been used for many years to manufacture coatings and linings. They offer a number of desirable physical properties, including weatherability, corrosion resistance, and chemical resistance. These properties arise from the nature of the chemical bond formed by fluorine with carbon. The strength of this bond is too great to be broken by ultraviolet (UV) radiation (weatherability) and by chemicals (chemical resistance). Examples of fluoropolymers used in coatings and linings include poly(tetrafluoroethylene) (PTFE), polyvinyl fluoride (PVF), ethylene tetrafluoroethylene (ETFE) and polyvinylidene fluoride (PVDF). Of these, PVDF has found the widest application in thin film coatings.

The use of most fluoropolymers in coatings has been limited to applications where they can be softened, solubilized, or melted to form coatings and linings. This requirement usually limits their use to shop or factory applied coatings. PVDF coatings are formed by melting and fusing a blend of PVDF and an acrylic resin (added to improve adhesion) at temperatures of about 300^{o} C. PVDF coatings are primarily found in architectural applications, where their excellent weatherability, in excess of 30 years, makes them particularly useful. However, traditional PVDF coatings are not usable for field application; rather, metal components are shop coated with PVDF, then fabricated in the field.

In the early 1980's, a new type of fluoropolymer resin was developed in Japan. These resins, generically known as fluoroethylene vinyl ether (FEVE) resins, combined the weatherability and corrosion resistance of fluoropolymers with the handling and reactivity of polyurethanes. Fluorourethane coatings made with FEVE coatings overcome the disadvantages of traditional fluoropolymers. They are extensively used in field applied coatings for industrial maintenance and architectural markets, matching the weatherability of PVDF coatings, but also providing excellent corrosion resistance.

PROPERTIES AND APPLICATIONS OF FLUOROETHYLENE VINYL ETHER RESINS

FEVE resins have a unique chemical structure that leads to the outstanding weatherability and corrosion resistance of fluorourethane coatings. The structure is shown below in Figure 1.

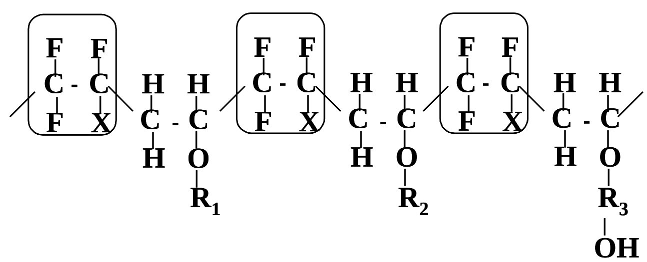


Figure 1: Structure of FEVE Resins

The FEVE polymer consists of regularly alternating fluoroethylene and vinyl ether units. The unique structure allows the fluorinated units to protect the polymer from degradation by UV light and chemicals. The vinyl ether units are chosen to make the FEVE polymer soluble in common solvents like xylene, MEK, MAK, MIBK, esters, and Oxsol® The vinyl ether units can be changed to affect coating properties like flexibility, toughness, and chemical resistance. The vinyl ether units can also be functionalized with hydroxyl groups. This enables the FEVE polymer to be crosslinked with aliphatic isocyanates to form fluorourethanes. These coatings can be sprayed with standard paint application equipment.

Properties of FEVE Resins

FEVE resins have a number of characteristics that offer improvements over conventional fluoropolymer resins. First, FEVE resins are solvent soluble. This makes them easy to handle and apply. The clear FEVE resin solution has excellent pigment compatibility, and enables the use of a wide range of pigments. Colors of the FEVE solutions are crisp and clean; this effect can be difficult to achieve with other fluoropolymer resins. FEVE resins can be used to make coatings with a wide range of gloss, from a high of 90 at 60^o down to a completely flat finish at a 5 gloss. FEVE resins are thermoset, which means they are crosslinked and cured during application. They can be used for field applied coatings where the use of other fluoropolymer coatings can be limited. However, they can also be factory or shop applied.

Types of FEVE Resins

Because the structure of FEVE resins can be varied so easily, a wide variety of resin types can be manufactured. Traditional FEVE resins are supplied in xylene. In order to meet VOC regulations throughout the U. S., these resins have been supplemented by solid resins which can be dissolved in VOC-free and HAPS exempt solvents. Both traditional and solid FEVE resins have been used in military aerospace applications; the Air Force's Advanced Performance Coating is based on FEVE resins. A flexible resin dissolved in special solvents is used for coil coatings, where precoated metal components are coated at high temperatures and fabricated in the field. There are also powder coating FEVE resins. While these resins are typically used for architectural applications, there is increased use of powder coatings for military applications, mostly for interior coatings at present. FEVE resins can be designed as water emulsions. These resins usually don't weather as well as solvent or powder grade resins. The primary applications for FEVE water emulsion resins have been as modifiers for other water based resins to improve weatherability. An FEVE water dispersion resin was introduced several years ago. This resin appears to offer weatherability that matches that of traditional FEVE resins.

Applications for FEVE Resins

FEVE resins are usually formulated into fluorourethane coatings. Markets for fluorourethane coatings include architectural, industrial maintenance, aerospace, automotive, and alternative energy. Typical applications are summarized below.

Architectural. FEVE resins are used to make coil coatings that are applied to metal composite panels for commercial and industrial buildings. These are shop applied coatings. FEVE resins can also be used for field applied air dry fluorourethane coatings useful for repainting existing buildings and other structures. FEVE powder coatings are used to replace liquid fluoropolymer coatings for aluminum extrusions, used mainly for window frames. Fluoropolymer architectural coatings are usually formulated to meet the American Architectural Manufacturer's Association (AAMA) specification AAMA 2605. The coating portion of this specification requires at least 10 years of South Florida weathering with >50% gloss retention, and a maximum color change of 5ΔE.

Industrial Maintenance. FEVE resins are widely used on industrial structures, including water towers, bridges, and offshore platforms. For these applications, corrosion protection in addition to coating weatherability is critical. These coatings are usually 3-coat systems, with a zinc rich primer, an epoxy or urethane mid-coat, and a fluorourethane topcoat. In most cases, these are field applied coatings, although new bridge construction involves shop coating a significant portion of the components.

Aerospace. FEVE resins are used in the Air Force's Advanced Performance Coating system. Because military aircraft are repainted on a regular basis, 30 years of durability are not required. For this application, FEVE resins are blended with standard aerospace coating resins to improve weatherability and color retention. The resins are beginning to find a place in commercial aerospace as well. Fluorourethanes have been approved for use on the Boeing 787, which contains large amounts of composite materials.

Automotive. FEVE resins because of their high cost are not typically used in the automotive industry. However, they have found a place used in blends as an appearance coating for the Chevrolet Cruze, General Motors' new small car platform.

Alternative Energy. Fluorourethane coatings are finding application in solar and wind power markets. Both solar panels and wind towers have an expected life of around 30 years. FEVE coatings are capable of meeting and exceeding this lifetime requirement. On solar panels, FEVE coatings are used to coat polymers that are used to protect the solar panels from damage. Many times these polymers are not stable to UV light. On wind towers, FEVE coatings can be used to protect metal and fiberglass components from corrosion and degradation by UV light.

WEATHERABILITY OF FEVE FLUOROURETHANE COATINGS

FEVE resin based coatings, when exposed in both accelerated and natural weathering tests, offer durability that exceeds that of most competitive coatings. In many cases, FEVE coatings applied to structures such as bridges are meant to last for almost 60 years.

The weatherability of FEVE resins is due to the nature of the fluorine chemical bond, and to the unique alternating polymer structure. Fluorine forms extremely strong bonds with other elements. In addition, fluorine also increases the bond strength of chemical bonds adjacent to it. In all cases, the bond strengths exceed the maximum energy available in UV light, 411 kJ/mol. Table 1 below illustrates this point.

Table 1: Chemical Bond Strengths in FEVE Polymers

Resin Type	C-C Bond Type	C-C Bond Strength, kJ/mol	C-F, C-H Bond Types	C-F, C-H Bond Strength, kJ/mol
Fluoropolymer	CF_3-CF_3	414	F-CF_2-CH_3	523
Fluoropolymer	CF_3-CH_3 (1)	424	CF_3-CH_2-H (3)	447
Hydrocarbon	CH_3-CH_3 (2)	379	CH_3-CH_2-H (4)	411

Data from Table 1 show that in all cases, the fluorine bonds exceed the maximum UV energy of 411 kJ/mol. However, it also shows that the bond strength of C-C bond (1) is much higher than that of C-C bond (2). The C-H (3) bond strength is also higher than that of the C-H bond (4). In the FEVE polymer, each fluorinated unit alternates with a vinyl ether unit. In all cases, there is a fluorinated unit adjacent to each vinyl ether unit. This serves to increase the relatively weaker bond strengths in the vinyl ether, reducing the ability of UV light to degrade the polymer.

Accelerated Weathering Tests

Accelerated weathering tests for coatings have been developed to accelerate the screening process for coatings. The goal is to gather information in months in an accelerated test compared to years required for natural weathering. Two such tests are discussed below.

QUV-A Weathering Test. A fluorourethane coating was submitted for exposure in the QUV-A weathering test. In this test, a coating is exposed to a specific wavelength of UV radiation, as well as cyclic exposure to moisture to simulate exposure to rain. Results are shown below in Figure 2.

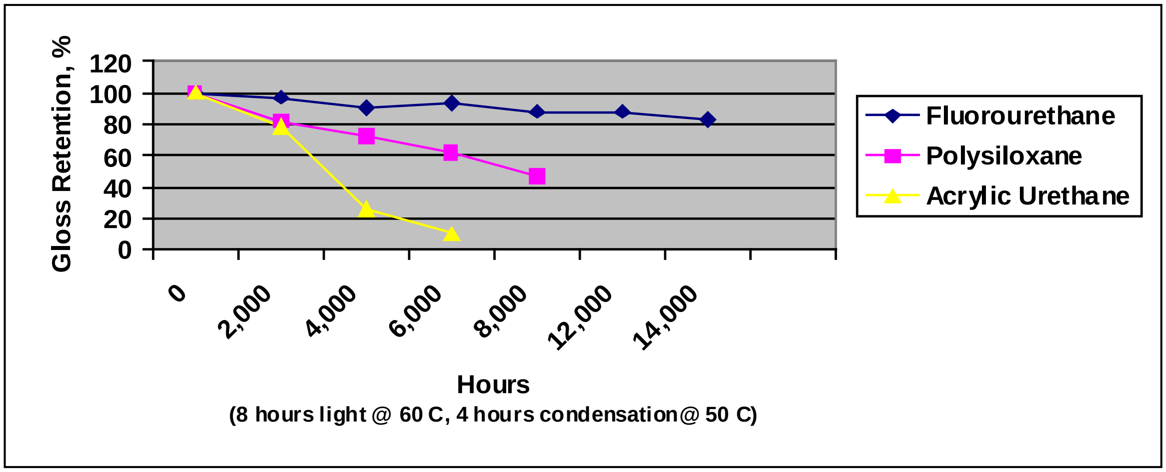


Figure 2: QUV-A Weathering of Fluorourethane Coatings

The fluorourethane coating offers excellent gloss retention even after 15,000 hours of exposure in the QUV-A Weatherometer. It outperforms both the standard acrylic urethane and the polysiloxane coatings.

Equatorial Mount With Mirrors for Acceleration With Water (EMMAQUA Test). The major problem with many accelerated weathering tests is that they expose the coating to only one wavelength of light. Sunlight consists of many different wavelengths of UV light, some of which are more damaging than others. The EMMAQUA test was developed to solve this problem. In this test, 10 mirrors focus and concentrate sunlight on the sample panels. The coatings are exposed to the entire range of wavelengths of radiation found in natural light. They are periodically exposed to deionized water to simulate exposure to rain. Results are reported as energy exposure per unit area (megaJoules/m^2) rather than in time of exposure. Figure 3 below shows test results for a fluorourethane, an acrylic urethane, and a PVDF coating.

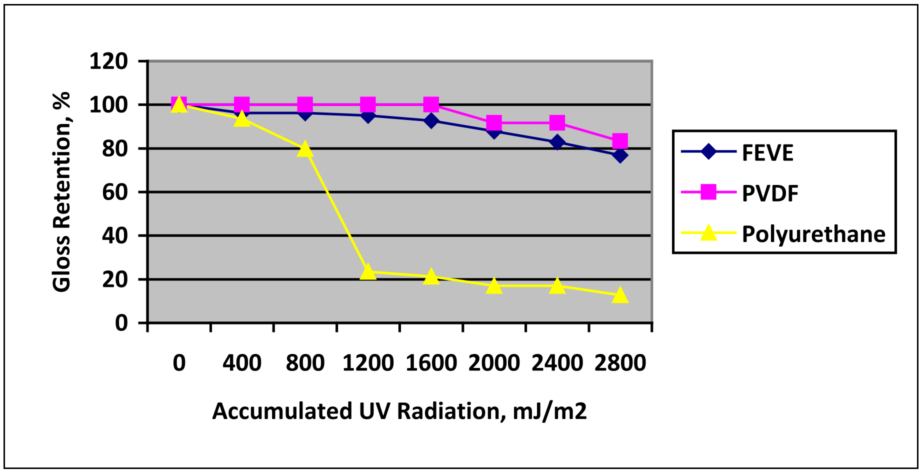


Figure 3: EMMAQUA Accelerated Test Results

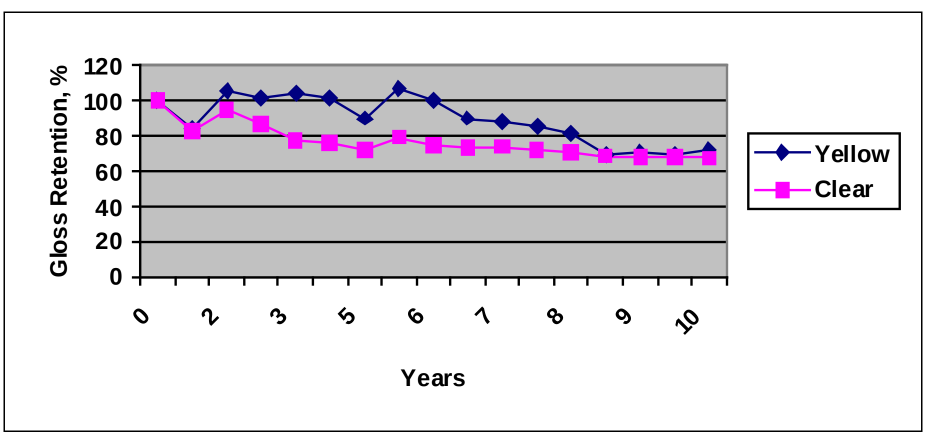
As expected, the fluorourethane and the PVDF coating outperformed the acrylic urethane.

Accelerated weathering tests all suffer from some disadvantages. Most likely, they are most useful for comparative testing rather than developing absolute performance standards. For that, natural exposure tests are required.

Natural Exposure Weathering Tests

Natural weathering is the best method of determining the long term performance of coatings. However, with extremely weatherable coatings like fluorourethanes, these tests take many years to develop useful data. Natural exposure tests for coatings are usually run in tropical, subtropical, or desert environments due to the strength and duration of UV exposure. In the U. S., the best known site for testing coatings is in South Florida.

<u>South Florida Weathering of FEVE Coatings.</u> Two FEVE fluorourethane coatings were placed in South Florida weathering. Figure 4 below shows gloss retention after 10 years of exposure there.



Location: Miami, FL
Exposure: Direct, 30 degrees South, Open Back

Figure 4: South Florida Exposure of Fluorourethane Coatings

Gloss retention exceeds 65% for both the clear and pigmented FEVE coating. Over the 10 year test period, the color change of the yellow coating was 1.5ΔE.

Okinawa Weathering of Fluorourethane Coatings. In Japan, coatings are usually tested on Okinawa, which lies at the same latitude as Jacksonville, FL. Since Okinawa is an island, it is thought that exposure there is more severe than that found in mainland based sites. Figure 5 below shows results from an extended exposure test of a fluorourethane coating.

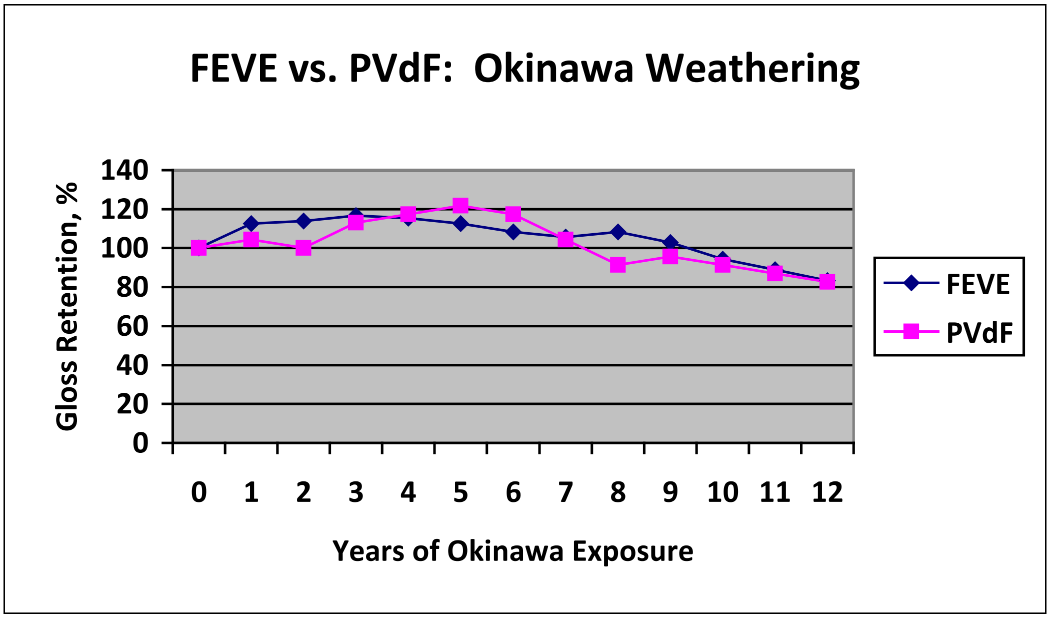


Figure 5: Okinawa Weathering of Fluorourethane Coating

Okinawa exposure results show that both the FEVE fluorourethane and the PVDF fluoropolymer coating weather extremely well in the harsh marine environment.

Weatherability of Fluorourethane Coatings on Structures

Ultimately, weatherability of a coating is determined by its performance in real life. FEVE coatings have been used for more than 25 years on hundreds of thousands of buildings, bridges, aircraft, and automobiles. A number of structures coated with fluorourethanes in Japan have been monitored for more than 20 years for gloss and color change.

Weathering of Fluorourethane Topcoat on the Tokiwa Bridge. The Tokiwa Bridge, located in a mountainous area of Japan, was recoated with a fluorourethane topcoat in 1986. Periodically, photos were taken to assess gloss and color retention. Photos of the bridge over time are shown below.

October 1988 April 1993 July 2007

Figure 6: Photos of Tokiwa Bridge Over 21 Years

The appearance of the coating the photograph taken after 21 years is indistinguishable from that taken two years after the topcoat was applied.

After the last photograph was taken, gloss and color of the coating were measured and compared to the original data obtained in 1986. Results are shown below in Table 1.

Table 2: Color and Gloss Change in Fluorourethane Topcoat after 21 Years

	Gloss Retention, %	Color Change, ΔE
Unwashed Coating	92%	3.5
Washed Coating	100%	2.3

As the figures in Table 1 shows, gloss and color of the fluorourethane topcoat is virtually unchanged after 21 years.

CORROSION PREVENTION BY FEVE FLUOROURETHANE COATINGS

While appearance is important on industrial structures like bridges, the main purpose for using protective coatings is to prevent corrosion. Regardless of appearance, corrosion will reduce the usefulness of the asset such as a bridge or offshore rig over a short period of time if not combated. In most cases, FEVE fluorourethane topcoats are used in coating systems including a zinc rich primer and an epoxy or urethane middle coat. The zinc rich primer is present to protect steel substrates by preferentially corroding in the presence of corrosion initiators such as oxygen and chloride ion. The primer is the last line of defense against corrosion; it is usually called into service when the topcoat and middle coat are damaged. The topcoat, in addition to maintaining good coating appearance, also acts as a barrier to corrosion initiators.

Degradation of Fluorourethane Coatings

Coatings are degraded when the chemical bonds in their constituent polymers are broken by UV light or chemical exposure. The coating polymers are broken into lower molecular weight entities that can be removed from the surface by wind, rain and other environmental factors. Over time, the coating loses thickness which reduces its effectiveness to act as a barrier to corrosion.

Fluorourethane coatings do degrade over time, but only very slowly. As mentioned earlier, the carbon-fluorine chemical bonds are extremely strong. The energy it takes to break these bonds exceeds that available from UV light. In addition, the unique alternating structure of the FEVE polymer protects other bonds in the polymer from degradation.

There have been both accelerated and natural weathering tests performed on FEVE coatings that demonstrate the superiority of the coatings to conventional urethane coatings.

Comparative Degradation of Fluorourethane Coatings: Sunshine Weatherometer Test. Four different types of coatings were exposed to 2,000 hours in the Sunshine Weatherometer Accelerated Weathering test. The coating surfaces were inspected using scanning electron microscopy before and after exposure. Results are shown below in Figure 7.

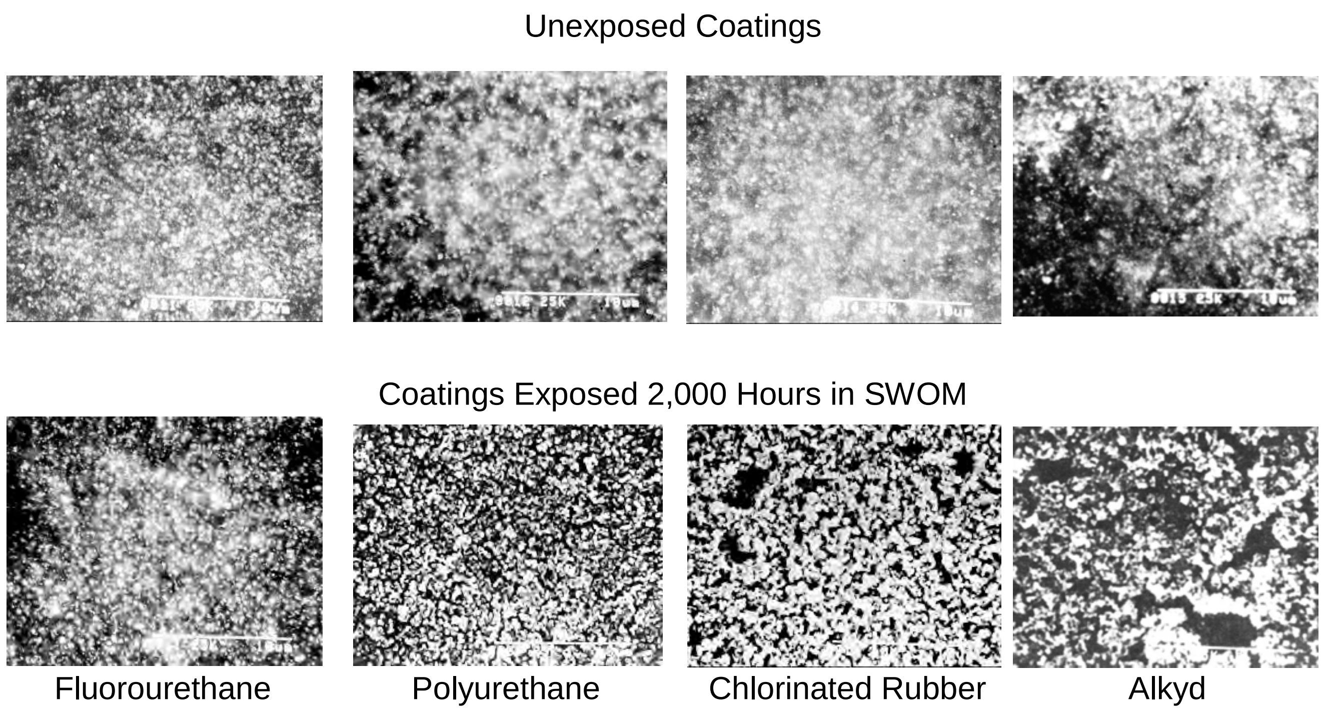


Figure 7: Comparative SEM Scans: 2,000 Hours SWOM Exposure

In the scans, the constituent polymers of each topcoat are seen as fuzzy white areas. After exposure, the fluorourethane coating shows a small amount of change at the surface. In contrast, the scan of the polyurethane topcoat shows virtually no polymer remaining at the surface. The white seen in the scan is the TiO_2 pigment unsupported by any polymer. The chlorinated rubber and alkyd are even more degraded. Cracks and crevices can be seen in the surfaces of these topcoats. Topcoat degradation leads to increased penetration of corrosion initiators like oxygen, water, and chloride ion, reducing the ability of the coating to prevent corrosion.

Corrosion Prevention by FEVE Coatings

Sunshine Weatherometer Test: Permeability of Coatings to Oxygen. Materials like oxygen, chloride ion, and water can help initiate corrosion of metals. Accelerated weathering of a fluorourethane coating and an acrylic urethane coating demonstrate the superiority of the fluorourethane in allowing permeation of oxygen through the coating. In this experiment, oxygen permeability of a 25 µm topcoat of an FEVE coating and an acrylic urethane coating were measured before and after accelerated weathering. Results are shown below in Table 2.

Table 3: Permeability of FEVE Coatings to Oxygen

Oxygen Permeability	Fluorourethane	Acrylic Urethane
0 Hours (Before Exposure)	4.2×10^{-11}	2.6×10^{-10}
After 5,000 Hours SWOM	4.5×10^{-11}	Coating failure at 2,000 hours
Exposure in Sunshine Carbon Arc Weatherometer, cycling exposure temperature, humidity, and water spray. Permeability coefficients in cc•cm/cm•cm•sec•cm Hg.		

The initial permeability of the FEVE coating is about a factor of 10 lower than that of the acrylic urethane. After 5,000 hours in the SWOM, the fluorourethane experiences only a small increase in permeability to oxygen, while the acrylic urethane is completely degraded after only 2,000 hours of exposure. The test results indicate that the fluorourethane will continue to prevent oxygen to penetrate the coating even after weathering.

Natural Weathering: Change in Fluorourethane Coating Thickness. Test panels with an inorganic zinc rich primer (75 µm, 3 mils), epoxy middle coat (150 µm, 6 mils), and either a fluorourethane or acrylic urethane topcoat (25 µm, 1 mil) were placed on an offshore platform and exposed in this natural environment for over 15 years. The test site in Japan is one of 47 around the world used in ISO 9223, which classifies the corrosivity of an environment based on elements of exposure like time of wetness, and pollution (sulfur dioxide, etc.). Changes in topcoat thickness were measured over the life of the test. The results of the test are shown below in Table 3.

Table 4: Natural Weathering Test Results: Fluorourethane vs. Acrylic Urethane

Coating Type	Change in Coating Thickness, µm/year	Coating Thickness After 15 Years	Theoretical Topcoat Life
Fluorourethane	0.20	22 µm	>60 Years
Acrylic Urethane	2.1	0	12 Years
Test platform at Suruga Bay, Japan, 34.85 North Latitude.			

During the first seven years of the test, the fluorourethane topcoat lost no thickness. For the remaining eight years, the FEVE coating lost an average of 0.38 µm (0.015 mils) per year, for an overall annual thickness loss of 0.20 µm (0.008 mils). The theoretical life of this 25 µm topcoat is more than 60 years. Of course, topcoat thickness is typically 50-100 µm, which means lifetimes exceeding 60 years are easily achieved with fluorourethanes.

In contrast to the fluorourethane, the acrylic urethane began losing coating thickness in the second year of the test. The urethane had completely degraded after only 12 years, losing an average of 2.1 µm per year. The loss of coating thickness means that corrosion initiators can move more quickly through the urethane coating potentially reducing the life of the asset.

Electrochemical Impedance Spectroscopy Testing of Fluorourethane Coatings. Several different topcoats were testing using Electrochemical Impedance Spectroscopy (EIS). EIS involves sending an alternating current between two electrodes, where the main electrode is in a 3% salt water solution and the counter electrode on the metal coupon substrate. The change in impedance at a constant frequency of 1 kHz is then measured. The smaller the change in impedance during the test, the better the corrosion protection offered by the coating system. The configuration of the tested coating systems was zinc rich primer/epoxy/topcoat. Since the only difference between each coating system was the topcoat, the EIS test yields data that can be used to compare the relative performance of each topcoat. Relative coating performance is usually measured by determining the tangent of the angle θ, which is the acute angle each line makes with the vertical.

In the version of the test used in this case, impedance of each coating system was measured just after application. The coatings were exposed for 1,000 hours in the Sunshine Weatherometer, and then impedance measured again. Finally, each coating system was exposed to 500 and 1,000 hours in the ASTM B-117 salt fog test, and impedance measured after each exposure. Results of the test are shown below in Figure 8.

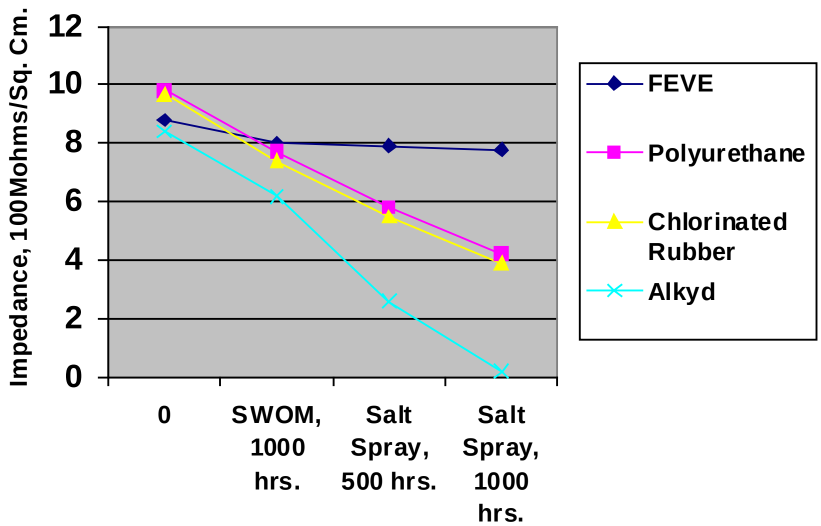


Figure 8: Electrochemical Impedance Spectroscopy Test Results for Coating Systems

Test results indicate that the FEVE fluorourethane coating outperforms the other coating systems. After exposure, the alkyd topcoat had decomposed completely. The chlorinated rubber and polyurethane topcoats showed a significant change in impedance.

Measuring the tangent of the angle to the vertical of each coating type yields the data shown below in Table 4.

Table 5: Determination of Relative Coating Life from EIS Results

Topcoat Type	Tangent θ	Relative Life (Polyurethane=1)
Fluorourethane	5.7	2.2
Polyurethane	2.6	1.0
Chlorinated Rubber	1.8	0.7
Alkyd	1.3	0.5

Per the EIS test results, the lifetime of a fluorourethane coating can be expected to be at least double that of a standard polyurethane topcoat. The difference in performance in the EIS test is believed to be due to the difference in levels of degradation of each topcoat.

Coating degradation allows penetration of corrosion initiators like chloride and water into the topcoat, accelerating the degradation process.

DOD APPLICATIONS FOR FEVE RESINS

Advanced Performance Coating

The Air Force, and later the Navy, have approved a fluorourethane topcoat for use on a range of aircraft, including the C-17, the KC-135, the F-15 and F-16, and the C-130 under MIL-PRF-85285. According to the Air Force, the use of the fluorourethane has saved around $600 million in life cycle costs on the KC-135 and C-17 alone. Advantages of the APC include improved gloss and color retention, resulting in less frequent repainting, improved fluid and heat resistance, better flexibility after aging, improved cleanability, and lower dry film density. Improvement in density is likely due to the fact that lower levels of flattening agents are required with the APC. Because the APC offers 2-3 times the life of conventional 85285 coatings, a significant reduction in pollution is also observed.

Other DOD Applications

There are several additional DOD applications where FEVE-based coatings are under consideration. The first is for an exterior coating for fuel tanks for the Navy. A specification is currently under development for this application. Weatherability and chemical resistance are the key attributes required for this application. A fluorourethane coating system is also being tested for use on the superstructure of naval vessels. Low solar absorbance and weatherability are desired.

CONCLUSIONS

Coatings based on FEVE resins, mainly fluorourethanes, have been in use in the field for almost 30 years. The primary use for FEVE coatings has been for preservation of appearance. It has been found that because of the extreme weatherability of the FEVE polymer, fluorourethane coatings maintain their thickness for many years more than standard polyurethanes or other coatings. This enables them to resist penetration by corrosion initiators like chloride ion, water, or oxygen. Thus, improved topcoat properties prevent corrosion of the metal substrate.

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