Title

Environmental Effects on a Protective Coating used to Mitigate Composite Degradation

Author

Akwari, Nelson Ndukwe

Publication Date

2021

UNIVERSITY OF CALIFORNIA

Los Angeles

Environmental Effects on a Protective Coating used to Mitigate Composite Degradation

A dissertation submitted in partial satisfaction of the requirements for the degree Doctor of Philosophy in Materials Science and Engineering

by

Nelson Ndukwe Akwari

2021

ABSTRACT OF THE DISSERTATION

Environmental Effects on a Protective Coating used to Mitigate Composite Degradation

by

Nelson Ndukwe Akwari

Doctor of Philosophy in Materials Science and Engineering

University of California, Los Angeles, 2021

Professor Jenn-Ming Yang, Chair

Composite materials are widely used in various applications and industries around the world. In recent years, commercial airplane companies have used composite materials for critical load bearing structures such as fuselage sections. During the lifetime utilization of composite materials, they can be subjected to extreme environmental conditions such as ultraviolet light, temperature fluctuations, humidity, and moisture. Depending on the amount of exposure to a composite material, the environmental conditions can negatively affect the chemical and mechanical properties of the fibers and matrix. Companies are investing substantial amounts of money and time to prevent composite materials from deteriorating by protecting them with protective materials such as coverings or coatings specifically designed to mitigate any damage to the underlying composite material.

This project focused on the effect of environmental conditions on a thermoset composite material and the evaluation of protective coatings to mitigate ultraviolet (UV) degradation of an underlying carbon fiber reinforced polymer (CFRP) composite substrate. Standardized laboratory test methods were used to replicate aerospace production in-service environments commonly seen in commercial aerospace factories. Additional considerations were given to the manufacturing processes and transport of composite parts. Tests and evaluations focused on the durability of the protective coating to environmental effects such as moisture, heat, and ultraviolet radiation. In order to evaluate the effectiveness of the coating to prevent degradation to the underlying composite substrate, mechanical and analytical evaluations of the coated composite substrate were performed. The composite substrate used throughout testing contained a co cured surfacing film with an epoxy polymer matrix chemistry. Consideration was given to the effects of environmental exposure on the surfacing film and potential subsequent effects on the composite structure as a whole.

A goal of the project was to down select a non-chromate protective coating that could effectively protect the underlying composite material from UV degradation. A second goal of this project was to quantify the UV and Condensing Humidity (moisture) degradation mechanisms and kinetics of the protective coating. A kinetic model was utilized to evaluate the independent degradation mechanisms due to UV exposure and Condensing Humidity (moisture) respectively. These independent mechanisms were evaluated for synergistic effects and compared to Outdoor exposure of protective coated composite panels with the goal of trying to determine if a cumulative model can be used to predict the overall degradation kinetics.

One commercial off-the-shelf non chromate epoxy-based coating was down selected after the completion of screening tests and proved to successfully protect the underlying composite material throughout a wide range of extreme environmental conditions. The independent evaluation of the degradation mechanisms due to UV exposure and Condensing Humidity (moisture) yielded results which correlated to industry and literature results. This research was unable to accurately develop a cumulative model that could consistently predict the overall degradation kinetics of a protective coated composite panel subjected to outdoor exposure conditions.

The dissertation of Nelson Ndukwe Akwari is approved.

Richard Kaner

Qibing Pei

Dwight Streit

Jenn-Ming Yang, Committee Chair

University of California, Los Angeles

2021

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Acknowledgments

I am thankful for the support and guidance of my doctoral committee members, Professors Jenn-Ming Yang, Richard Kaner, Qibing Pei, and Dwight Streit. Their feedback helped me to focus the scope of my work on qualitative and quantitative theoretical background information needed to complete my research.

Thanks to the Boeing Company for the sponsorship of not only my research but the patent that was earned as a part of this research. There are numerous Boeing teammates that guided me through my pursuit of a PhD, all of which made a significant impact on me throughout my journey.

Thanks to the UCLA Athletic Department and the Rose and Sam Gilbert family who offered additional sponsorship of my graduate studies. In particular, thanks to Joanne Suechika, Linda Lassiter, and Ric Coy for opening their doors and listening to my story when I returned to UCLA after a 10-year break to pursue a professional soccer career.

I am extremely grateful for the support, prayers, and constant encouragement of family and friends over the years. A pursuit of a PhD would not have been possible without the prayers, love, and constant support of my incredible wife, Mandy Akwari. There were many long days and nights, and you were always by my side encouraging me and reminding me to “focus my focus and finish strong”. Additionally, I’m grateful to my children, Nehemiah, Gideon, and Naomi who similarly were my biggest fans along my journey through graduate studies. To my parents, Bill and Hannah Akwari, thank you for challenging me to work hard, stay humble, and follow my dreams. You built a foundation that I could stand on and you provided me with more than enough to ensure I could succeed and not want for anything. To my brothers, Theo and

Stephen Akwari, thanks for pushing me and reminding me that nothing is more important than family. To my in-laws and extended family who prayed with me and for me over the years, I will be forever grateful. In particular, thanks to Tom and Chris Hlad, Josh and Whitney Hlad, Jeff Hlad, Gabe and Allie Coyle, and Leyna Akwari. It truly takes a village, so to the extended Akwari, Ubani, Hlad, and Moore families, I am truly blessed to call you family. Your constant encouragement and prayers, timely text messages, and family vacations were just what I needed. Some family members have passed away, but the impact they made and the impression they left on me will never be forgotten. Theo Akwari, Uncle Onye Akwari, Auntie Ngozi Obineche, Tom "Poppy" Hlad, David "Paka" and Bertha "Nini" Moore, I will see you again and we will celebrate.

To my close friends Matt and Wendy MacGinnis, thanks for always providing the encouragement I needed throughout my studies. You were a part of this journey from the beginning and you were a constant source of encouragement. Dr John Tracy, thanks for your willingness to share your story about returning back to school to pursue graduate studies while working full time. Your practical advice and willingness to share you experience are truly appreciated.

There are a number of brothers and sisters in Christ who prayed with me and for me throughout my journey. Thanks for reminding me of Proverbs 3:5-6, "Trust in the LORD with all your heart, And lean not on your own understanding; In all your ways acknowledge Him, And He shall direct your paths."

And to my Lord and Savior, Jesus Christ. Thanks for saving me and giving me the promise of eternal life. You have shown me that "I can do all things through Christ who strengthens me” (Philippians 4:13). I hope and pray that this degree ultimately honors and glorifies you. “Praise the Lord! Praise the Lord, O my soul! I will praise the Lord as long as I live; I will sing praises to my God while I have my being” (Psalm 146:1-2).

Vita

Education

University of California, Los Angeles

Master of Science, Engineering – Structural Materials December 2016
Bachelor of Science, Chemistry/Materials Science June 2014

Employment

The Boeing Company

Operations Superintendent and Site Leader September 2020 – Present
Interiors Responsibility Center South Carolina
Boeing Commercial Airplanes – Fabrication
Charleston, South Carolina

Materials & Process Fuselage Manager June 2019 – September 2020
787 and Propulsion South Carolina
Boeing Research and Technology
Manufacturing Technology Integration
Charleston, South Carolina

Materials, Process, and Physics Engineer June 2013 – June 2019
Boeing Research and Technology
Materials and Manufacturing Technology – Chemical Technology
Charleston, South Carolina
Huntington Beach, California

Academic Student Services Mentor	September 2013 – June 2014
UCLA Athletics Department	
UCLA Materials Science Undergraduate Student Researcher	January 2013 – June 2014
Professional Soccer Player	January 2007 – December 2011
United Soccer League	
Professional Soccer Player	August 2002 – December 2006
Major League Soccer	

Chapter 1 Introduction and Significance of the Research

Introduction

Composite materials have garnered plenty of attention since their arrival onto the scene. They offer a diverse range of material properties which allows them to be extensively used for various applications. Composite materials are utilized in industries such as the automotive, aerospace, biomedical, and sporting industries. As the utilization of composite materials continues to expand, there is a growing need to protect composite materials to ensure they properly function throughout their intended lifetime. Varying environmental conditions can negatively affect the chemical and mechanical properties of a composite material. Experimental and in-service data of composite materials is providing opportunities for manufacturers to better understand the life expectancy of the products being developed within their organizations. In an attempt to extend the lifetime of composite materials, companies are spending a significant amount of money to research formulations and chemistries that are suited to protect composite materials utilized in varying environmental conditions. Cost effective protective materials used to mitigate degradation to composite materials are coverings and coatings. Protective coatings are used throughout the biomedical, automotive, and aerospace industries. As the aerospace industry continues to grow in its utilization of composite materials, there is interest in identifying protective coating materials that can be utilized to protect large composite structures.

Background Information on Composite Materials

Composite materials are divided into two main categories – thermoplastics and thermosets. Thermoplastic composites have a chain structure of atoms which are linear or branched. Upon heating to a characteristic temperature for a specific thermoplastic, the polymer chains become moldable and flexible. With subsequent cooling, thermoplastics will return to a solid state. Alternatively, thermoset polymers are cross-linked. During the curing process of thermosets, irreversible chemical bonds are created. When a thermoset polymer is heated to a characteristic temperature for a specific thermoset, the chemical bonds will be broken and unable to reform upon cooling. Main characteristics, Advantages, and Disadvantages of thermoset and thermoplastic materials are below:

Main Characteristics

Thermoset	Thermoplastic
▪ Undergoes chemical change when cured	▪ Non-reacting, no cure required
▪ Low strain to failure	▪ High strain to failure
▪ Low fracture energy	▪ High fracture energy
▪ Processing is irreversible	▪ Very high viscosity
▪ Very low viscosity is possible	▪ Processing is reversible
▪ Absorbs moisture	▪ Absorbs little moisture
▪ Highly resistant to solvents	▪ Limited resistance to organic solvents, in some cases

Advantages

Thermoset

- Relatively low processing temperature
- Good fiber wetting
- Formable into complex shapes
- Liquid-resin manufacturing feasible
- Resistant to creep

Thermoplastic

- Short processing time possible
- Reusable scrap
- Post-formable can be reprocessed
- Rapid Processing
- Unlimited shelf life without refrigeration
- High delamination resistance

Disadvantages

Thermoset

- Long processing time
- Long cure (approximately 1-2 hours)
- Restricted storage life (requires refrigeration)

Thermoplastic

- Lower resistance to solvents
- Requires high temperature (300-400°C) and pressure processing
- Can be prone to creep
- Poor drapability and tack

Carbon Fiber/Epoxy Composite Manufacturing

The focus of this research was on thermoset composite materials utilized in the aerospace industry. Test panels were fabricated with an epoxy/carbon laminate in an autoclave. Pre-impregnated composite fibers were used for fabrication of the composite panels. The pre-impregnated composite fiber material consisted of composite fibers with an epoxy resin prepared from the reaction of bis-phenol A and epichlorohydrin with reactive functional groups in their molecular structure. The epoxy material used in this fabrication process required that the material be stored at freezing temperatures to ensure that the partially cured material could be handled easily. In general, resin curing goes through three stages of cure – A-stage, B-stage, and C-stage. During A-stage, the resin curing stage is in its initial stage, and no reaction has occurred. During B-stage, the intermediate stage, the material softens when heated and becomes plastic and fusible. During C-stage, the final stage, the reaction of the thermoset resins causes the material to become insoluble and infusible. The curing stage of the resin at the time of fabrication was B-stage. Cure temperatures generally range from 200-400°F. Autoclaves are capable of curing large airplane fuselage sections or wings. An industrial lay-up tool, sample industrial autoclave, and fully cured fuselage sections out of autoclave can be seen in Figure 1[1] and Figure 2[2] respectively.

Figure 1: Composite autoclave and lay-up cure tool

Figure 2: Fully cured fuselage sections out of autoclave

Applications in Aerospace Industry

The Aerospace Industry has benefited from the introduction of both thermoset and thermoplastic composite materials into the design build of airplanes. When compared to aluminum and metal airplane parts, noted benefits of aerospace composite materials are durability, reduction in fatigue, improved corrosion protection, and higher strength lightweight materials. For some airplane models, composite materials comprise approximately 50% of the commercial airplane. Figure 3[3] provides a nominal breakdown of a current commercial airplane utilized for long range global flights:

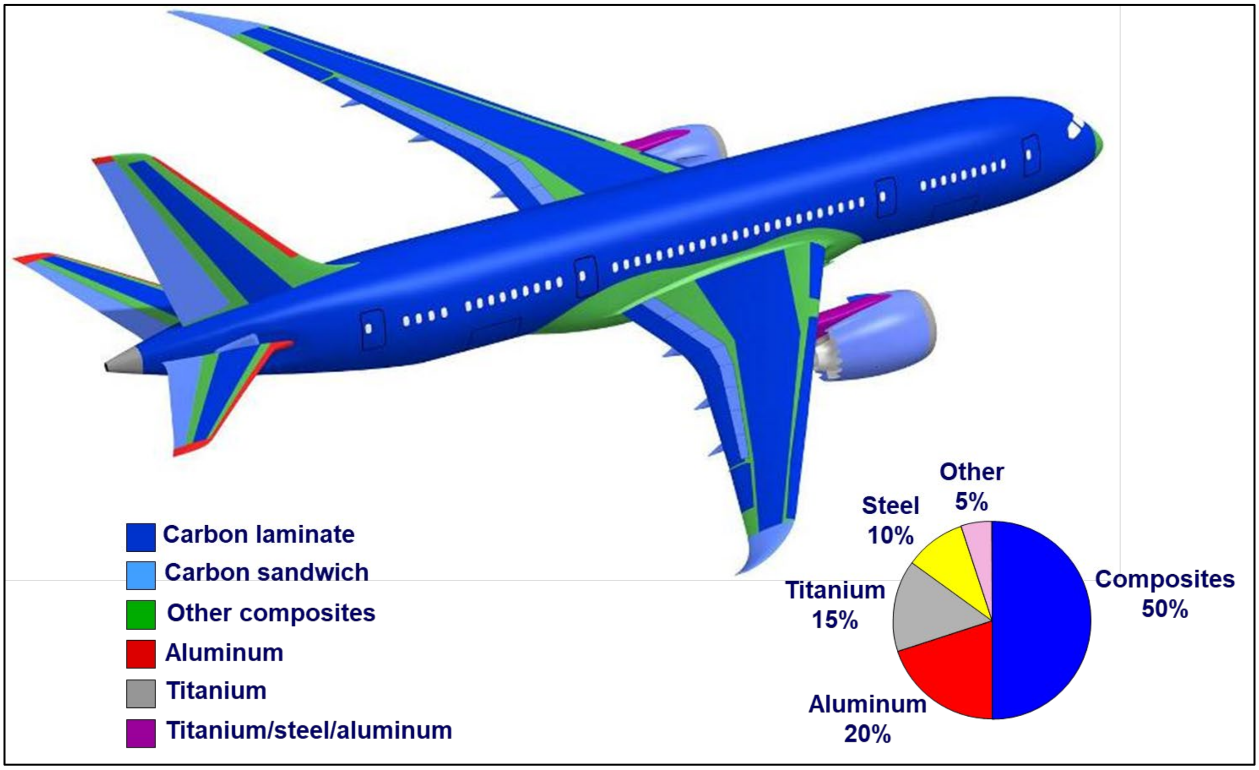


Figure 3: Percentage of varying materials utilized in a commercial airplane

The availability of composite materials globally has provided an opportunity for the manufacturability of airplane components to occur throughout the globe. In particular, the 787 Boeing airplane is manufactured by global partners. The assembly and transport of large individual aircraft sections occurs in a large airplane structure specifically manufactured for the transportation of airplane parts. The manufacturing and assembly sites vary in size and scale which requires additional consideration of the environmental impacts on the individual composite parts. Figure 4[4] highlights some of the manufacturing global partners for the Boeing 787 airplane.

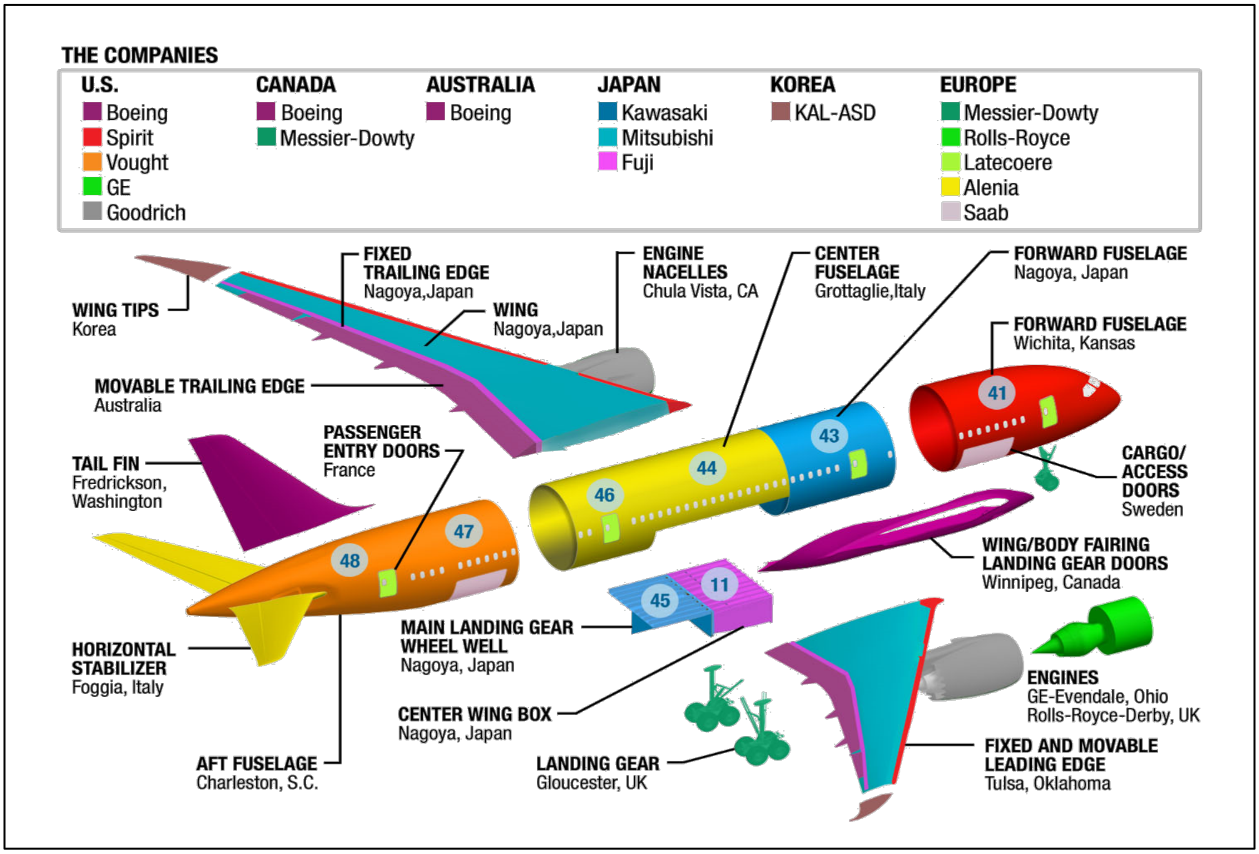


Figure 4: Manufacturing global partners for the Boeing 787 airplane

An identified challenge during transport of the composite materials is to ensure the environmental conditions during manufacturing, assembly, and transport do not negatively impact the properties of the composite materials. Ultraviolet (UV) radiation from sunlight and factory light has been identified as one of many critical environmental factors that must be monitored throughout the lifetime of composite materials. In an attempt to minimize any impacts of UV radiation on composite materials, it is very common to wrap and store composite parts with large protective coverings or coatings. The cost associated for utilizing a protective covering can be substantial throughout the lifetime of an airplane program. Figure 5[5] highlights composite airplane fuselage parts covered with large plastic coverings.

Figure 5: Protective coverings used for composite parts during transport

The environmental effects on aerospace parts are not unique to composite materials. Even aluminum and metal parts on airplanes must be protected in order to prevent corrosion of critical parts. Figure 6[6] highlights a Boeing 737 during transport that is being protected with a coating. Some critical parameters that must be considered for the utilization of protective coatings are the formulation chemistries of the coating, application constraints (sprayability, material thickness, environmental regulations) of the coating, and the compatibility of the coating to the underlying structure. For protective coatings utilized in the aerospace industry, it’s very common to use a temporary protective coating during assembly and transport. The protective coating must be removed at the completion of all processes prior to the application of a final coating scheme that will be used for the lifetime of the airplane.

Figure 6: Protective coating utilized to prevent corrosion on metal airplane parts during transport

Chapter 2 Literature Survey on Degradation Mechanism of Composite Materials and Kinetics of UV Degradation

Introduction

The effectiveness of composite materials or coatings to resist degradation due to environmental effects can be characterized by non-destructive evaluation methods utilized to analyze properties such as color, gloss, surface wetting, chalking, or weight.[7] Destructive evaluation methods such as toughness, glass transition temperature, tensile strength, and flexural tests can also be utilized to further understand microscopic and macroscopic changes in a material system due to relative lengths of environmental exposure times. A firm understanding of the chemistry of a composite material and a coating system is needed in order to identify a degradation mechanism.

Composite Degradation – Chemistry

Degradation of composite materials can be evaluated by the chemical reactions which occur during environmental exposure. Specifically, for ultraviolet radiation, the mechanism of dissociation energies of polymeric covalent bonds is prevalent. Polymeric epoxy covalent dissociation bonds energies lie in the range of 280-460 kJ/mole. The equation for the Planck-Einstein law:

$$E = hv = \frac{hc}{\lambda}$$

where E is energy, h is Planck's constant, v is the frequency, c is the speed of light, and λ is the wavelength. The value of Planck's constant (h) is $6.626 \times 10^{-34}\ m^2kg/s$. The value of the speed of light (c) is $3 \times 10^8\ m/s$. Using Planck's constant and the relevant variables, calculation of the relative range of wavelengths which would initiate polymeric epoxy covalent bond dissociation is straightforward. The calculation can be seen below:

$$\lambda = \frac{hc}{E} = \frac{6.626 \times 10^{-34}\ (m^2kg/s)\ \cdot\ 3 \times 10^8\ (m/s)}{280\ (kJ/mole)} = 427.5\ nm$$

$$\lambda = \frac{hc}{E} = \frac{6.626 \times 10^{-34}\ (m^2kg/s)\ \cdot\ 3 \times 10^8\ (m/s)}{460\ (kJ/mole)} = 260.2\ nm$$

The value for mole is $6.022 \times 10^{23}\ mol^{-1}$. The range of wavelengths calculated fall within the Ultraviolet spectrum given off by sunlight. Figure 7[8] is an image of the UV radiation and light spectrum. The formulations of the composite and coating materials evaluated for this research were predominantly made up of polymeric chemical bonds such as C-C, C-O, C-N, and H-O. Table 1 contains the Bond Dissociation Energy and Wavelength of common polymeric chemical bonds. These bonds have large absorbance in the UV range. Figure 8[9] is a graph of the Absorbance vs. Wavelength in nanometers of common polymeric bonds.

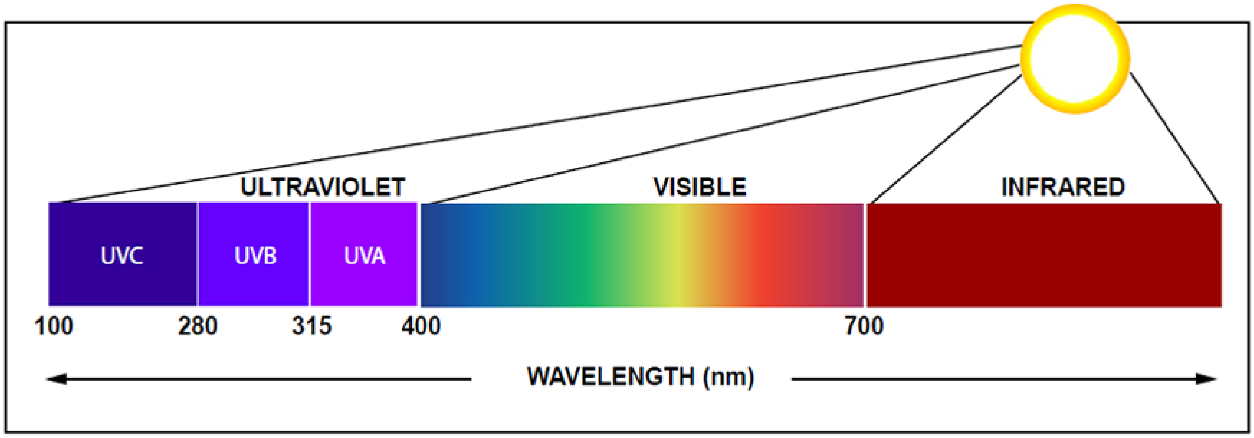


Figure 7: Ultraviolet radiation and light spectrum

Table 1: Bond Dissociation Energy (eV) and Wavelength (nm)

	Bond Dissociation Energy (eV)	Wavelength (nm)
C-C	3.48	356
C-O	3.60	344
C-N	3.08	403
H-O	3.66	339

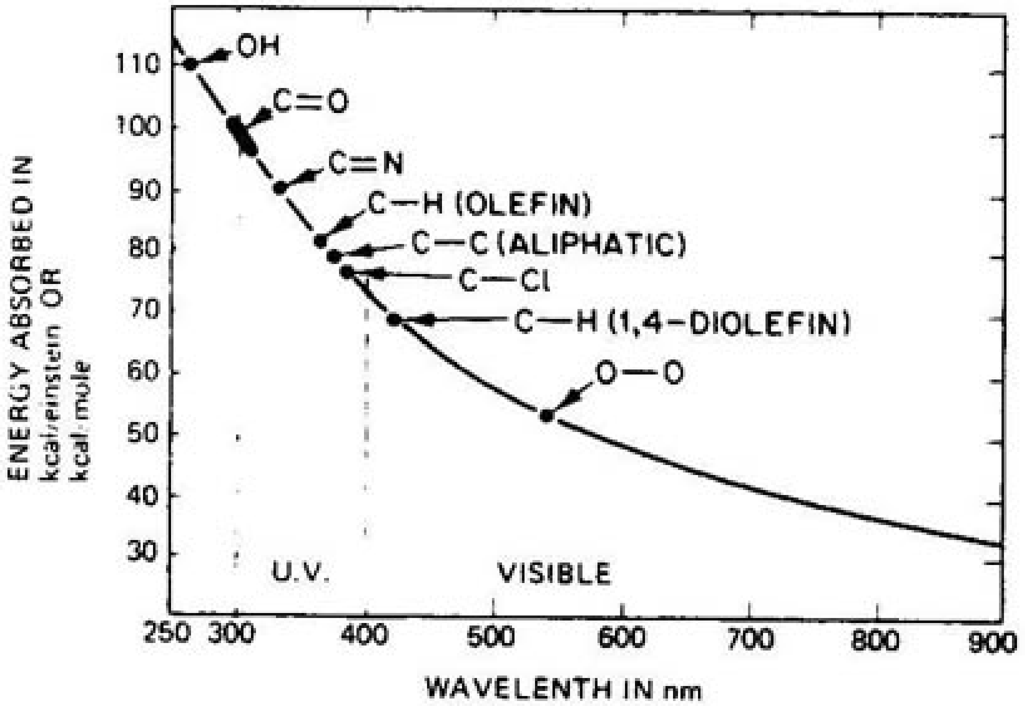


Figure 8: Absorbance vs. Wavelength (nm) of polymeric chemical bonds

An organic compound used in this research and commonly used in epoxy resins is Bisphenol A diglycidyl ether (DGEBA). Figure 9[10] is the chemical structure of DGEBA. Since epoxy resins contain aromatic groups within the referenced UV range containing high absorbance, the expectation is that the overall structure will degrade over time when exposed to UV. There are various UV degradation mechanisms that have been proposed for DGEBA and they are highlighted in Figure 10[10].

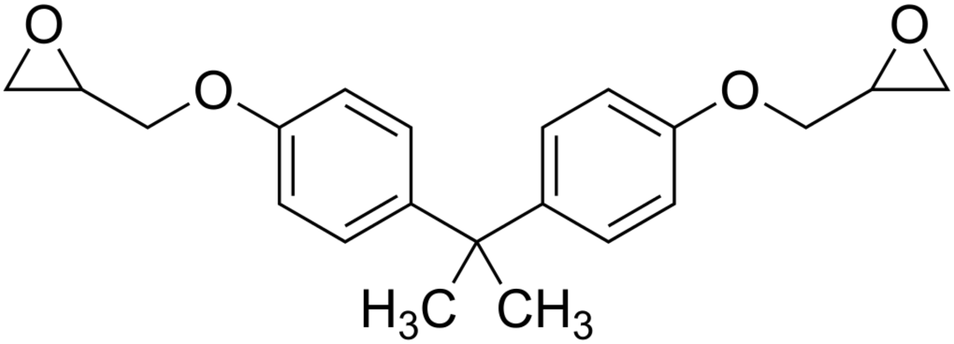


Figure 9: Chemical structure of Bisphenol A diglycidyl ether (DGEBA)

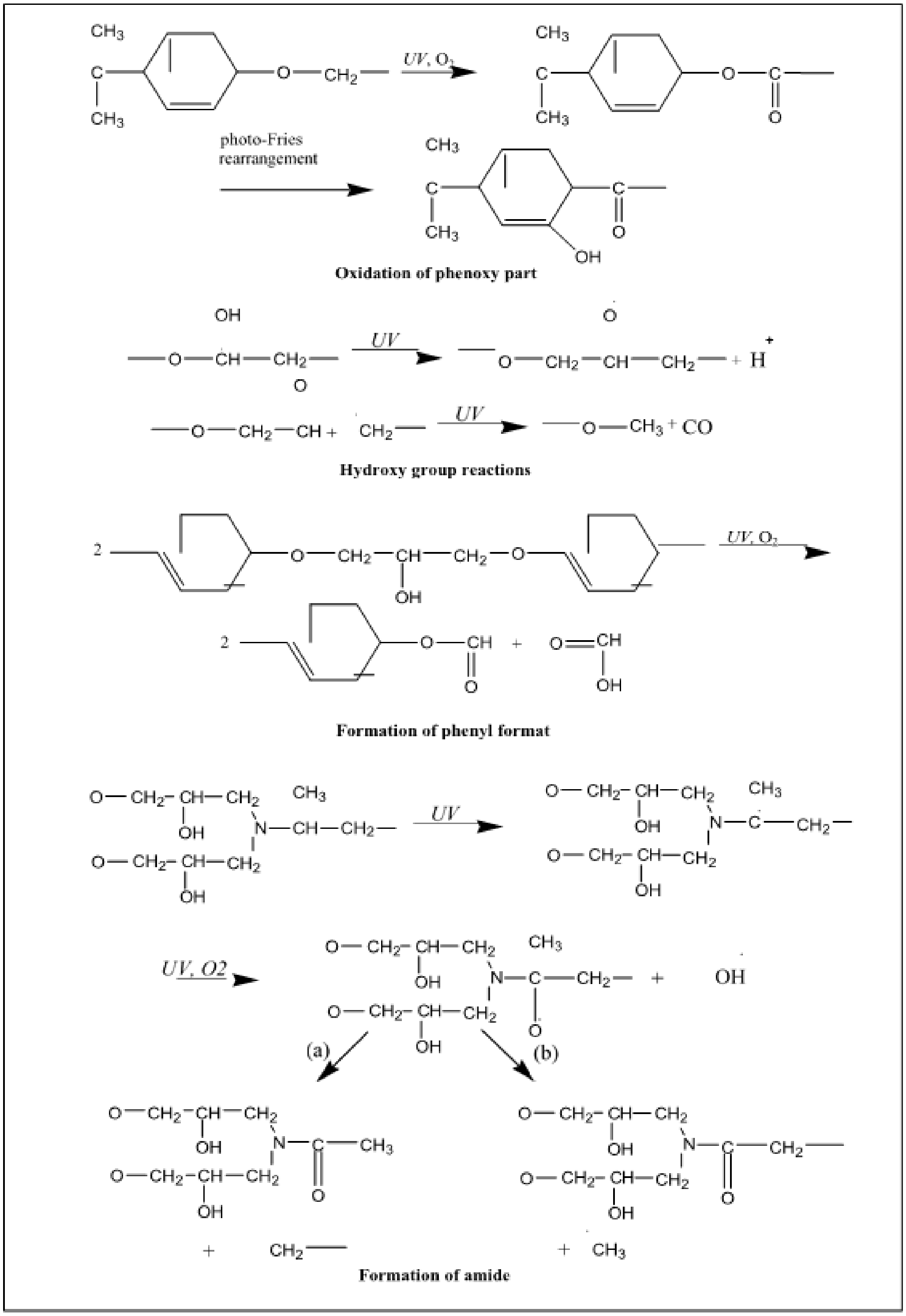


Figure 10: Mechanisms of UV degradation of DGEBA epoxy system

Composite Degradation – Weight Change

As the field of composites continues to grow, so does the understanding of the effects of the environment on composites. Lv and all[11] documented that cyclic environmental exposure of carbon fiber/epoxy composite by xenon lamp and humidity shows a correlation between weight change rate as a function of exposure duration. The engineers reached a conclusion that the UV radiation exposure formed microcracks on the epoxy matrix and the interface. Optical micrographs reinforced their conclusion and can be seen in Figures 11 and 12.

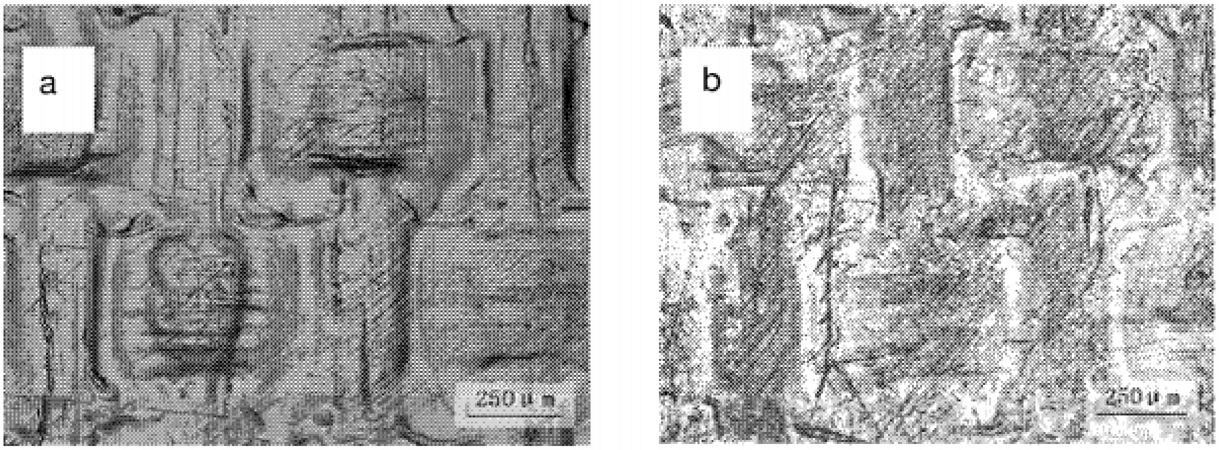


Figure 3 Surface of woven composite (a) prior to environmental exposure (b) after 672 h of sequential exposure.

Figure 11: Optical micrograph of woven composite pre- and post-environmental exposure showing microcracking

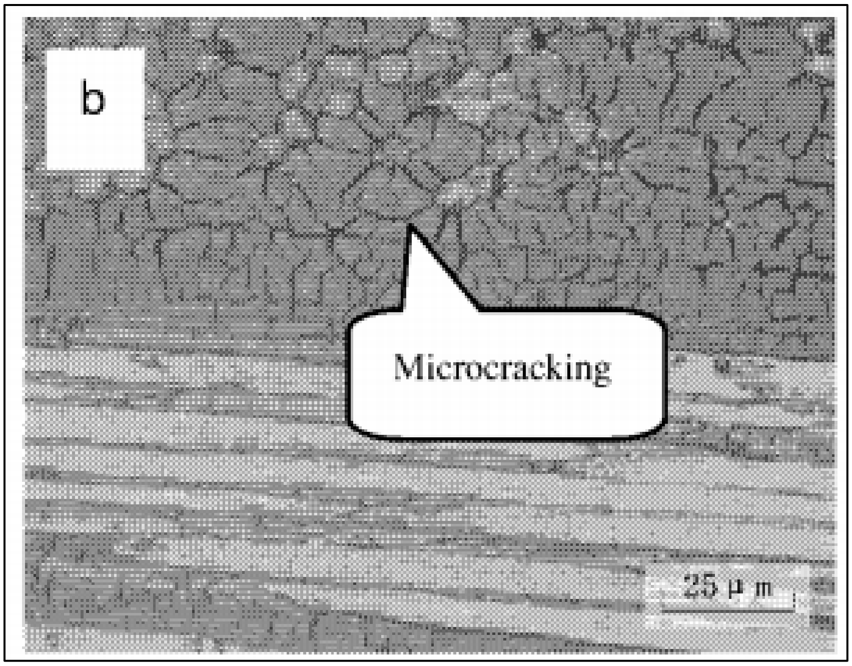
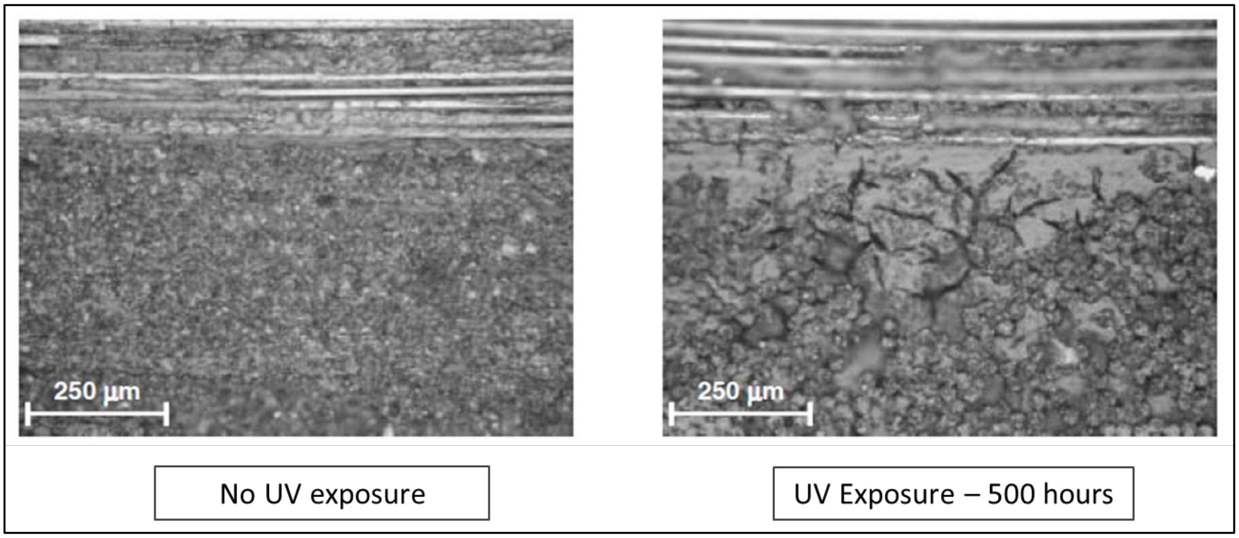


Figure 12: Optical micrograph of cross section of woven composite post environmental exposure showing microcracking

In addition to microcracking, UV radiation and humidity exposure resulted in a decrease of interlaminar shear strength and transverse tensile strength. This paper doesn't deal with the standard test methods of interlaminar fracture toughness, but ASTM D5228[12] is the recommended test method outlined in numerous materials. Nguyen et al. recorded the effects of UV and temperature on mechanical properties of carbon fiber and carbon fiber reinforced polymer[13].

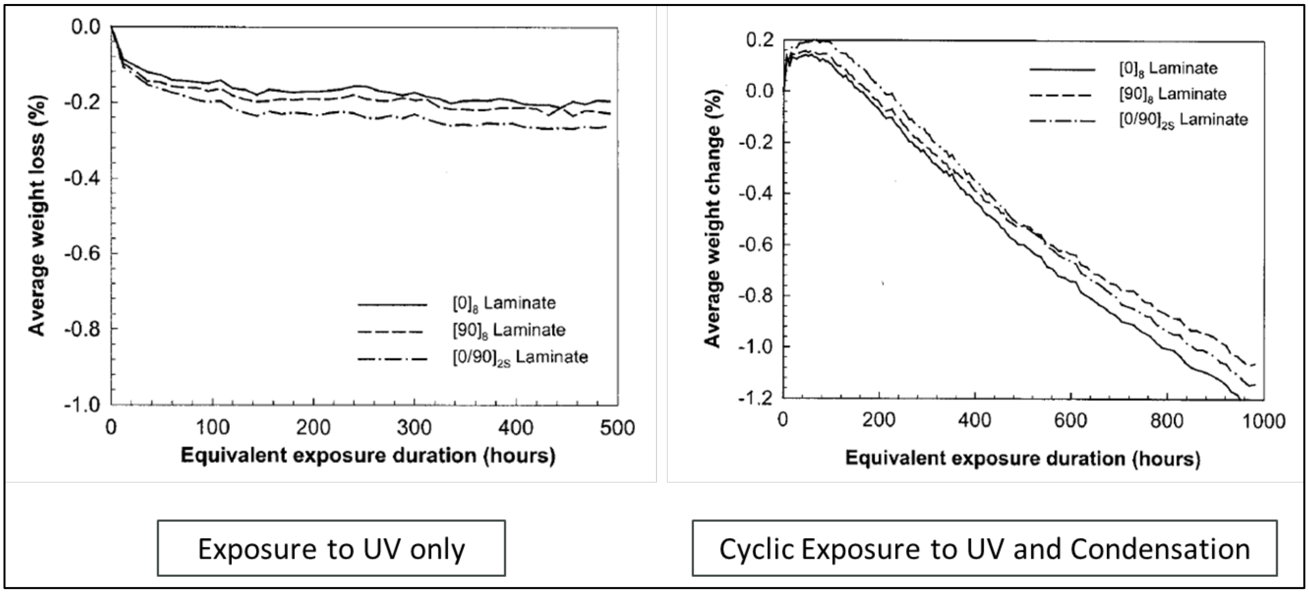
Kumar et al.[14] researched composite materials irradiated by UV light sources and correlated weight loss over exposure times. The decrease in weight of the composite samples tested were attributed to the expulsion of volatiles and residual moisture on the surface of the samples. Additionally, their research attempted to identify synergistic effects of UV light, moisture, and humidity. Comparable results were identified by N. Guermazi et al who noted that for an increase in weight content for samples tested, there was a linear increase correlating to rapid absorption of water during the initial stages of composite degradation[15]. At a certain point though, the expectation would be that the composite material would reach a saturation point dependent on environmental temperatures and exposure durations. Optical micrographs in Figure 13 demonstrate their results for accelerated composite degradation due to the synergistic effects of UV light, moisture, and humidity. Figure 14 highlights their results of Environmental Exposure Duration (UV Exposure only and UV Exposure and Condensation) vs. Average Weight Change.



No UV exposure

UV Exposure – 500 hours

Figure 13: Optical micrographs of a composite material with no UV Exposure and degradation of the composite material after UV Exposure (500 hours)



Exposure to UV only

Cyclic Exposure to UV and Condensation

Figure 14: Environmental Exposure Duration (UV Exposure only and UV Exposure and Condensation) vs. Average Weight Change (%)

Kinetic Models of UV Degradation

UV Degradation effects on coatings occur at the surface layer of the coating and can be represented as multiple damage events over the exposed area. An assumption within this research is that all damage events occur evenly throughout the samples. Additionally, for each damage event, there will be a complete removal of the UV degraded layer. Any effects to layers below the surface are negligible and are not accounted for in the degradation models. Examples of some commonly used models are the Monte Carlo Model[16], the Cumulative Dosage Model[17], and a modified Specular Reflectance (Gloss) model by Bennet and Porteus[18] that relates the exponential decay of the gloss with exposure time.

Monte Carlo Model

The Monte Carlo Model for a coating utilizes a simulation which erodes randomly and cumulatively over time as the degradation and exposure times are increased. This model proves useful for the degradation of polymeric materials due to the combination of a high number flux of photons and the low quantum yield. The degradation process model can provide successful results if the degradation mechanism can be defined by an algorithmic equation. Croll, S. G., Hinderliter, B. R., and Liu, S[7] provided an example below where *N* is the average number of pieces of the coating removed on the surface layer and Kt is a rate parameter:

$$N = Average\ number\ of\ pieces\ removed\ at\ a\ point\ on\ the\ coating$$

$$= Flux\ of\ photons\ at\ that\ position\ \left[\frac{photons}{time}\right]$$

$$\times\ Absorption\ \left[\frac{photons\ absorbed\ per\ polymer\ segment\ volume}{photon\ incident\ on\ the\ segment\ area}\right]$$

$$\times\ quantum\ efficiency\ of\ creating\ a\ polymer\ fragment\ \left[\frac{probability}{photon\ absorbed}\right]$$

$$\times\ probability\ that\ the\ polymer\ segment\ is\ removed\ prior\ to\ recommendation\ into\ the\ existing\ polyer$$

$$\times\ exposure\ time, t\ [e.g. years] = Kt$$

Specular Reflectance (Gloss) Model (Bennett and Porteus)

Specular Reflectance, “gloss”, is a measure of a smooth specular reflectance $R_s(t)$ at a time (t) to a black glass standard R_{std}.

$$Gloss = \frac{R_s(t)}{R_{std}}$$

The Specular Reflectance (Gloss) Model assumes an exponential decay that can be modeled using the start experimental variable of a measured specular reflection of a smooth surface $R_s(0)$ and the specular reflection of a rough surface $R_s(t)$ of the same material after a defined amount of time. This model assumes a Gaussian distribution and Root Mean Square (R.M.S.) deviation, σ, of the surface from its mean. The wavelength of light, λ, and specular angle φ.

$$\frac{R_s}{R_0} = exp\left[-\left(\frac{4\pi cos\varphi\sigma}{\lambda}\right)^2\right]$$

The expression relating the change in reflectance due to changes in surface roughness over exposure time are below:

$$R_s(t) = R_0 exp\left[-\left(\frac{4\pi cos\varphi\sigma}{\lambda}\right)^2 (\sigma_{initial}^2 + kt)\right]$$

$$R_s(t) = R_0 exp\left[-\left(\frac{4\pi cos\varphi\sigma inital}{\lambda}\right)^2\right] \times exp\left(\frac{4\pi cos\varphi\sigma}{\lambda}\right)^2 kt$$

Algebraic manipulation allows for an expression for the function of exposure time to correlate linearly to the logarithm of gloss:

$$log_e R_s(t) - log_e R_s(0) = -\left(\frac{4\pi cos\varphi\sigma}{\lambda}\right)^2 kt$$

Cumulative Dosage Model

The Cumulative Dosage Model relates photodegradation of polymeric surfaces by radiation fields[17]. The equation is below:

$$D_{total}(t) = \int_0^{\tilde{t}} \int_{\lambda_{min}}^{\lambda_{max}} E_0(\lambda, t)\left(1 - e^{-A(\lambda)}\right)\phi(\lambda) d\lambda dt$$

where λ_{min} and λ_{max} are the minimum and maximum effective wavelengths (nm). $A(\lambda)$ is the absorbance of the sample at a specified UV wavelength, (dimensionless). $E_0(\lambda)$ is the incident dose (Wm^{-2}) of UV radiation which interacts with the polymeric coating. $\phi(\lambda)$ is the quantum yield which is the number of times a specific event occurs per photon absorbed by the material. The elapsed time (t) and the total radiation time $\tilde{t}$ have units of seconds. $D_{total}(t)$ is the total effective dosage (J/m^2). Some key assumptions for usage of the cumulative dosage model when applied to this research are below:

1) All areas of the coating and composite material receive the same environmental exposure (dosage of UV, moisture, condensation, humidity) throughout the respective time of exposure.

2) The specimens are “flat” and any changes in surface roughness are negligible when compared to the effect of the environmental exposure on the coating and composite materials.

3) Where applicable, there is no UV radiation transmitted through both the protective coating and composite material. This was confirmed through UV spectroscopy of the protective coating showing less than 0.1% UV Transmittance over UVA range (315-400 nm) for coatings of various thickness (0.5, 1.0, 2.0, 3.0 mils). The conclusion is that the protective coating fully absorbs all effective wavelengths making the probability of the absorption of UV photons equal to 1. Mathematically, this is represented: $e^{-A(\lambda)} \approx 0$.

4) Based on the chemistry of the protective coating, the effective UV wavelength causing primary photodegradation is within the UVA range (315-400 nm). The quantum efficiency number[19] for this range of wavelengths is 10^{-4}.

5) Photodegradation due to UV photons results in the complete removal of the uppermost layer of the coating and chemical changes of the coating are minimal.

The resulting assumptions provide a simplified equation:

$$D_{total}(t) = E_0 \times \phi \times t$$

A relationship between UV damage and polymeric materials can be approximated by a linear equation, exponential equation, or a power law equation. A quantitative critical material characteristic can be measured and evaluated to build an equation and model damage kinetics. For this research, the critical material characteristic was specimen weight and a linear dependence was identified yielding the equations below:

$$\Omega = \left(\frac{W_i - W_f}{W_i} \times 100 \right)$$

$$\Omega = cD_{total}$$

$$\left(\frac{W_i - W_f}{W_i} \times 100 \right) = cD_{total}$$

where Ω is the critical material characteristic which is the percent weight change. W_i is the initial weight (grams) before environmental exposure, W_f is the final weight after environmental exposure, and c is an empirical constant.

This research utilized the cumulative dosage model due to the easily identifiable critical parameters of weight change, the ability to evaluate UV Transmittance over periods of UV exposure, and the ability to monitor photodegradation through optical microscopy.

Proposed Work

This project focused on the ability of a protective coating to assist in mitigation of environmental effects on carbon fiber reinforced polymer (CFRP) composite material. Tests and evaluations focused on the durability of the protective coating to environmental effects such as moisture, heat, and ultraviolet radiation. In order to evaluate the effectiveness of the coating to prevent degradation to the underlying composite substrate, mechanical and analytical evaluations of the composite substrate were performed. Test methods used to replicate in-service effects in a lab environment are discussed. While the combination of environmental effects such as moisture, heat, and ultraviolet radiation are considered, a priority throughout individual tests was to focus on a specific property of the coating and its ability to protect the underlying composite substrate. By focusing on a singular property, the goal was to correlate an UV Degradation mechanism in order to model the kinetics of UV degradation. Four commercial off the shelf coatings with differing formulations were evaluated and one coating was down selected for further testing. All coatings were non chromate polymer-based coatings. The composite substrate used throughout testing contained a co cured surfacing film with an epoxy polymer matrix chemistry. Consideration was given to the effects of environmental exposure on the surfacing film and potential subsequent effects on the composite structure as a whole.

Despite the advancement in technology, environmental exposure tests run in a laboratory environment do not always directly correlate to in-service conditions. For example, a common in-service issue seen in composite materials is the degradation of the epoxy resin matrix on the surface of the composite material due to ultraviolet (UV) radiation. UV radiation sources include sunlight and factory lights. Factors such as the length of exposure and distance between the source and the composite material will affect the amount of UV damage that will occur to a given composite material. Overexposure to these sources causes the surface chemistry of the composite to be negatively impacted. Microcracking, water absorption, hardening of the surface, and discoloration of the composite are all indicators and consequences of degradation to the epoxy resin. Issues during the manufacturing process such as paint and sealant adhesion failure may occur due to degradation of composite fiber reinforced polymer. Replicating these failures seen during the manufacturing process can be difficult so whenever possible, subsequent tests were run on the same sets of panels to accelerate the environmental conditions and simulate in-service conditions. Additional qualitative tests were performed to simulate the manufacturing processes and transport of composite parts.

An additional focus of this research is to utilize theoretical equations to model UV Degradation and degradation kinetics quantitatively. Based on literature and fundamental theoretical polymer chemistry principles, critical material characteristics can be measured and evaluated throughout differing environmental exposure conditions. The data collected can be used to build an equation and model damage kinetics. By identifying the critical material characteristics, this research aims to optimize the coating design parameters. Design parameters that play an integral role in the protective coatings evaluated, are the protective coating chemistry, protective coating thickness, and percent transmittance through the coating.

Chapter 3 UV Resistant Coating Formulation and Evaluation

Composite Panel Fabrication

Test panels were fabricated with an epoxy/carbon laminate in an autoclave. Pre-impregnated composite fibers were used for fabrication of the composite panels. The pre-impregnated composite fiber material consisted of composite fibers with an epoxy resin prepared from the reaction of bis-phenol A and epichlorohydrin with reactive functional groups in their molecular structure. The epoxy material used in this fabrication process required that the material be stored at freezing temperatures to ensure that the partially cured material could be handled easily. A composite lay-up tool approximately 6 feet by 12 feet was used to fabricate a singular large sheet of composite material. Prior to use of the lay-up tool, the tool was solvent cleaned, wiped thoroughly, and left to dry at ambient conditions for 45 minutes. A mold release was applied to the tool in thin coats and excess mold release was removed from the tool. The mold release was then baked onto the tool for approximately 4 hours at 60 – 120°F. Next, a thin coat of tackifier solution was mixed and spray applied to the composite panel lay-up tool. Ten minutes were allowed to pass prior to the laying of materials in order to ensure solvent had time to evaporate. The composite fiber orientation was layered in [-45/90/+45/0]s. A schematic of the composite lay-up orientation is shown in Figure 15.

Figure 15: Schematic of composite lay-up

After fiber placement, the composite fibers were vacuum bagged and prepared for cure. Thermocouples were placed in identifiable locations inside the autoclave in order to monitor the curing cycles of the composite material. After the autoclave cycles were complete, final trimming of the large composite sheet occurred via a water jet cutting tool. Panel dimensions were 4 inches by 6 inches, 3 inches by 12 inches, and 4 inches by 4 inches.

Prior to the application of coatings onto the composite test panels, the panels were cleaned in order to remove contaminates, such as dust, dirt, foreign particles, release agents, or oils. Suitable solvents that were utilized to properly clean the surface of the composite coatings were Methyl Ethyl Ketone (MEK), Methyl Propyl Ketone (MPK), or a blend of 70% MPK and 30% MEK.

Coating Formulations (Role of UV Stabilizer)

A protective coating can comprise of a number of varying molecular chemistries that will affect the final product. Three critical components of the protective coating formulation are UV Blocking Chemistry, Silicate Fillers, and Solvents. Details of the critical components and examples of each component are listed below.

UV Blocking chemistry

In order to block both UV-A and UV-B light radiation, UV absorbers can be incorporated in organic coatings. Lowry et. al. researched the effectivity of using ZnO on UV degradation polyurethane coatings and found that the presence of ZnO at 2.0 g/m^2 can block more than 99% UV radiation[20]. Organic absorbers can degrade over time and potentially migrate out of the coating matrix. Hang et. al noted that inorganic absorbers do not migrate and can provide effective degradation mitigation due to their small sizes[21]. Inorganic absorbers roughly the size of 10-30 nanometers can be dispersed throughout coatings to improve blocking efficiency and not inhibit mechanical properties or coating durability. An example of another commonly used UV Blocking material is Titanium dioxide. Pang et. al studied the dispersion of nano-Titanium Dioxide and recorded the surface morphological changes and improved UV blocking correlate to heterogeneity of the nanoparticle dispersion throughout the material[22].

Silicate Fillers

Silicate fillers can be utilized in large percentages in coating formulations. Its primary function is to diminish light scattering in the visible range while maintaining the same UV absorbance. Particle sizes are approximately 1-5 micrometers and their hardness provides strong abrasion resistance. Additional key characteristics of silicate fillers is to provide resistance to acid, chemical, and heat. Examples of silicate fillers are Barium Sulfate, Calcium Carbonate, and Silica.

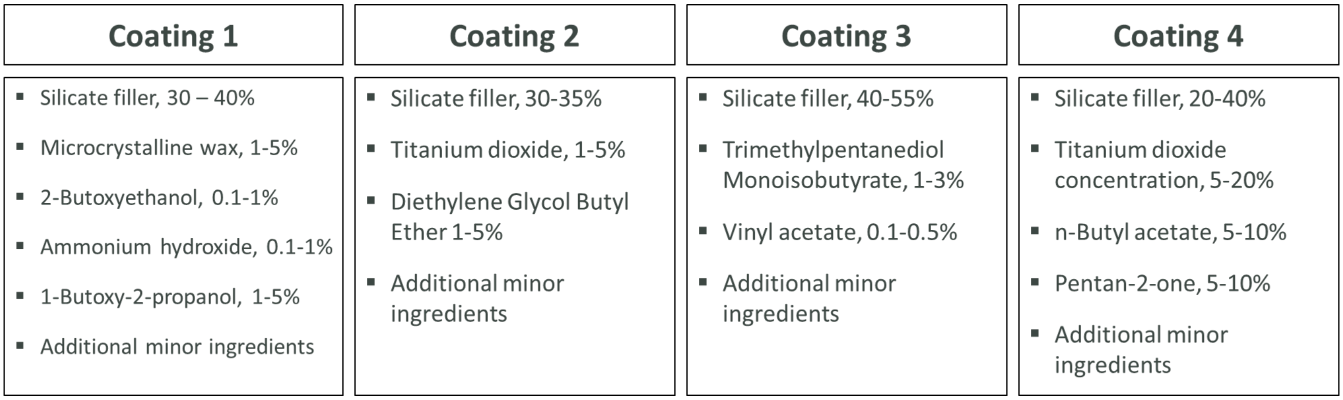
Solvents

Solvents are utilized to reduce the viscosity of paints and coatings which allows for optimal spray properties. A high volatile content allows for quick solvent evaporation after spraying which is generally needed to ensure that the coating can properly wet the intended surface and dry without leaving streaks. Examples of solvents in paint and coating formulations are 2-Butoxyethanol; Diethylene Glycol Butyl Ether; 2,2,4-Trimethyl-1,3-pentanediol monoisobutyrate.

Formulations

The correct ratio of UV Blocking Chemistry, silicate fillers, and solvents can minimize the effect of UV energy on an underlying substrate which is why these key parameters are essential for an effective protective coating. Four coatings with varying percentages of critical components that would influence the identified key parameters were evaluated for this research.

Below is a figure highlighting the main components of the four coatings. Additional key parameters within the formulations were the effective dispersion of the UV blocking and silicate filler components.



Coating 1	Coating 2	Coating 3	Coating 4
▪ Silicate filler, 30 – 40% ▪ Microcrystalline wax, 1-5% ▪ 2-Butoxyethanol, 0.1-1% ▪ Ammonium hydroxide, 0.1-1% ▪ 1-Butoxy-2-propanol, 1-5% ▪ Additional minor ingredients	▪ Silicate filler, 30-35% ▪ Titanium dioxide, 1-5% ▪ Diethylene Glycol Butyl Ether 1-5% ▪ Additional minor ingredients	▪ Silicate filler, 40-55% ▪ Trimethylpentanediol Monoisobutyrate, 1-3% ▪ Vinyl acetate, 0.1-0.5% ▪ Additional minor ingredients	▪ Silicate filler, 20-40% ▪ Titanium dioxide concentration, 5-20% ▪ n-Butyl acetate, 5-10% ▪ Pentan-2-one, 5-10% ▪ Additional minor ingredients

Figure 16: Main Components of Four Protective Coatings under evaluation

Coating Preparation

Three of the coatings were one component coatings which did not need to be mixed by ratio prior to application. One of the coatings (Coating 4) was a two-component coating (Base/Thinner). The ratio of Base to Thinner was 1.0 to 0.2 respectively. Coating 4 was mixed in accordance with the provided recommendations in the technical data sheet provided by the Supplier. In general, 30 minutes of mixing on a standard paint mixer ensured homogeneity of the coating. After mixing, the coating was poured through a strainer and into a pressure pot. The strainer was used to prevent any solid content or particulate that may be introduced and allowed for the reduction of air prior to spray application.

Coating Evaluation

Spray Parameters (Application Temperature, Humidity, Cure Time)

For industrial use of a protective coating, there must be ease of application through utilization of a spray gun. The preferred spray gun was a High Volume-Low Pressure (HVLP) spray gun. Figure 17[23] is an image of a standard HVLP gun in industry.

Figure 17: High Volume-Low Pressure (HVLP) spray gun

Key parameters for a coating are its viscosity and sprayability, and the main factor affecting these parameters are the percentage of solid contents in the coating formulation. Ideally, the coating will not require additional lubricants to become sprayable and after the spray application, there will be no remaining buildup of residue. For environmental considerations, a water reducible coating formulation was a primary down selection requirement as this characteristic would ensure easier maintenance of the spray gun components (nozzle, tip) and pressure pots which contained the coating.

For all four coatings, minimal amounts of solvent (MEK or MPK) were used to optimize sprayability of the coating. The composite panels were labeled and sprayed only on one side of the composite test panel. The coatings were sprayed using a High Volume-Low Pressure (HVLP) spray gun in a manner which gave a smooth, continuous, and uniform film, free of bubbles and particulate matter. Recommended pressure during sprays and material overlap for the protective coatings were provided by technical data sheets. Application of the coatings occurred at ambient conditions between 70-80°F and Relative Humidity between 30-50%.

Dry Time and Cure Time

The Dry Time of the coating is an important parameter in a production environment. A desired dry to handle time for the coating is approximately 1 hour. For each coating, three composite test panels were coated, and the test panels were tested in 10-minute intervals for dust free, dry-to-touch, and dry hard times in accordance with ASTM D 1640[24]. The first test interval began 10 minutes after the spray application was complete. All panels were dust free, dry-to-touch, and dry hard in approximately 40 minutes. For full cure of the coatings, guidance provided by the technical data sheets was cure time ranged from 6-7 hours for ambient temperatures between 70-80°F. Accelerated cure times could be achieved between 1-2 hours for temperatures between 140-150°F. The Dry Film Thickness of the coatings was between 0.5-3.0 mils.

Visual Appearance

Throughout mixing, spray application, and curing, the coatings were visually inspected for uniformity, smoothness, and to ensure no foreign object debris was present. The coatings were tested for visual appearance as a Pass or Fail. For each coating, composite test panels were coated and cured at ambient temperatures and accelerated cure temperatures. Additionally, large composite cutouts were coated in order to better visualize any potential imperfections. A total of six composite test panels were evaluated per coating. All coatings passed the Visual Appearance requirement. Figure 18 is an image of the four coatings.

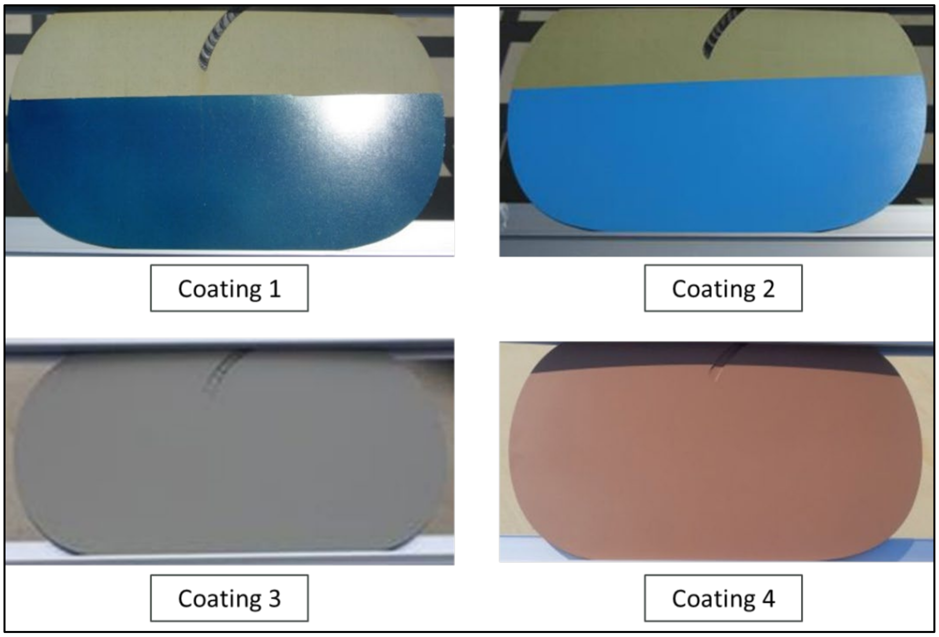


Figure 18: Coatings evaluated for Visual Appearance

UV Transmittance

In order to determine the UV Blocking Efficiency of each protective coating, a fused quartz microscope slide was coated with the respective coating and transmittance of light from 250 – 700 nm was evaluated using a Cary 5000 UV-Vis NIR Spectrophotometer. The main region of interest for the screening test was the UVA region which is between 280 – 400 nm. Bond dissociation energies in epoxy systems occur in the UVA wavelength region, approximately 280-400 nm. Bond dissociation due to UV absorption can initiate a process which ultimately leads to microcracking, water absorption, and hardening of the surface. UV Blocking results shows that Coating 3 transmitted more than 1% light for the majority of the UVA region. Coating 1, 2, and 4 transmitted less than 1% light throughout the UVA region (280 – 400 nm). The results of UV Blocking testing can be seen in Figure 19.

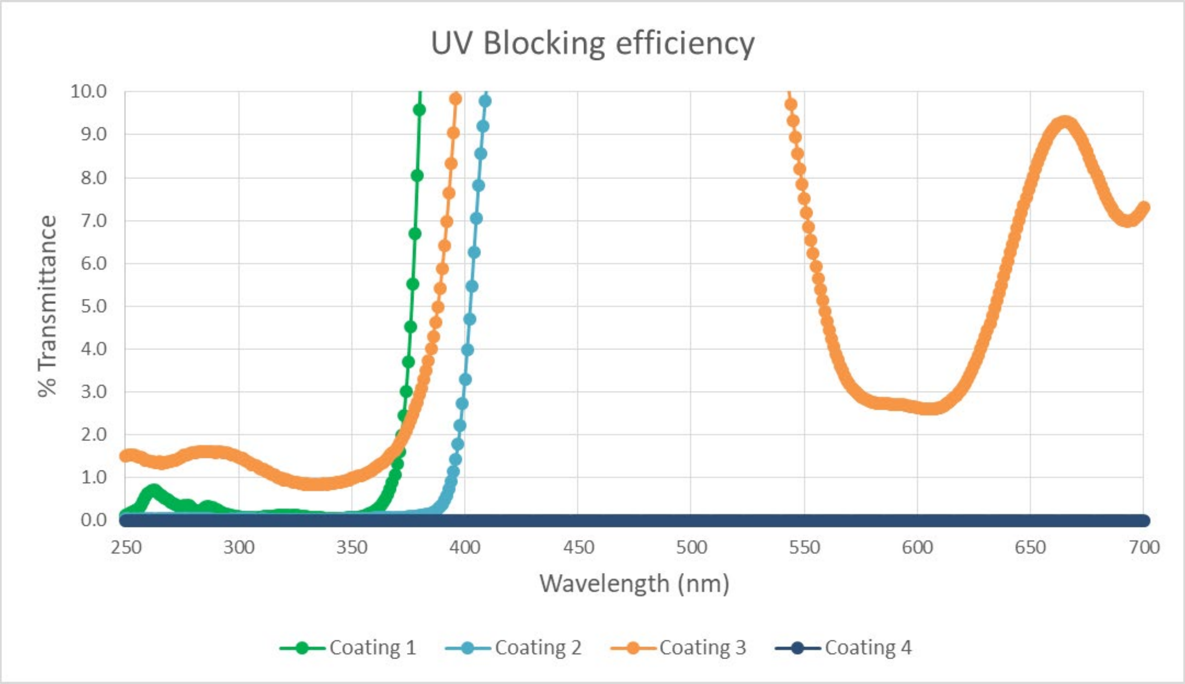


Figure 19: UV Spectroscopy of Four Protective Coatings

Environmental Exposure – Thermal Cycling Resistance

An Industrial specification that outlines the requirements of Thermal Cycling Resistance is ASTM D6944 – Standard Practice for Determining the Resistance of Cured Coatings to Thermal Cycling. “The purpose of this test is to obtain information on the ability of a coating system to adhere to substrates under thermal stress. It is assumed that the coating systems tested are applied and cured according to the coating manufacturer’s instructions.[25]” For each coating, three – 3 inch x 6 inch composite test panels were coated and cured at ambient temperatures. After cure, the panels were exposed to thermal cycling for 7 days ± 6 hours. Each cycle lasted 24 ± 1 hours consisting of 16 ± 1 hours at -10 ± 5 F followed by 8 ± 1 hours at 120 ± 5 F. After the thermal cycling was complete, the panels were visually inspected for signs of blistering, flaking, or cracking. The coatings were tested for visual appearance as a Pass or Fail. Thermal Cycling Resistance testing for each coating can be seen in Figures 20-23.

Figure 20: Thermal Cycling Resistance – Coating 1

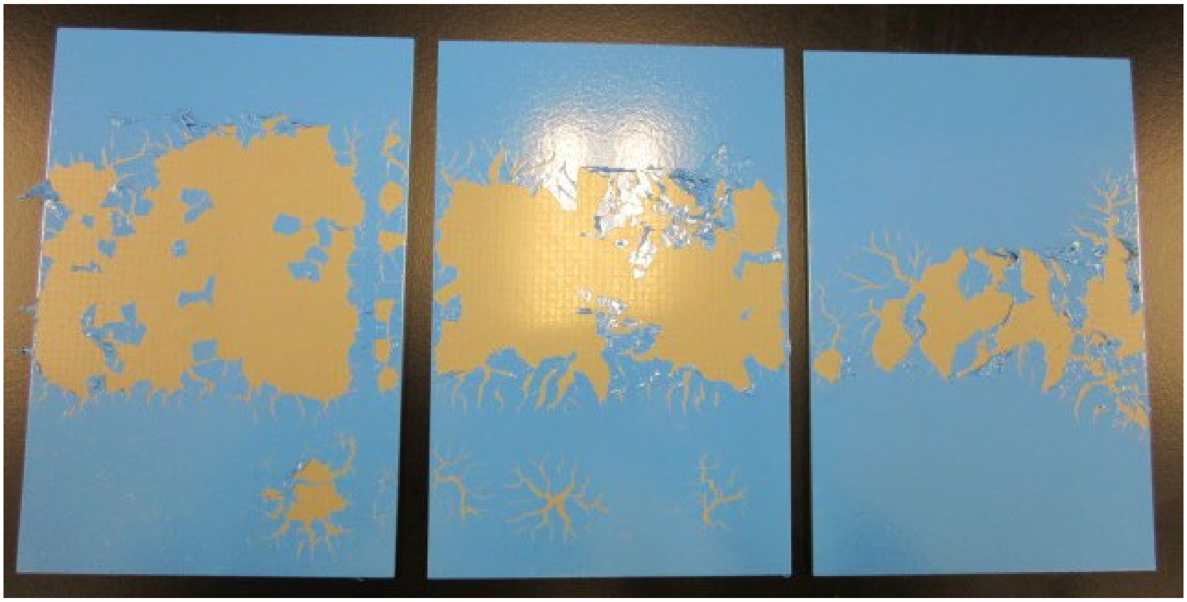


Figure 21: Thermal Cycling Resistance – Coating 2

Figure 22: Thermal Cycling Resistance – Coating 3

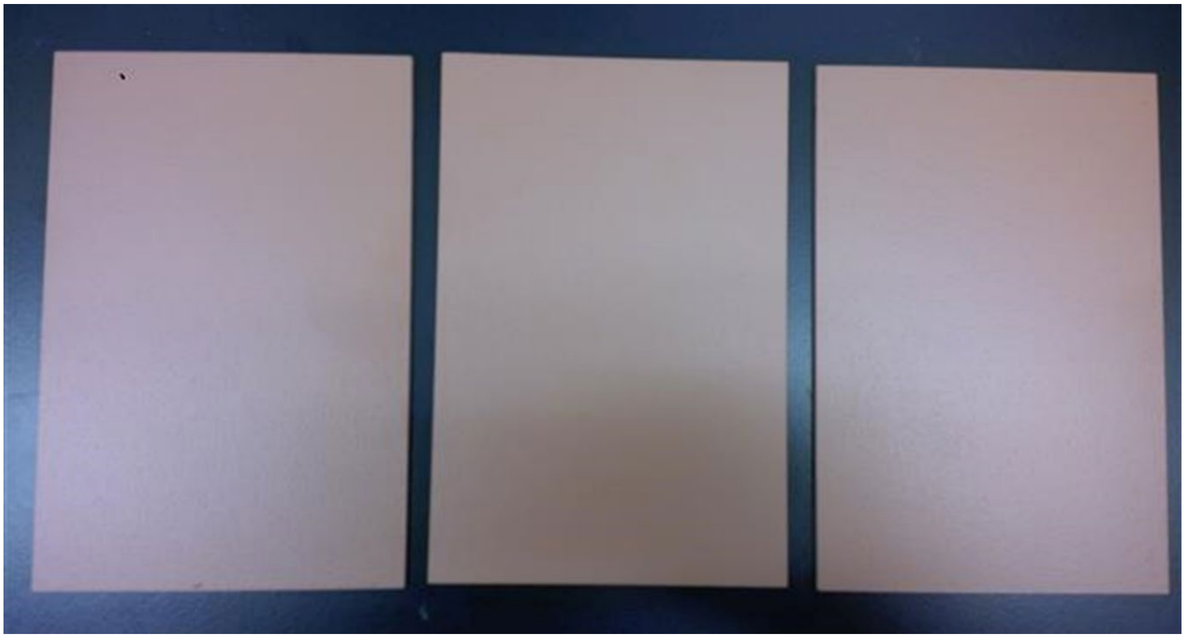


Figure 23: Thermal Cycling Resistance – Coating 4

Coating 1 and Coating 2 exhibited extreme cracking, flaking, and adhesion loss after Thermal Cycling exposure was completed. Coating 3 and Coating 4 did not exhibit any cracking, flaking, or adhesion loss, but Coating 3 showed signs of discoloration of the protective coating. Table 2 highlights the results of all Screening tests.

Table 2: Screening Test and Requirements for Coating Down Selection

Screening Test	**Requirement**
Spray Application	Smooth, continuous, uniform film free of particulate matter
Dry Time	Dry to touch in 1 hour
Visual Appearance	Uniform, smooth, free of foreign object debris
UV Transmittance	Less than 1% light throughout UVA region (280-400 nm)
Environmental Exposure - Thermal Cycling	No blistering, flaking, or cracking after thermal cycles

Table 3: Screening Test and Results for Coating Down Selection

Screening Test	**Coating 1**	**Coating 2**	**Coating 3**	**Coating 4**
Spray Application	Pass	Pass	Pass	Pass
Dry Time	Pass	Pass	Pass	Pass
Visual Appearance	Pass	Pass	Pass	Pass
UV Transmittance	Pass	Pass	Fail	Pass
Environmental Exposure - Thermal Cycling	Fail	Fail	Pass	Pass

Conclusion – Down Selection

Four commercial off the shelf coatings were evaluated by qualitative and quantitative screening tests: Spray Application, Dry Time, Visual Appearance, UV Transmittance, and Environmental Exposure – Thermal Cycling. All coatings were designed to protect individual aircraft parts in storage, during the assembly process, and for outdoor storage. Coating 4 was selected due to the optimal conditions seen throughout the down selection criteria. In particular, the dry time, combination of good UVA blocking chemistry of Titanium dioxide, and solvent ingredients already inherent in the formulation provided higher performance sprayability of the protective coating.

Chapter 4 Composite and Coating Evaluation

Introduction

After the completion of screening tests: Spray Application, Dry Time, Visual Appearance, UV Transmittance, and Environmental Exposure – Thermal Cycling, Coating 4 was down selected for further evaluation. Additional quantitative tests were performed to further evaluate the coating and to determine its ability to mitigate composite degradation of the underlying composite substrate. These tests included UV Transmittance at varying coating thickness, Weight Change due to Accelerated UV Exposure, Weight Change due to Condensing Humidity, Weight Change due to Outdoor Exposure and Optical Microscopy of a coated composite sample before and after Outdoor Exposure. Additional data was collected to determine the %Weight Change of the coated composite samples during the varying Environmental Exposure conditions.

UV Transmittance

Coating 4 showed high levels of UV Blocking Efficiency throughout the light spectrum. Further evaluations of UV Transmittance were performed for Coating 4 at varying Dry Film Thicknesses – 0.5, 1.0, 2.0, and 3.0 mils. Four fused quartz microscope slides were coated at varying thicknesses – 0.5 mils, 1.0 mils, 2.0 mils, 3.0 mils. The slides were evaluated for the transmittance of light across the UV and visible light spectrum (200 – 750 nm) using a Cary 5000 UV-Vis NIR Spectrophotometer. UV Blocking results show that Coating 4 transmits less than 1% light in the UV and visible light spectrum at all Dry Film Thicknesses. After evaluations, the coated quartz slides were mounted and exposed in an Atlas Ci4000 xenon arc weatherometer in accordance with ASTM D7869[26]. The slides were exposed in block cycles equivalent to a dosage of 250 kJ/m^2 per block. After each block, the panels were evaluated for their effective transmittance. The total exposure of the coated slides was 500 kJ/m^2. Throughout all evaluations, Coating 4 transmitted less than 1% light in the UV and visible light spectrum. The results of UV Blocking testing can be seen in Figures 24-26:

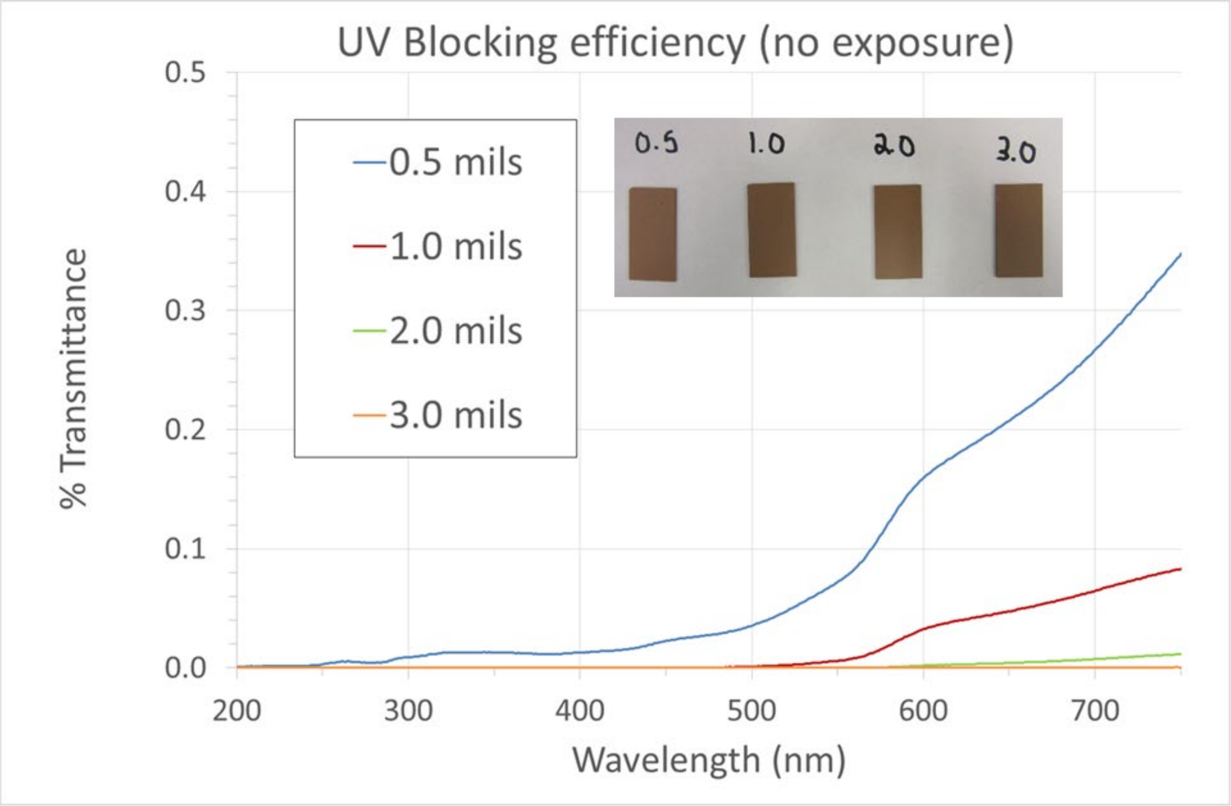


Figure 24: UV Blocking efficiency for varying coating thicknesses (no exposure)

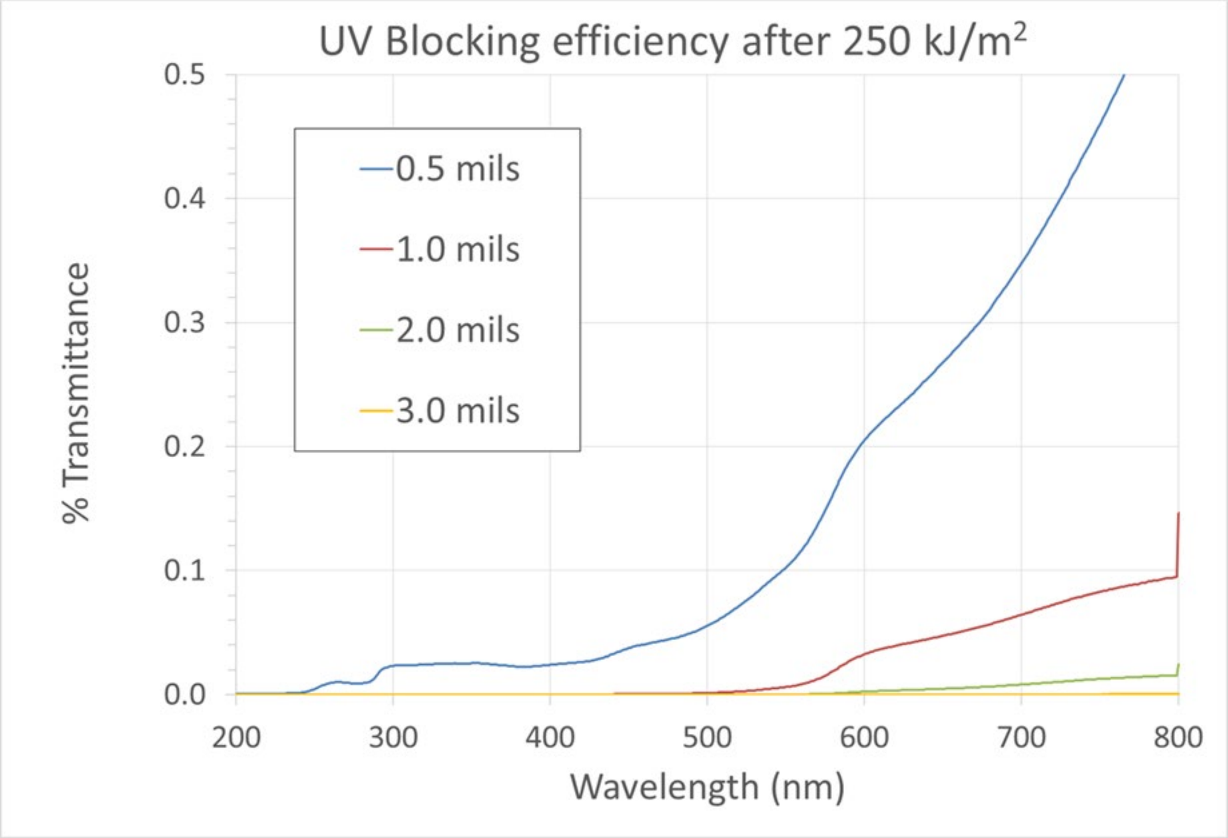


Figure 25: UV Blocking efficiency for varying coating thicknesses after 250 kJ/m^2

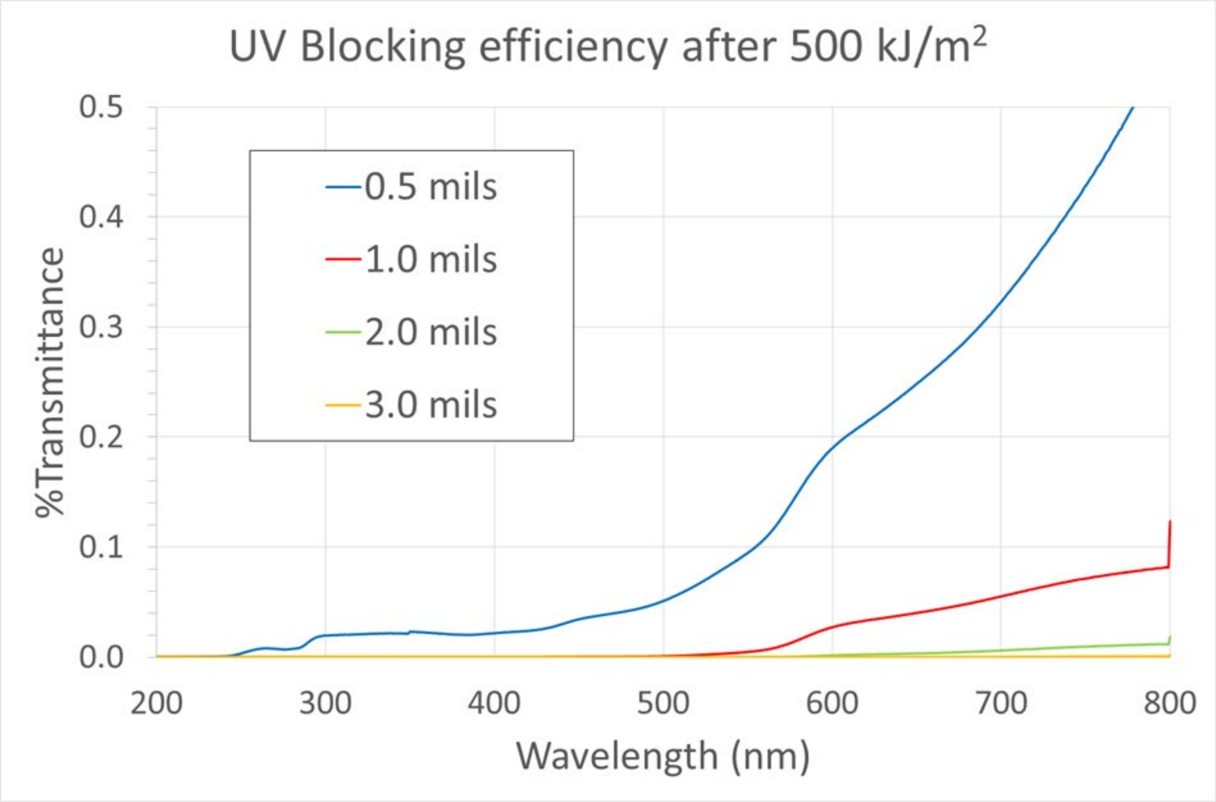


Figure 26: UV Blocking efficiency for varying coating thicknesses after 500 kJ/m^2

Weight change due to Environmental Exposure

Accelerated UV Exposure

One composite test panel was coated with Coating 4 and cured at ambient temperatures. The test panel was exposed in an Atlas Ci4000 xenon arc weatherometer in accordance with ASTM D7869 for block cycles equivalent to a total dosage of 500 kJ/m^2. Figure 27[27] is an image of an Atlas UV weatherometer.

Figure 27: Atlas UV weatherometer utilized for Accelerated UV Exposure

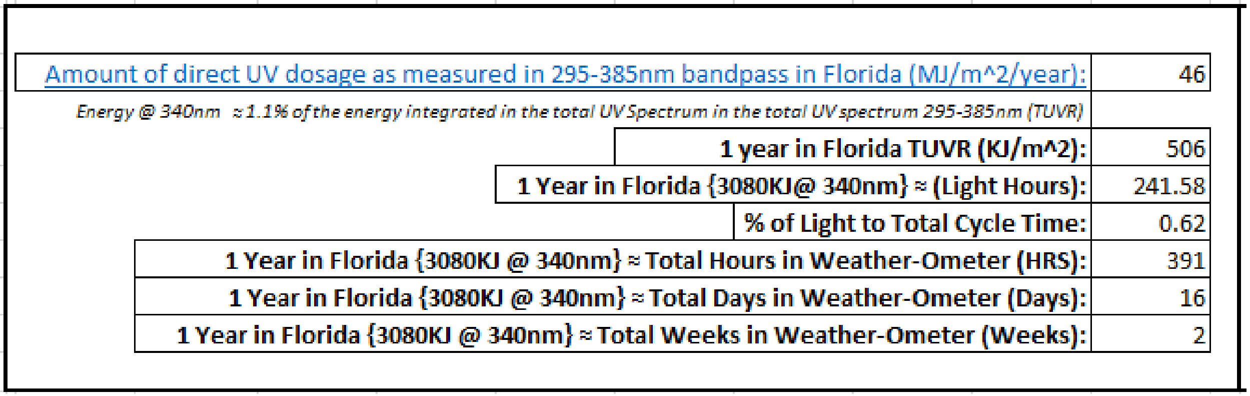
Testing panels in an accelerated environment provides an opportunity to simulate outdoor exposure conditions in a real-world environment. Calculations to correlate the amount of Accelerated UV Exposure to Outdoor sunlight are below:

$$Light\ Hrs = \frac{KJ}{Irradiance\ \times 3.6\, Ksec/hr}$$

$$\%\ of\ Light\ To\ Total\ Cycle\ Time = \frac{Light\ Hours\ per\ Cycle}{Total\ Cycle\ Time}$$

$$Total\ Hours\ in\ Weatherometer = \frac{Light\ Hours\ per\ Cycle}{\%\ of\ Light\ to\ Total\ Cycle\ Time}$$

Table 4: Correlation between Accelerated UV Exposure and Outdoor Exposure



Amount of direct UV dosage as measured in 295-385nm bandpass in Florida (MJ/m^2/year):	46
Energy @ 340nm ≈1.1% of the energy integrated in the total UV Spectrum in the total UV spectrum 295-385nm (TUVR)	
1 year in Florida TUVR (KJ/m^2):	506
1 Year in Florida {3080KJ@ 340nm} ≈ (Light Hours):	241.58
% of Light to Total Cycle Time:	0.62
1 Year in Florida {3080KJ @ 340nm} ≈ Total Hours in Weather-Ometer (HRS):	391
1 Year in Florida {3080KJ @ 340nm} ≈ Total Days in Weather-Ometer (Days):	16
1 Year in Florida {3080KJ @ 340nm} ≈ Total Weeks in Weather-Ometer (Weeks):	2

At regular intervals, the composite test panel was weighed on an analytical balance to monitor any changes in weight. Figure 28 is an image of the coated composite test panel being weighted on an analytical balance.

Figure 28: Coated composite test panel being weighted on an analytical balance

After the completion of all block cycles and weight measurement time intervals, the data was plotted. Figure 29 is the Environmental Exposure duration (hours) vs. Weight (grams) and Figure 30 is the Environmental Exposure duration (hours) vs. Percent Weight Change.

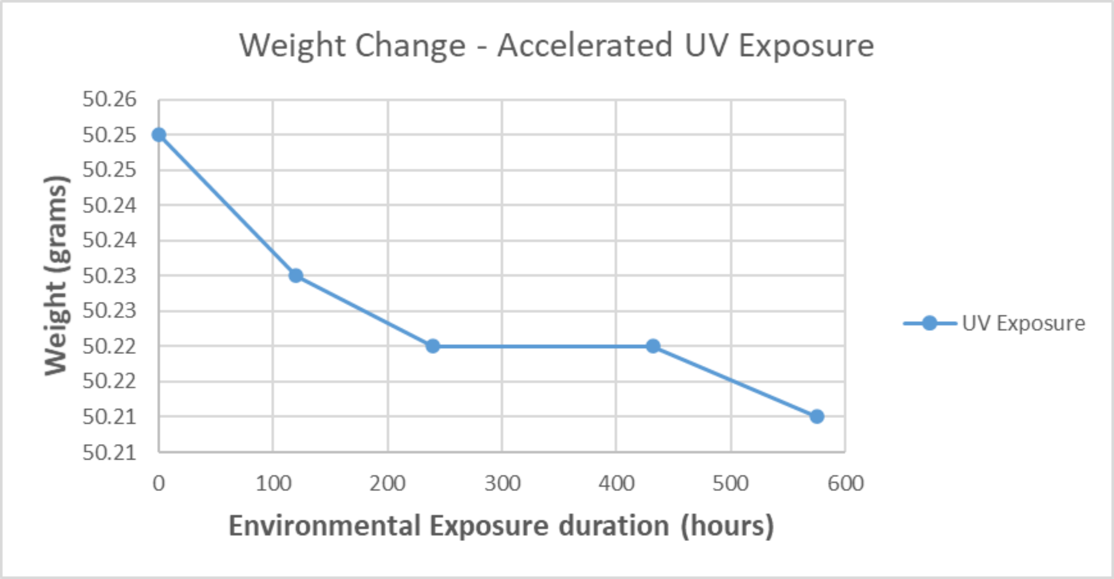


Figure 29: Environmental Exposure duration (hours) vs. Weight (grams)

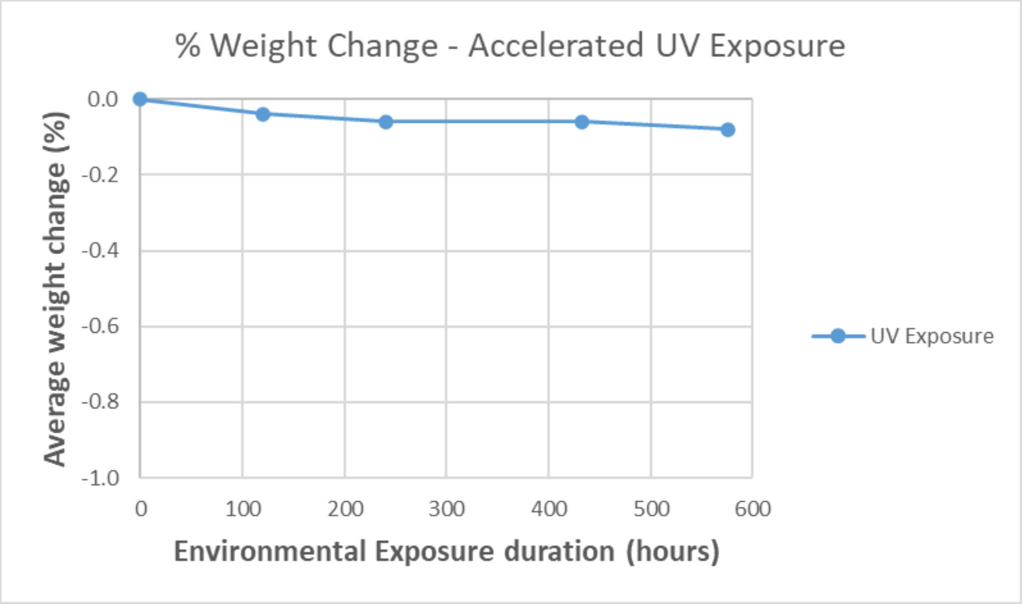


Figure 30: Environmental Exposure duration (hours) vs. Average (%) Weight Change

The panel exposed to Accelerated UV in a weatherometer demonstrated a decrease in weight over time. The overall percent weight change decreased approximately 0.1% after 600 hours. This data is consistent with additional research of the independent variable of UV light exposed to a polymeric coating or composite material. Yousif and Haddad noted that UV radiation causes photooxidative degradation which results in breaking of polymer chains, produces radicals and reduces the molecular weight[28].

Condensing Humidity

One composite test panel was coated with Coating 4 and cured at ambient temperatures. The test panel was exposed in a Thermotron humidity chamber to Condensing Humidity in accordance with ASTM D 2247[29]. ASTM D 2247 Condensing Humidity requirements outline

100 % Relative Humidity, but the humidity chamber utilized for this research was only capable of reaching 95 ± 5 % Relative Humidity throughout the duration of testing. The temperature of the humidity chamber was thermostatically maintained at a temperature of 120 ± 5°F. The test panel was placed into the humidity chamber for approximately 1000 hours. The panel was removed from the chamber and blotted with an absorbent cloth prior to being weighted on an analytical balance. At regular intervals, the composite test panel was weighed to monitor any changes in weight. After the completion of all weight measurement time intervals, the data was plotted. Figure 31 is the Environmental Exposure duration (hours) vs. Weight (grams) and Figure 32 is the Environmental Exposure duration (hours) vs. Percent Weight Change.

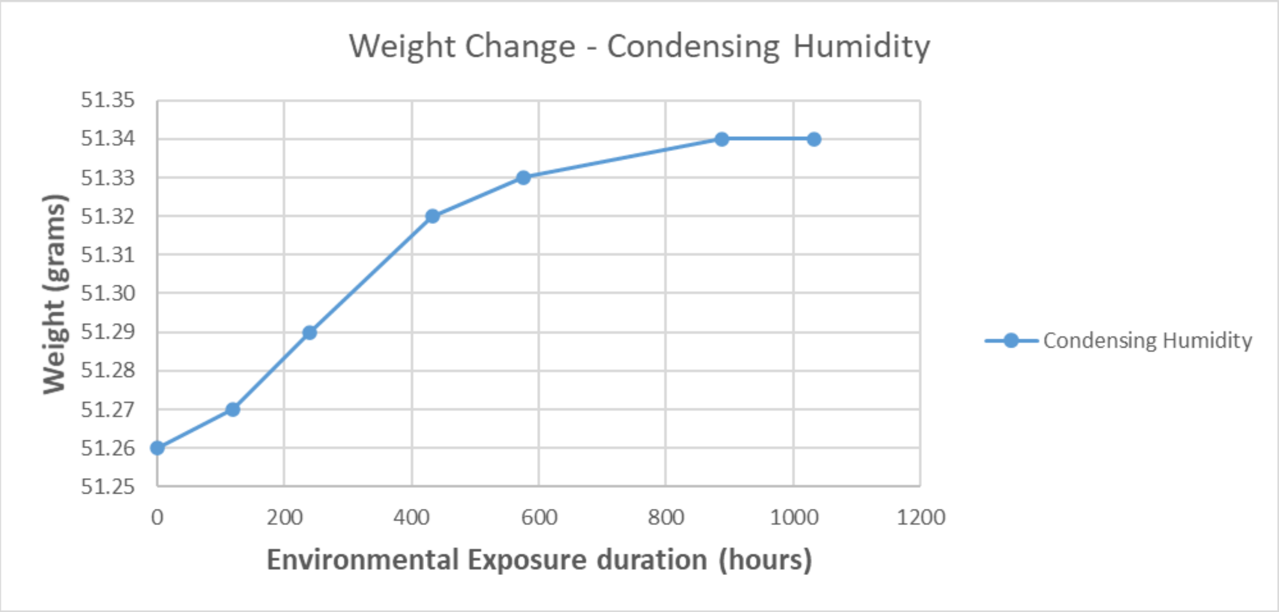


Figure 31: Environmental Exposure duration (hours) vs. Weight (grams)

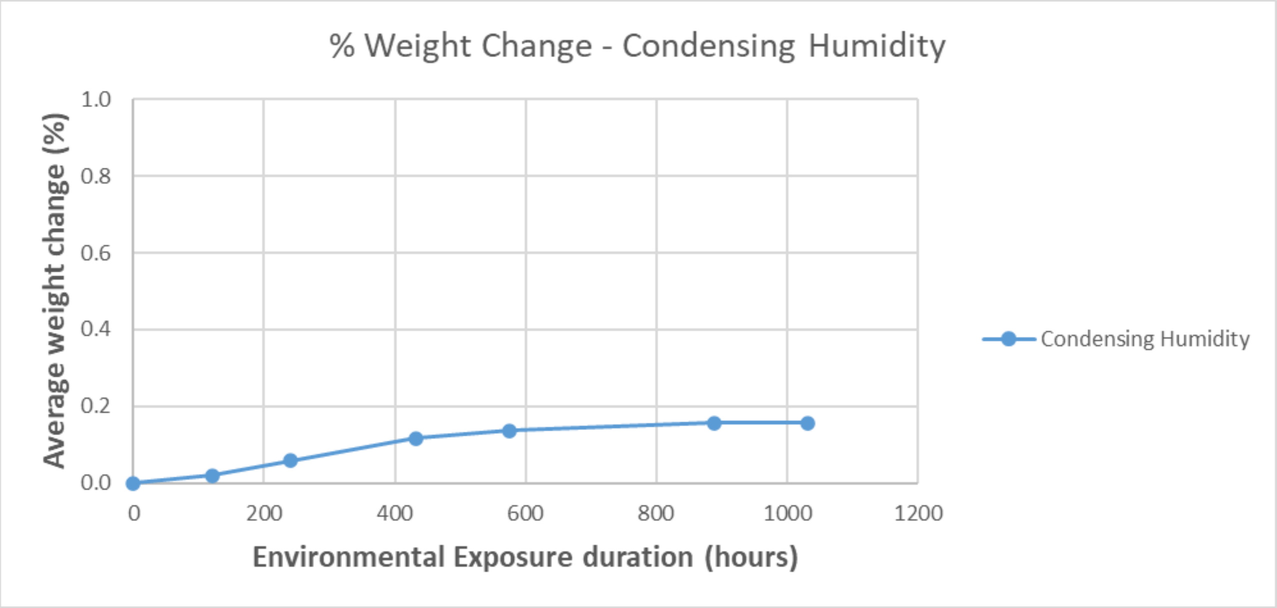


Figure 32: Environmental Exposure duration (hours) vs. Percent Weight Change

The panel exposed to Condensing Humidity in the Thermotron humidity chamber demonstrated an increase in weight over time. The overall percent weight change increased approximately 0.2% after 1000 hours. This data is consistent with additional research of the independent variable of the exposure of moisture content to a polymeric coating or composite material[14].

Outdoor Exposure

Six – 3 inch by 6 inch panels were coated with Coating 4 and cured at ambient temperatures. The test panel were placed on an outdoor exposure rack in Charleston, SC for 1000 hours. Figure 33 is an image of the Outdoor Exposure rack.

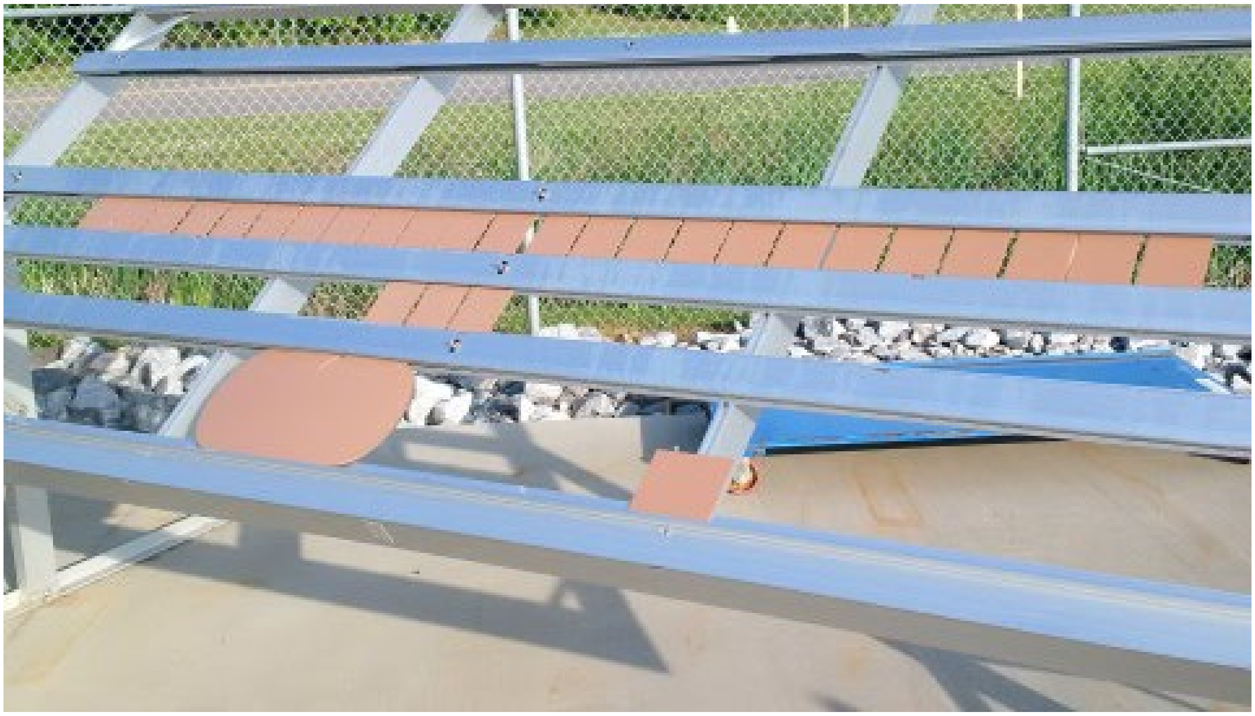


Figure 33: Coated composite panels on Outdoor Exposure rack

At regular intervals, the composite test panels were weighed on an analytical balance to monitor any changes in weight. After the completion of all weight measurement time intervals, the data was plotted. Figure 34 is the Environmental Exposure duration (hours) vs. Weight (grams) and Figure 35 is the Environmental Exposure duration (hours) vs. Percent Weight Change.

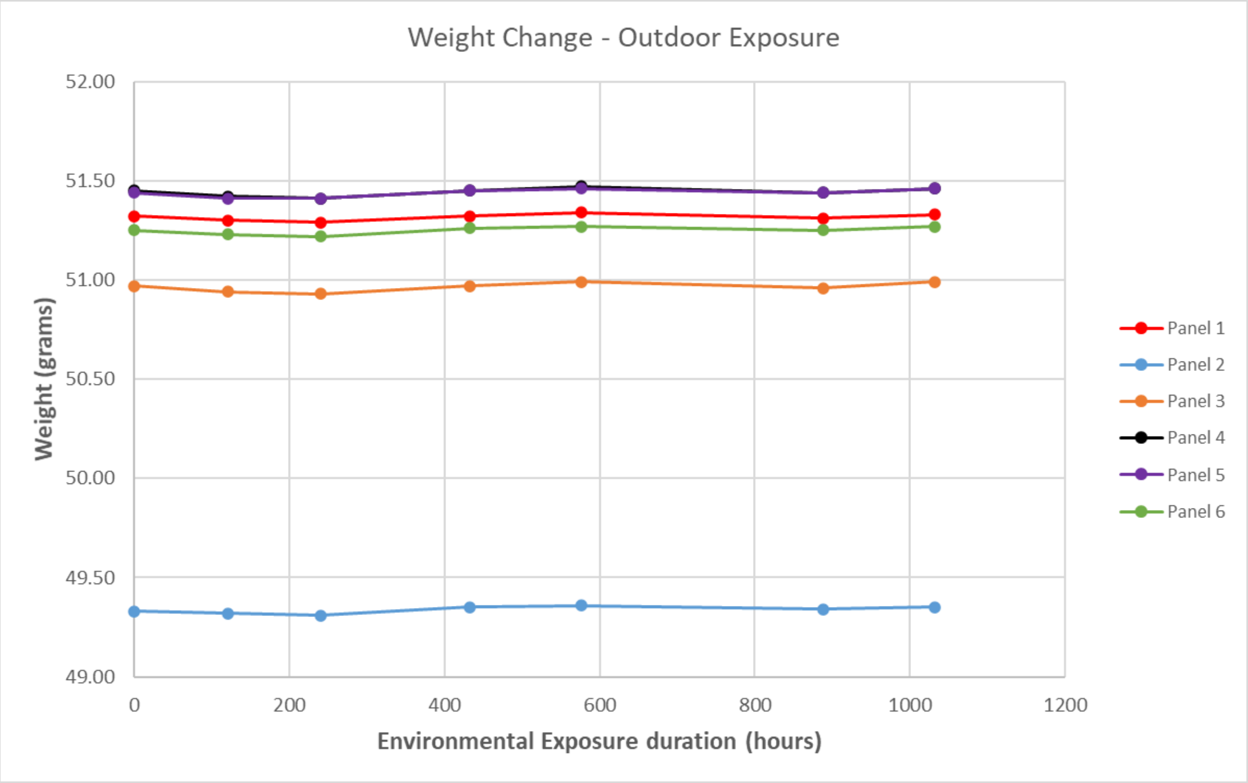


Figure 34: Environmental Exposure duration (hours) vs. Weight (grams)

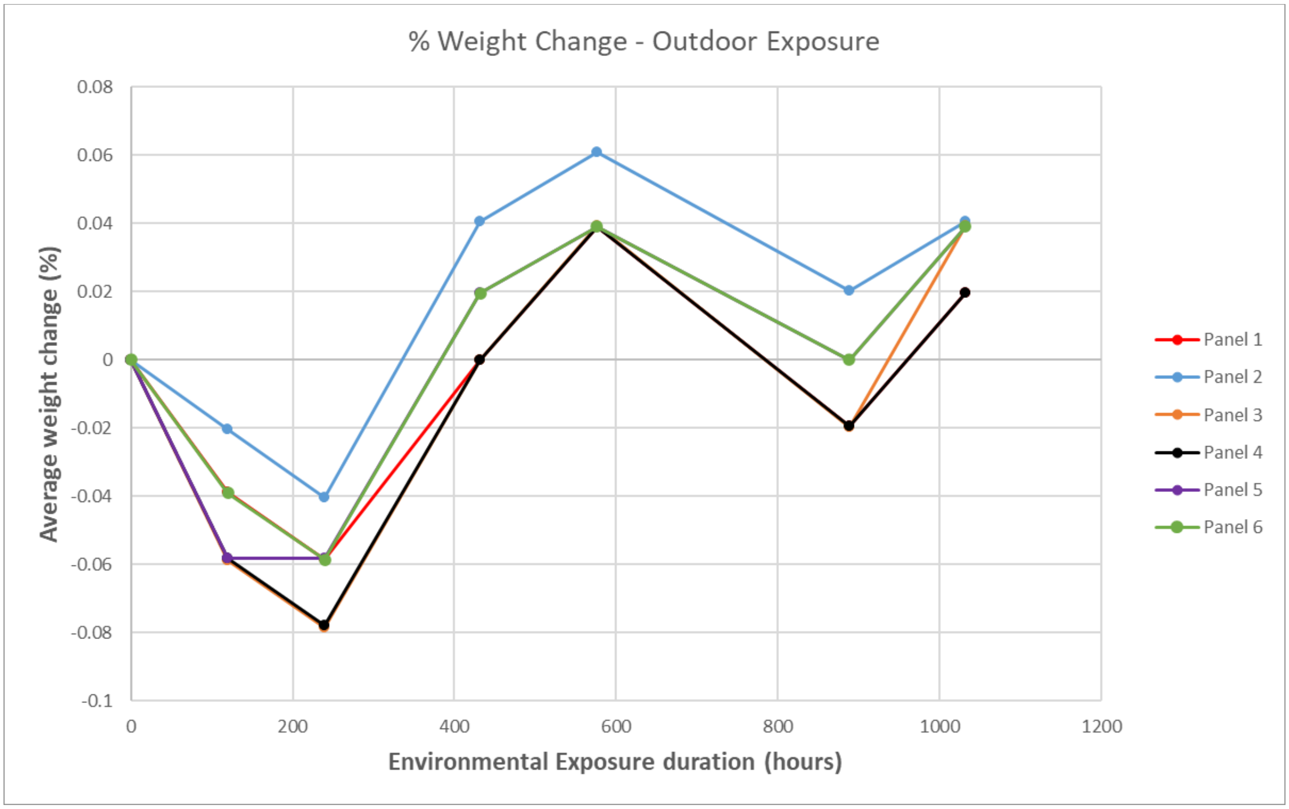


Figure 35: Environmental Exposure duration (hours) vs. Percent Weight Change

The panels exposed to Outdoor Exposure endured a combination of simultaneous environmental conditions including extreme temperatures, humidity, and moisture. In studying the relative weight change from start time to finish time of 1000 hours, it appears as if there is very minimal weight change. Evaluation of the percent weight change over 1000 hours shows that all six panels demonstrated an initial percent weight loss during the first 200 hours of exposure. For approximately 250 hours to 550 hours of exposure, all six panels demonstrated a percent weight gain. From approximately 600 hours to 900 hours of exposure, all six panels again demonstrated a percent weight loss, but the percent weight loss was smaller than the initial percent weight loss. The last 100 hours of exposure for a total of 1000 hours of exposure demonstrated a slight percent weight gain. The cyclical nature of the weight loss vs weight gain is consistent with literature. Boer et. al noted that moisture damage begins near the surface of the material and spreads inwards over time[30]. The timing of the weight measurements and the unpredictability of the weather during Outdoor Exposure were critical factors considered during this research.

Optical Microscopy

One composite test panel was coated with Coating 4 and cured at ambient temperatures. The coated panel was evaluated using a Keyence Optical Microscope prior to Outdoor Exposure. Figure 36 is the initial surface profile showing a smooth continuous texture. Figure 37 is the final surface profile of coated composite panel after Outdoor Exposure. The surface profiles appear relatively unchanged by the environmental effects over the length of exposure.

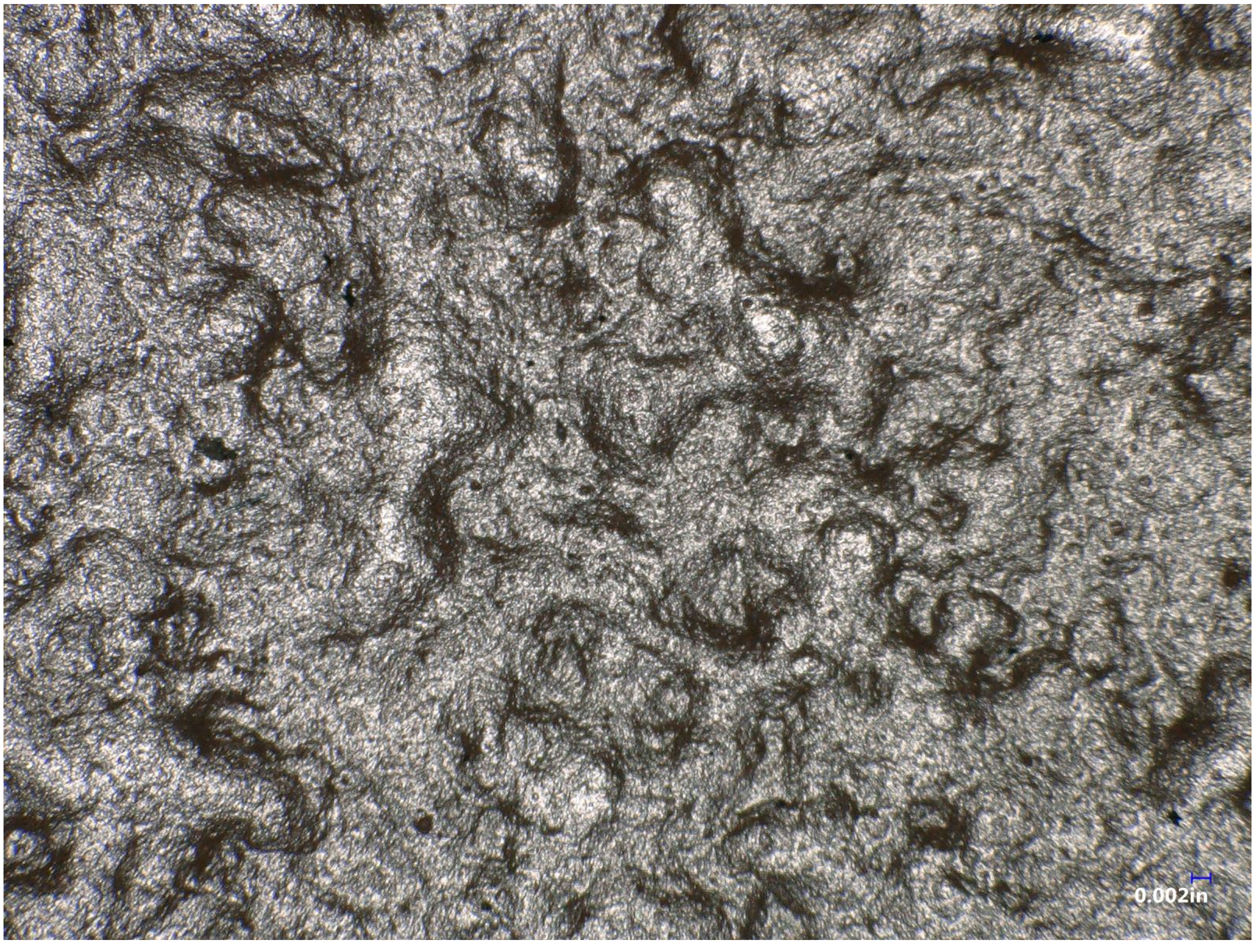


Figure 36: Initial surface profile of coated composite panel

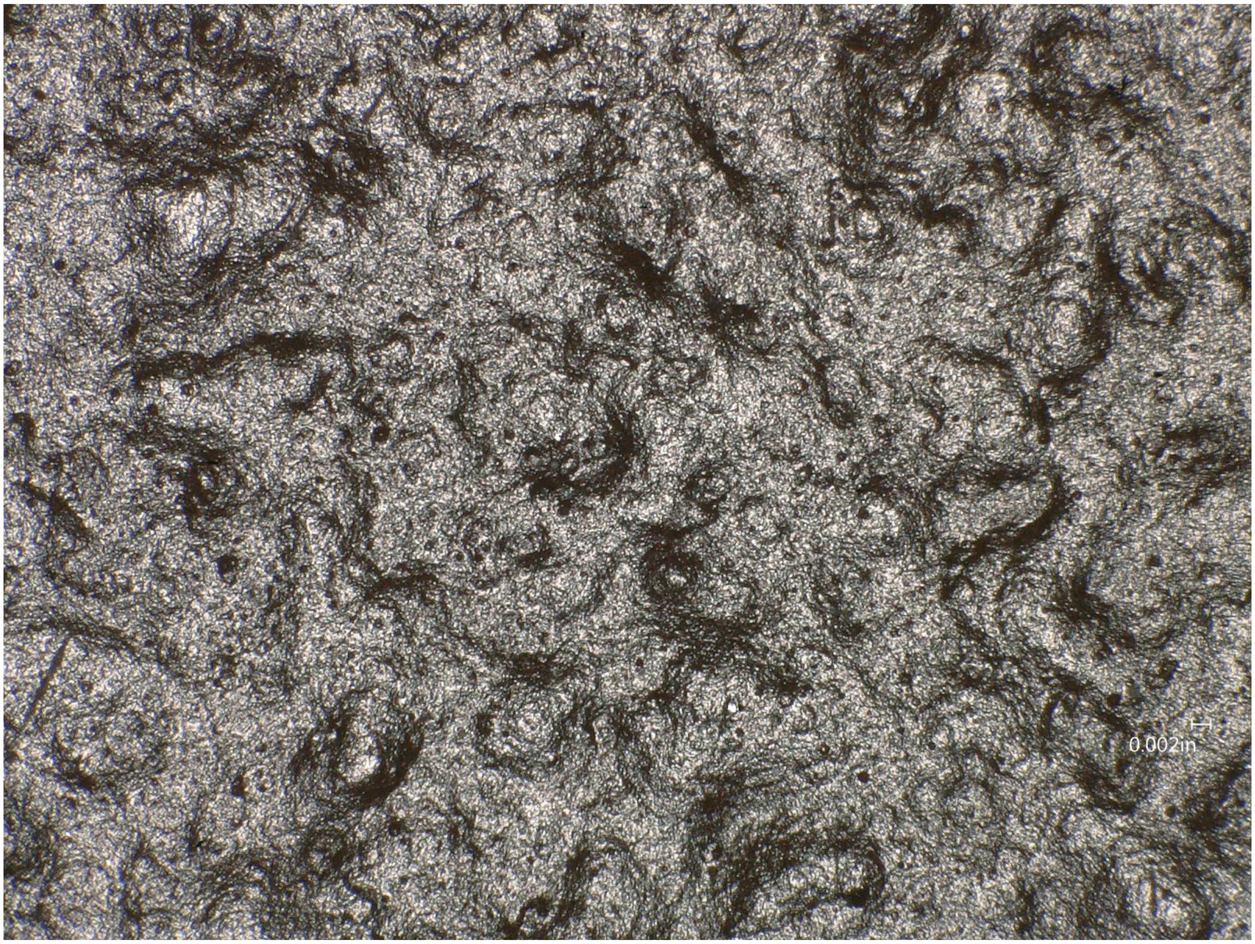


Figure 37: Final surface profile of coated composite panel after Outdoor Exposure (1000 hours)

Conclusion

Quantitative tests were performed to further evaluate the capability of Coating 4 to mitigate composite degradation of the underlying composite substrate. These tests included UV Transmittance at varying coating thickness, Weight Change due to Accelerated UV Exposure, Weight Change due to Condensing Humidity, Weight Change due to Outdoor Exposure and Optical Microscopy of a coated composite sample before and after Outdoor Exposure. Additional data was collected to determine the Percent Weight Change of the coated composite samples during the varying Environmental Exposure conditions. UV Transmittance for Coating 4 was less than 1% light in the UV and visible light spectrum for all thickness ranges 0.5, 1.0, 2.0, and 3.0 mils. This was initially missed during the UV Transmittance screening tests for all four coatings due to the fact that the UV Transmittance scale required that UV Transmittance Percent was captured up to 10%. After the coated quartz slides were exposed to a total of 500 kJ/m^2, the UV Transmittance remained below 1% indicating that the Coating 4 formulation with a combination of UV stabilizers and silicate fillers effectively blocks and absorbs UV and visible light. Additionally, the optical micrographs showed minimal changes to the surface morphology of the coating pre- and post-UV exposure conditions. The quantitative weight change witnessed throughout testing provides a foundation to develop and verify a UV Degradation Kinetic Model.

Chapter 5 Development and Validation of Kinetic Model

Introduction

The cumulative dosage model[31] [32] has been widely accepted in various industries and provides suitable parameters to independently verify the overall effect of a system. Synergistic effects and assumptions can be hard to predict, and where applicable, assumptions have been provided for the UV Degradation modeling and Condensing Humidity modeling of the protective coating.

UV Degradation Modeling

In the development of a kinetic model for UV degradation exposure of composite materials and polymeric coatings, the modified cumulative dosage model[17] is below.

$$D_{total}(t) = \int_0^{\tilde{t}} \int_{\lambda_{min}}^{\lambda_{max}} E_0(\lambda, t)\left(1 - e^{-A(\lambda)}\right)\phi(\lambda)\,d\lambda\,dt$$

where λ_{min} and λ_{max} are the minimum and maximum effective wavelengths (nm). $A(\lambda)$ is the absorbance of the sample at a specified UV wavelength, (dimensionless). $E_0(\lambda)$ is the incident dose (W/m^2) of UV radiation which interacts with the polymeric coating. $\phi(\lambda)$ is the quantum yield which is the number of times a specific event occurs per photon absorbed by the material. The elapsed time (t) and the total radiation time $\tilde{t}$ have units of seconds. $D_{total}(t)$ is the total effective dosage (J/m^2). Some key assumptions for usage of the cumulative dosage model for this experiment are below:

1) All areas of the coating and composite material receive the same environmental exposure (dosage of UV, temperature, moisture, condensation, humidity) throughout the respective time of exposure.

2) The specimens are “flat” and any changes in surface roughness are negligible when compared to the effect of the environmental exposure on the coated composite material.

3) Where applicable, there is no UV radiation transmitted through both the protective coating and composite material. This was confirmed through UV spectroscopy of the protective coating showing less than 0.1% UV Transmittance over UVA range (315-400 nm) for coatings of various thickness (0.5, 1.0, 2.0, 3.0 mils). The conclusion is that the protective coating fully absorbs all effective wavelengths making the probability of the absorption of UV photons equal to 1. Mathematically, this is represented: $e^{-A(\lambda)} \approx 0$.

4) Based on the chemistry of the protective coating, the effective UV wavelength causing primary photodegradation is within the UVA range (315-400 nm). The quantum efficiency number[19] for this range of wavelengths is 10^{-4}.

5) Photodegradation due to UV photons results in the complete removal of the uppermost layer of the coating and chemical changes of the coating are minimal.

The resulting assumptions provide a simplified equation:

$$D_{total}(t) = E_0 \times \phi \times t$$

The incident spectral UV radiation dose, E_0, was provided by the Atlas UV chamber specification, and the quantum efficiency for the effective absorbed wavelength, ϕ, was provided by literature. A relationship between UV degradation and polymeric materials can be approximated by a linear equation, exponential equation, or a power law equation. A quantitative critical material characteristic can be measured and evaluated to build an equation and model damage kinetics. For this research, the critical material characteristic was specimen weight and a linear dependence was identified yielding the equations below:

$$\mathit{\Omega} = \left(\frac{W_i - W_f}{W_i} \times 100\right)$$

$$\mathit{\Omega} = cD_{total}$$

$$\left(\frac{W_i - W_f}{W_i} \times 100\right) = cD_{total}$$

where Ω is the critical material characteristic which is the percent weight change. W_i is the initial weight (grams) before environmental exposure, W_f is the final weight after environmental exposure, and c is an empirical constant.

In order to evaluate the UV degradation model of the protective coating, one test panel was exposed in an Atlas UV chamber in accordance with ASTM G154[33] for block cycles equivalent to a total dosage of 500 kJ/m^2. At regular intervals, the composite test panel was weighed on an analytical balance to monitor any changes in weight. The data points at the regular intervals were plotted and an approximate linear fit was matched to the data. Figure 38 is the Percent Weight Change of the coated composite panel after UV exposure for 600 hours with a linear fit.

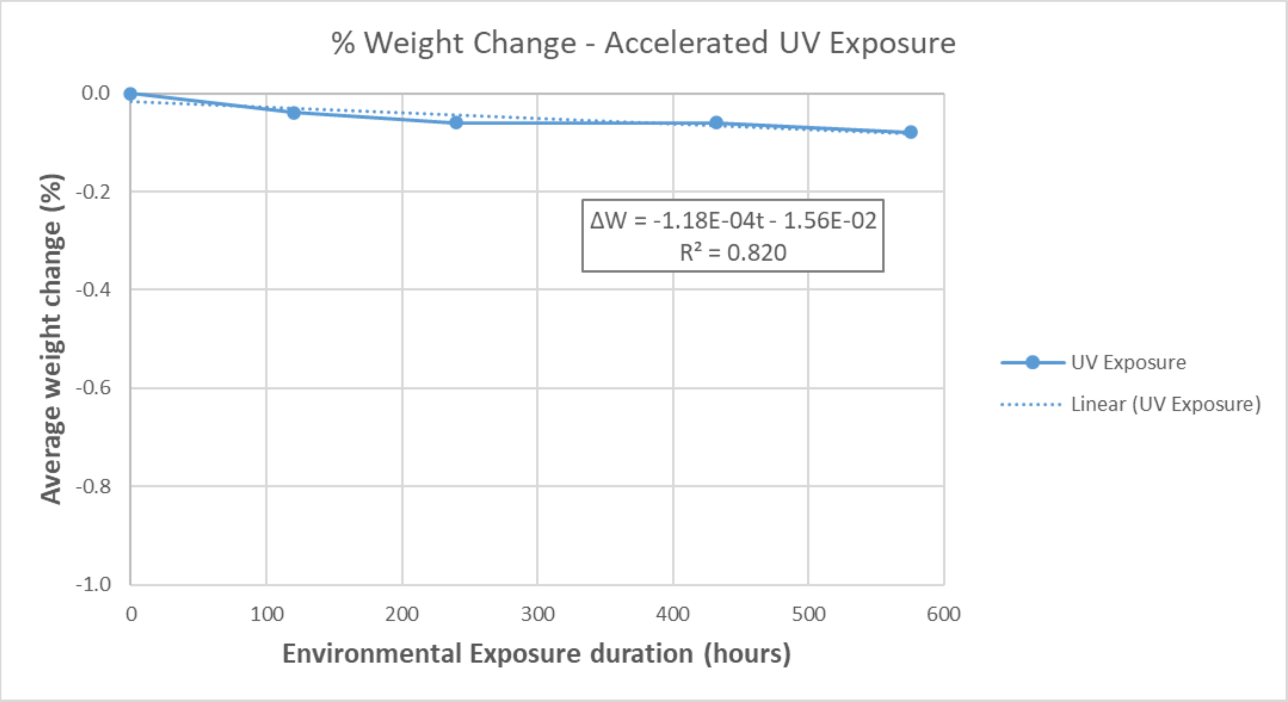


Figure 38: Linear Trend of % Weight Change of coated composite panel exposed to UV light for 600 hours

The UV degradation model has a c value of 1.18×10^{-4} for an equation:

$$\left(\frac{W_i - W_f}{W_i} \times 100\right) = cD_{total}$$

$$\left(\frac{W_i - W_f}{W_i} \times 100\right) = -1.18 \times 10^{-4} D_{total}$$

The physical parameters for the UV exposure modeling are listed in Table 5:

Physical Parameters	**Value**
Sensitive wavelength, λ	315-400 nm
Radiation intensity, Eo	1.5 W/m2
Quantum yield φ	10-4
Exposure time, t	600 h (2.6 x 106s)
Exposure area	11.6 x 10-3 m2

Condensing Humidity Modeling

In the development of a kinetic model for Condensing Humidity exposure to composite materials and polymeric coatings, the modified cumulative dosage model is below:

$$D_{total}(t) = \int_0^{\tilde{t}} \int_{\varphi_{min}}^{\varphi_{max}} E_0(\varphi, t)\left(1 - e^{-A(\varphi)}\right)\phi(\varphi)d\varphi dt$$

where φ_{min} and φ_{max} are the minimum and maximum effective moisture content (g/m). $A(\varphi)$ is the absorbance of the sample at a specified moisture content level, (dimensionless). $E_0(\varphi)$ is the incident dose (g/m^3) of moisture content which interacts with the polymeric coating. $\phi(\varphi)$ is the relative humidity in the chamber and is a constant. The elapsed time (t) and the total exposure time $\tilde{t}$ have units of seconds. $D_{total}(t)$ is the total effective dosage (g/m^2) of moisture into the samples. Some key assumptions for usage of the cumulative dosage model for this experiment are below:

1) All areas of the coating and composite material receive the same environmental exposure (temperature, moisture, condensation, humidity) throughout the respective time of exposure.

2) The specimen is "flat" and any changes in surface roughness are negligible when compared to the effect of the environmental exposure on the coated composite materials.

3) The specimen is "dry" upon entering the Thermotron chamber and there is a negligible effect on the specimen by moisture in the air or in the chamber. The conclusion is that the incident, E_0, moisture content is equal to a non-zero constant.

4) Where applicable, there is no moisture that transmits through both the protective coating and composite material. The conclusion is that the protective coating and composite material fully absorb all effective moisture making the probability of the absorption of moisture equal to 1. Mathematically, this is represented: $e^{-A(\lambda)} \approx 0$.

5) The Percent Relative Humidity in the chamber was calculated to be a constant 95 ± 5 %.

6) Where applicable, degradation due to moisture content occurred primarily in the uppermost layer of the coating and chemical changes of the coating are minimal[34 35].

The resulting assumptions provide a simplified equation:

$$D_{total}(t) = E_0 \times \phi \times t$$

A relationship between Condensing Humidity moisture content and polymeric materials can be approximated by a linear equation, exponential equation, or a power law equation. A quantitative critical material characteristic can be measured and evaluated to build an equation and model damage kinetics. For this research, the critical material characteristic was specimen weight and a linear dependence was identified yielding the equations below:

$$\Omega = \left(\frac{W_i - W_f}{W_i} \times 100\right)$$

$$\Omega = cD_{total}$$

$$\left(\frac{W_i \ - \ W_f}{W_i} \times 100\right) = cD_{total}$$

where Ω is the critical material characteristic which is the percent weight change. W_i is the initial weight (grams) before environmental exposure, W_f is the final weight after environmental exposure, and c is an empirical constant.

In order to evaluate the Condensing Humidity model of the protective coating and composite panel, one test panel was exposed in a Thermotron humidity chamber to Condensing Humidity in accordance with ASTM D 2247. ASTM D 2247 Condensing Humidity requirements outline 100 % Relative Humidity, but the humidity chamber utilized for this research was only capable of reaching 95 ± 5 % Relative Humidity throughout the duration of testing. The temperature of the humidity chamber was thermostatically maintained at a temperature of 120 ± 5°F. The test panel was placed into the humidity chamber for approximately 1000 hours. The panel was removed from the chamber and blotted with an absorbent cloth prior to being weighted on an analytical balance. At regular intervals, the composite test panel was weighed to monitor any changes in weight. Figure 39 is the Percent Weight Change of the coated composite panel after Condensing Humidity exposure for 1000 hours with a linear fit.

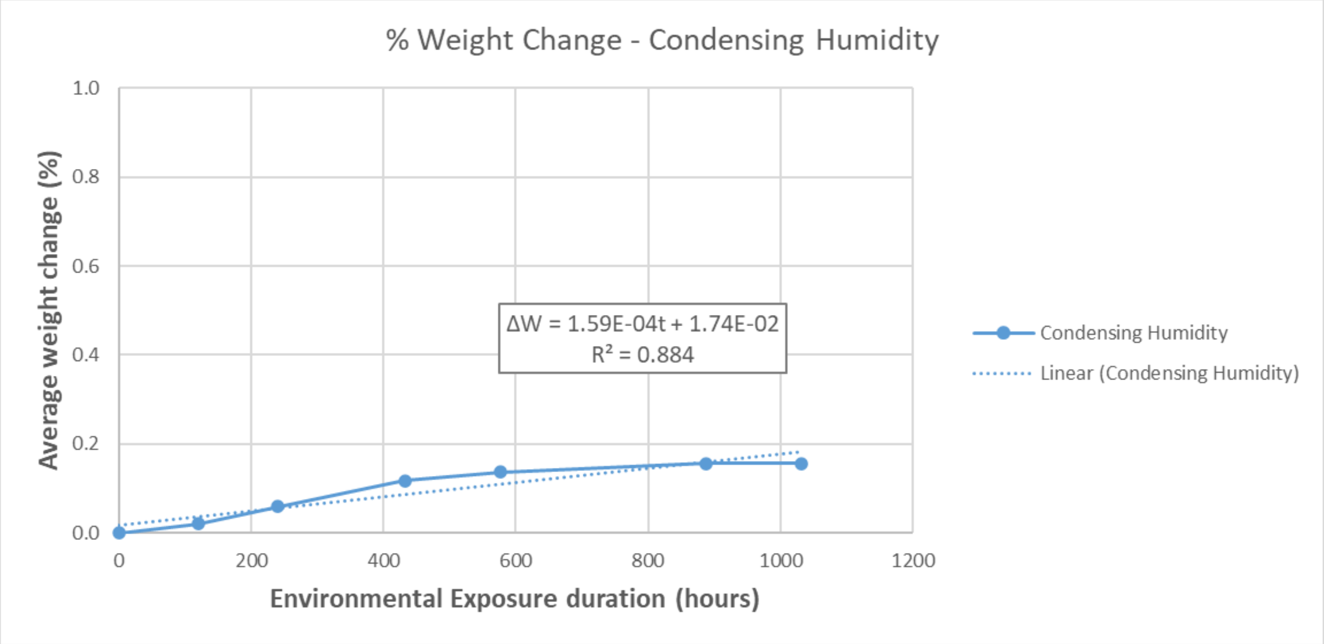


Figure 39: Linear Trend of % Weight Change of coated composite panel exposed to Condensing Humidity for 1000 hours

The Condensing Humidity model has a c value of 1.59×10^{-4} for an equation:

$$\left(\frac{W_i - W_f}{W_i} \times 100\right) = cD_{total}$$

$$\left(\frac{W_i - W_f}{W_i} \times 100\right) = 1.59 \times 10^{-4} D_{total}$$

The physical parameters for the Condensing Humidity modeling are listed in Table 6:

Table 5: Physical parameters for the Condensing Humidity modeling

Physical Parameters	**Value**
Relative Humidity, ϕ	95 ± 5 %
Moisture Content, φ	0-0.08 g/m^2
Incident moisture content, E_0	Non-zero constant
Exposure time, t	1000 h (3.6 x 10^6 s)
Exposure area	11.6 x 10^{-3} m^2

Fitting the Model

A significant change in weight of the coated composite panel due to UV Degradation or Condensing Humidity would allow for validation of the model using the provided linear equations. For a small change in weight comparable to the data in this research, the estimated rate of degradation can be verified by directly measuring the weight change of individual specimens[17]. The cumulative dosage model utilizing the aforementioned assumptions for the UV Degradation equation and Condensing Humidity equation were combined to validate synergistic effects. A theoretical cumulative dosage model equation encompassing the UV Degradation equation and Condensing Humidity equation yielded the final equation below.

UV Degradation Model: $$\left(\frac{W_i - W_f}{W_i} \times 100\right) = -1.18 \times 10^{-4} D_{total}$$

Condensing Humidity Model: $$\left(\frac{W_i - W_f}{W_i} \times 100\right) = 1.59 \times 10^{-4} D_{total}$$

Cumulative Dosage Model: $$\left(\frac{W_i - W_f}{W_i} \times 100\right) = 0.41 \times 10^{-4} D_{total}$$

$D_{total}(t)$ is the total effective dosage (g/m^2) of UV and moisture respectively into the samples and correlates to the relative exposure time (hours).

Validation of the Model

The Cumulative Dosage Model used to predict the synergistic parameters of UV Degradation and Condensing Humidity were compared to experimental data. Coated composite panels were placed on an outdoor exposure rack in Charleston, SC for 1000 hours. The exposed panels endured a combination of simultaneous environmental conditions including extreme temperatures, humidity, and moisture. At regular intervals, the panels were weighed on an analytical balance to monitor any changes in weight. The data points at the regular intervals were plotted, but a linear fit could not be established based on the fluctuation of weight changes seen in the coated composite panels over time (Figure 35). There are four distinct phases in the Outdoor Exposure graph that were fit linearly to evaluate the rate constants to the UV

Degradation and Condensing Humidity constants identified in Chapter 4. The outdoor exposure rate constants identified for the parameter “c” were larger than the identified rate constants in the earlier experiments. Figure 40 is the Environmental Exposure duration (hours) vs. Percent Weight Change graph highlighting the four phases of one of the coated composite panels exposed outdoors for 1000 hours.

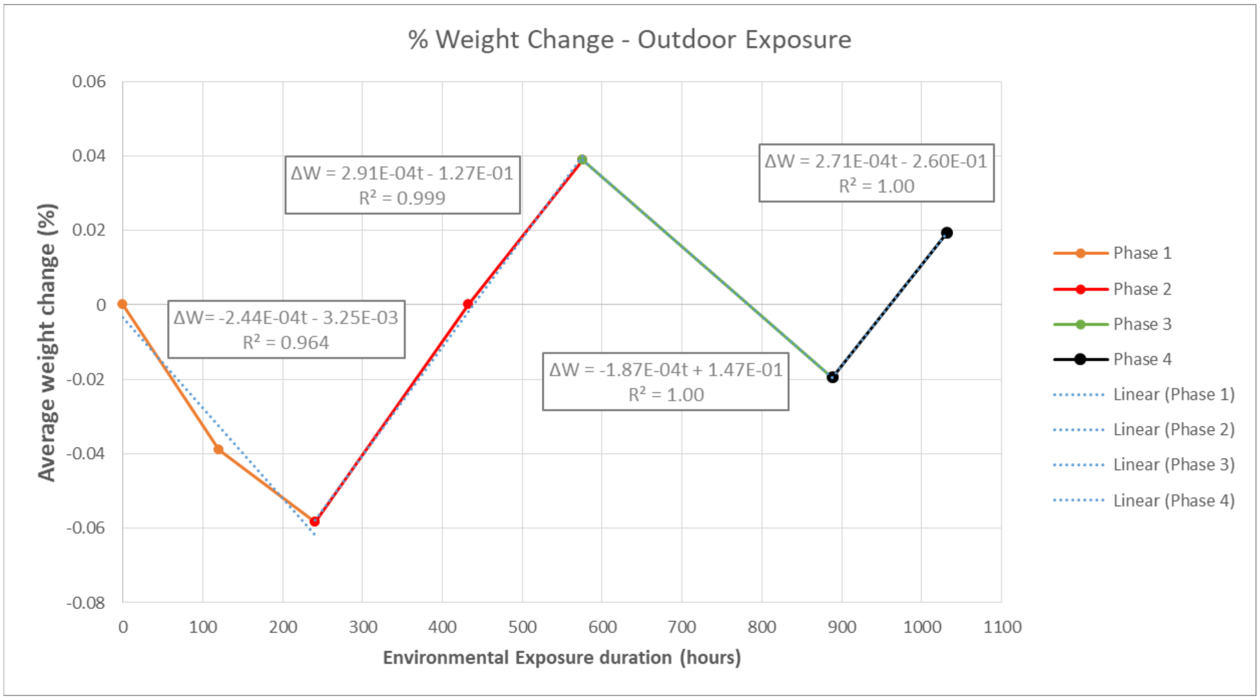


Figure 40: Environmental Exposure duration (hours) vs. Percent Weight Change linearly fit in four phases

Conclusion

The data plotted for the coated composite panels did not allow for a linear fit. The assumption based solely on the synergistic UV Degradation equation and Condensing Humidity equation suggests that the rate of weight loss over time due to UV degradation ($-1.18 \times$

$10^{-4}D_{total}$) would be smaller than the rate of weight gain over time due to Condensing Humidity ($1.59 \times 10^{-4}D_{total}$). While the data agrees with the theoretical prediction of overall weight gain, there are additional factors that ultimately challenge the predictive model and complicate the ability to fully model all aspects of environmental outdoor exposure. The experimental data aligns with predicted behavior of composite materials exposed to outdoor environmental conditions of varying temperatures, humidity, moisture, and condensation in that the initial primary weight change is a reduction in overall weight. The first noted mechanism of deterioration to a sample will occur predominantly through UV radiation causing the breakage of polymer chains through photooxidative aging. The physical removal of a UV degraded material from the surface as assumed in the model rarely occurs as prescribed. Additional factors such as surface adhesion could prevent full removal of particles from the surface. Lu et. al noted that combined UV and water condensation creates an effective condition for small polymer particles formed by UV to be subsequently removed by water condensation exposing fresh still undamaged surfaces to further UV degradation[36]. The undamaged surfaces provide an opportunity for additional surface erosion to occur greater than a damaged surface not exposed to moisture or condensation.

Chapter 6 Evaluation of Additional Aerospace Production Environmental Effects

Introduction

Qualitative tests performed on protective coated composite panels can help with providing a better understanding of the capability of the coating to withstand environmental effects experienced in an aerospace production facility. A selection of identified tests was chosen to replicate differing environments and production processes consistent with the manufacturing, assembling, and transport of large composite airplane sections across the globe. The tests performed were Water Resistance, Abrasion Resistance, Flammability, Impact Resistance, and Evaluation of Adhesion between the protective coating and an assembly coating. The adhesion evaluation included conditioning of the protective coated composite panels in differing environments – Condensing Humidity, Outdoor Exposure, and Accelerated UV Exposure and water. Preliminary results identified during the quantitative evaluation of the protective coating in Chapter 4 determined that the optimal coating thickness should be approximately 1.0 mil. For all future tests, this was the assumed dry film thickness.

Water Resistance

Three – 3 inches by 6 inches test panels were immersed vertically in distilled water for 7 days +/- 6 hours at 70 to 80°F. The water covered one-half of the panel length and the coated surfaces did not contact each other during immersion. Figure 41 shows the test panels removed immediately after the 7-day immersion. The panels were allowed to dry for 24 hours at 70 to 80°F. The panels were visually examined and tested for adhesion in accordance with ASTM

D3359[37], an industry standard cross hatch scribe test. A scribe tool is utilized to cut parallel lines through the coating and into the composite panels. Another set of parallel lines is cut perpendicularly to the first set of parallel lines, through the coating, and into the composite panels. An industry standard adhesive tape is affixed to the cut lines, and pulled in an upward abrupt motion perpendicular to the test surface in order to induce failure of the coating to adhere to the composite panel. Evidence of adhesion failure between the coating and composite panel will be seen on the adhesive tape. Relative levels of adhesion can be scored per industry standards. All test panels passed adhesion testing indicating no loss of the coating along scribe lines and very slight loss of the coating beyond scribe lines. Adhesion testing results can be seen in Figure 42.

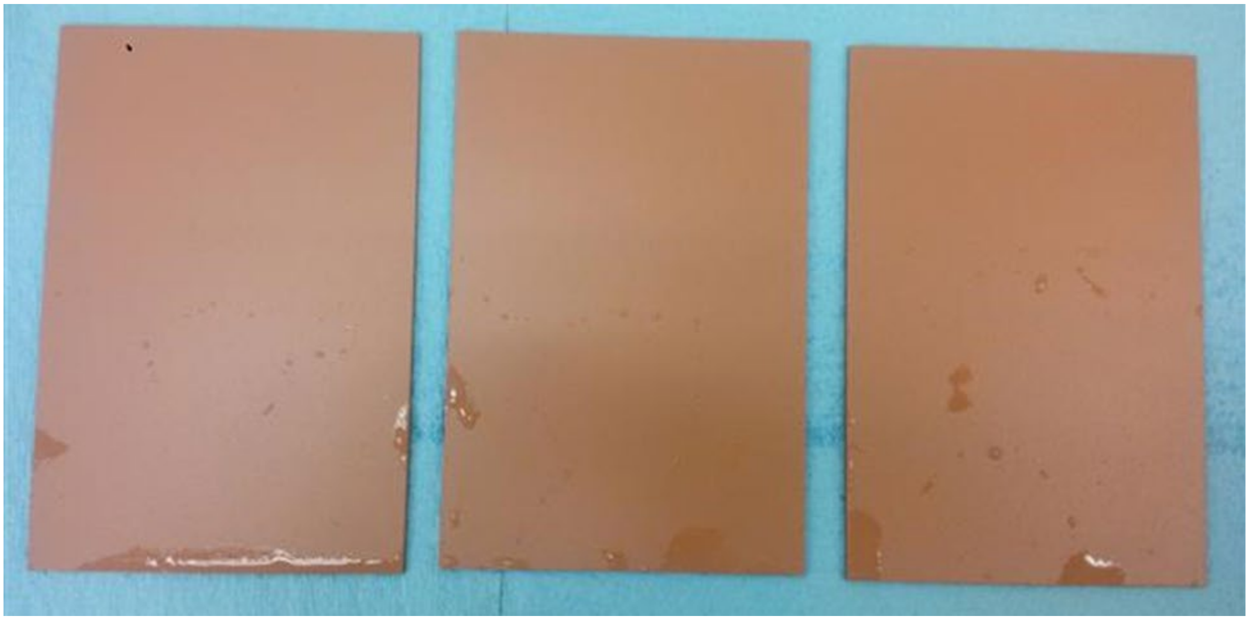


Figure 41: Coated composite panels removed from distilled water after 7 days

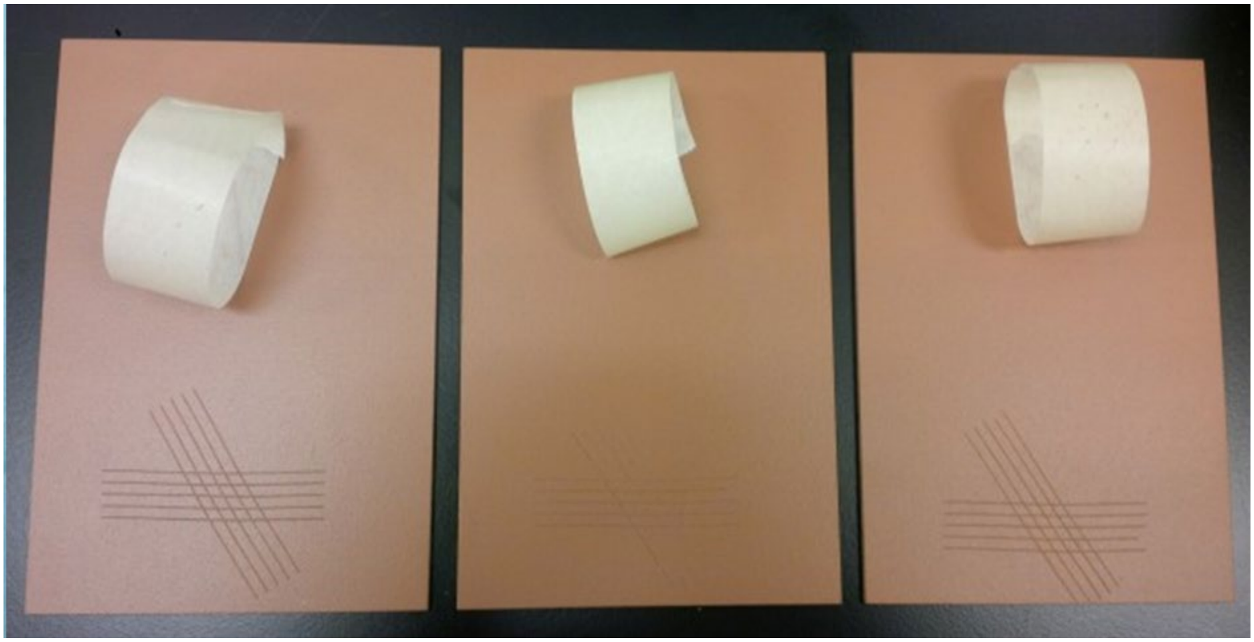


Figure 42: Coated composite panels after adhesion testing

Abrasion Resistance

Three – 3 inches by 6 inches test panels were tested in accordance with ASTM D4060[38] using a Taber Abraser 5155. The load weight was 250 grams and two test panels were cycled for 1000 cycles using a CS-10 wheel. The third test panel received the same load weight and 1000 cycle time using a CS-10F wheel. After 500 cycles, the wheels were resurfaced with a resurfacing stone for 25 cycles. Visible wear down to the bare composite surface could not be seen. Abrasion resistance testing results can be seen in Figure 43.

Figure 43: Coated composite panels after abrasion testing

Flammability

Three – 3 inches by 12 inches panels were coated and tested for flammability using a 12 second ignition Vertical Burn test. An additional uncoated test panel was tested as a control. All test panels passed flammability with a burned length of 0.1 inches or less. Flammability testing results can be seen in Figure 44.

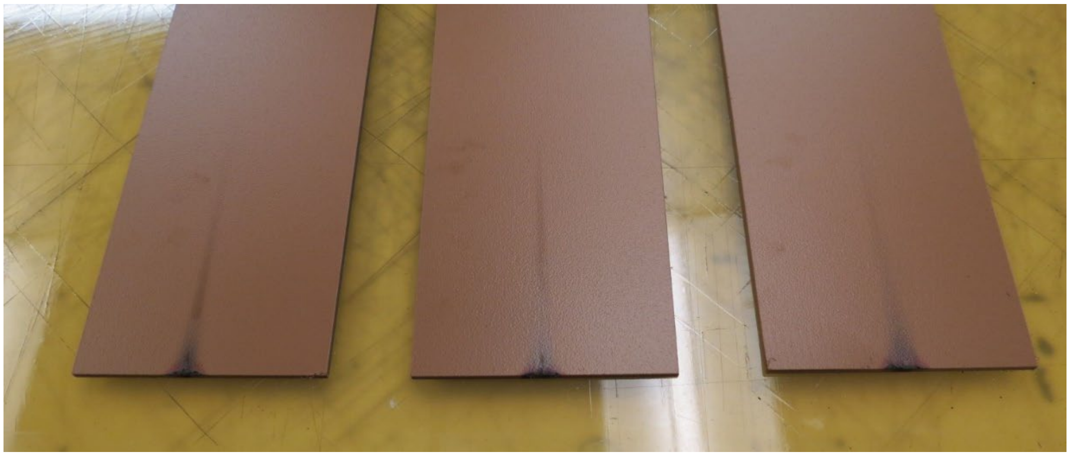


Figure 44: 12 second ignition Vertical Burn Test

Impact Resistance

Three – 3 inches by 6 inches test panels were coated and subjected to an impact of 20 ± 2 inch pounds using a Gardco Universal Impact Tester in accordance with ASTM D2794[39]. An industry standard adhesive tape was applied over the impacted spot and firmly pressed down. The tape was pulled in an upward abrupt motion perpendicular to the test surface in order to induce failure of the coating to adhere to the composite panel. Evidence of adhesion failure between the coating and composite panel will be seen on the adhesive tape. Relative levels of adhesion can be scored per industry standards. All test panels were visually examined and showed no removal of the coating. Impact Resistance testing results can be seen in Figure 45.

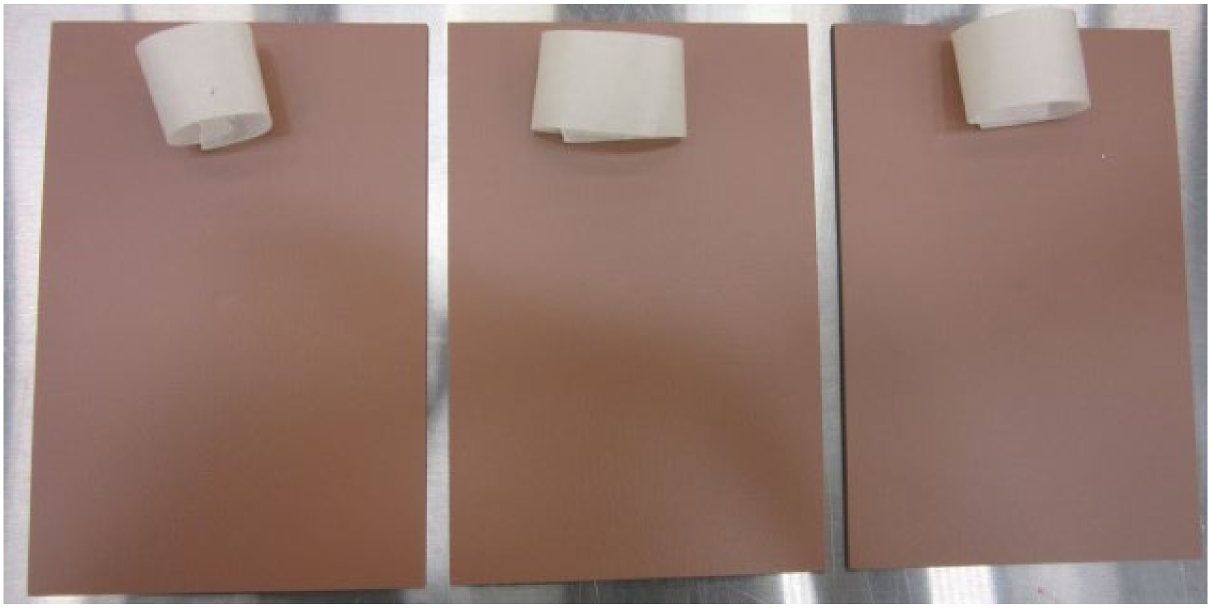


Figure 45: Coated composite panels after impact and adhesion testing

Evaluation of Adhesion between protective coating and assembly coating

Condensing Humidity

Three – 3 inches by 6 inches test panels were exposed to Condensing Humidity in accordance with ASTM D 2247[29] using a Thermotron humidity chamber. Condensing Humidity requirements outline 100 % Relative Humidity, but the humidity chamber utilized for this research was only capable of reaching 95 ± 5 % Relative Humidity throughout the duration of testing. The temperature of the humidity chamber was thermostatically maintained at a temperature of 120 ± 5°F. Test panels were placed into the humidity chamber for 30 days. The panels were removed from the chamber and blotted with an absorbent cloth prior to an initial visual examination. The panels were then allowed to stand for 24 hours in an atmosphere at 77 ± 5°F having a Relative Humidity of 50 ± 10 %. The panels were then visually re-examined. During examinations, the coatings showed no signs of blistering, flaking, or cracking. Condensing Humidity testing results can be seen in Figure 46.

Figure 46: Visual inspection of coated composite panels after Condensing Humidity exposure

After inspection, the panels were coated at ambient temperatures with a white epoxy high solid non chromated assembly topcoat used in aerospace production environments. The dry film thickness of the assembly topcoat was 0.8 mils. The panels were allowed to cure at ambient conditions for 24 hours and were visually examined and tested for adhesion in accordance with ASTM D3359, an industry standard cross hatch scribe test. A scribe tool is utilized to cut parallel lines through the coating and into the composite panels. Another set of parallel lines is cut perpendicularly to the first set of parallel lines, through the coating, and into the composite panels. An industry standard adhesive tape is affixed to the cut lines, and pulled in an upward motion to induce failure of the coating to adhere to the composite panel. Evidence of adhesion failure between the coating and composite panel will be seen on the adhesive tape. Relative levels of adhesion can be scored per industry standards. All test panels passed adhesion testing indicating no loss of paint along scribes and very slight loss of paint beyond scribes. Adhesion testing results can be seen in Figure 47.

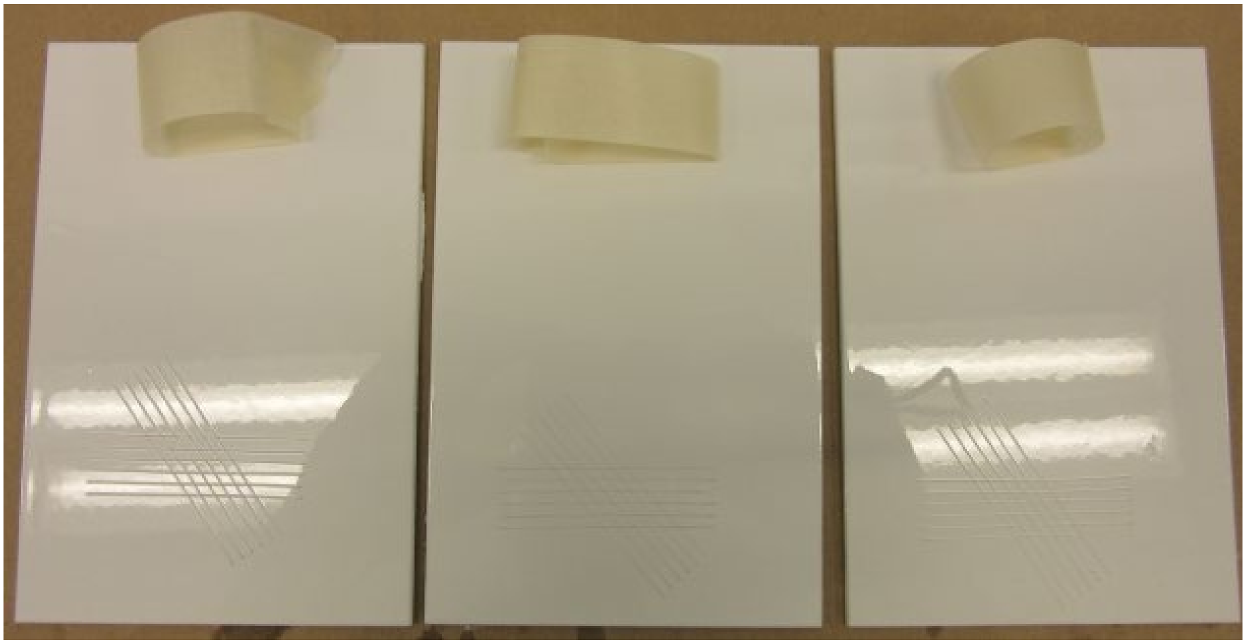


Figure 47: Adhesion testing of composite panels coated with protective coating and an assembly topcoat

Outdoor Exposure – 30, 60 days

Six – 3 inches by 6 inches panels and one large curved composite panel were placed on an outdoor exposure rack. Three – 3 inches by 6 inches panels were removed after 30 days and the remaining three – 3 inches by 6 inches panels and large curved composite panel were removed after 60 days. Where applicable, any degradation to the composite panels was evaluated by using sand paper and gently removing the protective coating to the point where the underlying composite surface was visible. Caution was taken to ensure that there was no removal of any UV degraded surfaces by the sand paper. The test panels were further evaluated via a solvent wipe test. A clean wiper saturated with acetone was wiped 15 times (forward and backward is one wipe) with vigorous heavy hand pressure. Yellowish residue seen on the wiper indicated failure and composite degradation. All test panels for 30 days of outdoor exposure passed the solvent wipe test. All test panels for 60 days of outdoor exposure passed the solvent wipe test. After the solvent wipe test, the composite panels were coated with a white epoxy high solid non chromated assembly topcoat used in aerospace production environments. The dry film thickness of the assembly topcoat was 0.8 mils. The panels were allowed to cure at ambient conditions for 24 hours and were visually examined and tested for adhesion in accordance with ASTM D3359, an industry standard cross hatch scribe test. All test panels passed the adhesion test with scores of 10 “Perfect” or 9 “Very Slightly” indicating no loss of paint along scribes and very slight loss of paint beyond scribes respectively. Outdoor exposure and adhesion testing results can be seen in Figure 48-52.

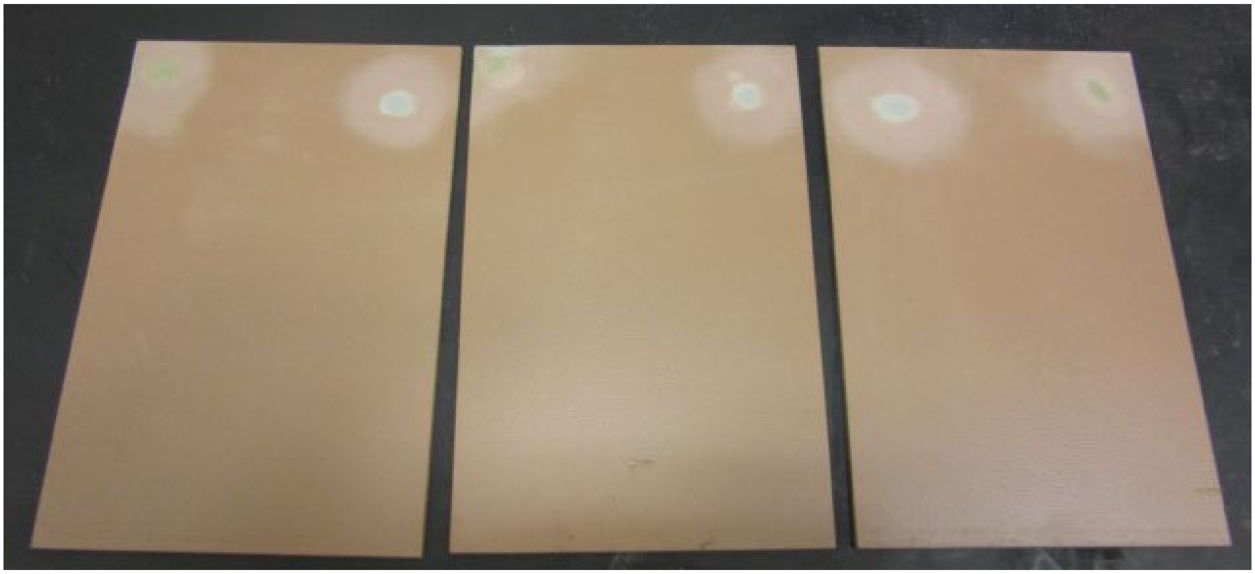


Figure 48: Protective coated composite panels after 30 days of outdoor exposure

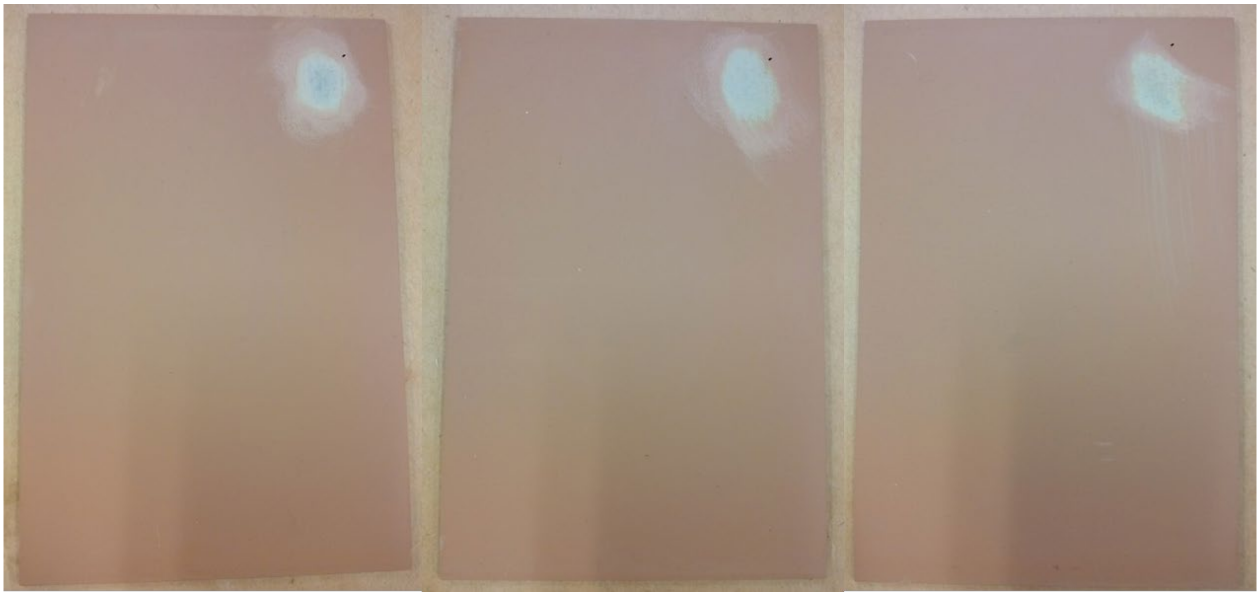


Figure 49: Protective coated composite panels after 60 days of outdoor exposure

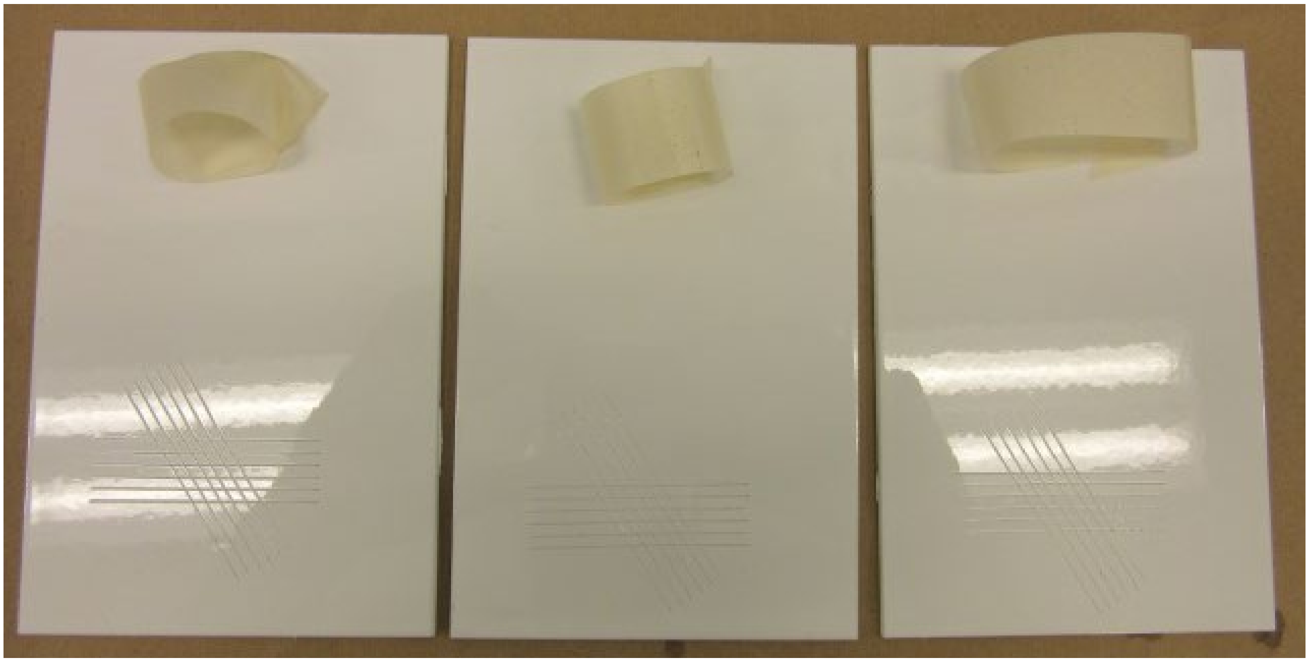


Figure 50: Adhesion testing of composite panels coated with protective coating and an assembly topcoat after outdoor exposure

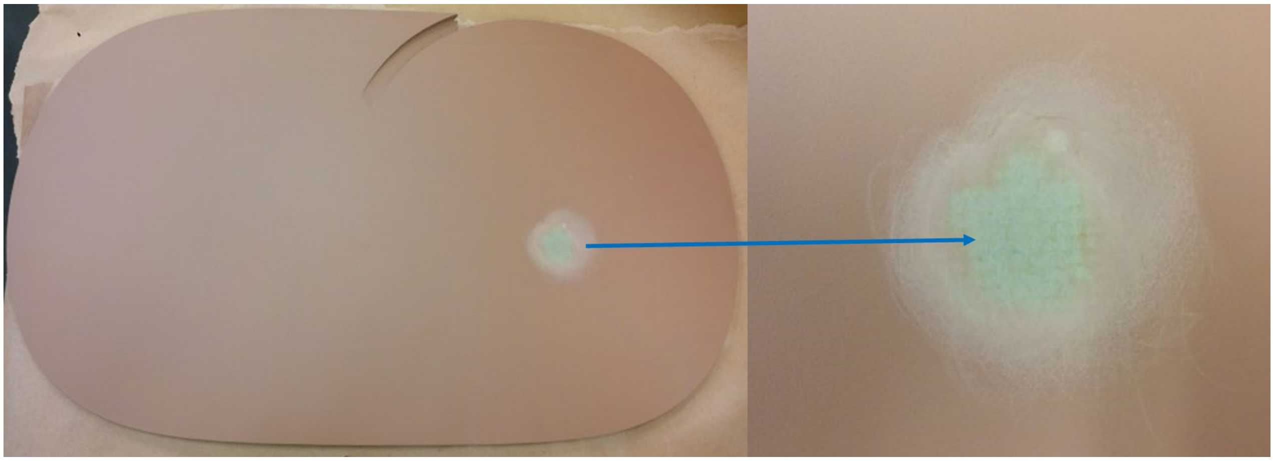


Figure 51: Protective coated large curved composite panel after 60 days of outdoor exposure

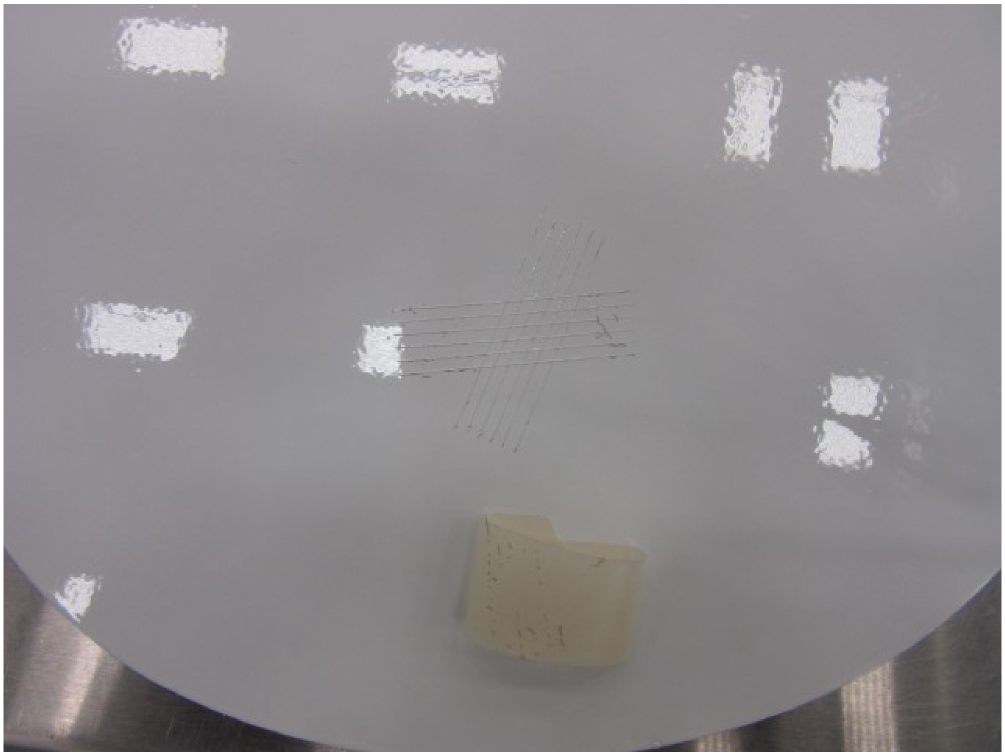


Figure 52: Adhesion testing of large curved composite panel coated with protective coating and an assembly topcoat after 60-day outdoor exposure

After using sand paper and gently removing the protective coating to the point where the underlying composite surface was visible, the panels were placed back on the outdoor exposure rack. Further evaluation at timed intervals highlighted yellowing and chalking in the area of the unexposed portions of the composite panel. In order to replicate the conditions seen, a bare composite panel was placed on the outdoor exposure rack for 48 hours. After 48 hours of exposure, degradation of the composite surface layer was evident. Additionally, a powdery yellowish material was present on the surface of the panel and the composite fiber matrix telegraphed through the panel. A clean wiper saturated with acetone was wiped 15 times (forward and backward is one wipe) with vigorous heavy hand pressure. The powdery yellowish material was removed from the surface of the composite panel and deposited onto the wiper. Figure 53 is an image of the composite panel after outdoor exposure and the solvent saturated wiper with yellowish powdery residue.

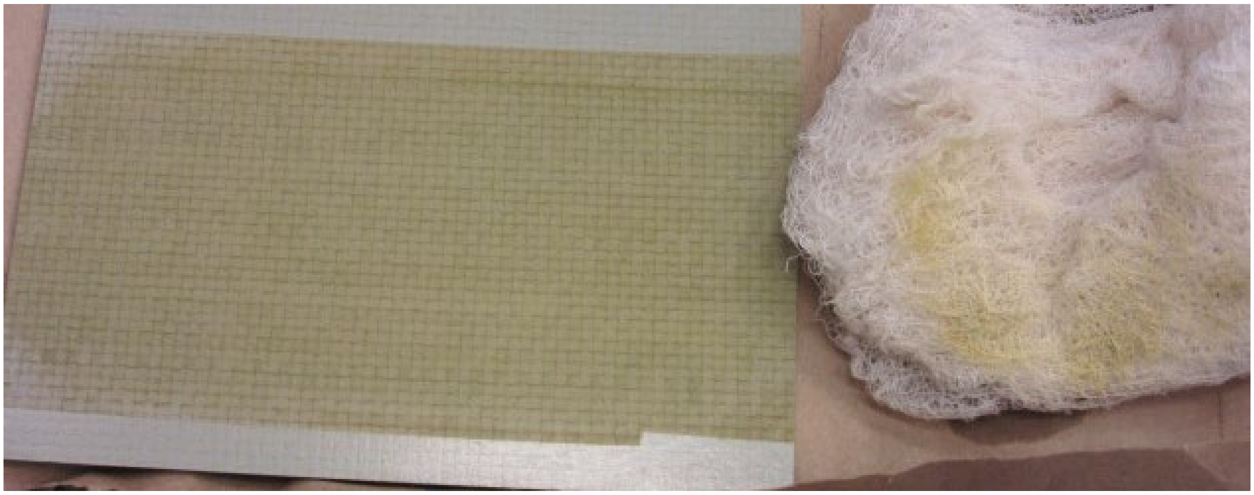


Figure 53: Bare composite panel after 48 hours of outdoor exposure and solvent wipe tested

Accelerated UV Exposure and water – 250, 500, 750, 1080 kJ/m^2

Four – 3 inches by 6 inches test panels were exposed in an Atlas Ci4000 xenon arc weatherometer in accordance with ASTM D7869[26] for block cycles equivalent to a total dosage of 250 kJ/m^2 per block. After each block, the panels were evaluated for degradation to the composite panels by using sand paper and gently removing the protective coating to the point where the underlying surface was visible. Caution was taken to ensure that there was no removal of any UV degraded surfaces by the sand paper. The test panels were further evaluated via a solvent wipe test. A clean wiper saturated with acetone was wiped 15 times (forward and backward is one wipe) with vigorous heavy hand pressure. Yellowish residue seen on the wiper indicated failure and composite degradation. The test panels passed the solvent wipe test for 250, 500, and 750 kJ/m^2. Three additional 3 inches by 6 inches test panels were exposed for a total of 1080 kJ/m^2 without removing the panels from the weatherometer. The test panels were evaluated via the solvent wipe test in the same manner as the first three test panels and all panels passed. After the solvent wipe test, all the test panels were coated with a white epoxy high solid non chromated assembly topcoat used in aerospace production environments. The dry film thickness of the assembly topcoat was 0.8 mils. The panels were allowed to cure at ambient conditions for 24 hours and were visually examined and tested for adhesion in accordance with ASTM D3359, an industry standard cross hatch scribe test. All test panels passed the adhesion test with scores of 10 "Perfect" or 9 "Very Slightly" indicating no loss of paint along scribes and very slight loss of paint beyond scribes respectively. UV light and water exposure and adhesion testing results can be seen in Figures 54-58.

Figure 54: Protective coated composite panels after 250 kJ/m^2 UV light and water exposure

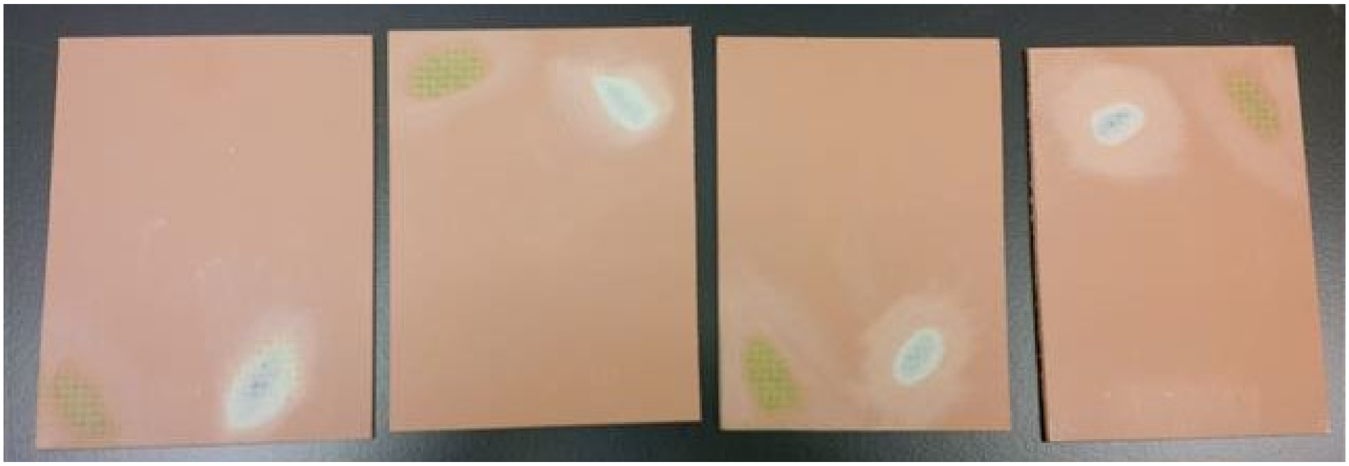


Figure 55: Protective coated composite panels after 500 kJ/m^2 UV light and water exposure

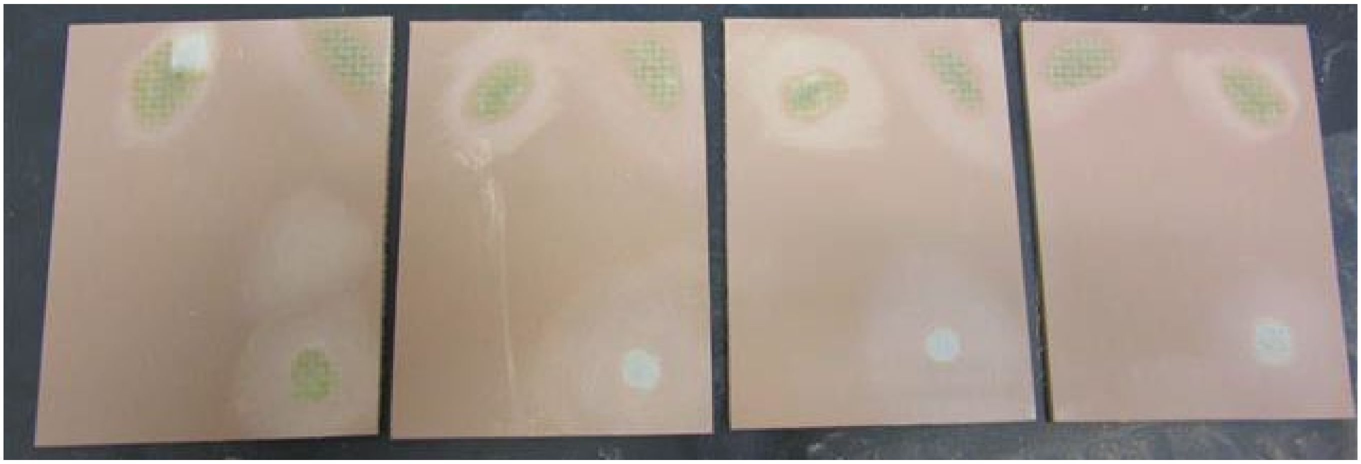


Figure 56: Protective coated composite panels after 750 kJ/m^2 UV light and water exposure

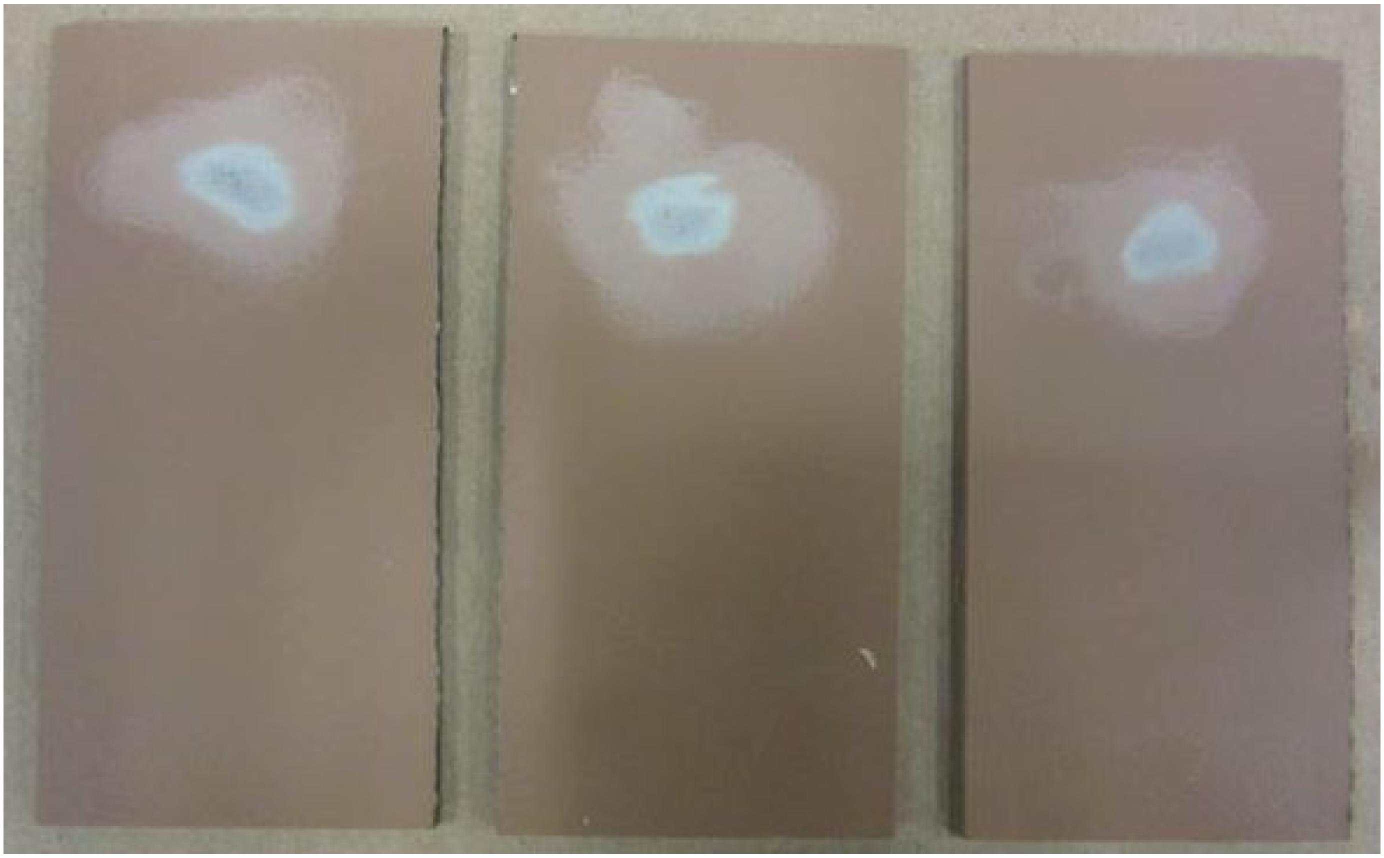


Figure 57: Protective coated composite panels after 1080 kJ/m^2 UV light and water exposure

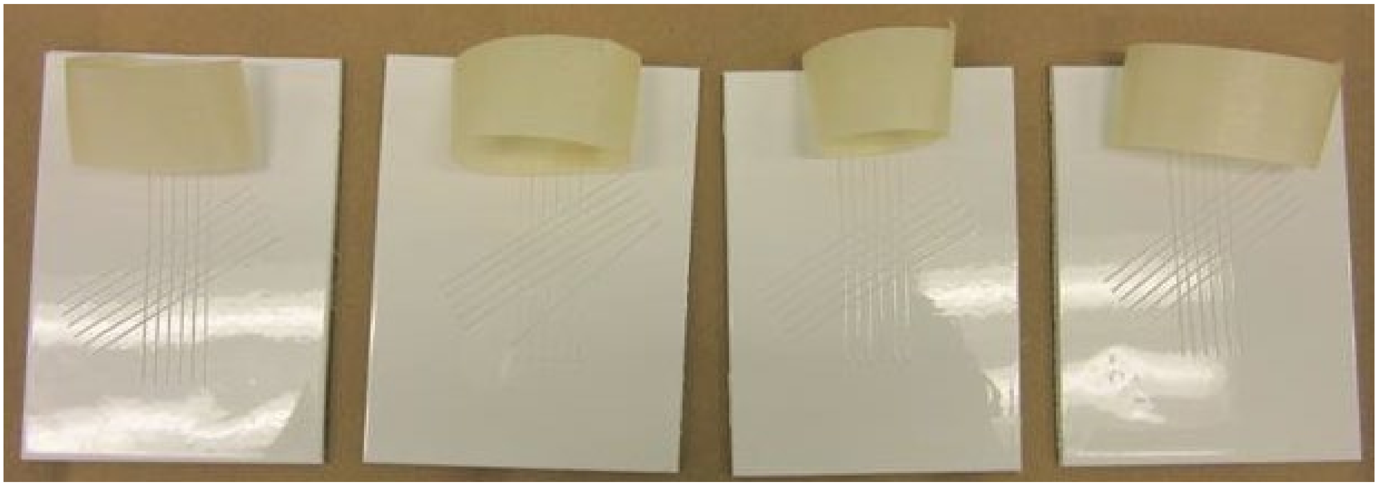


Figure 58: Adhesion testing of composite panels coated with protective coating and an assembly topcoat after UV light and water exposure

Conclusion

Qualitative tests were performed on protective coated composite panels to evaluate the capability of the coating to withstand environmental effects experienced in an aerospace production facility showed promising results. The identified tests – Water Resistance, Abrasion Resistance, Flammability, Impact Resistance, and Evaluation of Adhesion between the protective coating and an assembly coating all passed. Chalking, yellowing, and flaking of composite material due to UV degradation was witnessed on an unprotected composite panel. The areas witnessed on unprotected portions of the protective coated composite panels showed similar results indicating that qualitatively, the protective coating is protecting the underlying composite materials as expected. Industry standards provided the specification requirements that can be utilized to ensure testing results can be reproduced and evaluated in future investigations.

Chapter 7 Summary and Conclusion

The availability of composite materials globally has provided an opportunity for the manufacturability of airplane components to occur throughout the globe. The manufacturing and assembly sites vary in size and scale which requires additional consideration of the environmental impacts on the individual composite parts. An identified challenge during transport of the composite materials is to ensure the environmental conditions during manufacturing, assembly, and transport do not negatively impact the properties of the composite materials. Ultraviolet (UV) radiation from sunlight and factory light has been identified as one of many critical environmental factors that must be monitored throughout the lifetime of composite materials. The cost associated for utilizing a protective covering or coating can be substantial throughout the lifetime of an airplane program.

The aim of this dissertation was to identify a protective coating suited for various production environmental conditions that could protect composite parts utilized in the aerospace industry. Additionally, the goal of this dissertation was to identify a degradation model that could account for two synergistic and prevalent degradation mechanisms (UV and Condensing Humidity) applicable to the environment that the coating would be utilized. After quantitative and qualitative screening tests, a protective coating was down selected and evaluated for degradation modeling. The cumulative dosage model was chosen as the most appropriate model to evaluate UV degradation and Condensing Humidity degradation. The model provided suitable results for the individual parameters of UV and condensation, but was not suitable to full replicate all of the independent parameters seen in an outdoor exposure environment (UV, temperature variations, moisture, condensation, humidity).

The conclusion of this research led to the granting of United States Patent US 2018/0320004 A1 – Ultraviolet Protective Coating For Fabricating Epoxy-Based Components. The referenced patent can be found in the Appendix.

US 20180320004A1

(19) **United States**

(12) **Patent Application Publication**

Gibson et al.

(10) **Pub. No.: US 2018/0320004 A1**

(43) **Pub. Date: Nov. 8, 2018**

(54) **ULTRAVIOLET PROTECTIVE COATING FOR FABRICATING EPOXY-BASED COMPONENTS**

(71) Applicant: **The Boeing Company**, Chicago, IL (US)

(72) Inventors: **Loren F. Gibson**, Charleston, SC (US); **Nelson N. Akwari**, Summerville, SC (US); **Heath D. Kaufman**, Moncks Corner, SC (US)

(73) Assignee: **The Boeing Company**, Chicago, IL (US)

(21) Appl. No.: **15/584,915**

(22) Filed: **May 2, 2017**

Publication Classification

(51) **Int. Cl.**

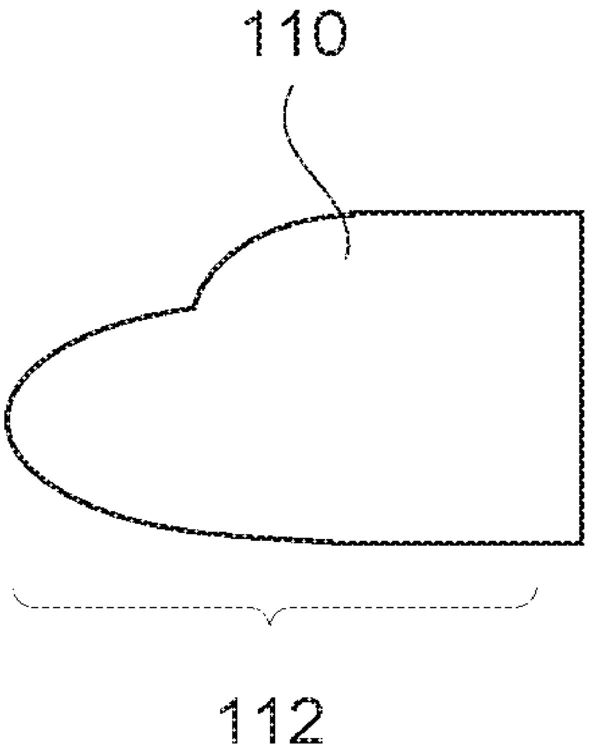
C09D 5/32	(2006.01)
C09D 183/02	(2006.01)
C09D 175/04	(2006.01)

(52) **U.S. Cl.**

CPC ***C09D 5/32*** (2013.01); ***C09D 175/04*** (2013.01); ***C09D 183/02*** (2013.01)

(57) **ABSTRACT**

Provided are epoxy-based components and methods of fabricating such components. Specifically, an component comprises an epoxy-based composite part and a UV protective coating disposed over the part. This coating allows for the component to be exposed to UV radiation without any additional coating and without deterioration of the epoxy-based composite part. Specifically, the component may be exposed to interior lights and direct sun during its subsequent fabrication and/or transportation. The UV protective coating comprises polyurethane and silicate filler, such as hydrated aluminum silicate and/or hydrated magnesium silicate. The coating may have a transmittance of less than 1% or even less than 0.1% in the UV range. An epoxy primer layer may be formed directly over the UV protective coating followed by various other coatings, including a decorative finish.



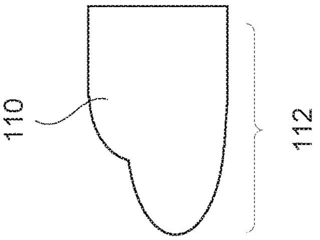


FIG. 1A

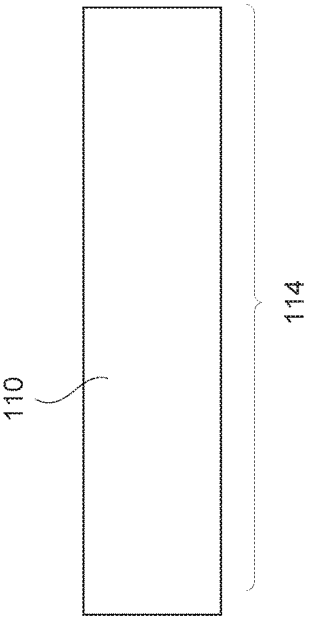


FIG. 1B

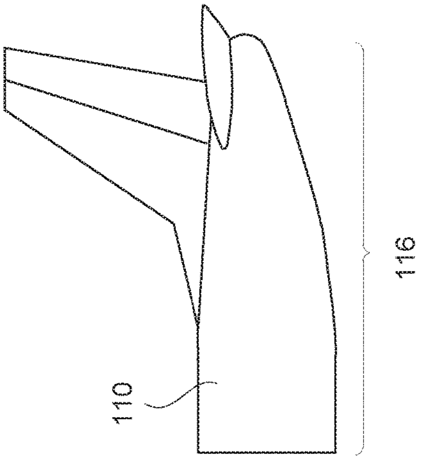


FIG. 1C

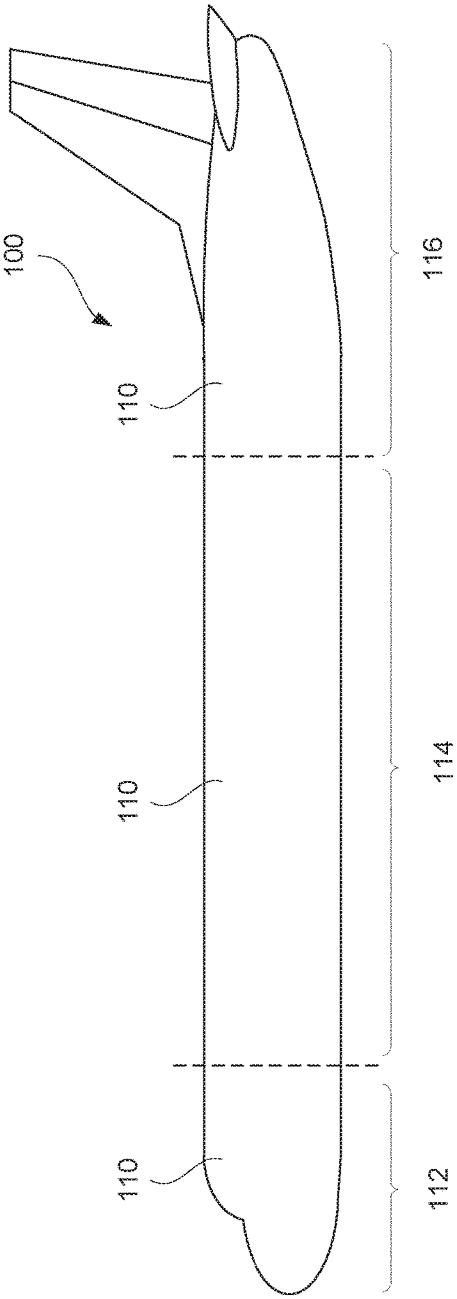


FIG. 1D

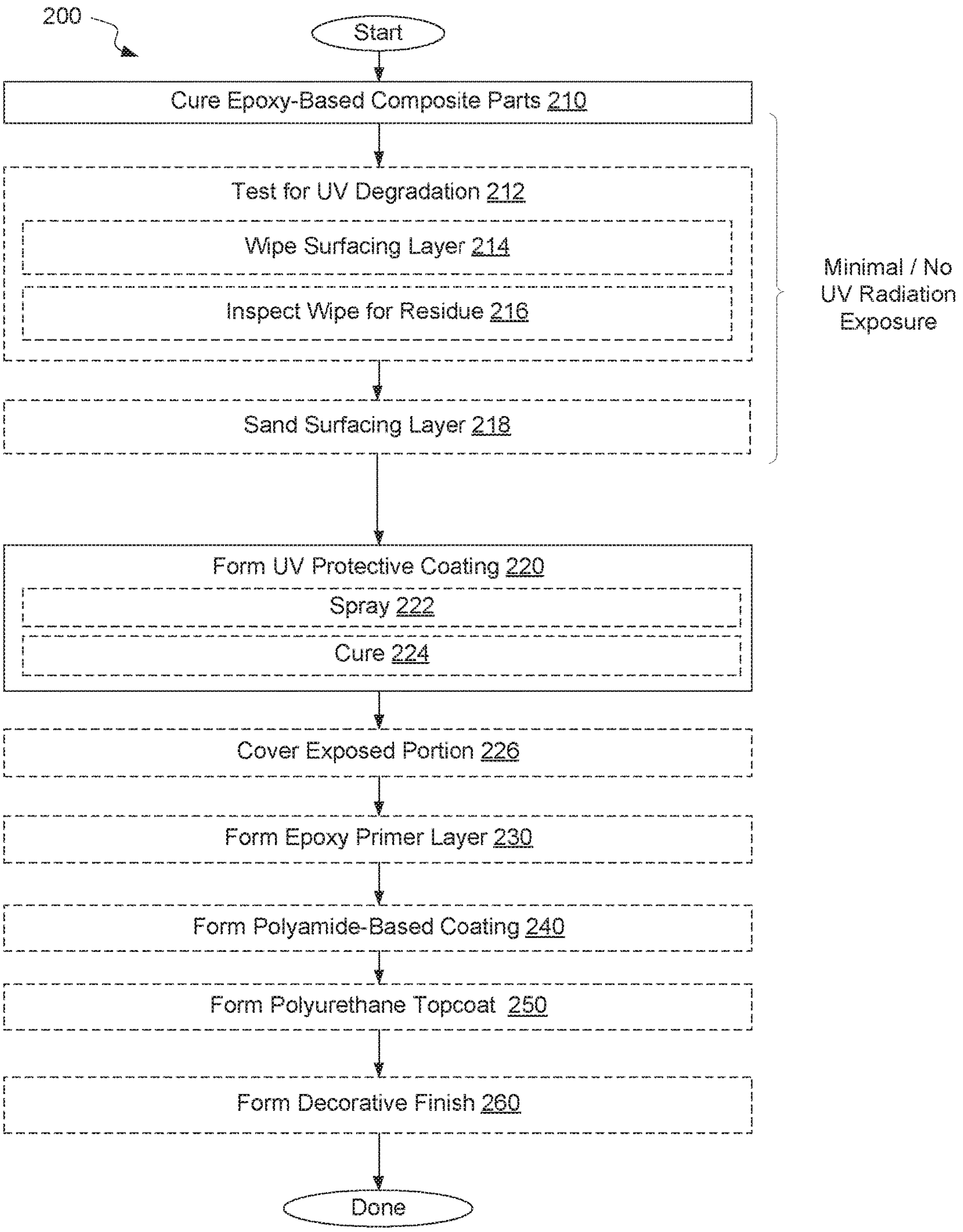

200
Start
Cure Epoxy-Based Composite Parts 210
Test for UV Degradation 212
Wipe Surfacing Layer 214
Inspect Wipe for Residue 216
Sand Surfacing Layer 218
Minimal / No UV Radiation Exposure
Form UV Protective Coating 220
Spray 222
Cure 224
Cover Exposed Portion 226
Form Epoxy Primer Layer 230
Form Polyamide-Based Coating 240
Form Polyurethane Topcoat 250
Form Decorative Finish 260
Done

FIG. 2

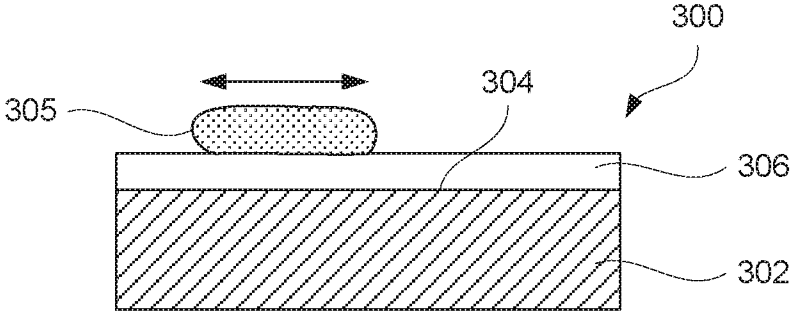


FIG. 3A

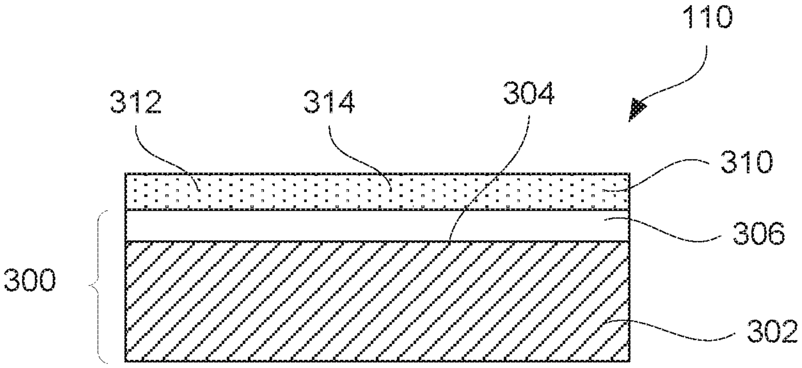


FIG. 3B

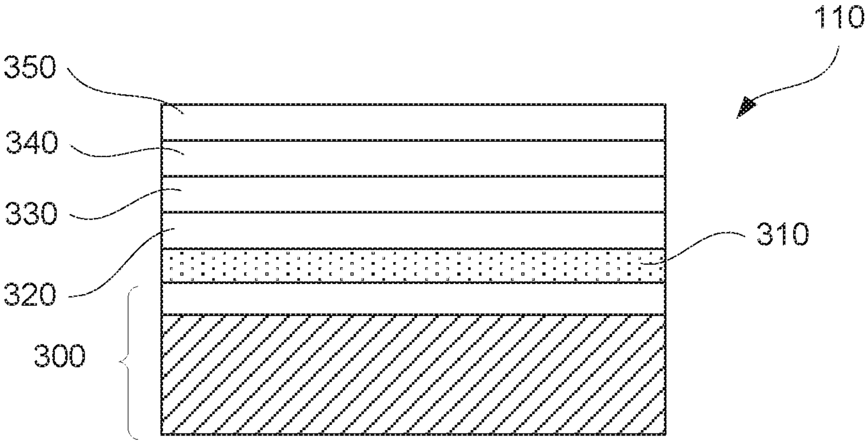


FIG. 3C

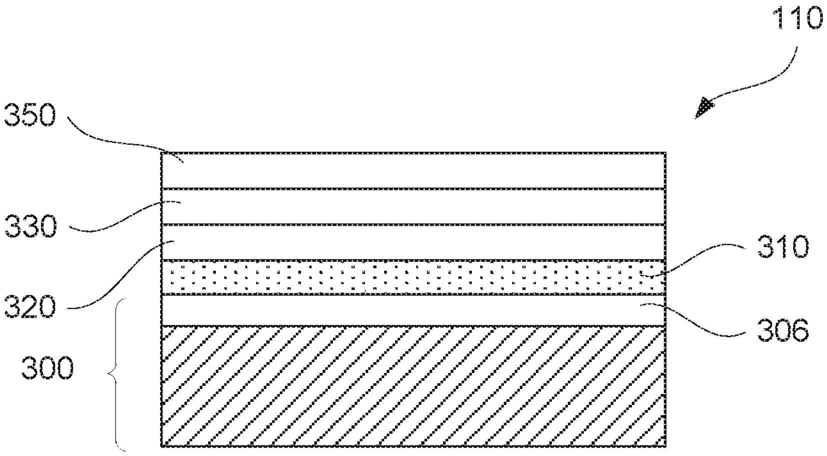


FIG. 3D

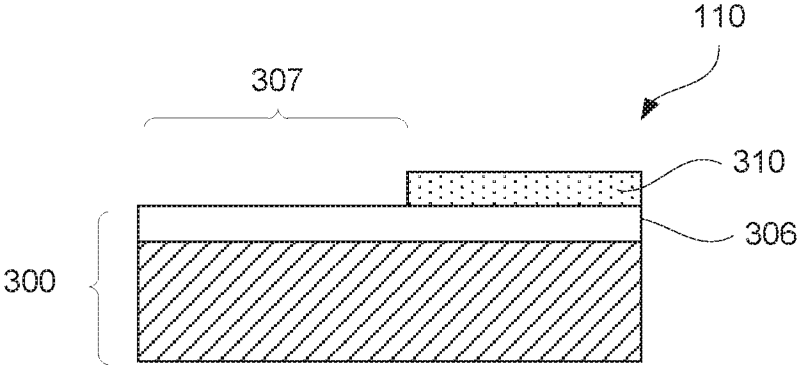


FIG. 3E

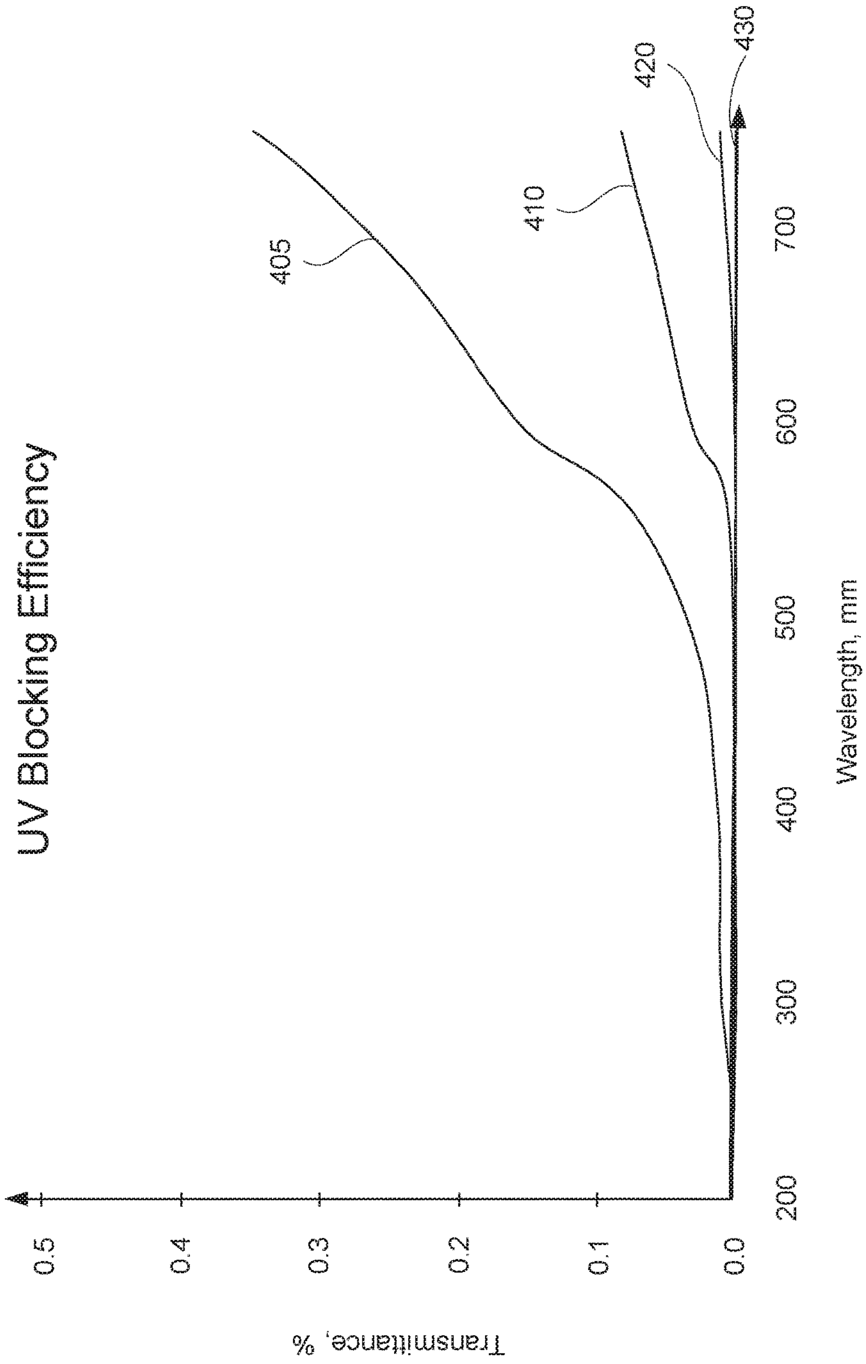

UV Blocking Efficiency
Transmittance, %
0.5
0.4
0.3
0.2
0.1
0.0
200
300
400
500
600
700
Wavelength, mm
405
410
420
430

FIG. 4

ULTRAVIOLET PROTECTIVE COATING FOR FABRICATING EPOXY-BASED COMPONENTS

BACKGROUND

[0001] Composite materials, such as carbon fiber-reinforced polymers (CFRP), are widely used for fabricating various components due to high strength and rigidity, low weight, corrosion resistance, and other favorable properties of these composites. Specifically, many composite materials are increasingly used in aircraft fabrication, e.g., to form fuselage, wings, tail sections, skin panels, and other components. However, some composite materials, especially epoxy-based composites such as epoxy-graphite composites, are ultraviolet (UV) sensitive. These composite materials may degrade if not protected and exposed UV radiation, such as direct sun exposure and/or interior lighting used on manufacturing floor, both of which may include some UV radiation.

SUMMARY

[0002] Provided are epoxy-based components and methods of fabricating such components. Specifically, an component comprises an epoxy-based composite part and a UV protective coating disposed over the part. This coating allows for the component to be exposed to UV radiation without any additional coating and without deterioration of the epoxy-based composite parts. The UV protective coating protects an epoxy based surfacing film as well as epoxy pre-impregnated carbon fiber composite. Specifically, the component may be exposed to interior lights and direct sun during its subsequent fabrication and/or transportation. The UV protective coating comprises polyurethane and silicate filler, such as hydrated aluminum silicate and/or hydrated magnesium silicate. The coating may have a transmittance of less than 1% or even less than 0.1% in the UV range. An epoxy primer layer may be formed directly over the UV protective coating followed by various other coatings, including a decorative finish.

[0003] In some examples, a method of fabricating an epoxy-based component comprises curing an epoxy-based composite part. The epoxy-based composite part comprises a composite base having a surface. The composite base comprises epoxy. The epoxy-based composite part further comprises a surfacing film disposed over the surface of the composite base.

[0004] The method proceeds with forming an ultraviolet (UV) protective coating directly over and in contact with at least a portion of the surfacing film of the epoxy-based composite part. The UV protective coating comprises polyurethane and silicate filler. The silicate filler comprises a silicate selected from the group consisting of hydrated aluminum silicate and hydrated magnesium silicate. In some examples, both hydrated aluminum silicate and hydrated magnesium silicate are present in the UV protective coating. Furthermore, the UV protective coating may also comprise titanium oxide. The concentration of the silicate filler in the UV protective coating may be at least about 20% by weight or, more specifically, at least about 40% by weight.

[0005] In some examples, the method further comprises forming an epoxy primer layer directly over and in contact with the UV protective coating. The method may proceed with forming a polyamide-based coating directly and in contact with over the epoxy primer layer. The method may further comprise forming a decorative finish directly and in contact with over the polyamide-based coating. Alternatively, the method may comprise forming a polyurethane topcoat directly over and in contact with the polyamide-based coating and before forming the decorative finish. The method then proceed with forming the decorative finish directly over and in contact with the polyurethane topcoat.

[0006] In some examples, the surfacing film remains substantially unexposed to UV radiation after curing the epoxy-based composite part and prior to forming the UV protective coating. For example, the maximum exposure between curing the epoxy-based composite part and forming the UV protective coating may be less than 200 kJ/m^2 ultraviolet (UV-A) radiation. Furthermore, forming the UV protective coating is performed in an environment substantially free from UV radiation such that the epoxy-based composite part is not exposed to UV radiation while forming the UV protective coating or UV exposure is minimal such that the epoxy-based composite part remains unaffected. Once the UV protective coating is formed over the epoxy-based composite part, the assembly can be exposed to UV radiation without risk of damaging the epoxy-based composite part.

[0007] In some examples, the method further comprises, prior to forming the UV protective coating, testing the surfacing film of the epoxy-based composite part for UV degradation. For example, this testing may involve wiping the surfacing film with a wipe saturated with acetone and inspecting the wipe for residues. In some examples, the method further comprises, prior to forming the UV protective coating, sanding the surfacing film.

[0008] Forming the UV protective coating may comprise spraying a UV protective liquid material onto the epoxy-based composite part. Furthermore, forming the UV protective coating may comprise curing the UV protective liquid material. Curing the UV protective liquid material may be performed at a room temperature.

[0009] In some examples, the UV protective coating has a thickness of between 10 micrometers to 100 micrometers or, more specifically, between 30 micrometers to 65 micrometers. Even at such small thicknesses, the UV protective coating may sufficiently block UV radiation. In some examples, the UV protective coating has a transmittance of less than 1% in a wavelength range of 100 nanometers and 400 nanometers or, more specifically, less than 0.1% in a wavelength range of 100 nanometers and 400 nanometers.

[0010] In some examples, the ultraviolet (UV) protective coating is formed over a portion of the surfacing film of the epoxy-based composite part, while another portion of the surfacing film remains exposed. In these examples, the method may comprise covering the exposed portion of the surface layer of the epoxy-based composite part with a protective sheet.

[0011] Also provided is an epoxy-based component. The epoxy-based component comprises an epoxy-based composite part and an ultraviolet (UV) protective coating. The epoxy-based composite part comprises a composite base comprising epoxy and having a surface. The epoxy-based composite part further comprises a surfacing film, disposed over the surface of the composite base. The ultraviolet (UV) protective coating may be disposed directly over and in contact with at least a portion of the surfacing film of the epoxy-based composite part. The UV protective coating may comprise polyurethane and silicate filler. The silicate filler may comprise a silicate selected from the group consisting of hydrated aluminum silicate and hydrated magnesium silicate. The concentration of the silicate filler in the UV protective coating is at least about 20% by weight.

[0012] In some examples, the epoxy-based component further comprises an epoxy primer layer, disposed directly over and in contact with the UV protective coating. The epoxy-based component may also comprise a polyamide-based coating disposed directly over and in contact with the epoxy primer layer. In some embodiments, the epoxy-based component further comprises a decorative finish disposed directly over and in contact with the polyamide-based coating. The epoxy-based component may also comprise a polyurethane topcoat and a decorative finish. The polyurethane topcoat may be disposed directly over and in contact with the polyamide-based coating. The decorative finish is disposed directly over and in contact with the polyurethane topcoat.

[0013] In some examples, the UV protective coating has a thickness of between 30 micrometers to 65 micrometers. The silicate filler may comprise both hydrated aluminum silicate and hydrated magnesium silicate. In some examples, the UV protective coating further comprises titanium oxide. The UV protective coating may have a transmittance of less than 1% in a wavelength range of 100 nanometers and 400 nanometers or, more specifically, less than 0.1% in a wavelength range of 100 nanometers and 400 nanometers. The epoxy-based component may be selected from the group consisting of a nose section, a tail section, and a middle section.

[0014] The features and functions that have been discussed can be achieved independently in various examples or may be combined in yet other examples further details of which can be seen with reference to the following description and drawings.

BRIEF DESCRIPTION OF THE DRAWINGS

[0015] FIGS. **1**A-**1**C are schematic illustrations of different epoxy-based components, in accordance with some examples.

[0016] FIG. **1**D is a schematic illustration of the epoxy-based components of FIGS. **1**A-**1**C forming a fuselage of an aircraft, in accordance with some examples.

[0017] FIG. **2** is a process flowchart corresponding to a method of fabricating an epoxy-based component, in accordance with some examples.

[0018] FIGS. **3**A-**3**E are examples of epoxy-based components at different stages of the method presented in FIG. **2**.

[0019] FIG. **4** illustrates UV blocking efficiency of UV protective coatings having different thicknesses.

DETAILED DESCRIPTION

[0020] In the following description, numerous specific details are set forth in order to provide a thorough understanding of the presented concepts. The presented concepts may be practiced without some or all of these specific details. In other instances, well known process operations have not been described in detail so as to not unnecessarily obscure the described concepts. While some concepts will be described in conjunction with the specific examples, it will be understood that these examples are not intended to be limiting.

Introduction

[0021] Epoxy-based composites or, more specifically, epoxy-based materials comprising graphite, which are sometimes referred to as carbon fiber-reinforced plastics, are very strong, lightweight, and have other desirable properties. Such composites may be used to fabricate various types of components, such as aircraft components where a combination of mechanical stress and low weight is highly desirable. However, other types of epoxy-based components are also within the scope, such as automotive parts. The fabricated components can be made into various geometric forms and sizes.

[0022] The process may involve layering sheets of carbon fiber cloth into a mold. The internal cavity of the mold may be shaped as the fabricated component. The process continues with filling the mold with epoxy and curing the composite. The epoxy may be a phenol or cresol (e.g., bisphenol-A) and a crosslinking agent (e.g., epichlorohydrin). The type and alignment of carbon fibers may be selected to optimize the strength and stiffness properties of the fabricated component.

[0023] One drawback of epoxy-based composites is their UV degradation, in particular at a 290-400 nm of UV spectrum. This spectrum is comparable to dissociation energies of polymer covalent bonds found in epoxies used for composite materials. Specifically, UV radiation absorbed by epoxies causes photo-oxidative reactions resulting in material degradation, which corresponds molecular weight reduction as well as reduction of mechanical strength and heat resistance.

[0024] Even short periods of UV exposure can change surface morphology of epoxy-based composites. For example, the surface of a composite may exhibit a distinct color change from black to dark green. This color change may serve as a way for monitoring the degradation process. Changes in surface smoothness may also be observed.

[0025] It should be noted that UV radiation is present, at some level, in the sun light and artificial light sources that illuminate manufacturing facilities. Protecting of epoxy-based fabricated components can be difficult, in particular when these composites are large structures, such as fuselage components of aircraft. FIGS. **1**A-**1**C are schematic illustrations of different aircraft epoxy-based components **110**, in accordance with some examples. In these examples aircraft epoxy-based components **110** are nose section **112**, tail section **116**, and middle section **114** of aircraft fuselage **100**. Aircraft epoxy-based components **110** may be processed and receive a UV protective coating prior to assembling these components together, e.g., to form aircraft fuselage **100** as, for example, schematically shown in FIG. **1**D.

Processing Examples

[0026] FIG. **2** is a process flowchart corresponding to method **200** of fabricating epoxy-based component **110**, in accordance with some examples. Some examples of epoxy-based components are described above and may include a nose section, a tail section, and/or a middle section, in some examples. For example, epoxy-based component **110** may be a wing-box. Other examples of epoxy-based components are also within the scope.

[0027] In some examples, method **200** involves curing epoxy-based composite part **300** during operation **210**. Operation **210** may involve layering sheets of carbon fiber cloth (e.g., a prepreg material) into the mold and filling the mold with epoxy. In some examples, the sheets are layered using a layup machine, such as an automated tape layup machine. The surface of the mold, which received the sheets, may be treated with a release agent or a film. Multiple sheets may be applied one on top of the other until a desired thickness is achieved and desired orientation of the reinforcement fibers is achieved for maximum strength and efficiency. In some examples, the layered structure is subjected to an optional pre-cure vacuum hold under a vacuum pressure. Operation **210** may then involve heat curing epoxy-based composite part **300** in a heating apparatus (e.g., an autoclave). The heat curing may be performed under vacuum. The temperature and duration of heat curing depends on epoxy used for epoxy-based composite part **300** and other factors.

[0028] Referring to FIG. **3**A, epoxy-based composite part **300** may comprises composite base **302** having surface **304**. Composite base **302** may be an epoxy prepreg resin having carbon fibers disposed within. The carbon fibers may be in a woven fabric form. More specifically, epoxy-based composite part **300** may be a carbon fiber reinforced polymer composite (CFRP). In some examples, epoxy-based composite part **300** comprises surfacing film **306**. The composition of surfacing film **306** may be different from the composition of composite base **302**. For example, surfacing film **306** may have a higher concentration of epoxy than composite base **302**, e.g., a resin-rich shell that blocks fiber read-through and enhances paint adhesion. In some examples, surfacing film **306** may comprises a different polymer than composite base **302**, e.g., polyurethane/polycarbonate, polyester, and the like.

[0029] Method **200** may also involve testing surfacing film **306** of epoxy-based composite part **300** for UV degradation during optional operation **212**. For example, operation **212** may involve wiping surfacing film **306** with wipe **305**, referring to block **214** in FIG. **2** and a schematic illustration in FIG. **3**A. Wipe **305** may be saturated with acetone. For example, the same spot on epoxy-based composite part **300** may be wiped between 10-20 times using a heavy hand pressure.

[0030] Operation **212** may also involve inspecting wipe **305** for residues, referring to block **216** in FIG. **2**. FIG. **3**A is s schematic illustration of surfacing film **306** being wiped with wipe **305**. It would be understood that if optional operation **212** is performed, operation **212** is performed prior to forming UV protective coating **310** during operation **220**.

[0031] Without being bound to any particular theory, it is believed that UV degradation of epoxy-based composite part **300** may produce quinone, hydroquinone, or alkyl ketone products on the surface. This may result in discoloration of the surface or, more specifically, producing green residue on the surface of epoxy-based composite part **300**. Wiping during operation **214** may transfer this residue to from the surface to wipe **305**. Furthermore, small amounts of residue may not detectable directly on the surface but when concentrated on wipe **305**, this residue may be more detectable. For example, the same wipe **305** may be used to wipe a large area of the surface.

[0032] Method **200** may also involve sanding surfacing film **306** during optional operation **218**. Sanding surfacing film **306** may be used to remove any residue resulting from UV degradation of epoxy-based composite part **300**. Furthermore, sanding surfacing film **306** may increase the surface roughness of surfacing film **306** to improve bonding to UV protective coating **310**. It would be understood that if optional operation **218** is performed, operation **218** is performed prior to forming UV protective coating **310** during operation **220**.

[0033] Returning to FIG. **2**, method **200** may proceed with forming UV protective coating **310** during operation **220**. UV protective coating **310** may be formed directly over and in contact with at least a portion of surfacing film **306** of epoxy-based composite part **300** as, for example, shown in FIG. **3**B. FIG. **3**B is a schematic illustration of epoxy-based component **110** after completing operation **220**. At this stage, epoxy-based component **110** comprises epoxy-based composite part **300** with UV protective coating **310** disposed over epoxy-based composite part **300**. If epoxy-based composite part **300** comprises surfacing film **306**, then UV protective coating **310** may be disposed over surfacing film **306** or, more specifically, directly over and in contact with surfacing film **306**. If epoxy-based composite part **300** does not have surfacing film **306**, then UV protective coating **310** may be disposed over composite base **302** or, more specifically, directly over and in contact with composite base **302**.

[0034] UV protective coating **310** comprises polyurethane **312** and silicate filler **314**, such as hydrated aluminum silicate and/or hydrated magnesium silicate. The concentration of silicate filler **314** in UV protective coating **310** may be at least about 20% by weight or, more specifically, at least about 40% by weight. In some examples, silicate filler **314** is uniformly distributed throughout the entire volume of UV protective coating **310**. Likewise, polyurethane **312** may be uniformly distributed throughout the entire volume of UV protective coating **310**. For purposes of this disclosure, the term "uniformly distributed" means that the concentration of a component varies by less than 10 weight % throughout the entire volume.

[0035] UV protective coating **310** may have a thickness of between 10 micrometers to 100 micrometers of, more specifically, between 30 micrometers to 65 micrometers. As further described below with reference to FIG. **4**, UV protective coating **310** provides sufficient blockage of UV radiation even at such low thicknesses. Furthermore, a lower thickness of UV protective coating **310** corresponds to a lower added weight to a subassembly comprising UV protective coating **310**, which may be important for aircraft applications.

[0036] Silicate filler **314** of UV protective coating **310** may comprise one or both hydrated aluminum silicate (e.g., kaolin) and hydrated magnesium silicate (e.g., talc). In some examples, UV protective coating **310** further comprises titanium oxide. UV protective coating **310** may comprise silica.

[0037] Forming **220** UV protective coating **310** may comprise spraying a UV protective liquid material (block **222** in FIG. **2**). However, other disposition techniques, such as brushing, rolling, and the like are also within the scope. In some examples, the UV protective liquid material includes one or more acetates, such as n-butyl acetate and/or 2-methoxy-1-methylethyl acetate.

[0038] Forming **220** UV protective coating **310** may also comprise curing the UV protective liquid material (block **224** in FIG. **2**). Curing operation **224** may be performed at a room temperature. The curing duration may be between about 0.5 hours and 2 hours.

[0039] In some examples, surfacing film **360** remains substantially unexposed to UV radiation after curing of epoxy-based composite part **300** during operation **210** and prior to forming UV protective coating **310** during operation **220**. For example, maximum exposure between the curing operation and the UV protective coating forming operational may be less than 200 kJ/m^2 ultraviolet UV-A radiation or even less than 100 kJ/m^2 ultraviolet UV-A radiation. Furthermore, forming UV protective coating **310** during operation **220** may be performed in an environment substantially free from UV radiation (e.g., a manufacturing facility with a special lighting). These features ensure that epoxy-based composite part **300** does not experience UV degradation before forming UV protective coating **310**.

[0040] Method **200** may proceed with forming epoxy primer layer **320** during optional operation **230**. Epoxy primer layer **320** may be formed directly over and in contact with UV protective coating **310**. Epoxy primer layer **320** may comprise one or more polyfunctional amine-containing compounds or a bisphenol-A-diglycidyl ether (e.g., cured with triethylene tetramine). For example, an epoxy resin dissolved in tert-butyl acetate may be used to form epoxy primer layer **320**.

[0041] Method **200** may proceed with forming polyamide-based coating **330** during optional operation **240**. Polyamide-based coating **330** may be formed directly and in contact with over epoxy primer layer **320**.

[0042] Method **200** may involve forming polyurethane topcoat **340** during optional operation **250**. Polyurethane topcoat **340** may be formed directly over and in contact with polyamide-based coating **330**

[0043] Method **200** may involve forming decorative finish **350** during optional operation **260**. Decorative finish **350** may be formed directly and in contact with polyamide-based coating **330**. Alternatively, decorative finish **350** may be formed directly and in contact with polyurethane topcoat **340**, if polyurethane topcoat **340** was previously formed.

Epoxy-Based Component Examples

[0044] FIG. **3**C is a schematic illustration of epoxy-based component **110** comprising epoxy-based composite part **300**, UV protective coating **310** disposed over epoxy-based composite part **300**, epoxy primer layer **320** disposed over UV protective coating **310**, polyamide-based coating **330** disposed over epoxy primer layer **320**, polyurethane topcoat **340** disposed over polyamide-based coating **330**, and decorative finish **350** disposed over polyurethane topcoat **340**. It should be noted that even though one or more of polyamide-based coating **330**, polyurethane topcoat **340**, and decorative finish **350** may provide UV protection once these layers are formed, UV protective coating **310** remains as a part of epoxy-based component **110**. As such, UV protective coating **310** provides UV protection until though one or more of polyamide-based coating **330**, polyurethane topcoat **340**, and decorative finish **350** are formed. Furthermore, UV protective coating **310** allows using additional options for one or more of polyamide-based coating **330**, polyurethane topcoat **340**, and decorative finish **350** that may not have been previously available since epoxy-based composite part **300** is already protected from UV degradation by UV protective coating **310**.

[0045] FIG. **3**C is a schematic illustration of another example of epoxy-based component **110** that does not have polyurethane topcoat **340**. In this example, UV protective coating **310** is also disposed over epoxy-based composite part **300**, epoxy primer layer **320** is disposed over UV protective coating **310**, polyamide-based coating **330** is disposed over epoxy primer layer **320**. However, decorative finish **350** is disposed directly over and interfaces with polyamide-based coating **330**.

[0046] FIG. **3**D illustrates another example where epoxy-based composite part **300** is only partially covered with UV protective coating **310**. As such, epoxy-based composite part **300** has exposed portion **307**, which may be protected from UV degradation by other means.

[0047] In general, epoxy-based component **110** may comprises at least epoxy-based composite part **300** and UV protective coating **310**. Epoxy-based composite part **300** may comprise composite base **302**, having surface **304**. In some examples, epoxy-based composite part **300** also comprises surfacing film **306**, disposed over surface **304** of composite base **302**. Alternatively, epoxy-based composite part **300** may not have surfacing film **306**.

[0048] UV protective coating **310** may be disposed directly over and in contact with at least a portion of surfacing film **306**, if surfacing film **306** is present. If surfacing film **306** is not present, then UV protective coating **310** may be disposed directly over and in contact with at least a portion of composite base **302**.

[0049] UV protective coating **310** may comprise polyurethane **312**. Furthermore, UV protective coating **310** may comprise silicate filler **314**, such as hydrated aluminum silicate and hydrated magnesium silicate. In some examples, UV protective coating **310** both hydrated aluminum silicate and hydrated magnesium silicate. The concentration of silicate filler **314** in UV protective coating **310** may be at least about 20% by weight or, more specifically, at least about 40% by weight. In some examples, UV protective coating further comprises titanium oxide.

[0050] UV protective coating **310** may have a thickness of between 10 micrometers and 100 micrometers or, more specifically, between about 30 micrometers to 65 micrometers. UV protective coating **310** may have a transmittance of less than 1% in a wavelength range of 100 nanometers and 400 nanometers or, more specifically, less than 0.1% in a wavelength range of 100 nanometers and 400 nanometers.

Experimental Data

[0051] FIG. **4** illustrates experimental data showing UV blocking efficiency of UV protective coatings having different thicknesses. All UV protective coatings had the same composition and were applied using the same spray technique. Specifically, the UV protective coatings included both hydrated aluminum silicate and hydrated magnesium silicate and titanium oxide.

[0052] Line **405** corresponds to a UV protective coating having a thickness of 13 micrometers (0.5 mils). Line **410** corresponds to a UV protective coating having a thickness of 25 micrometers (1.0 mils). Line **420** corresponds to a UV protective coating having a thickness of 50 micrometers (2 mils). Finally, line **430** corresponds to a UV protective coating having a thickness of 76 micrometers (3 mils). It has been found that even very thin UV protective coatings sufficiently block UV radiation for the entire range. Specifically, the thinnest test sample was only 13 micrometers thick and has a transmittance rate of less than 0.5% for the entire UV range.

CONCLUSION

[0053] Although the foregoing concepts have been described in some detail for purposes of clarity of understanding, after reading the above-disclosure it will be apparent that certain changes and modifications may be practiced within the scope of the appended claims. It should be noted that there are many alternative ways of implementing the processes, systems, and self-aligning riveting tools. Accordingly, the present examples are to be considered as illustrative and not restrictive.

[0054] In the above description, numerous specific details are set forth to provide a thorough understanding of the disclosed concepts, which may be practiced without some or all of these particulars. In other instances, details of known devices and/or processes have been omitted to avoid unnecessarily obscuring the disclosure. While some concepts will be described in conjunction with specific examples, it will be understood that these examples are not intended to be limiting.

1. A method of fabricating an epoxy-based component, the method comprising:
- forming an ultraviolet (UV) protective coating directly over and in contact with at least a portion of a surfacing film of an epoxy-based composite part, wherein:
  - the UV protective coating comprises polyurethane and silicate filler,
  - the silicate filler comprises a silicate selected from the group consisting of hydrated aluminum silicate and hydrated magnesium silicate, and
  - a concentration of the silicate filler in the UV protective coating is at least about 20% by weight.

2. The method of claim **1**, further comprising curing the epoxy-based composite part, wherein the epoxy-based composite part comprises a composite base having a surface, wherein the composite base comprises epoxy, and wherein the epoxy-based composite part further comprises the surfacing film disposed over the surface.

3. The method of claim **1**, further comprising forming an epoxy primer layer directly over and in contact with the UV protective coating and forming a polyamide-based coating directly and in contact with over the epoxy primer layer.

4. The method of claim **3**, further comprising forming a decorative finish directly and in contact with over the polyamide-based coating.

5. The method of claim **3**, further comprising forming a polyurethane topcoat directly over and in contact with the polyamide-based coating and forming a decorative finish directly over and in contact with the polyurethane topcoat.

6. The method of claim **1**, wherein the surfacing film remains substantially unexposed to UV radiation prior to forming the UV protective coating.

7. The method of claim **1**, wherein a maximum exposure prior to forming the UV protective coating is less than 200 kJ/m^2 ultraviolet (UV-A) radiation.

8. The method of claim **1**, wherein forming the UV protective coating is performed in an environment substantially free from UV radiation.

9. The method of claim **1**, further comprising, prior to forming the UV protective coating, testing the surfacing film of the epoxy-based composite part for UV degradation.

10. The method of claim **9**, wherein testing for the UV degradation comprises wiping the surfacing film with a wipe saturated with acetone and inspecting the wipe for residues.

11. The method of claim **1**, further comprising, prior to forming the UV protective coating, sanding the surfacing film.

12. The method of claim **1**, wherein forming the UV protective coating comprises spraying a UV protective liquid material and curing the UV protective liquid material.

13. The method of claim **12**, wherein curing the UV protective liquid material is performed at a room temperature.

14. The method of claim **1**, wherein the UV protective coating has a thickness of between 30 micrometers to 65 micrometers.

15. The method of claim **1**, wherein the silicate filler comprises both hydrated aluminum silicate and hydrated magnesium silicate.

16. The method of claim **1**, wherein the UV protective coating further comprises titanium oxide.

17. The method of claim **1**, wherein the concentration of the silicate filler in the UV protective coating is at least about 40% by weight.

18. The method of claim **1**, wherein the UV protective coating has a transmittance of less than 1% in a wavelength range of 100 nanometers and 400 nanometers.

19. The method of claim **1**, wherein the UV protective coating has a transmittance of less than 0.1% in a wavelength range of 100 nanometers and 400 nanometers.

20. (canceled)

21. An epoxy-based component comprising:
- an epoxy-based composite part, comprising a composite base and a surfacing film, wherein:
  - the composite base comprises epoxy and a surface, and
  - the surfacing film is disposed over the surface of the composite base; and
- an ultraviolet (UV) protective coating, disposed directly over and in contact with at least a portion of the surfacing film of the epoxy-based composite part, wherein:
  - the UV protective coating comprises polyurethane and silicate filler,
  - the silicate filler comprises a silicate selected from the group consisting of hydrated aluminum silicate and hydrated magnesium silicate, and
  - a concentration of the silicate filler in the UV protective coating is at least about 20% by weight.

22-29. (canceled)

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