Study the Effects of UV Radiation, High Temperature and Moisture on Epoxy with and without Titanium Metallic Coating

Kevin Culp, Ryan Alyamani, Abdulaziz Aldhubaie, Pengyu Chen, Steven Schroeder, Students
Dorina Mihut, Arash Afshar, Stephen Hill, Faculty
Mercer University School of Engineering

Abstract

Epoxy based composites have a wide range of applications for aviation, automotive and house construction due to their lightweight, corrosion resistance and good mechanical properties. During their exposure to harsh environmental conditions (UV radiation, high temperature and moisture) the epoxy-matrix part suffers significant degradation with detrimental effects on the mechanical properties. Within an independent study class, student teams worked together on the current research that offered them the opportunity to learn engineering skills through hands-on experiences. They developed the manufacturing process for producing standardized size epoxy samples and designed the testing procedures for assessing their mechanical properties. They also investigated the benefits of using a thin layer of metallic (titanium) coating deposited on epoxy by using the DC magnetron sputtering technique as an effective barrier to environmental degradation. The present research tested the epoxy behavior under accelerated weathering conditions (ASTM G154 standard) and offered methods for improving the mechanical performances. Results of the research are presented along with the students experiences.

Keywords

Epoxy, ASTM, UV radiation, Titanium coating, Independent Study

Introduction

The independent study class provides the opportunity for students to apply knowledge acquired in the classroom for exploring and solving real-world engineering problems. The first assignment for the independent study class is that each student research specialized knowledge from internationally recognized and recently published scientific journals. The students then review the article and present to a team of faculty. This procedure allows the students to become acquainted with current trends and findings in specific areas of research. Within this study students learn about the application and characterization of polymer based composite materials. The demand of polymer composites is increasing in automotive, aerospace and marine industries due to their desirable properties such as: lightweight, high strength to weight ratio, good corrosion resistance, good mechanical properties, design flexibility and cost efficiency. Most notable applications of polymer composites are: aircrafts (i.e. Boeing 787) and Zumwalt-Class Destroyer[1]. Epoxy resins for example, are among one of the most common matrix types used in the composite industry. However, long time environmental exposure to UV radiation, moisture and heat may lead to a significant degradation in the mechanical properties of the epoxy matrix which can ultimately lead to the deterioration of the entire composite material[2,3]. The simulation of outdoor environmental conditions can be obtained by exposing the specimens to moisture, UV light and high temperature in an accelerated weathering chamber (QUV)[4]. UV light initiates photo-degradation reactions in polymer matrix which deteriorates the surface integrity of matrix by enhancing the brittleness and creating surface microcracks[5,6]. More deleterious effects on the mechanical properties of composite materials will result after synergistic exposures of epoxy to both UV light and moisture[7,8]. The present research analyzes the degradation effects created by the outdoor environmental exposures and determine methods to limit degradation of the epoxy as the matrix part of the composites. The use of proper metallic coating layers and layer thicknesses on composites[9,10] may prevent the degradation of the polymer matrix under environmental exposures and consequently increase the life span of the composite materials. The current research offers students the opportunity to understand the importance of creating new materials and improving existing advanced materials with enhanced properties. Also, students develop a good understanding of the impact of engineering solutions on safety and economical related problems. For example, the usage of durable, strong but light materials in construction of airplanes, ships, cars or buildings is an important national and global need.

Methods and Experimental Plan

The epoxy polymer used in this study should maintain its mechanical properties during outdoor applications. This is tested by exposing the polymers to aggressive environmental conditions. During the independent study class, students develop plans for manufacturing of epoxy samples. Also, the students are fully involved in the design of experiments, performing of experiments, and interpreting the results. The epoxy resin samples were manufactured according to the ASTM D790 standard[11] with 0.5 x 5 x 0.125 inch final dimensions and prepared for further evaluations using three-point bending test in order to determine their initial mechanical properties (e.g. flexural modulus and ultimate tensile strength). In order to produce multiple samples, two molds were designed and built containing the inverse shape of multiple specimens (16), by using room temperature vulcanization rubber mold system (US Composites Inc.). Initially, sixteen specimens of 7075-T6 aluminum alloy with dimensions similar to the final polymer dimensions were created and secured with adhesive glue on the bottom of a 16.5 x 11.25 x 0.6 inch aluminum container. The rubber mold material was selected based on criteria of durability, flexibility and ease of production and it was created with 2:1 ratio of RTV urethane rubber and hardener respectively with a total volume of 1563 mL (as calculated). A releasing agent spray was applied over the surface of the aluminum container and aluminum glued samples and later the rubber mixture was poured into the container and allowed to dry for 24 hours. In order to obtain the specimens, a mixture of both resin and hardener (4:1 ratio, respectively) was poured into a glass container and the ratio was monitored by weight measurements. The mixture was continuously stirred for five minutes, ensuring a uniform mixture. Figure 1 shows one of the two molds that were manufactured, where there are sixteen cavities with standardized dimensions for the epoxy specimens' production.

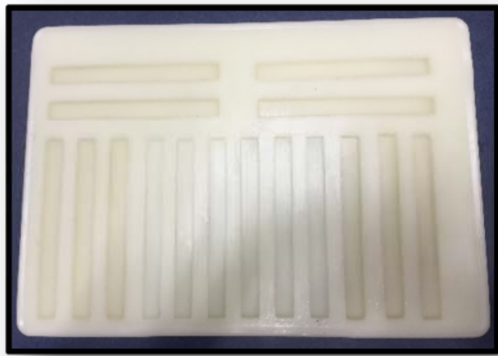

Figure 1: Rubber mold used for manufacturing epoxy specimens

A releasing spray agent was than applied to the mold before filling the mold in order to ensure that the specimens will be easily removed. Syringes with 5 ml capacity were used to accurately transfer the required mixture amount of epoxy mixture from the container to fill each mold slot. All specimens were left to fully cure at room temperature for a total time of 24 hours. The manufacturing process took place in a well-ventilated room and personal protective equipment (e.g. goggles, rubber gloves, respiratory masks, lab coats) was used at all times.

In order to improve the mechanical behavior twelve epoxy samples were further prepared for the coating with metallic titanium layers of 400 nm thickness. Samples were initially cleaned with water, soap and dried, then they were placed in a container filled with isopropyl alcohol and everything was placed in an ultrasonic bath for 20 minutes. The specimens were dried and transferred to another container with methanol following another ultrasonic cleaning for 20 more minutes. After drying, they were placed on a rotating plate and loaded in the high vacuum DC magnetron sputtering chamber. The base pressure in the chamber was monitored using an ion gauge and it was set to 1.066 x 10^{-3} Pa (8.0 x 10^{-6} torr). In order to achieve the desired pressure, a dry roughing pump and a turbomolecular high vacuum pump was used. The argon flow during the coating process was maintained at 47 sccm, and the pressure used during the deposition was established at 2.66 x 10^{-1} Pa (2.0 x 10^{-3} torr). High purity (99.99%) titanium target was used for coating the polymer specimens, and the target deposition power was maintained at 250 W during deposition. The coating thickness was in-situ monitored using a quartz crystal microbalance device and ex-situ measured using the KLA Tencor Profiler. The epoxy specimens were continuously rotated during the deposition process in order to ensure uniformity of the coating, and both sides of the specimens were coated with a 400 +/- 50 nm thickness titanium layer. The simulation of the extreme outdoor environmental conditions was produced by placing the specimens inside the QUV environmental chamber. All epoxy and titanium coated epoxy specimens were under multiple cycling exposures consisting of: 8h UV cycles at 60°C followed by 4h condensation cycles (%100 humidity) at 50°C based on ASTM G154 standard [12] for different times. Based on the time intervals presented in Table 1, the specimens were monitored for changes in microstructure and flexural properties in order to understand the time dependent changes. Table 1 is also presenting the number of epoxy and titanium coated epoxy samples and the time used for synergistic accelerated weathering conditions.

Table1. Exposure Times and Specimens

Specimen	0 h	300 h	600 h	1200 h	Number of Specimens
Epoxy	5	5	5	5	20
Titanium coated epoxy (400 nm)	3	3	3	3	12

After each time interval presented in Table 1 the surface microstructure was analyzed using digital optical microscopy (Keyence), and the flexural properties of the specimens were measured using the Mechanical Testing System (MTS). A supplementary fixture was designed and manufactured to meet the standard specifications for performing the three point bending test and then mounted on the MTS machine. The span length was set to 2 inch and crosshead rate was set to 0.06 in/min in order to produce a strain rate of 0.01in/in. in specimens following the ASTM D790 standard. An important outcome of the independent study class provides the students the opportunity to work with advanced equipment not typically used in standard classes. The students become familiar using the environmental testing equipment, DC magnetron sputtering equipment, MTS testing machine for three point bending testing evaluation, Rockwell hardness tester for hardness evaluations, and the optical microscope. Students also learn about the importance of standardized procedures for mechanical and environmental testing.

Students are required to keep a note book and record observations each day on experiments, measurements, comments on progress, and interesting observations during experiments. An important outcome for students of the independent study class is learning how to effectively communicate the results and research findings. The students' grade for the class consist of different assignments such as: literature review presentation in the area of study, presentations of the technical aspects of measurements, standards, and equipment used for the research, and presentation of results. The independent study class' final grade requires preparations of a PowerPoint presentation and a research poster containing the main project results in order to be presented either internally at Mercer's undergraduate research symposium or externally at a conference. Student observations, figures, and plots from the experiments in this study are included here.

Results and Discussions

The optical microscopy pictures for pristine epoxy after different hours of exposure (0, 300, 600 and 1200) are shown in Figure 2. It can be observed that during the first 600 h of exposure, pits and cavities form on the surface of the epoxy due to the combined UV and moisture conditioning. However, after 600h a recovery process occurs in the matrix due to the localized flow after prolonged exposure.

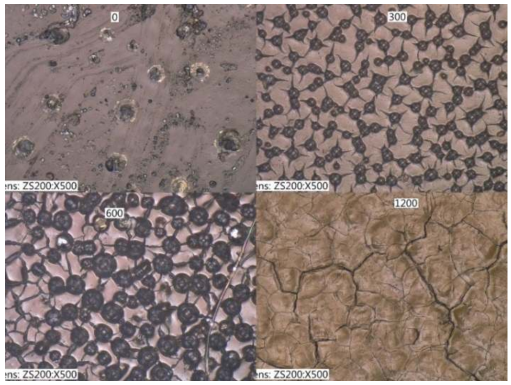

Figure 2: Optical microscopy images of epoxy surface after 0, 300, 600 and 1200 hours of exposure (500 X magnification)

The optical microscopy images for titanium coated epoxy samples after different hours of exposure (0, 300, 600 and 1200) are shown in Figure 3. It can be observed that the damage in the coating layer progresses during the course of exposure with a significant change in the microstructure after 1200 h of exposure.

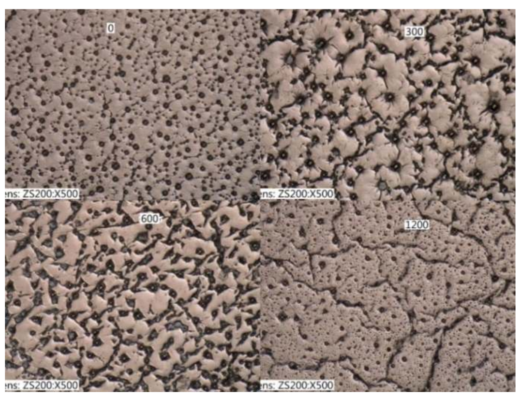

Figure 3: Optical microscopy images of titanium coated epoxy after 0, 300, 600 and 1200 hours of exposure (500 X magnification)

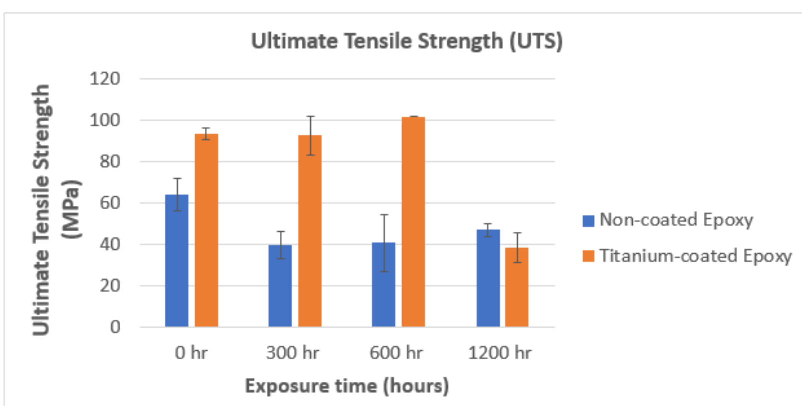

Figure 4: The ultimate tensile strength of epoxy and titanium coated epoxy after 0, 300, 600 and 1200 hours of exposures

The ultimate tensile strength values obtained after performing the flexural tensile test for epoxy and titanium coated epoxy are shown in Figure 4. A substantial decrease was observed in the flexural strength for the uncoated epoxy after 300 h of exposure and it remained relatively unchanged up to 1200 h exposure. It was observed that titanium coating worked as an effective barrier up to 600 h of exposure. However, the testing performed at 1200 h of exposure showed a significant decrease in the flexural strength showing that titanium coating lost its protective ability. It is possible that higher titanium coating thickness could lead to a better mechanical performance of the structure and a better surface behavior.

Conclusions

The independent study class allowed the students to investigate the polymeric matrix (epoxy) of a composite material. The students' involvement in this research allowed them to use advanced tools to analyze the behavior of the epoxy as a function of exposure duration to environmental conditions. They found that epoxy can significantly degrade during environmental exposures, ultimately affecting the lifetime of the entire composite. A significant decrease in the flexural strength of the pristine epoxy was observed after 300 h synergistic environmental exposure due to the substantial damage in the surface microstructure. However the titanium coatings were an effective barrier during 600 h of exposure but it lost its protection ability after 1200h of exposure. Applying a proper metallic coating with an appropriate thickness can significantly increase the lifetime of the epoxy based composites. In future classes, different metallic coatings will be investigated (e.g. zirconium, aluminum, copper) in order to evaluate different coating materials or coating thicknesses for environmental protection.

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Dorina Mihut, Ph.D.

Dorina M. Mihut graduated with Ph.D. Materials Science from the University of Nebraska, Lincoln, Ph.D. Technical Physics from Babes-Balyai University, Cluj-Napoca, Romania and M.S. Mechanical Engineering from the University of Nebraska, Lincoln. Research interests are in the area of Physical Vapor Depositions (PVD) using Thermal Evaporator, DC and RF Magnetron Sputtering Equipment, evaluations using X-Ray Diffraction, Scanning Electron Microscopy, and Atomic Force Microscopy: Development of thin films for wear, corrosion and erosion protection Manufacture of metallic and ceramic nanotubes using Physical Vapor Deposition via polymer template. Study of metallic nanoparticles deposited on filter paper, polymer nanofibers and their antibacterial properties Structures composed of metallic PVD thin films on carbon nanofiber reinforced polymer substrates for protection against electromagnetic interference. Evaluation of mechanical properties of thin films and materials: hardness, micro hardness, wear, fatigue and fracture analysis.

Arash Afshar, Ph.D.

Dr. Afshar is currently an assistant professor in the School of Engineering at Mercer University. He earned his B.S and M.S in applied mechanics from Amirkabir University of Technology in Tehran, Iran. He also received his M.S in systems and design and Ph.D. in solid mechanics from the State University of New York at Stony Brook. His teaching and research interests are in the area of solid mechanics including composite materials, finite element analysis, mechanical design and experimental mechanics. Prior to joining Mercer University he taught at Saginaw Valley State University and also worked as a design engineer in oil and gas and injection molding industries.

Stephen Hill, Ph.D.

Stephen Hill earned his Ph.D. from the Georgia Institute of Technology. He is currently an associate professor in the School of Engineering at Mercer University. He worked for the oilfield services giant Schlumberger for 14 years before returning to academia in 2013 to pursue his goal of educating the next wave of engineers entering the work force. His experience in the work force was in product development of downhole tools related to the extraction of oil and natural gas from various reservoirs. His current research interests include impact erosion, wear, two phase flow phenomena, solid/liquid phase change, desalination, and highly ionized plasma.

John K. Culp

Kevin Culp graduated from Mercer University with a bachelor's degree in mechanical engineering in the spring of 2017. His interests lie in structural design and materials, specifically related to the aeronautical field. He is currently pursuing his master's degree in mechanical engineering at Mercer University

Ryan Alyamani

Ryan Alyamani is currently a researcher at King Abdullah Petroleum Studies and Research Center (KAPSARC). He earned his B.S in Mechanical Engineering from Mercer University, where he was involved in several research projects. In April 2015, one of the research projects was awarded 1st place at the American Society of Engineering Education's Student Poster Competition (held in the University of Florida, Gainesville, FL.). He also has been selected to be a member of Sigma Xi Research Honor Society for Engineers and Scientist.

Abdulaziz Aldhubaie

Mr. Aldhubaie works as a nuclear energy engineer at King Abdullah City for Atomic and Renewable Energy (K.A.CARE). He earned his B.S in mechanical engineering from Mercer University in the spring of 2017. He is currently involved in an intensive program to expand his experience as a design engineer by the Korea Atomic Energy Research Institute (KAERI) in South Korea. He is part of a team that aims to transfer the nuclear power technology as well as the first nuclear power reactor to Saudi Arabia.

Pengyu Chen

Pengyu Chen is currently in his junior year attending Mercer University as a mechanical engineering student. He is interested in material science with a concentration on polymers and ceramics.

Steven Schroeder

Steven Schroeder is currently attending Mercer University as a mechanical engineering student. He is presently pursuing both his bachelor's and master's degrees in mechanical engineering through the schools 4+1 program. He is interested in materials science and how material properties can improve current designs.