



Time-dependent changes in mechanical properties of carbon fiber vinyl ester composites exposed to marine environments



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ABSTRACT

Carbon fiber–vinyl ester (CFVE) composites are good candidates for use in civil and naval vessels. CFVE composites offer high strength and stiffness-to-weight ratio and superior resistance to electrochemical corrosion. However, during application CFVE composites are exposed to harsh marine environments which can significantly degrade the mechanical properties. Here, the time dependent changes in flexural mechanical properties of CFVE composites due to exposure from marine environments are investigated. Exposure to marine environments are simulated by indoor water immersion, accelerated aging in environmental chambers, and outdoor exposure in a tidal pond. Comparison between the conditions will be discussed in the context of long-term outdoor exposure with controlled laboratory conditions.

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1. Introduction

A wide range of marine environmental elements can adversely affect the mechanical properties of fiber-reinforced polymeric composites including humidity, sea water, thermal variations, and ultraviolet radiation, to name a few. To employ fiber reinforced composites in naval and civil vessels, a detailed characterization of the effects of marine environments on mechanical properties of the materials is essential.

The effect of moisture on the mechanical properties of fiber-reinforced polymeric composites has been investigated in recent years [1–3]. Moisture can be absorbed into the fiber–matrix interface and replace or weaken resin/fiber chemical bonds. Moisture reduces the strength of the fiber–matrix interface via irreversible hydrolysis and plasticization. In composite materials with a brittle matrix, the damage mode can change from being matrix-dominated before moisture absorption to a fiber–matrix interfacial mode after saturation. This leads to a significant change in the strength of composites. In composite materials with a very brittle matrix, the moisture's plasticizing effect may cause a small increase in the material toughness [4,5]. Different water chemistry effects the moisture absorption of composite materials. The weight gain of specimens immersed in sea water is lower than that of ones immersed in pure water. This is due to the fact that polymers can

be considered as a semi-permeable membrane and the osmotic pressure reduces the driving force for moisture absorption. The hydrostatic pressure in deeply immersed specimens may not have a significant effect on the quantity of moisture absorption. The increased driving force for moisture absorption due to high pressure will be eliminated by the simultaneous reduction in free volume by hydrostatic pressure [4,5].

Ultraviolet (UV) radiation is another environmental factor in degrading polymeric composite materials [6–8]. UV light is not able to penetrate deep into fiber-reinforced composite laminates, and as a result, most of the matrix remains undamaged and fibers maintain their bonds to the matrix. Composite stiffness varies slightly due to UV radiation, as the change induced by UV radiation is a surface phenomenon, while modulus is a bulk material property. In many polymers, UV irradiation initiates chain scissions on the exposed surfaces, which produce small free molecules. These molecules are capped by oxygen and can be removed from the specimen, resulting in polymer weight loss [9]. The energy of UV photons is almost the same as the energy of polymer covalent bonds, which is about 290–460 kJ/mole. UV photons initiate photo-oxidative reactions, which deteriorate polymers by changing their chemical structures. Molecular chain scission and/or chain cross-linking are typically produced by photo-oxidative reactions. Chain scission reduces the molecular weight of the polymer, and lowers strength and heat resistance. Chain cross-linking increases the brittleness of the polymer, which may result in microcracking [10,11].

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Combined exposure to UV radiation and moisture from environmental conditions synergistically exacerbates composite property degradation and has gained much attention in recent years. The microcracks at the UV-irradiated surface of polymeric composites, can be propagated into the laminates in the presence of thermal variations [9]. The microcracks can also facilitate the ingress of moisture into the laminate. Moisture is able to dissolve and remove products of photo-oxidation reactions from a UV-irradiated surface and provide a fresh surface for further degradation by UV radiation [11–13]. The effects of combined exposure on the mechanical properties can be more deleterious compared to individual exposures. The transverse strength is the most susceptible property of polymeric composites to environmental elements and may decrease substantially after long-time exposures [11].

CFVE composites are promising candidates for naval and marine applications [14–16]. Carbon fibers are hydrophobic materials and aqueous environments have a minimal effect on the mechanical properties. Vinyl ester exhibits high corrosion resistance as well as superior resistance against water absorption. To employ CFVE composites in marine environments, it is necessary to characterize time-dependent changes in the mechanical properties and also to seek a method to approximate real-life outdoor exposures with indoor controlled conditioning. This provides valuable property values to predict the life expectancy of CFVE composites in harsh marine environments.

2. Materials

Carbon-fiber reinforced vinyl ester unidirectional composite laminates (Graphtek LLC) were used for all sets of experiments and conditions. Laminates have an isotropic vinyl ester matrix with a modulus of 3.2 GPa and carbon fibers with longitudinal and transverse moduli of 220 GPa and 22 GPa, respectively. Laminates have a fiber volume fraction of 0.56, and consist of 8 plies. Composite laminate sheets with nominal thickness of 1.4 mm were machined using a diamond wet saw into 12.5×77 mm (width \times length) coupons. Two sets of unidirectional specimens were prepared, to represent the two material principal directions of the laminate. The two sets are referred to, based on fiber orientation, as $[0^\circ]$ and $[90^\circ]$. For the former (i.e., longitudinal) the fibers are aligned with the length of specimens (i.e., the 77 mm long side), while for the latter (i.e., transverse) the fibers are aligned with the width of specimens (i.e., the 12.5 mm long side).

3. Exposure conditions

Sample coupons were divided into three groups. The first group of samples ($[0^\circ]$ and $[90^\circ]$) were exposed up to 2000 h of combined and individual accelerated aging using three chambers: (i) Moisture and heat in a Tenney Benchmaster BTRS temperature and humidity chamber, (ii) UV Radiation in a Q-Lab QUV/se accelerated weathering chamber, and (iii) Salt spray and heat in a Bemco P700XL chamber. UV radiation simulates natural sunlight using fluorescent UV bulbs at a 340 nm wavelength. Intensity is monitored by real-time UV irradiance sensors. One-half the samples in the first group were exposed to individual exposures and the other half were rotated every 48 h between two sets of chambers: salt spray and UV chambers, and heat and humidity and UV chambers to produce combined exposures. Because UV radiation exposure is only on one side of the coupon in the QUV/se chamber, sample sides were rotated every 24 h to receive uniform UV radiation. In the temperature and humidity chamber, moisture was set at 85% relative humidity (RH) and temperature at 35°C. In the QUV chamber, the UV irradiance was set at 0.6 W/m^2 at 60°C. The salt spray chamber was fed with 5% NaCl solution at constant flow rate using

~ 70 kPa air pressure and with a chamber temperature set to 35°C. The conditions in the chambers remained constant for the duration of exposures.

The second group of samples (all $[0^\circ]$) were immersed in distilled water, salt water, and sea water for a one year duration at room temperature, in closed containers. The third group of samples (all $[0^\circ]$) were mounted on a ladder frame with four different levels which was placed in a tidal pond for one year. Based on the position of samples on the ladder frame, they were exposed to varying amounts of sea air (which contains sunlight and atmospheric moisture) and sea water. The four levels of the frame are as follows: The upper level (Row 1) samples always remained out of water and only exposed to the sea air for the entire duration. The samples in the second level (Row 2) were exposed to 18 h of sea air and 6 h of sea water immersion during each day; two cycles of 9 h sea air followed by 3 h sea water. The samples in the third level (Row 3) were exposed to 6 h of sea air and 18 h of sea water immersion for each day; two cycles of 3 h sea air followed by 9 h sea water. The samples in the lower level of the frame (Row 4) were immersed for the entire duration in sea water. Five specimens per condition were used in the experiments.

4. Experimental setup

As a result of the samples exposure to moisture and photo-degradation conditions, the samples mass will change during the exposure. The mass of the composite samples were measured using a precision balance at every 48 h of exposure for the environmental chambers. In the case of combined exposure to moisture and UV radiation, mass of the samples were tracked at the end of the UV cycles, such that condensate was not present on the samples surfaces. Three point bending tests were performed on the composite coupons following the ASTM D790 standard [17] using a screw-driven mechanical loading frame (TiraTest 26005) with a 0.5 kN load cell. Support geometry followed ASTM D790, with the support span set for 60 mm, resulting in a span/thickness ratio of ~ 43 . A crosshead rate of 4.25 mm/min. was used to give a strain rate of 0.01 mm/min. The morphology of the fracture surfaces of broken samples from three-point bending test were examined next using a scanning electron microscope (Hitachi, HD2700C).

5. Results and discussion

5.1. Time-dependent weight change

Weight measurements of composite specimens were performed every 48 h during exposure to salt spray and combined UV and salt spray from the environmental chambers. Percent weight change was calculated by taking the difference between the original average sample weight and the average measured weight at each measurement time, divided by the average original weight and multiplied by 100. The results indicate that samples constantly exposed to salt spray had mass gain, reaching a steady-state due to moisture uptake ($\sim 1\%$). There was a significant difference between the specimens in the combined UV-salt spray condition versus those in the constant moisture condition, particularly for the salt spray cases (Fig. 1). The decrease in the weight after a peak value of ~ 700 h for the combined UV-salt spray cases indicates loss of surface material due to degradation and erosion of the matrix. It is worth noting that no change in volume was detected during exposure of samples to salt spray condition. For the combined exposure to UV and salt spray, no significant volume change occurred during the first 700 h of exposure. For the rest of the exposure time, the decrease in the volume was proportional to the weight loss in samples.

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