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Accelerated aging effects on carbon fiber/epoxy composites

Ana P. Cysne Barbosa [a, *], Ana Paula P. Fulco [a], Erick S.S. Guerra [a], Franscisco K. Arakaki [b], Márcio Tosatto [b], Maria Carolina B. Costa [a], José Daniel D. Melo [a]

[a] Department of Materials Engineering, Federal University of Rio Grande do Norte, Av. Senador Salgado Filho 3000, CEP 59078-970, Natal, RN, Brazil
[b] Empresa Brasileira de Aeronáutica – EMBRAER, Av. Brigadeiro Faria Lima 2170, CEP 12227-901, São José dos Campos, SP, Brazil

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ABSTRACT

The influence of operational environments on the long-term durability of structural components fabricated with carbon fiber reinforced composites is an ongoing concern. Exposures to ultraviolet radiation, temperature cycles and moisture are known to degrade the polymeric matrix. In this work, carbon-epoxy composites were subjected to accelerated aging in an aging chamber with controlled conditions of temperature, humidity and UV-radiation. Changes within the material are evaluated by Fourier-Transform Infrared (FTIR) Spectroscopy, Dynamic Mechanical Analysis (DMA), interlaminar shear strength and compressive strength, Scanning Electron Microscopy (SEM), and also in terms of mass variation. Although significant changes in mechanical properties were not observed, the effects of accelerated aging on the composite material were evidenced by mass loss, fiber exposure, chemical alterations, increased crack density in interlaminar shear tests and fiber buckling in fractured specimens after compression testing.

1. Introduction

Carbon fiber reinforced polymer (CFRP) composites have emerged as the materials of choice in many aeronautic structures due to their combination of properties, which include high strength and modulus-to-weight ratio and corrosion resistance. With the ever-increasing use of these materials, the influence of environmental service conditions on their durability has become an ongoing concern to the aeronautic industry and airline companies. Such structures must be designed to withstand the operational conditions imposed throughout the service life of the structural component. Thus, the influence of environmental factors such as moisture, temperature and UV radiation on the durability, as well as the reliability and safety of CFRP aeronautic structures must be better understood and considered in the design.

Traditionally, polymer aging has been classified into two main categories: physical and chemical aging. Physical aging [1] may be characterized by changes in molecular conformation of the material without changing the structural integrity of the molecules. This type of aging process is therefore reversible. It is the manifestation of a slow evolution of the polymer towards thermodynamic equilibrium with time dependent changes in volume, enthalpy, entropy and mechanical properties. Physical aging may occur during long time exposures of the material to elevated temperatures below the Tg. The changes in molecular conformation result in increases in Young's modulus, yield stress, density, viscosity, and reductions in creep and stress relaxation. Previous studies [2] have shown that physical aging may cause material embrittlement as well. In the case of chemical aging, there is an irreversible degradation of the molecular structure caused by mechanisms such as chain scission, changes in crosslink density, oxidation and depolymerization. In most applications, the two types of aging (physical and chemical) occur simultaneously.

High temperatures are known to accelerate the degradation process of polymers [3]. Also, thermal cycling is known to produce cracks in laminates and crack growth may be accelerated in oxidative atmospheres [4]. Elevated air and oxygen pressures accelerate the rate of thermo-oxidative degradation in polymeric composite materials as compared to samples tested at atmospheric pressure [5].

Light exposure may increase the concentration of free radicals in the polymer, which is a sign of degradation [6]. Photodegradation initiated by solar radiation results in absorption of UV radiation by chromophores and in activation of excited states in macromolecules. This degradation process may affect mechanical properties of

* Corresponding author.
E-mail address: anacysne@ct.ufrn.br (A.P. Cysne Barbosa).

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polymeric films due to chain scission and crosslinking of macromolecules [7].

The consequences of water absorption on polymer properties have been investigated in many studies and some of the effects reported include plastification, a reduction in glass transition temperature, an increase in creep and stress relaxation, and a decrease in mechanical strength and elastic modulus [8–13]. The absorbed moisture may also react with unreacted groups of the epoxy resin structure and therefore influence the aging process [14]. For composites without protective painting, UV-radiation may degrade the polymeric surface of the composite, thereby exposing the fibers while further increasing moisture absorption [15,16]. This combination of moisture absorption and UV degradation may produce significant effects on mechanical properties, particularly on elastic modulus and interlaminar shear strength [15,16].

In a previous study of the mechanisms of hydrothermal degradation in hot-wet conditions of graphite-epoxy laminates, interlaminar shear strength tests indicated that the combination of high relative humidity with high temperature may produce a significant reduction in strength [15]. In another study, hydrothermal aging of carbon-epoxy caused a decrease in modulus and strength, an increase in strain to failure and a reduction in wear resistance [17]. Hydrothermal cycling has also been reported as causing delamination [18].

Aging is normally associated with the deterioration of CFRP properties, although slight improvements in mechanical properties as a result of aging have also been reported. In one of these studies reported in the literature, interlaminar Mode I fracture toughness of a carbon-epoxy composite showed a variable trend towards slight decreases or slight increases caused by hydrothermal aging [19]. Another study [20] suggests that tensile and compressive properties of carbon-epoxy composites may improve during aging at the initial consolidation stage. In this case, the improvement in mechanical properties was attributed to post-curing reactions. A degradation stage followed when the composite properties are significantly decreased; this is attributed to a deterioration of the matrix and weakening of the fiber-matrix interface.

Although many studies regarding aging of carbon-epoxy composites are available in the literature, the effects of aging and the extent of those effects on properties of these materials are not fully understood. In this study, carbon-epoxy composites were exposed to alternating cycles of UV-A radiation and water condensation in an accelerated aging chamber, and the effects of aging on the material were evaluated by Fourier-Transform Infrared (FTIR) Spectroscopy, mechanical tests (interlaminar shear strength and compressive strength), Scanning Electron Microscopy (SEM) and also in terms of mass variation.

2. Experimental

The material tested in this investigation was HexPly® AS4/8552, a carbon/epoxy unidirectional prepreg tape from Hexcel Corporation, with a fiber volume fraction of 57% and a fiber weight ratio of 65%. The laminates were vacuum bagged and cured in an autoclave using a two-step cure cycle based on manufacturer specifications. The temperature was first increased to 110 °C and held for 1 h at a pressure of 700 kPa. Then, the temperature was increased to 180 °C and held for 2 h at the same pressure, before cooling to room temperature. All temperature ramps were controlled at 2 °C/min. Specimens with dimensions of 2.13 mm × 9.87 mm x 19.90 mm (thickness x width x length) were used for interlaminar shear strength (ILSS) tests. Composite specimens with dimensions 1.10 mm × 15.15 mm x 79.91 mm (thickness x width x length) were used for the compression tests.

2.1. Accelerated aging

The composite samples were exposed to UV radiation (UVA-340) and water condensation in an accelerated aging chamber (Equilam). Alternating cycles of UVA radiation (8 h) at 80 °C of 0.89 W/m^2/nm and water condensation (4 h) at 50 °C, for a period of 3 months (2160 h) were employed. Tests were conducted according to ASTM G 154–00a (Standard Practice for Operating Fluorescent Light Apparatus for UV Exposure of Nonmetallic Materials). The accelerated aging test was initiated with the moisture step and the test was concluded after 2160 h of the UV-radiation step.

2.2. Mass variation

In order to observe mass variation, samples were weighted before and after the accelerated aging procedure. The mass variation results presented for the composite material are the average values of 10 ILSS samples and 10 compression samples. Mass variation was calculated by using Equation (1):

$$M(\%) = \frac{Final\ \ mass(g) - Initial\ \ mass(g)}{Initial\ \ mass(g)} * 100 \quad (1)$$

2.3. Scanning Electron Microscopy (SEM)

Material morphology was analyzed using a Hitachi TM-3000 scanning electron tabletop microscope. Before the accelerated aging process, the cross-sectional area of the ILSS samples were polished using silicon carbide grinding paper (400–2000 mesh) and afterwards polished with alumina suspension of 1.0 μm. This procedure was carried out in order to allow visualization of the carbon fibers at the cross-section of the samples so that the effect of aging on sample morphology could be better assessed.

Care was taken to take images of the same specimen at the same spot, before and after the aging procedure. This procedure allowed direct comparison of the same surface and the effects of accelerated aging could be more reliably assessed.

2.4. Fourier-Transform Infrared (FTIR) Spectroscopy

Fourier-Transform Infrared (FTIR) Spectroscopy of ILSS samples was performed using a Shimadzu IRTracer – 100 Spectrometer, in order to compare the spectra of unaged and aged material. The analysis conditions were 32 scans, at a range of 700–4000 cm^{-1} and 4 cm^{-1} resolution. Samples were conditioned in a desiccator before analysis. Two specimens for each condition (aged and unaged) were analyzed in Attenuated Total Reflectance (ATR) mode.

2.5. Dynamic Mechanical Analysis (DMA)

DMA samples were cut from the tab area of the samples for compression tests. In case of aged samples, care was taken to take the material from the side of the specimen which was exposed to UV-radiation. Sample dimensions were 1.00 mm × 3.15 mm x 17.50 mm (thickness x width x length).

Aged and unaged samples were conditioned for 48 h in an oven at 50 °C and placed inside a desiccator for at least 5 h with calcium chloride as absorbing material prior to the DMA tests.

These tests were conducted following the recommendations of ASTM D 7028-07 for determining glass transition temperature. Aged and unaged samples were analyzed using a DMA Q 800 TA under single cantilever mode, and strain amplitude of 0.1%. Temperature scan measurements were performed within the