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Hygrothermal effects in aeronautical composite materials subjected to freeze-thaw cycling

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Abstract. Fiber-reinforced composites (FRC) are becoming increasingly popular in aerospace, automotive and energy sectors. Despite the advantages owed to their strength and lightweight, understanding their behavior in different environments poses challenges. Particularly, humidity, temperature, and freeze-thaw cycles can significantly affect the durability of FRC components. This study investigates the impact of humidity, temperature, and freeze-thaw cycles on FRC inter-laminar areas and the matrix/fiber interface. Experimental methods, including heat analysis, X-Ray tomography and mechanical testing will assess the material's response to changing environmental conditions. This research enhances our understanding of FRC behavior, crucial for designing and maintaining FRC components.

Introduction

Fiber-reinforced composites (FRC) are increasingly utilized in various industries, including aerospace, automotive and energy. However, the extensive application of these advanced materials introduces new challenges in understanding their component behavior throughout their operational lifespan. Environmental factors, such as humidity and temperature, can adversely affect the properties of the matrix and weaken the fiber/matrix interface. Research indicates that the interlaminar regions and the matrix/fiber interface are particularly susceptible to the influence of humidity and temperature. Moreover, the mechanical properties of the composite are significantly impacted by the combined presence of moisture and the freeze-thaw cycle, surpassing the effects of individual environmental factors. Microcracks are induced by freeze-thaw cycles, which increases moisture penetration into composite structures [1,2].

Case studies

A/c structures, designed to withstand challenging conditions while prioritizing strength, weight and safety, are highly specialized and technologically advanced. Keeping this in mind, two case studies are conducted to establish accurate and meaningful thermal cycles. One of them considers the environmental conditions of commercial flights, typically cruising at altitudes between FL320 and FL400. At these altitudes, the International Standard Atmosphere (ISA) indicates temperatures ranging from -48°C to -55°C. The atmospheric pressure varies between 27450Pa and 18750Pa. Due to the compression of air by the aircraft's body, the skin temperature of the aircraft is higher than the static air temperature of -55°C as described in the following equation (1):

$$T_s = T_\infty - T_\infty \cdot \frac{(k-1)\cdot Ma^2}{2} \quad (1)$$

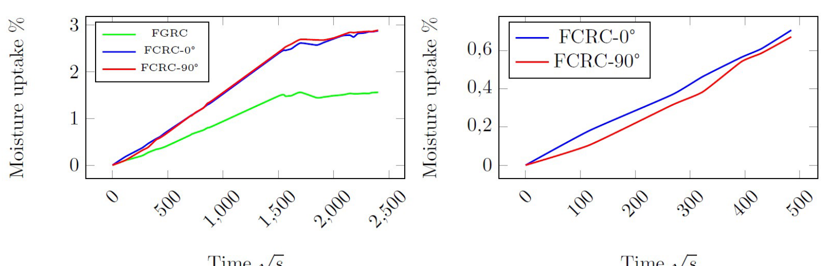
Where T_s is the stagnation point temperature of an ideal gas at temperature T_∞ impacting an object with a Mach number Ma. The temperature cycle is performed considering the ASTM-D7792 each one constituted of a 3-hour static phase at 30°C and 90% relative humidity, followed by a 3-hour thermal ramp to -22°C. General aviation planes typically cruise between 4500 and

9500 feet in altitude (data collected considering the data-sheet of a Cessna-172). For the ISA at this altitude, temperature values span between 6°C and -3°C, far less in respect of a commercial flight. Each cycle consists of 3-hour static phase at 30°C and 90% relative humidity, followed by 3-hour thermal ramp to -22°C.

Interlaminar shear strength
The relevant literature shows how composite materials behave when conditioned and frozen-thawed [3,4]. To understand the phenomena, one must investigate the adhesion between reinforcing fibre and epoxy matrix. Internal microcracks, voids and water inclusion can reduce the material's interlaminar characteristics, lowering its mechanical properties and operational life. The interlaminar shear strength is a matrix-dominated feature that plays a significant role in a variety of applications, as interlaminar shear is frequently the mechanism for load transmission across various composite components. The ASTM D2344 is adopted for manufacturing and test.

Test specimens
Two types of aeronautical composite materials are investigated using 0° ASTM specimens. One consists of carbon-reinforced fibre, while the other consists of glass-reinforced fibers. In both material a differential scanning calorimetry test is performed to assess the glass transitional glass temperature, T_g values span between 140°C and 145°C.

Conditioning process
Temperature and relative humidity influence composite materials degradation. Moisture affects resin and fiber-resin interface under humid conditions. Temperature affects moisture absorption as well, expanding it in freezing conditions and contracting it at room temperature. Moisture absorption is a diffusion process, where water molecules move from high to low concentration until equilibrium. In moisture uptake conditioning, ASTM D5229's "B" procedure is followed. Specimens are immersed in distilled water at 80°C and weighed at defined intervals until saturation. Another parameter variation was investigated to better define composite material humidity absorption. To study carbon fiber-reinforced composites' humidity absorption, two types of specimens were created. The first type, according to ASTM D5229, is made of 0° fibers, while the second is made of 90° fibers.



(a) Moisture uptake confrontation: FGRC; FCRC-0°; FCRC-90°
(b) Close-up moisture uptake confrontation FCRC-0° vs. FCRC-90°

Figure 1: Conditioning results in different experimental conditions

Fig.1 data shows that both materials reached saturation in approximately 30 days. Fig.1.b demonstrates an opposite initial humidity absorption trend than expected, considering the higher concentration of fiber ends on the long side of the specimen. Specifically, FCRC-0° exhibits a higher absorption coefficient compared to FCRC-90°. However, after an initial transient, both specimen types (FCRC-0° and FCRC-90°) show similar behaviors regarding the rate and nature of moisture absorption. Ultimately, both reach saturation simultaneously, indicating that the fiber orientation in the plies has minimal impact on the absorption process.

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Computed Tomography

A North Star Imaging X25 CT X-Ray Inspection is employed to inspect intralaminar damage due to conditioning and thermal cycling. The techniques works well also if material to be analyzed is not radioopaque (as for example carbon fibers). In this case it previously moistened with a Zinc Iodide solution, which penetrates defects by capillarity, making them visible to X-rays. For non-radio-opaque composite materials an opaque enhanced dye penetrant was been used

Experimental results

Thermal and hygrothermal behavior is studied by separating the effects of water inclusion from those due to environmental temperature variation. The effects of thermal cycles are investigated as a final step.

Thermal effects. Temperature significantly affects the mechanical properties of composites, causing microstructural and property transformations including thermal expansion, degradation, and softening. To establish accurate testing conditions, dry specimens are referenced at a standard temperature of 23°C as a baseline. Shear strengths of carbon-reinforced and glass-reinforced composites are determined as 91.77±0.37MPa and 72.52±1.72MPa, respectively, exhibiting a linear elastic phase followed by sudden load decrease until fracture, in accordance with ASTM D2344. Temperature-dependent behavior is assessed from tests conducted at 23°C, 60°C, 80°C, 110°C and 125°C; the results are presented in Fig.2. Samples undergo controlled heating in a convection oven aided by infrared lights to maintain desired temperatures. Interpolation curves generate mean shear stress summary graphs. Macroscopic examination and X-Ray Tomography confirm the expected fracture behavior of composites across different temperatures.

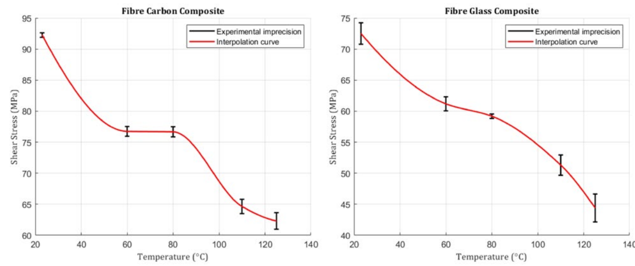
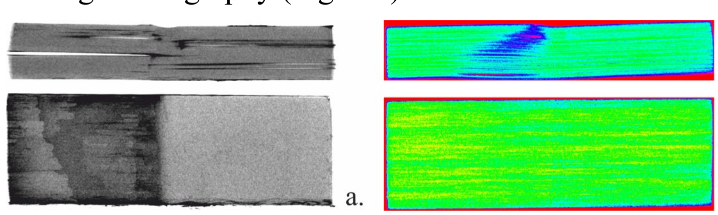


Figure 2: Interpolation ILSS results for FCRC and FGRC specimen at different temperatures.

Hygrothermal effects. Plasticization occurs when water molecules are absorbed into a material, transitioning it from a glassy to a rubbery state. This results in enhanced chain mobility and weaken intermolecular bonds. Consequently, there is a decrease in strength modulus and stiffness, while toughness and strain capacity increase. The ILSS test demonstrates that the carbon-reinforced composite exhibits plastic behavior with a less pronounced fracture compared to the dry material. Fracture occurs due to shear stress at mid-plane, confirmed by macroscopic examination, tomographic analysis (Fig. 3.a), and slow-motion videos. In contrast, the glass fiber reinforced composite initially shows an elasto-brittle behavior and the expected failure mode according to ASTM D2344. However, further investigation reveals an incorrect failure mode during the ILSS test. The wet glass-reinforced specimen collapses at the center until buckling occurs, as observed through tomography (Fig. 3.b).



a. b.

Figure 3: Tomography results on an FCRC and FGRC specimen tested in "wet" condition.

Thermal cycling effects. A drying cycle is included in both types of cycles to restore the material to a dry condition once the cycle is completed. By removing the internal water in the composite, the impacts of cycles can be separated from the plasticization effects of the matrix. Fig.4.a and Fig.4.b compare the "Freeze-Thaw" and "Hot-Hot" cycling outcomes. According to previous hypotheses, the results should demonstrate a distinction between the two curves indicating the severity of degradation caused by the "F-T", due to the nature of water expansion within the material. As a matter of fact, the results obtained cannot demonstrate this phenomenon.

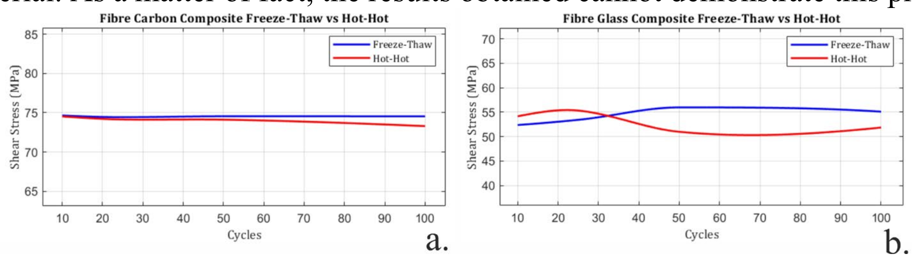


Figure 4: ILSS data confrontation for FCRC and FGRC "F-T" vs. "H-H" cycling.

Conclusions

The study is aimed to establish a representative coefficient indicating mechanical deterioration of materials under harsh environmental conditions, for timesaving in aeronautical industry destructive tests and standardized strength loss parameters in project planning. Two materials commonly used, carbon-reinforced and glass-reinforced composites, were selected. ILSS test investigated the phenomenon. Findings revealed significant shear strength reduction in both materials compared to the dry condition, with the "Hot-Wet" condition showing the most pronounced reduction. Further research is needed to understand failure mechanisms. Existing literature suggests a -25% shear strength reduction [1]. Research on multiple cycles and humidity absorption in composite materials coated with aeronautical paint is of interest, as it provides a more realistic understanding of aeronautical component behavior under hygrothermal effects.

References

[1] Mohammad Abedi, S. Ebrahim Moussavi Torshizi, and Roohollah Sarfaraz. Damage mechanisms in glass/epoxy composites subjected to simultaneous humidity and freezethaw cycles. Engineering Failure Analysis, 120:105041, 2 2021. https://doi.org/10.1016/j.engfailanal.2020.105041

[2] Laurent Cormier and Simon Joncas. Effects of cold temperature, moisture and freeze thaw cycles on the mechanical properties of unidirectional glass fiber-epoxy composites. American Institute of Aeronautics and Astronautics, 4 2010. https://doi.org/10.2514/6.2010-2823

[3] Pietro Aceti, Luca Carminati, Paolo Bettini and Giuseppe Sala. Hygrothermal ageing of composite structures. Part 1: technical review. Composite Structures, 117076, 2023. https://doi.org/10.1016/j.compstruct.2023.117076

[4] Pietro Aceti, Luca Carminati, Paolo Bettini and Giuseppe Sala. Hygrothermal ageing of composite structures. Part 2: mitigation technique, detection and removal. Composite Structures, 117076, 2023. https://doi.org/10.1016/j.compstruct.2023.117076