

PAPER • OPEN ACCESS

## Is Hydrothermal Aging of Construction Polymer Composites a Reversible Process?

To cite this article: Sotirios Grammatikos *et al* 2020 *IOP Conf. Ser.: Mater. Sci. Eng.* **842** 012004

View the [article online](#) for updates and enhancements.

### Recent citations

- [Basalt Fibre Composite with Carbon Nanomodified Epoxy Matrix under Hydrothermal Ageing](#)  
Tatjana Glaskova-Kuzmina *et al*

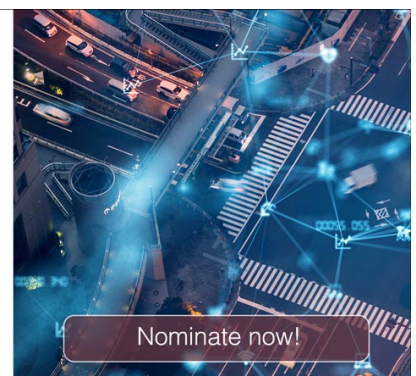


**The Electrochemical Society**  
Advancing solid state & electrochemical science & technology

The ECS is seeking candidates to serve as the  
**Founding Editor-in-Chief (EIC) of ECS Sensors Plus,**  
a journal in the process of being launched in 2021

The goal of ECS Sensors Plus, as a one-stop shop journal for sensors, is to advance the fundamental science and understanding of sensors and detection technologies for efficient monitoring and control of industrial processes and the environment, and improving quality of life and human health.

*Nomination submission begins: May 18, 2021*



# Is Hygrothermal Aging of Construction Polymer Composites a Reversible Process?

Sotirios Grammatikos<sup>1, a)</sup>, Styliani Papatzani<sup>2, 3, b)</sup> and Mark Evernden<sup>4, c)</sup>

<sup>1</sup>Group of Sustainable Composites, Department of Manufacturing and Civil Engineering, Norwegian University of Science and Technology, Gjøvik, Norway

<sup>2</sup>Hellenic Army Academy, Department of Mathematics and Engineering Sciences, Vari, Greece

<sup>3</sup>Hellenic Ministry of Culture, Directorate of Restoration of Medieval & Post-medieval Monuments, Athens, Greece

<sup>4</sup>Department of Architecture & Civil Engineering, University of Bath, Bath, United Kingdom

<sup>a</sup> sotirios.grammatikos@ntnu.no

**Abstract.** This paper presents the effects of wet/dry cycling loading on the moisture uptake behavior of a Fibre Reinforced Polymer (FRP) composite used in the civil engineering sector. FRP samples of various dimensions were cut from an ‘off-the-shelf’ pultruded flat sheet and conditioned in a cyclic hygrothermal environment. A series of 3 consecutive moisture absorption-desorption cycles lasting for 153 days were carried out to investigate the moisture uptake behavior of FRPs. The hygrothermal procedure consisted of immersion in 60°C distilled water until saturation and consecutively drying in a 60°C oven until equilibrium was reached. After the 1<sup>st</sup> desorption cycle, it was found that FRP samples lose a significant amount of mass due to chemical decomposition, the extent of which increases as wet/dry cyclic loading progresses. The effective mass loss leads to a subsequent significant increase in the rate of moisture uptake. Mechanical behavior of the FRPs aged at 40°C, 60°C and 80°C for 224 days is examined at both ‘wet’ and ‘dry’ states to reveal the reversible and irreversible effects of moisture uptake. It was revealed that the effects of 40°C hygrothermal aging on mechanical performance are reversible when moisture is removed.

## 1. Introduction

Pultruded FRPs (FRPs) are commonly used materials in the construction sector. The common cross section of a FRP consists of E-glass fibre reinforcement in a thermoset matrix [1]. FRPs are being employed as primary and secondary structural elements providing high resistance to extreme environmental conditions such as freeze-thaw, UV, etc. [2]. In addition to bridge engineering, civil FRPs [3] are used in a variety of other engineering applications including facades, off-shore and on-shore platforms, wind turbine blades, ladders and composite utility poles [4, 5], as well as reinforcement of concrete and steel structures [6].

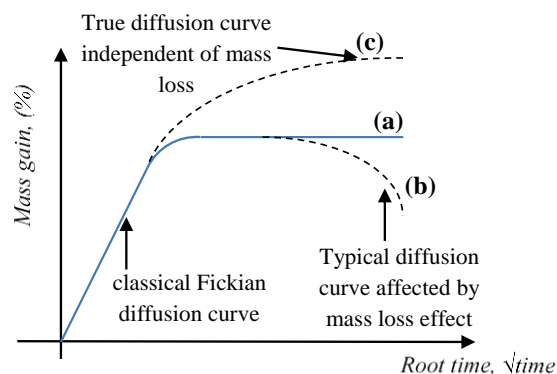
In off-shore applications, the FRPs would be partly or fully submersed in water and subjected to wetting and drying cycles. It is therefore, essential that an experimental regime of simulated accelerated aging process is devised, assessing the behavior of FRPs during absorption and desorption cycles.



Thus far, the diffusion coefficients in the three principal directions have been determined for selectively aging conditions at 60 °C through the application of one-dimensional Fickian theory. Mechanical, viscoelastic and physico-chemical analyses evaluated the moisture uptake of the FRPs and verified the decomposition taking place during the aging of FRP samples after 224 days of immersion [7, 8]. Elemental analysis has showed no chemical degradation incidents on the fibre reinforcement surfaces and infrared spectroscopy revealed superficial chemical alteration in the aging matrix. Therefore, it was reported that chemical decomposition, which is simply seen as mass loss of the initial dry mass of samples, stems from the ‘weak’ fibre sizing [1, 7]. Moreover, a sensing system based on Electrical Impedance Spectroscopy (EIS) has been developed to reveal ‘true’ moisture uptake characteristics of FRPs, independent of mass loss due to decomposition effects [9]. It is of particular interest though, to assess the effect cyclic hygrothermal loading has on FRPs and the possibility to reverse the effects of aging. The results are significant for the development of durable composites exposed in wetting and drying environments, while research can be extended to loading combinations including harsh environment and extreme loading requirements.

## 2. Background

When FRP samples are exposed to moist environments, moisture concentration increases with time and reaches a saturation point after an extended period of time which is always dependent on the exposure temperature, the type and thickness of the material. Generally, moisture absorption in polymer composites follows a Fickian diffusion trend [10]. However, Fickian theory is unable to describe the moisture diffusion process when significant mass loss due to chemical decomposition occurs simultaneously with moisture absorption [1]. Figure 1 depicts 3 moisture uptake vs. time curves that are representative of a) a classical Fickian diffusion three-stage curve with no mass loss, b) a diffusion curve affected by mass loss taking place, and, c) a ‘true’ diffusion curve when mass loss takes place without affecting the moisture uptake measurements.



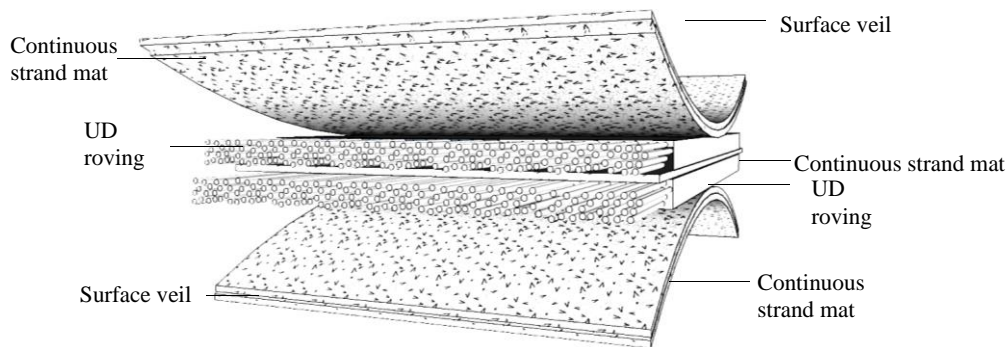
**Figure 1.** Representative plot of a) a classical Fickian diffusion three-stage curve with no mass loss, b) a diffusion curve affected by mass loss and c) a ‘true’ diffusion curve when mass loss takes place without affecting moisture uptake measurements for Mass gain (%) vs.  $\sqrt{t}$

Water uptake monitoring conducted via gravimetric measurements often results into case ‘b’ curves of Figure 1. It is therefore interesting to study the effect of cyclic hygrothermal loadings on the water uptake characteristics of FRPs.

## 3. Experimental procedure

### 3.1. Materials

A 5-ply commercially available glass FRP pultruded profile [FS040.101.096A] was tested provided by ‘Creative Pultrusions Inc., PA, USA’.



**Figure 2.** FRP profile schematic representation.

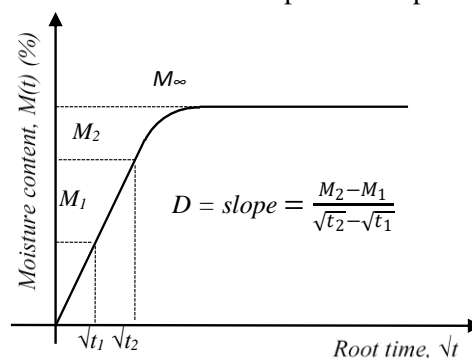
The nominal thickness of the profile was approximately 6.4 mm. The outer surfaces of the laminates are covered by a protecting and non-structural polyester layer (veil) which has the dual functions of retarding moisture ingress and protecting the FRP material from UV radiation. E-glass fibres served as the reinforcement and an isophthalic polyester resin formed the matrix of the composite. Figure 2 shows the structure of the tested FRP material which consisted of 3 continuous strand mats (CSM) and 2 unidirectional (UD) layers with 33.3% fibre and 54.5% fibre volume fractions, respectively.

### 3.2. Hygrothermal aging

Cyclic hygrothermal loadings were conducted on 40x40mm samples immersed in 60°C distilled water. All samples were dried in an oven for 48hrs at 30°C to remove any moisture absorbed by the environment. Samples were subjected to a total of 3 absorption-desorption cycles. Moisture absorption cycles continued up until samples had gained 0.5% of mass compared to their initial dry state. Then samples were dried in an oven at 60°C until equilibrium, prior to water immersion for the next cycle. Gravimetric measurements were conducted using a digital analytical scale with  $\pm 0.001\text{mg}$  accuracy. Relative ( $M(\%)$ ) moisture uptake increase was determined using Eq. 1 (ASTM D5229), see figure 3:

$$M(\%) = \frac{M_t - M_0}{M_0} * 100\% \quad (1)$$

where  $M_t$  is the measured moisture mass at time  $t$  and  $M_0$  the initial dry mass (reference state). Moisture uptake measurements were conducted over a period of up to 224 days [1].



**Figure 3.** Diagram of a classical Fickian diffusion three-stage curve for  $M(t)$  vs.  $\sqrt{t}$  and the maximum absorbed moisture content  $M_\infty$ .

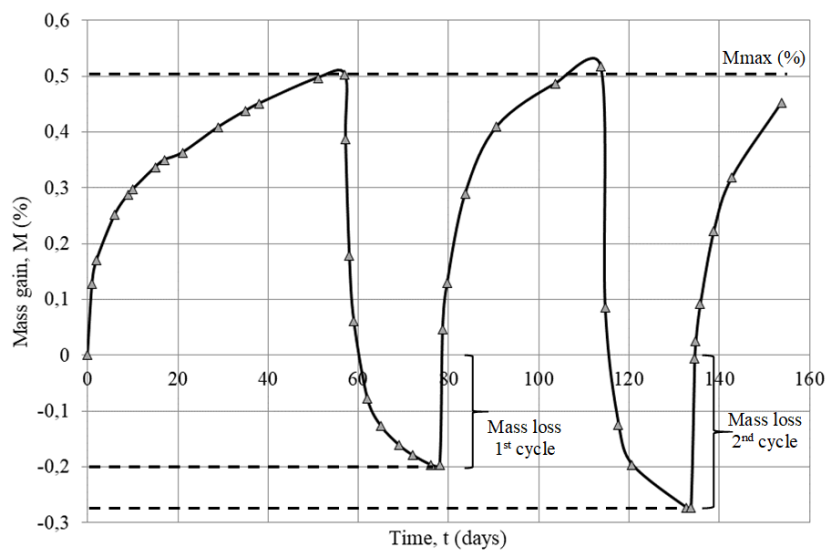
### 3.3. Mechanical testing

In order to assess the effects of hygrothermal cyclic loadings on the behaviour of the FRP material, 'Plate twist' testing was adopted. Plate twist is a standard in-plane shear mechanical test for polymer composites. During plate twisting, samples are subjected to minimal flexural loads which practically

do not affect the structural performance of the material. For practicality reasons, it was assumed that samples are not influenced by plate twisting and therefore the same samples were used for both wet and dry shear tests. Plate twist shear tests were conducted according to the ISO 15310 standard for testing [11]. Samples were cut in 200x200x6.4mm dimensions and subjected to water immersion at 40°C, 60°C and 80°C for a period of 224 days. After aging, all samples were tested in shear in the wet state. Subsequently, samples were dried in an oven (drying temperature identical to hygrothermal aging temperature), until equilibrium was reached. After drying, samples were then tested in their dry state.

#### 4. Results and discussion

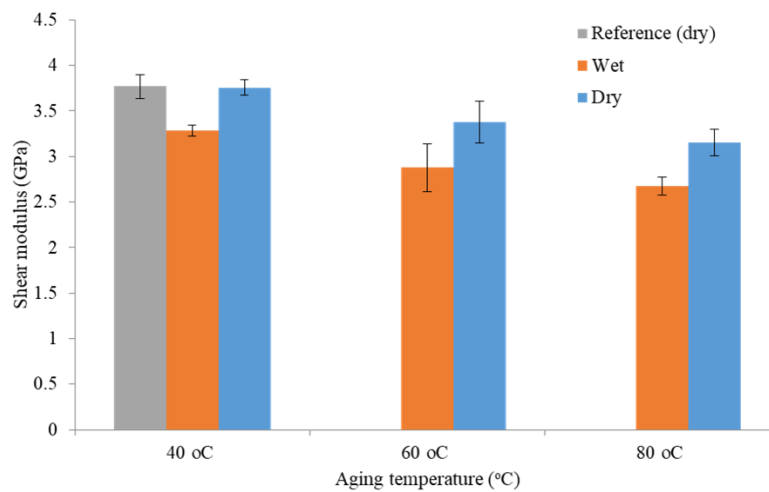
Figure 4 displays a representative 3-cycle moisture absorption-desorption curve. As can be seen, it takes less days for the samples to reach 0.5% mass gain, during the 2nd and significantly less during the 3rd cycle loading.



**Figure 4.** Moisture absorption and desorption curve for one sample hydrothermally aged at 60°C.

This effect is a result of significant mass loss that takes place during hygrothermal aging and is revealed during the drying phases of the material. Chemical decomposition leads to mass loss which practically allows for leachates to be washed out from the FRP structure. Chemical analysis of the aging medium has proved that leachates stem from the fibre/matrix interface paving the way for fibre/matrix interfacial failure [1, 7]. At the same time, hygrothermal aging along with wet/dry cyclic loading promote matrix cracking. The combination of interfacial failure and matrix-cracking leads to intrinsically more space or ‘reservoir’ for water molecules to penetrate altering the moisture uptake behavior of the material. Fibre/matrix interface failure, matrix-cracking and increasingly higher amount of moisture penetrating the structure are expected to promote a gradual deterioration of the FRP’s structural performance. It could be postulated that decomposition may cease when all the active compounds are totally consumed.

With respect to mechanical performance, figure 5 illustrates the ‘wet’ and ‘dry’ shear modulus of the studied FRP after 224 days of hygrothermal aging in comparison with the reference (unaged) dry state. As can be seen, hygrothermal aging induces detrimental effects to the material when tested at its wet state. However, this is not the case for aged samples tested at their dry state. It was revealed that, samples aged at 40°C for 224 days, recover completely (100%) their performance when tested at their dry state.



**Figure 5.** Shear modulus as a function of aging temperature for wet and dry samples after 224 days of aging.

In this respect, it becomes apparent that hygrothermal aging and mass loss due to decomposition become detrimental to the structural performance of FRPs at their wet state and less significant in the absence of moisture.

## 5. Conclusions

The effects of wet/dry cycling loading on the moisture uptake behavior of a commercially available pultruded polymer composite were investigated. The cyclic hygrothermal loadings demonstrate that the material losses mass from the initial dry mass due to chemical decomposition, which has an effect on the rate of moisture diffusion. Interestingly enough, the mechanical testing of wet and dry samples revealed that material decomposition is to a certain extent reversible when the material returns to its initial dry state.

## 6. References

- [1] Grammatikos, S.A., et al., Moisture uptake characteristics of a pultruded fibre reinforced polymer flat sheet subjected to hot/wet aging. *Polymer Degradation and Stability*, 2015. 121: pp. 407-419.
- [2] Pochiraju, K.V., G.P. Tandon, and G.A. Schoeppner, *Long-Term Durability of Polymeric Matrix Composites*, Springer, 2011.
- [3] Bakis, C., et al., Fiber-Reinforced Polymer Composites for Construction—State-of-the-Art Review. *Journal of Composites for Construction*, 6(2), (2002) pp. 73-87.
- [4] Shao, Y. and S. Kouadio, Durability of fiberglass composite sheet piles in water. *Journal of Composites for Construction*, 6(4), (2002) pp. 280-287.
- [5] *The New and Improved Pultex® Pultrusion Design Manual*, C.P. Inc., Editor. Alum Bank, PA, (2014) p. 255.
- [6] Linghoff, D., R. Haghani, and M. Al-Emrani, Carbon-fibre composites for strengthening steel structures. *Thin-Walled Structures*, 47(10), (2009) pp. 1048-1058.
- [7] Grammatikos, S.A., et al., On the response to hygrothermal aging of pultruded FRPs used in the civil engineering sector. *Materials & Design*, 96, (2016) pp. 283-295.
- [8] Ullah, Z., et al., Multi-scale computational homogenisation to predict the long-term durability of composite structures. *Computers and Structures*, 181, (2017) pp. 21-31.
- [9] Grammatikos, S.A., et al., Impedance spectroscopy as a tool for moisture uptake monitoring in construction composites during service. *Composites Part A: Applied Science and Manufacturing*, 2018. 105: pp. 108-117.

- [10] Surathi, P., et al., Hygrothermal Effects on Durability and Moisture Kinetics of Fiber-reinforced Polymer Composites. 2006: Department of Structural Engineering, University of California, San Diego.
- [11] Grammatikos, S.A., et al., Thermal cycling effects on the durability of a pultruded GFRP material for off-shore civil engineering structures. *Composite Structures*, 2016. 153: pp. 297-310.

### **Acknowledgments**

The EPSRC project DURACOMP (Providing Confidence in Durable Composites, EP/K026925/1) is greatly acknowledged for financial support.