# LONG-TERM EFFECTS OF MOISTURE ABSORPTION IN CF/PEKK COMPOSITES

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**Abstract**

Carbon fiber-reinforced polyetherketoneketone (CF/PEKK) thermoplastic composites were tested under various aging conditions to investigate the effects of moisture absorption on their mechanical properties, including static and fatigue performance. Accelerated moisture uptake was modeled up to saturation using Fick's law, enabling the derivation of the corresponding moisture absorption curve. This experimental procedure replicates the long-term environmental exposure typically experienced by high-performance composites. Both unconditioned (as-received) and moisture-conditioned CF/PEKK samples were analyzed to assess changes in static and fatigue responses under cyclic loading. The results demonstrated a reduction in overall mechanical performance for the conditioned samples compared to their unconditioned counterparts, indicating the detrimental impact of moisture on fatigue life and structural integrity.

## 1. INTRODUCTION

Thermoplastic polymers and composites offer several advantages over conventional thermosetting resins, such as epoxies. These advantages include superior chemical resistance, enhanced impact toughness, and usability across a broader temperature range (Ref. 1, Ref. 2). Furthermore, thermoplastic systems typically exhibit significantly lower saturation levels of moisture and water uptake compared to their thermosetting counterparts, resulting in reduced mechanical degradation under hot/wet environmental conditions (Ref. 3, Ref. 4). Moisture absorption remains one of the most prevalent aging mechanisms affecting polymer matrix composites in aerospace applications. It can induce physical swelling of the polymer matrix, leading to dimensional instability and the formation of microcracks, which in turn compromise the mechanical integrity and long-term performance of the material (Ref. 5).

This study investigates the impact of moisture-induced aging on the mechanical performance of a semi-crystalline carbon fiber-reinforced polyetherketoneketone (CF/PEKK) thermoplastic composite. Accelerated conditioning protocols were applied to simulate long-term environmental exposure, with a focus on moisture absorption effects. Static and fatigue tests were conducted on both unconditioned and conditioned specimens to assess degradation in mechanical response. The results revealed a notable reduction in both static strength and fatigue life following moisture uptake. These findings underscore the importance of accounting for environmental aging in the design and durability assessment of CF/PEKK-based aerospace components. A deeper understanding of aging evolution under cyclic loading is essential for accurately predicting long-term

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service performance in demanding operational environments.

The absorption and desorption of water molecules in thermoplastic composites play a critical role in determining the integrity of the fiber/matrix interface and the performance of the matrix itself (Ref 3). When exposed to humid environments, the composite absorbs moisture primarily through a combination of capillary action and diffusion, with the extent of absorption generally increasing with ambient humidity. Moisture ingress typically occurs along the fiber–matrix interphase, facilitated by the presence of microcracks, voids, and capillaries (Ref. 6). This process results in a reduction of the polymer's glass transition temperature, subsequently altering its mechanical, thermal, and thermo-mechanical properties. The plasticization of the thermoplastic matrix due to moisture uptake contributes to mechanical degradation, which is experimentally evaluated in this study.

## 2. EXPERIMENTAL METHODS

### 2.1. Sample preparation

This study investigates the properties of commercial CF/PEKK thermoplastic composite laminates. The material consists of AS4 carbon fibers embedded in a PEKK matrix, with a matrix content of 34 wt%. Laminates measuring 500 × 300 × 2.35 mm were fabricated using compression molding. A symmetrical lay-up consisting of 17 unidirectional plies oriented at 0°, 90°, +45°, and −45° was adopted, as detailed in Table 1. The average consolidated ply thickness was approximately 0.138 mm.

Table 1: Ply orientation distribution.

| Ply orientation | Number of plies |
|---|---|
| 0° | 7 |
| 45° | 4 |
| 90° | 2 |
| -45° (135°) | 4 |

Mechanical test coupons with dimensions of 250 × 25 × 2.35 mm were extracted from the molded laminates using water jet cutting. A total of 60 specimens were prepared for the experimental campaign.

### 2.2. Moisture absorption

To evaluate preliminarily the moisture absorption of the selected material, coupons with geometry used in the mechanical tests were used. The coupons were kept in an oven at 120 °C for 48 hours to dry them and be sure that no moisture was present prior to the conditioning. The coupons were then weighed using an analytical laboratory balance with 1 mg accuracy. The next step involved submersing the coupons in distilled water heated bath kept at the constant temperature of 70 °C. At regular intervals, the coupons were extracted from the conditioning bath and weighed again to measure water uptake. The initial weight (after drying) and saturation weight of the two coupons were recorded and used to calculate reference conditions (unconditioned and conditioned status). The experimental moisture absorption data was taken as an average of two coupons placed in the water bath. Before the initiation of the mechanical tests, the saturation limit (in days) was noted and the coupons for mechanical testing were placed in the water bath without any intermediate measures until reaching saturation time.

A total of 20 coupons (for both static and fatigue testing after saturation) were used for the moisture absorption saturation prior to mechanical testing. Accelerated moisture uptake experiments were then performed in the same water bath adopted for the preliminary study maintained at 70 °C where samples were kept until reaching saturation. The procedure was followed strictly adhering to ASTM D5229 standard.

### 2.3. Static and fatigue tests

The static and fatigue mechanical tests performed in this study are followed according to ASTM standards D3039 and D3479, respectively. All mechanical tests (static and fatigue) of unconditioned and conditioned coupons were conducted in MTS landmark universal hydraulic testing machine at room temperature (T=23±2 °C, 45±5% humidity). Composite tabs were attached to the coupons before the initiation of the mechanical tests, to avoid slips of the coupons within the mechanical grips and local damages during the testing protocol.

The stress-strain curves for static testing are derived from monotonic loading. The ultimate tensile strength and ultimate tensile strain of both unconditioned and conditioned batches of CF/PEKK coupons were derived from conducting quasi-static tensile tests with 3 coupons each. Each of these coupons were under increasing load until complete failure.

The measured mean value of unconditioned coupons at complete failure (failure within the grips in accordance with ASTM D3479) was kept as ceiling for initiation of fatigue tests. Three load levels (70%, 60% and 50%) of the maximum average stress measured in static mode were used for the fatigue testing for both batches (unconditioned and conditioned). At least 3 coupons were tested to derive the fatigue behavior at each load level explored. The stress ratio, R, for the loading cycles at each exploration load level was kept at 0.1. Frequency was kept at 3 Hz for 70% and 60% load levels and 5 Hz for 50% load level. The fatigue testing for 50% load level at 5 Hz frequency observed an increase in surface temperature of the coupons by less than 2 °C from the room temperature.

S-N diagrams were employed to represent experimental fatigue data (cyclic loading. To model fatigue life of a material, power model was adopted (Ref. 8) The experimental analysis is carried out as a log-log relation of stress applied and number of cycles. All coupons failed for load levels failed within the grips.

Residual performance after load cycling for the unconditioned batch (ultimate tensile strength after fatigue testing) were derived, for coupons completing the run-out phase of the loading cycle ($10^6$ cycles).

### 2.4. Damage investigation

Failure and damage at the microscopic level are closely related to the mechanical properties of the thermoplastic composites (Ref. 9). Hence, reliability and endurance for conditioned and unconditioned material was studied for long term applications. With the failure analysis, the stress level at the fiber, matrix and interface can be pinpointed during (and after) the mechanical tests. The damage evolution of the coupons tested for fatigue behavior was analyzed using an optical microscope (Nikon SMZ-U) with a maximum magnification of 11x and by a scanning electron microscope, SEM, (HITACHI TM 3000).

## 3. RESULTS AND DISCUSSIONS

### 3.1. Moisture absorption curve

The initial weight after drying and saturation weight of the two coupons used for the preliminary moisture absorption analysis are reported in Table 2.

Table 2: Moisture absorption data.

| Sample ID | Weight [g] (after drying) | Weight [g] (at saturation) |
|---|---|---|
| 1 | 22.832 | 22.892 |
| 2 | 22.716 | 22.780 |

The thermoplastic composite coupon was considered as a Fickian material, and therefore the absorption properties were obtained from the experimental data. The preliminary moisture absorption study is conducted with two coupons. The experimental data obtained reported that these CF/PEKK thermoplastic composites have a minimal absorption at moisture at saturation of 0.27% in weight, which is in accordance with literature data (Ref. 5, Ref. 7).

The through the thickness diffusivity is evaluated by:

(1) $$D_Z = \pi\left(\frac{h}{4M_m}\right)^2\left(\frac{M_2-M_1}{\sqrt{t_2-t_1}}\right)^2$$

where $h$ is the plate's nominal thickness, $M_m$ is the effective moisture content, and $\frac{M_2-M_1}{\sqrt{t_2-t_1}}$ is the slope of moisture absorption plot in the linear portion of the curve. The calculated through the thickness diffusivity for the material was equal to $5.51\cdot10^{-7}$ mm$^2$/s.

Fickian material is a material where the diffusion of moisture concentration follows the Fickian diffusion law. Assuming through the thickness diffusion within the material, the Fickian law can be ex-pressed as:

(2) $$\frac{\partial c}{\partial t} = D_Z\frac{\partial^2 c}{\partial Z^2}$$

where c is the moisture concentration and z is the special coordinate through the thickness. The moisture absorption, as a function of time can be approximated by:

(3) $$F(t) = 1 - \exp\left(-7.3\left(\frac{D_z t}{h^2}\right)^{0.75}\right)$$

The one-dimensional Fickian diffusion model, employing a diffusivity parameter derived from experimental data via equation (1), yields results presented in Figure 1. Despite the simplifying assumption of unidirectional (through-thickness) diffusivity, the model described by equation (3) accurately captures the experimentally observed moisture uptake behavior.

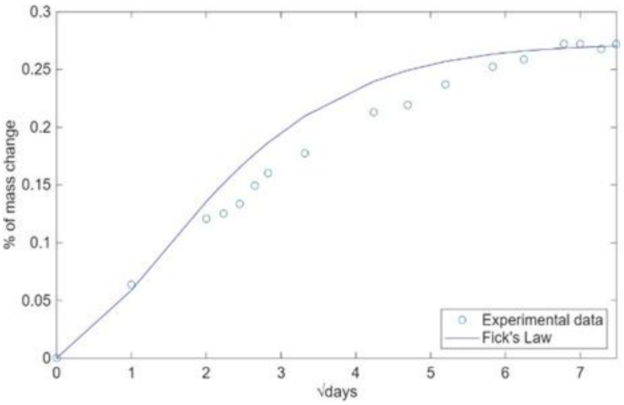


Figure 1: Experimental and modelled moisture absorption curve.

### 3.2. Static and fatigue tests

The mean ultimate tensile strength and ultimate tensile strain of CF/PEKK unconditioned coupons (3 coupons) are derived to be 1116.8±40.5 MPa and 0.0133±0.0012, respectively. Similar values were obtained for conditioned samples tested in static mode. The ultimate tensile strength and strain of conditioned coupon is derived to be 1091.5 MPa and 0.0143 respectively. The stress-strain curve for each tested coupon is presented in Figure 2. The similarity in static performance between unconditioned and conditioned samples indicates minimal moisture influence on monotonic loading.

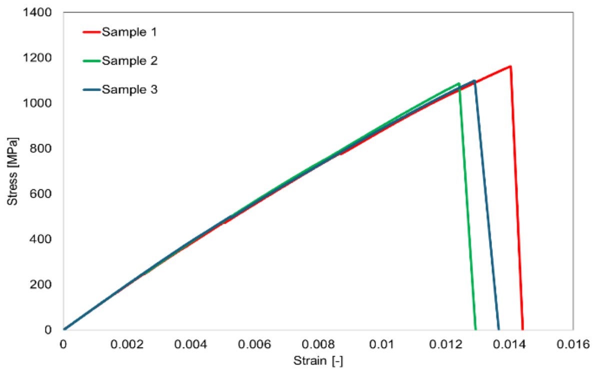


Figure 2: Stress-strain curves (Unconditioned).

The S-N curves for the three exploration load levels are derived and presented for both conditioned and unconditioned test coupons in Figure 3.

Despite the almost equal measured static properties for unconditioned and conditioned material, in fatigue mode, the conditioned samples clearly exhibit a decrease in mechanical performance, with a marked reduction of fatigue life in the high number of cycles regime (above $10^5$ cycles). In the case of conditioned material, at the lowest load level (50% of the maximin static tensile performance), fatigue life was always belove the runout set at $10^6$ cycles.

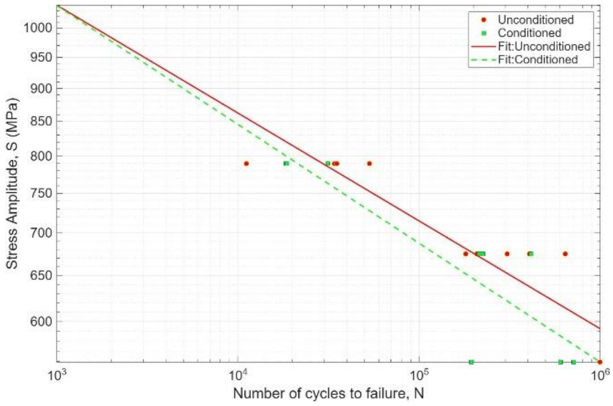


Figure 3: S-N curves (unconditioned and conditioned samples).

Hence, mechanical properties of the material are noted to deteriorate during accelerated aging/accelerated moisture uptake, and fatigue testing is more appropriate to mimicking the long-term performance of these materials in service life considering water absorption process.

On the other hand, static tensile testing of unconditioned samples that reached runout ($10^6$ cycles) in fatigue mode, showed a reduction in mechanical properties (Figure 4), indicating a damage mechanism affecting the static properties of the material.

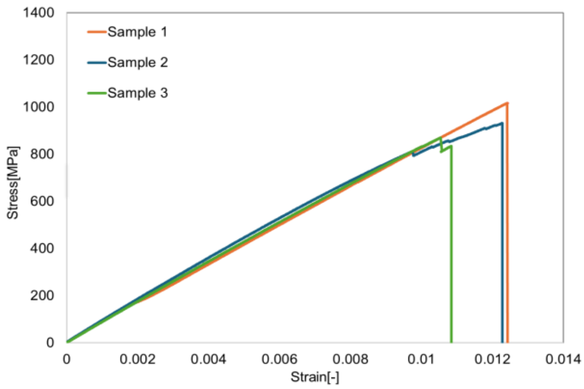


Figure 4: Stress-strain curves after fatigue life at 50% load for unconditioned samples.

The comparison for average ultimate tensile strength and ultimate tensile strain of the tested coupons for unconditioned, conditioned and after fatigue cycling (unconditioned coupons at 50% load level) is given in Table 3. A maximum stress reduction respect to unconditioned state of about 2% and 17% is detected for conditioned and fatigue cycled state, respectively.

Table 3: Ultimate tensile strength and ultimate tensile strain variation for different material conditions.

| Condition | Ultimate tensile strength [MPa] | Ultimate tensile strain [-] |
|---|---|---|
| Unconditioned | 1116.8 | 0.0133 |
| Conditioned | 1091.5 | 0.0143 |
| After fatigue cycling (unconditioned) | 927.7 | 0.0118 |

### 3.3. Failure analysis

Failure analysis were reported to understand the damage evolution across the test coupons for both conditioned and unconditioned cases. Different damage modes were highlighted depending on material conditions.

Unconditioned test coupon completing $10^6$ at 50% load level was analyzed and reported. As no single conditioned test coupon completed the runout phase for fatigue testing at each of the exploration load levels, tested samples at 60% load level was selected for the analysis completing about $10^5$ loading cycles (before complete failure).

Optical microscopy revealed that the unconditioned test coupon completing $10^6$ cycles has minimal damage. The visible damages were mainly fibers stripping or limited fiber breakage (Figure 5).


Figure 5: Optical image of unconditioned coupon completing $10^6$ cycles at 50% load level.

The conditioned coupon was subjected to fatigue testing at 60% load level for about $10^5$ cycles. The crack propagation was very prominent and symmetrical occurring largely at 90°/-45° plies and at 90°/45° plies interfaces (Figure 6). This aspect can be linked to an interlaminar weakness induced by moisture.

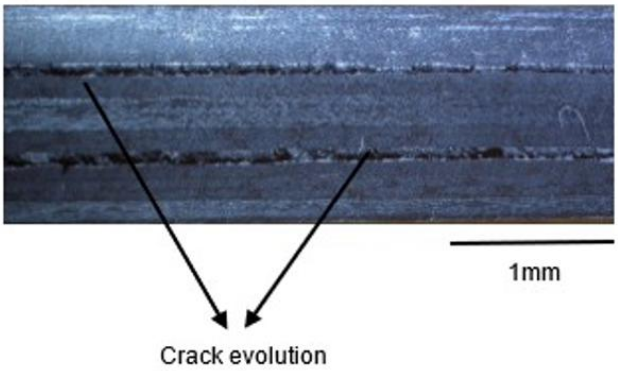


Figure 6: Optical image of conditioned coupon completing about $10^5$ cycles at 60% load showing crack localization at 90°/±45° interfaces.

Finally, SEM analysis of conditioned fatigue sample is reported (Figure 7). SEM imaging confirmed interfacial debonding and matrix cracking, consistent with the observed fatigue performance decline. Some extent of matrix-fiber interfacial damage was also observable.

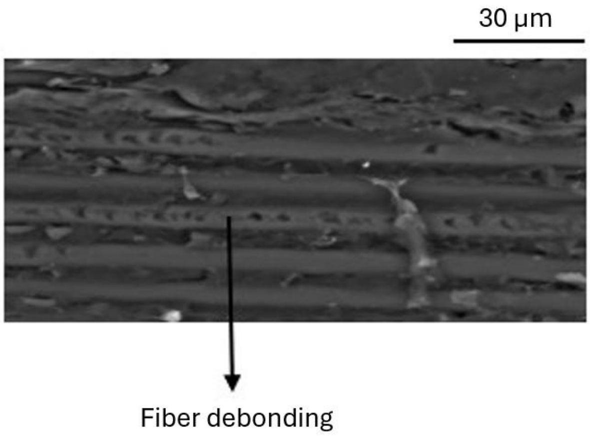


Figure 7: SEM image of open cracked surface of conditioned sample showing fiber debonding.

## 4. CONCLUSIONS

The study focused on the environmental effects on the long-term performance of CF/PEKK thermoplastic composites through mechanical testing of unconditioned and conditioned material. The long-term effects of moisture absorption on CF/PEKK thermoplastic composites were evaluated through an integrated approach combining diffusion modeling, mechanical testing, and microscopic failure analysis. Main results can be summarized in:

1. The moisture absorption saturation (through the thickness diffusivity) of the test coupons has been attained through accelerated water uptake. Moisture absorption followed Fickian kinetics,

reaching saturation at ~0.27 wt% after immersion in 70 °C water.

2. Static tensile performance was marginally affected by conditioning, with a ~2% reduction in UTS. This result indicates minimal moisture influence on monotonic loading.
3. The fatigue performance of material in different aging conditions were evaluated. Fatigue resistance decreased significantly under moisture-saturated conditions. No conditioned specimens survived $10^6$ cycles at 50% stress level. This suggests that fatigue damage is more sensitive to microstructural aging.
4. Optical and SEM observation were carried out to analyze the damage propagation of the test coupons during fatigue testing. Microscopy analysis revealed interfacial degradation and matrix cracking as primary damage mechanisms in conditioned specimens.

These findings emphasize the necessity of accounting for environmental aging effects, especially under cyclic loading, when designing CF/PEKK components for long-duration aerospace applications.

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