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Open-Hole Size and Thermal Cycling Effects on Mass Loss and Surface Degradation of Polymer Matrix Composites

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ABSTRACT

Degradation is a common problem for polymer matrix composites (PMCs) under low thermal cycling conditions. This paper investigates the effects of low thermal cycling on total mass loss (TML) and surface degradation of PMCs. Unnotched and open-hole specimens were weighed before and after low thermal cycling. The total mass loss and surface degradation of the specimens were studied over 250 cycles of 100˚C temperature difference. The experimental results showed that the mass loss linearly decreased during low thermal cycling. Also, it was found that laminates with smaller holes have higher percent mass loss than those with larger holes. Based on weight loss rates, a regression model is presented to evaluate the TML of laminated composite material samples. Also, under similar experimental conditions, the specimens exhibited 0.4% mass loss reduction after 250 cycles, and the incremental decrease of the hole diameter also decreased the TML. It was found that laminates with smaller holes have higher tensile strength variation than those with larger holes. The results showed that the incremental decrease of the hole diameter and number of cycles decreases the tensile strength of PMCs.

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1. Introduction

The degradation of mechanical properties, such as mass loss, during thermal cycling is one of the weaknesses of polymer matrix composites (PMCs). This weakness limits the applications of these high stiffness and high tensile strength materials in the aerospace industry. Virtually all the physical properties of PMCs, such as mass loss, may change with the passage of time. So, there is a need to develop a reliable model of the maximum lifetime for PMCs in aerospace service environments, such as in thermal cycling loading [1].

Different research has been done to identify the effects of different factors on the mass loss degradation of the PMCs [2–6]. These factors are mainly the effects of different kinds of radiation (ultraviolet or electron) [2], resin types [3], number of thermal cycles [4], environmental atmospheres (oxidative or neutral) [3, 5], fiber angle, and sample size [6].

Paillous et al. [2] subjected various graphite/epoxy laminate specimens to electron radiation combined with thermal cycling or to oxygen atom fluxes. The results showed that the synergistic action of electrons and thermal cycling degrades the matrix by chain scission, cross-linking, and microcrack damage.

Zhang et al. [3] evaluated the mass change in two carbon/epoxy +60˚/0˚/-60˚ triaxial braided composites, T700s/3502 and T700s/PR520, as well as the 3502 and PR520 pure resins were exposed to a thermal cycling environment. Based on their observations, the mass loss variation of both composites and pure resins were small (less than 1% after 160 cycles), and the mass loss became almost flat after 160 cycles. Also, it was found that the mass loss of composite specimens was larger than that of the pure resin specimens. This was probably due to the more volatile products that were trapped in the composites during the manufacturing process.

Shin et al. [4] evaluated the mass loss variation of composites in space. The mass of the graphite/epoxy composites after 80 thermal cycles of exposure under simulated conditions, including ultraviolet radiation, thermal cycling, and a high vacuum, was down almost 1.0% in comparison with the same mass loss in a vacuum environment at 125˚C. The results

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showed that the mass loss was a direct result of matrix loss and material out-gassing. The major out-gassed products were H_2O, N_2, and hydrocarbon (C_6H_5). Lafarie-Frenot et al. [5] considered the mass loss of cross-ply laminate samples in an oxidative and neutral environment. The main result obtained was that the test atmosphere, either neutral (nitrogen) or oxidative (air or oxygen), had a very significant influence on the damage processes and degradation rates of the composite materials. Also, they focused [6] on the characterization and understanding of the mechanisms of the thermo-oxidation of the epoxy matrix alone, both with and without mechanical stress. They used a neutral or oxidizing environment to subject specimens to thermal aging at 120˚C, 150˚C, and 180˚C. The results showed that the mass loss rate depended strongly on the stacking sequences and fiber orientation with respect to the exposed surface.

Recently, Ghasemi et al. [7–9] studied the mechanical and physical property degradation of unnotched glass/epoxy specimens. They used the Taguchi method [8–9] to consider a comprehensive experimental analysis to find the main effective factors and fracture behavior of specimens on glass/epoxy composite components subjected to thermal cycling. The unnotched specimens had various fiber volume fractions and stacking sequences. Statistical analysis was performed to study the contribution of each factor. Based on weight loss rates, a regression model was presented to evaluate the mass loss of laminated composite material samples.

One of the important factors affecting the design of composite materials is the load-carrying capability of the composite's joints. Holes are intentionally created to reduce structural weight or to facilitate joining and access. However, holes also undergo high stress concentration during loading, and, consequently, damage is often initiated from the hole area. During flight, aircraft structures are certainly subjected to fatigue loading. If the structure is made of composite laminates and some parts are reinforced with a stitching thread, it is imperative to investigate the fatigue characteristics of stitched laminates [10].

Persson et al. [11] studied the effects of open holes on the strength and fatigue life of carbon/epoxy composite specimens. Also, Tagliaferri et al. [12] investigated the tensile behavior of unidirectional glass fiber–reinforced polymer (UD-GFRP) laminates with drilled holes. Salleh et al. [13] reported the effects of drilled holes on the mechanical behavior of long kenaf composite with and without fiber glass reinforcement. The surface fracture, residual tensile strength, and stiffness of natural fiber/fiber glass hybrid composites were investigated.

Shimokawa et al. [14] evaluated the effect of isothermal aging on the ultimate strength of hole-notched and unnotched composites. The specimens were isothermally aged at 120°C and 180°C for up to 15,000 hours. Also, the effects of 5 oxidation-resistant treatments on open-hole composites' compressive strength at 180°C were investigated after isothermal aging for 5,000 hours at 180°C. The test results showed that the effects of isothermal aging on ultimate strengths and oxidation-resistant treatments on open-hole composites' compressive strength.

Nakamura et al. [15] subjected two kinds of carbon-reinforced composites up to 10,000 thermal cycles for use in the structures of the next-generation supersonic transport. Open-hole compressive (OHC) specimens with quasi-isotropic stacking sequences were considered. The number of microcracks initiated was counted, and the OHC strength was investigated by static mechanical testing at room temperature before and after thermal cycling tests. The study discussed the mutual relationship between the number of thermal cycles, number of microcracks initiated, and OHC strength. The results showed that the OHC strength before and after thermal cycles did not change significantly. Therefore, thermal cycles and the initiation of transverse microcracks did not affect OHC strength in their study.

However, despite the extensive studies on the mechanical behavior of open-hole laminates, there is a lack of studies on the effects of thermal cycling on mass loss and surface degradation of open-hole laminates. The goal of the present research is to predict the mass loss of PMCs as a function of thermal cycling exposure and composite material properties.

In the previous research, tensile tests and failure analysis have been investigated [16]. In this research, the mass loss behavior of specimens was investigated. The required specimens were prepared using glass fibers and epoxy resin. A two-chamber apparatus was used for thermal cycling tests on the specimens. The weight loss of specimens was measured. Based on a statistical approach, a correlation between the hole size and thermal cycles on the total mass loss was established, and a sensitivity analysis of each parameter on mass reduction was conducted during the 250 cycles.

2. Material and Experimental Conditions

All tests and observations were performed on glass/epoxy composite materials, which used the ML506 epoxy resin and polyamine hardener (HA-11) as the matrix. Due to mechanical properties and low viscosity, this resin is a suitable material for the composite applications. Also, unidirectional E-glass fibers (supplied by GuritTM) were used as the reinforcing material. The mechanical properties of the fiber and resin are shown in Table 1.

Table1. Mechanical and physical properties of epoxy resin and glass fiber

Material properties	Units	ML506 Epoxy	E-Glass
Tensile modulus	GPa	2.79	72
Shear modulus	GPa	15.24	15.24
Poisson's ratio	-	0.35	0.3
Density	g/cm^3	1.11	2.48
Coefficient of thermal expansion (CTE)	10^{-6}/°C	62	4.9

Laminates were prepared with [02/902]s stacking sequence. The hand lay-up method was used to fabricate composite laminates, and the specimens were allowed to cure for seven days at room temperature.

The test specimens were cut from laminates according to the standard ASTM D3039 [17]. The fiber volume fraction of the composites was 53%. Three rectangular cubic shape specimens were fabricated for each test. The length and width of specimens were 250 ± 2 and 25 ± 0.5 mm, respectively. Also, the thickness of each layer was 0.2 mm and the thickness of specimens was 1.6 ± 0.1 mm. The cross-ply glass/epoxy tabs were locally bonded on each side of the specimens. The size of the hole was 5 and 10 mm, as shown in Figure 1, and the specimens without holes were considered to compare the results.

For low thermal cycling experiments, a thermal cycling apparatus was assembled to provide the temperature cycle of the specimens [8]. The low thermal cycling tests consisted of 250 triangular thermal cycles. The minimum and maximum temperature of each cycle were 0 and 100°C, respectively. Also, the cooling and heating rates were constant (17°C/min). The glass transition temperature of the composite material was measured to be 157°C [18]. The maximum temperature of the thermal cycle, 100°C, was selected in order to accelerate the damage processes. This temperature is much higher than what could be supported by this material in real application, but is in the viscoelastic domain. Two sensors for the two sides of the specimen were used to measure their temperatures. An example of a temperature record is shown in Figure 2. The specimens were subjected to 0, 50, 100, 150, 200, and 250 thermal cycles.

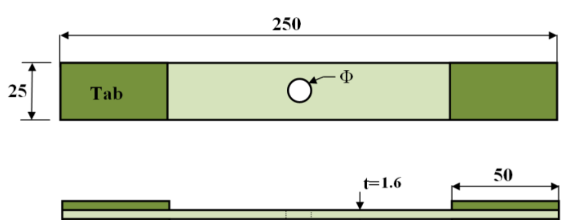

Figure 1. Test specimens with open hole

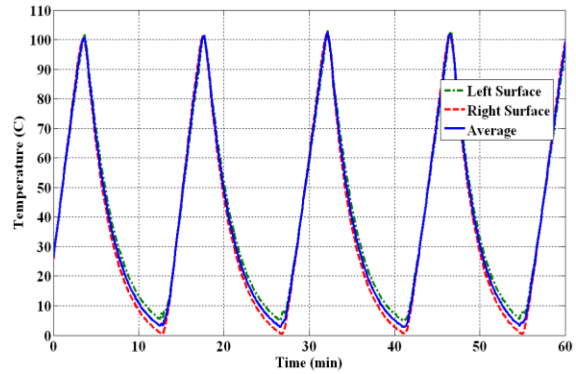

Figure 2. Different experimental thermal cycling profiles

The different profiles in Figure 2 show that the left and right sides of the samples and, consequently, all layers of the specimen have uniform temperataure any time, and are in the steady-state temperature distribution.

3. Results and Discussion

3.1. Total Mass Loss

For each specimen, total mass loss (TML) of the specimens was calculated as follows:

$$\%TML = \frac{M_b - M_a}{M_b} \times 100, \tag{1}$$

where M_b and M_a are the mass of the specimen before and after the thermal cycling experiments.

As shown in Table 2, incrementing the number of thermal cycling caused an increase in the mass loss. Also, the results show that the TML of the unnotched and open-hole specimens are close together, but the average values of unnotched specimens are greater than the open-hole specimens. One reason for this difference is the reduction of the oxidant-reactive surface of the composite in a harsh environment.

Table 2. Average total mass loss of the specimens

Number of thermal cycles	TML (%) (±SDV)		
	Unnotched	Φ=5 mm	Φ=10 mm
0	0.0 (0.0)	0.0 (0.0)	0.0 (0.0)
50	0.369 (0.05)	0.265 (0.03)	0.313 (0.07)
100	0.376 (0.05)	0.253 (0.03)	0.259 (0.03)
150	0.288 (0.06)	0.220 (0.03)	0.296 (0.04)
200	0.345 (0.04)	0.362 (0.02)	0.349 (0.02)
250	0.395 (0.06)	0.405 (0.04)	0.421 (0.05)

3.2. Surface Degradation

Studying the surface of the specimens after thermal cycling shows that surface matrix loss can be observed at a high number of thermal cycles (Figures 3–4). As shown in these figures, the surface matrix loss of unnotched and open-hole specimens increased slightly. Also, around the holes, the surface matrix loss matrix is more significant. This surface matrix loss could be an initial region for matrix de-bonding and crack propagation.

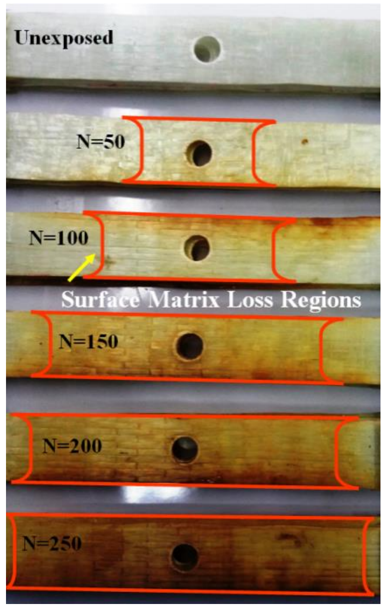

Figure 3. Growth of the surface-degradation regions during thermal cycle

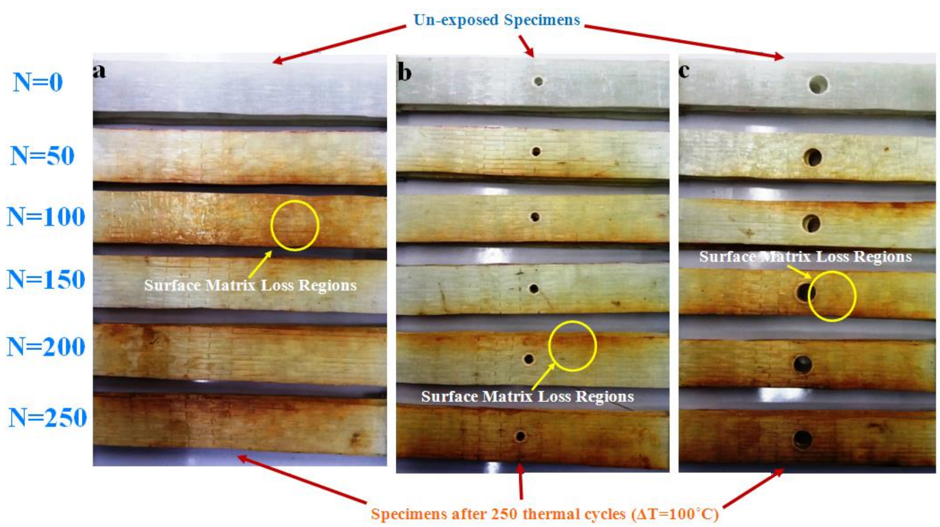

Figure 4. Surface degradation under the thermal cycling in a) unnotched, b) 5 mm open-hole, and c) 10 mm open-hole specimens.

3.3. A Proposed Regression Model for TML

Using case studies of two effective factors on the TML, a multiple linear regression model was obtained using the Minitab [19] commercial software during the low thermal cycling. The regression model function proposed to establish this correlation is as follows:

$$TML(\%) = A + BN + C\Phi , \quad (2)$$

where N and Φ are the number of thermal cycles and the hole diameter to width (D/W) ratio, respectively. Also, A, B, and C are constants that can be determined using experimental results. The values of the coefficients for this correlation are given in Table 3.

Table 3. The values of the correlation coefficients of the model

Properties	A	B	C
TML (%)	1.33×10^{-2}	1.21×10^{-3}	-2.22×10^{-3}

2.4. Sensitivity Analysis of the TML model

The mass loss behaviors of PMCs under different thermal cycling conditions were obtained (Figures 5–6) using the presented model (Equation 2) and the calibration factors presented in Table 3.

As shown in the statistical analysis, in a specific hole diameter, mass loss increases by almost 0.2414% after thermal cycling (0.0012% per each cycle), while in specific thermal cycling, the variation of hole-diameter mass loss increases by 0.0444% in large hole diameters (0.0005% per 0.01 D/W ratio). So, if each cycle and 0.01 D/W ratio is defined as a unit of parameters, each thermal cycle causes more mass loss than one unit of D/W ratio. The mass loss changes based on these parameters are shown in Figure 5. This figure indicates that increased thermal cycling causes increased mass loss at different hole diameters. One reason for this could be the incrementing oxidative reaction of the composite's molecular components in the harsh environment. As mentioned in another works [1], this mass and surface degradation in polymeric composites involves solid-gas reactions, and is associated with chain division. Also, as mentioned above, the TML slightly increases during thermal cycling, and the rate of mass loss increments by 0.0012% each cycle.

As shown in Figure 6, an increased D/W ratio has a low effect on the mass loss of the specimens. At a specific thermal cycle, the specimens with lower D/W ratios have more mass loss. Increased increments of this ratio cause decreased mass loss, slightly. It was found that the rate of mass loss decremented by 0.0005% for each 0.01 ratio.

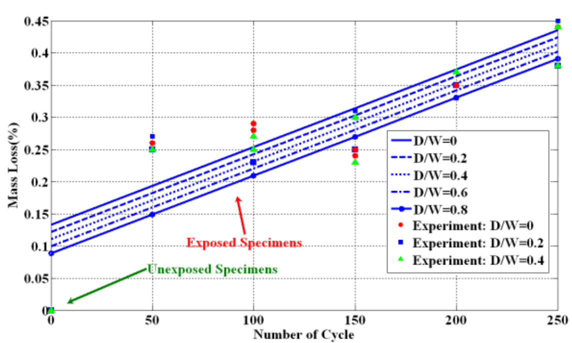

Figure 5. Variation of the mass loss as a function of the number of thermal cycles

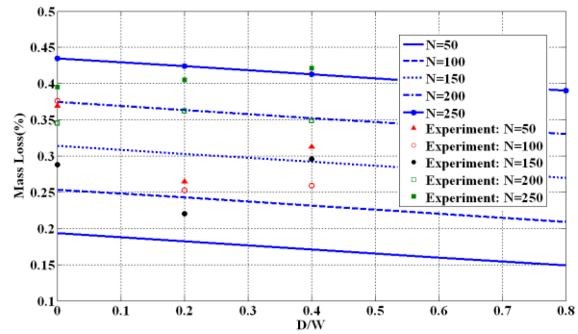

Figure 6. Mass loss of unnotched and open-hole $[0_2/90_2]_S$ specimens due to oxidation

4. Conclusions

In this study, the mass degradation of open-hole and unnotched glass/epoxy laminate composite materials subjected to thermal cycling loading was considered. A statistical approach and regression function were used to identify the effects of hole diameter and the number of thermal cycles on TML. Based on the results of the present study, the following conclusions can be drawn:

– Incremental thermal cycling causes an increase of TML. Also, TML in unnotched specimens was more than in open-hole specimens.
– Surface matrix loss of unnotched and open-hole specimens are increased slightly. Also, around the holes, the surface matrix loss was more significant.
– The TML increased slightly during thermal cycling, and rate of mass loss increment is 0.0012% per cycle. The maximum mass loss occured at the high thermal cycling load.
– Increased hole diameter to width ratio had a low effect on the mass loss of the specimens. At a specific thermal cycle, the specimens with lower D/W ratios had more mass loss.
– Increasing increments of D/W caused decreased mass loss, slightly. It was found that the rate of mass loss decremented by 0.05% per ratio.

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