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Environmental Effects on Mechanical Properties of Glass/Epoxy and Fiber Metal Laminates, Part I: Hygrothermal Aging

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A B S T R A C T

In this article, the effect of hygrothermal aging on mechanical properties of fiber metal laminates (FMLs) and E-glass/epoxy (GE) composites is investigated. First, FML and GE specimens were built using wet lay-up technique under vacuum pressure. Hygrothermal aging simulation was then carried out on both specimen types in distilled water at a constant temperature of 90 °C for 5 weeks. The resulting behavior of degradation for both types of specimens caused by hygrothermal aging was evaluated by bending and Charpy impact testing. As expected, because of the protective role of aluminum layers, FML specimens showed remarkably lower water absorption after hygrothermal aging compared to the glass/epoxy composites. Experimental results also revealed that the flexural properties of both the FML and GE laminates were affected by the hygrothermal aging, whereas a lower level of deterioration in impact strength was found.

1. Introduction

The recent development of advanced materials offering light-weight, high-strength, and high-stiffness, has been foundational for many engineering applications particularly in marine industries [1-3]. Polymer matrix composites (PMCs), as structural materials, are now widely used in marine applications including ships, patrol boats, fishing vessels, and diesel storage tanks [4]. The advantages of PMCs compared to conventional metallic materials include their low density, superior fatigue performance, corrosion resistance, and many more [5]. Despite these advantages, exposure to harsh marine environmental conditions such as hygrothermal influences, load cycling, ultraviolet radiation, and long-term water immersion have limited long-term application of these materials in the marine sector. Numerous studies have been conducted to evaluate the long-term performance of composite materials during their intended service lifes. However, there is little information available on strategies to improve long-term durability of marine composite materials. In this study, the hygrothermal behavior of fiber metal laminates (FMLs) is investigated, to evaluate their potential as water-resistant materials for marine applications.

FMLs, a new category of hybrid composite materials, were primarily developed at Delft University of Technology in 1980. They consist of alternating plies of PMC laminates and thin metal sheets, forming a hybrid material with enhanced mechanical properties [6]. They are demonstrably superior to the original component materials not only in weight reduction, but also in fatigue resistance and damage tolerance characteristics [7, 8]. Recently, Glass Laminate Aluminum Reinforced Epoxy (GLARE) as a type of FML based on high-strength glass fibers has been introduced as a material for many structural applications in aircraft such as the upper fuselage skin structure of Airbus A380 [9]. Most previous studies focused on primary advantages of FMLs such as impact resistance and fatigue tolerance [10, 11]. Yet, other potential characteristics of FMLs like their durability against specific environmental conditions have been investigated less.

As FMLs consist of aluminum and PMCs, they are likely to be susceptible to the same degradation effects as those two materials. However, the combina-

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tion of the two materials in FMLs leads to some differences that in turn lead to enhanced resistance to environmental conditions [12, 13].

Many researchers have investigated the durability of PMCs in various hygrothermal conditions. However, despite the fact that durability is a very important issue in the usage of FMLs in real environmental conditions, little work has been documented on the mechanical response of these materials subjected to hygrothermal conditions.

Karasek et al. [14] studied the effect of moisture and temperature on the impact resistance of graphite/epoxy composites. Results revealed that the damage initiation energy of composites decreased with increasing temperature. Ellyin and Maser [15] evaluated the effects of moisture absorption and exposure to an elevated temperature on mechanical properties of glass fiber reinforced epoxy composites. They concluded that the rate of moisture absorption was greater at elevated temperature (50 °C) compared to room temperature (20 °C). It also was observed that strength and stiffness decreased to some extent with the presence of moisture and increasing temperature. Nakai et al. [16] presented a work on the degradation of braided glass/epoxy composites in hot water conditions and showed that the combination of humidity and temperature can negatively affect the fiber/matrix interface properties.

Since the FMLs have a layered structure with completely different constituents, their response to hygrothermal conditions may be greatly different from conventional PMCs. In FMLs, only the external aluminum layers are exposed to environmental conditions. Therefore, because of the barrier function of aluminum layers, water penetration is limited to free edges or through bore holes [12, 13]. This can lead to a significantly lower rate of moisture absorption compared to conventional PMCs.

Botelho et al. [17] studied the effect of hygrothermal aging on mechanical properties of glass fiber/epoxy composites and FMLs. The results showed a decrease of nearly 22% in tensile strength and 10% in tensile stiffness values for conditioned glass fiber/epoxy composites, while no noticeable changes were observed in mechanical properties of FMLs.

This study reports the effect of hygrothermal aging on the mechanical properties of FML and E-glass/epoxy (GE) laminates. The hygrothermal aging (90 °C, immersed in distilled water) was used as an accelerated degradation tests. The impact strength and flexural properties of both the aged and dry specimens were measured experimentally to provide the knowledge of how the FMLs and PMCs act when exposed to hygrothermal conditions.

2. Experimental Procedure

2.1 Materials and Fabrication

The GLARE fiber metal laminate, referred to as 2/1 (2 layers of metal sheet and 1 layer of composite), in this study was built from aluminum alloy 2024-T3 sheets supplied by Alcoa Mill Products Inc., 200 gr/m^2 plain woven E-glass fabrics purchased from Colan Products Pty, and epoxy resin Araldite LY5052 and its hardener Aradur 5052 provided by Huntsman Advanced Materials Americas Inc. Specification of the aluminum alloy and resin used in this study, are listed in Table 1.

The FML plates were manufactured by stacking the three plies of plain weave E-glass/epoxy embedded between two 0.6 mm aluminum sheets in a simple mould. In the same way, E-glass/epoxy (GE) composite laminates were fabricated with 10 plies of the E-glass fabrics and epoxy resin.

After the wet lay-up process, both FML and GE plates were cured under vacuum of -60 kPa for one day at room temperature and then put into the autoclave for post-curing under pressure of 400 kPa and temperature of 100 °C for 4 h.

2.2 Hygrothermal Aging

Hygrothermal aging was conducted on both types of specimens in a controlled climatic chamber (Model CM120E, Fan Azma Gostar, Iran) by immersing the specimens in a distilled water bath at a constant temperature of 90 °C for a period of 5 weeks (Fig. 1). During the immersion, weight gain measurements were recorded weekly, using a digital balance with 0.0001 g resolution. The percentage of the water absorption (M_t) was calculated as weight gains related to the weight of the dry specimen accordance with ASTM D 5229/D 5229M [20], using the following formula:

$$M_t(\%) = (\frac{W_t - W_o}{W_o}) \times 100 \quad (1)$$

where W_t is the weight of the sample at time t and W_0 is the initial weight of the specimen.

Table 1. Specification of aluminum alloy and resin

Specification	Aluminum Alloy [18]	Epoxy Resin [19]
Density (g/m^3)	2.78	1.17
Ultimate tensile strength (MPa)	434-441	49-71
Tensile modulus (GPa)	73.1	3.35-3.55
Elongation at break (%)	10-15	1.5-2.5
Viscosity (cP) @ 25 °C	N/A	1000-1500

Figure 1. Hygrothermal test chamber

2.3 Flexural Testing

Flexural properties were evaluated using three-point bending tests according to ASTM D 790 [21] using a universal testing machine (STM-150 20kN, Santam Co., Iran). The nominal dimension for the flexural test specimens was 100 × 12.7 mm^2 (length × width). The test was conducted at crosshead speed 1 mm/min, the temperature 24±1 °C, and relative humidity of 36%.

2.4 Charpy Impact Testing

Charpy impact specimens were tested in a pendulum machine (Torsee, MFG. CO., Ltd., Japan) according to ASTM D 6110 [22]. This machine was equipped with a 750 mm length Charpy hammer having a maximum impact velocity of 5.1 m/s. The dimensions of the specimens were 127 × 12.7 mm^2. A V-notch with the angle of 45° and root depth of 2.54 mm was made on one side of the specimens. The experiments were conducted at ambient temperature 23±1 °C and relative humidity of 40%.

3. Results and Discussion

3.1 Moisture Absorption Behavior

Fig. 2 shows the variation of water absorption of FML and GE specimens as a function of the square root of exposure time during hygrothermal aging. Each point presented on the curves is an average from three specimens in gravimetric measurements and the error bars for each value indicate the upper and lower limits of the measured values.

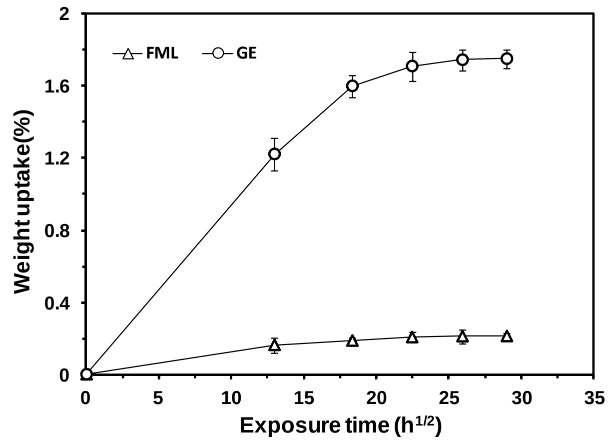

Figure 2. Water absorption curves of FML and GE laminates immersed in water at 90 °C

The water absorption of GE specimens reached the approximate limit of saturation after 5 weeks; thus, after this point, hygrothermal conditioning for all specimens was stopped. The FML and GE specimens show different trends in hygrothermal conditions. While the water absorption gradually increased over the period of time in FML specimens, GE specimens exhibited a sharp linear increase proportional to h$^{1/2}$ in the initial stages of water absorption, and then started to level off at ~1.6% water uptake. It was also observed that the maximum water absorption of GE composites was much more than that of the FMLs. The maximum water absorption recorded for FML and GE specimens was 0.212% and 1.751%, respectively. As expected, the presence of aluminum layers was found to decrease the water absorption of FMLs compared to GE composites. This can be attributed to excellent barrier properties of the aluminum layers that reduced area prone to moisture diffusion.

Moisture absorption is characterized by the migration of low molecular weight substances like water down the concentration gradient, which occurs through diffusion.

In the case of a PMC, the diffusion behavior is related to the orientation and amount of the fibers, properties of the matrix, and the nature of fiber–matrix interface. The water can diffuse between the fibers and the matrix and then capillary flow of water can occur through fiber–matrix interface, which in turn can lead to the weakening or destruction of the bond at the fiber–matrix interface, growth of microcracks or voids, and blistering formation [15]. In PMCs using a simple model which assumes that the moisture transport is proportional to the moisture gradient, is common [23].

When the temperature and humidity around the specimen are constant, the water diffusion inside the composites and overall water uptake at any given time can be can be determined by using Fick's second law, as shown in Eq. (2) [15]:

$$\frac{\partial C}{\partial t} = D\frac{\partial^2 C}{\partial z^2} \tag{2}$$

where C is the local water concentration, D is diffusivity and z the depth of the layer in the sample thickness.

The diffusivity, D, was calculated by assuming that the moisture absorption process followed Fick's law. Hence, D approximated from the initial slope of the percentage water absorption M_t versus $t^{1/2}$ curve [24]:

$$D = \frac{\pi}{16}\left(\frac{h}{M_m}\right)^2\left(\frac{M_{t2} - M_{t1}}{\sqrt{t_2} - \sqrt{t_1}}\right)^2 \tag{3}$$

where h is the thickness of the specimen, t is exposure time and Mm is the maximum water absorption. The results of water absorption and diffusion coefficient values of both FML and GE specimens are summarized in Table 2.

As can be seen in Table 2, the diffusion coefficient of GE laminate is 72% higher than that of the FMLs. As mentioned earlier, this indicates that the water flow in FMLs needs more time to penetrate inside the structure compared to the GE composites.

3.2 Flexural Properties

It is clearly noted in the literature [25, 26] that bending test results can be a very useful tool in the evaluation of the mechanical behavior of adhesively bonded joints. Based on this consideration, three-point bending tests were used to investigate the effect of hygrothermal aging on the mechanical properties of FML and GE specimens. Figs. 3 and 4 show typical stress-strain curves for the FML and GE specimens under dry and hygrothermal conditions. It is evident that the FML specimens, whether exposed to aging or not, exhibited an initial linear elastic response to the applied bending load; but when loading was increased, FMLs showed a similar pattern as aluminum alloys. The specimens exhibited a significant deviation from linearity associated with extended plastic deformation up to the ultimate stress. In contrast, GE/Dry specimens exhibited a linear deformation as well as a brittle response under the bending load before reaching ultimate stress. The stress/strain curve for the GE/Aged followed a different pattern which was nearly linear at the initial step of loading but then gradually exhibited a lower slope in relation to the initial linear step. This was associated with a slight increase in strain and relative softness compared to dry specimens.

Table 2. Hygrothermal aging test results

Material	Maximum moisture Absorption (%)	Diffusion coefficient (10^{-7} mm²/s)
GE	1.751	3.52
FML	0.212	0.97

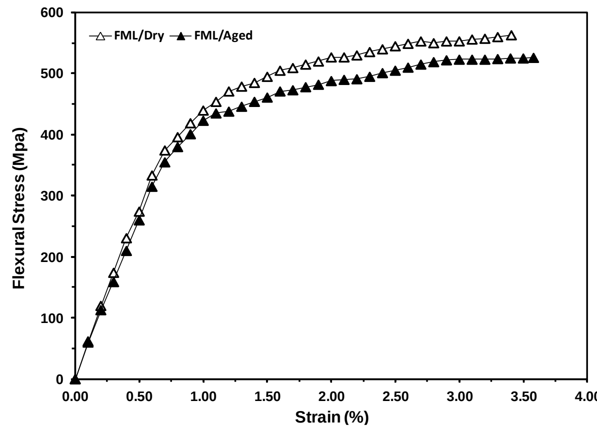

Figure 3. Typical flexural stress-strain curves for FML laminates before and after exposure to hygrothermal aging

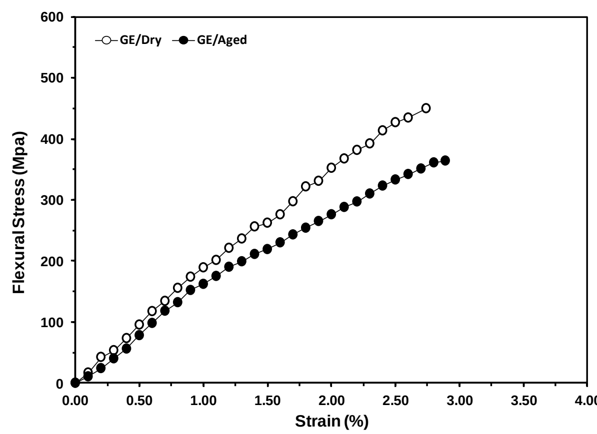

Figure 4. Typical flexural stress-strain curves for GE laminates before and after exposure to hygrothermal aging

Tables 3 and 4 summarize the average flexural properties of FML and GE laminates, which were derived from the specimens in both dry and aged conditions.

Table 3. Three-point bending test results for FML specimens before and after exposure to the hygrothermal aging

Property	FML/Dry	FML/Aged
Flexural stiffness (GPa)	49.52	48.55
Ultimate flexural strength (MPa)	563.00	526.01
Strain at ultimate strength (%)	3.41	3.58
Specific breaking energy (J/cm³)	15.22	15.13
Density (g/cm³)	2.47	2.47
Specific flexural stiffness (GPa/(g/cm³))	20.05	19.65
Specific flexural strength (MPa/(g/cm³))	227.94	212.96

Table 4. Three-point bending test results for GE specimens before and after exposure to the hygrothermal aging

Property	GE/Dry	GE/Aged
Flexural stiffness (GPa)	18.91	16.26
Ultimate flexural strength (MPa)	449.75	364.13
Strain at ultimate strength (%)	2.74	2.89
Specific breaking energy (J/cm^3)	6.62	5.88
Density (g/cm^3)	1.92	1.95
Specific flexural stiffness (GPa/(g/cm^3))	9.85	8.34
Specific flexural strength (MPa/(g/cm^3))	234.24	186.73

Normalized flexural properties degradation due to hygrothermal aging is presented in Figs. 5. As can be observed from Fig. 5, the water absorption has a negative influence on flexural properties of both sets of specimens. However, this effect is more prominent in the GE laminates. This reduction in the flexural properties can be attributed to diffusion of water into the whole of the material structure and subsequent possible plasticization of the polymer matrix. Because of the structural heterogeneity of PMCs, the water absorption process is rather complex. The non-uniform concentration of water in hygrothermal conditions can generate stresses in a PMC laminate even at constant temperatures. These stresses can produce several damages such as matrix cracking, microvoid generation, outer-ply delamination, or surface blistering during rapid heating [23]. It is noted that matrix cracks often initiate in surface plies and progress deeply in the laminate over time [17, 23, 27]. The micro-cracks which form in the PMC laminate subsequently promote new pathways for moisture uptake or fiber–matrix debonding which can lead to a drop in flexural values [28].

As shown in Fig. 5, the degradation of flexural strength and stiffness of GE/Aged laminates compared to dry specimens were 18.93% and 14.01%, respectively. In contrast most of the flexural properties of the FMLs did not show a significant reduction after hygrothermal aging. The normalized values showed that the flexural strength and stiffness of FML/Aged specimens were decreased by only 6.57% and 1.95%, respectively, which is negligible compared to GE composites.

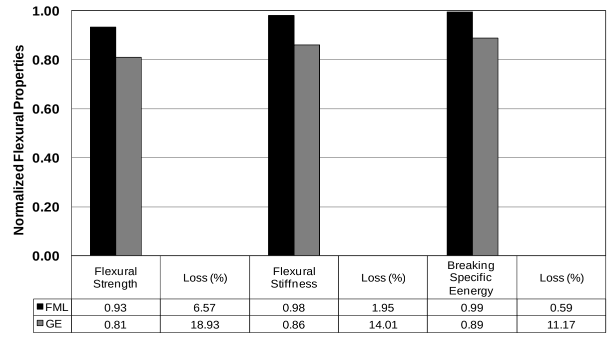

Figure 5. Degradation of flexural properties of FML and GE laminates after exposure to the hygrothermal aging

The conclusion drawn here is that the aluminum layers served as barriers to water penetration during the hygrothermal conditioning and only a small amount of water diffused into the full thickness of material. Because of this, the values of specific breaking energy of FML/Dry and GE/Dry laminates after hygrothermal aging decreased by 0.59% and 11.17%, respectively. In this study, the specific breaking energy which represents the flexural toughness of materials was calculated by the total area under the flexural stress-strain curve till the load dropped. It is worth noting, that although the flexural strength and stiffness values of FML/Aged specimens were decreased after hygrothermal aging, increasing the ductility of material due to plasticization effect caused by water absorption has previously led to no significant change in specific breaking energy values. Buehler and Seferis [29] reported an increase in fracture toughness values of carbon/epoxy composites after exposure to water for 1200 h. Plasticization effect and increased fiber bridging were reported to be the reasons of the enhancement in fracture toughness. On the other hand, in the case of GE composites, even though the conditions of hygrothermal aging were the same with FMLs, the values of specific breaking energy dropped dramatically. A possible explanation is that despite the increase of 5.47% ultimate stain due to ductility caused by hygrothermal aging, the weakening effects of hygrothermal aging on flexural strength and stiffness values dominated over the substantial improvement of ductility in the aged GE composites. Consequently, a reduction of specific breaking energy resulted.

To understand the nature of the failure mechanism which causes the differences between the two material types, the morphology of fracture surfaces of specimens was carefully examined. The fractographs of FML and GE surfaces before and after hygrothermal aging are shown in Figs. 6-9. As observed in Figs. 6 and 7, the fracture appearance of both GE/Dry and GE/Aged composites is somewhat similar to each other. The fracture morphology of GE/Dry composites was relatively smooth and both the matrix cracking and the fiber breakage which are characteristics of a brittle fracture had occurred. However, a rougher fracture surface and increased fiber pull-out were observed for GE/Aged composites, which is slightly different from the typical brittle failure observed with GE/Dry composites. A more ductile behavior in GE/Aged composites due to water absorption is implied.

For FML specimens, the fracture behavior of the material before and after hygrothermal aging is significantly different. Closer examination of fracture surfaces revealed that almost no delamination at the composite/metal interface of FML/Dry took place, indicating an excellent interfacial bonding between constituents of FMLs. The FML/Dry specimens exhibited a high flexural strain during bending test and until the ultimate flexural strain of 3.41% only the aluminum sheets in the tension zone were completely fractured (Fig. 8). The composite layer considerably failed throughout the thickness of the FML/Dry specimens with matrix cracking and broken fibers near the rupture site.

However, for the FML/Aged specimens, the specimens failed with an extended delamination in composite/metal interface, as shown in Fig. 9. In this case, no rupturing or tearing was observed in aluminum layers of FMLs. A limited amount of fiber bridging was also observed in the composite/metal interface, which indicated the lower values of interfacial bonding compared to FML/Dry specimens. This can be attributed to the plasticization effect caused by water absorption which has been associated with a reduction in interfacial strength of composite/metal interface which leads to the composite/metal interface being prone to delamination.

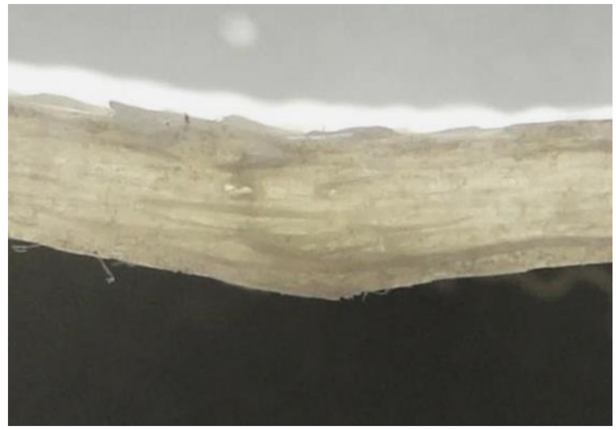

Figure 6. Photographs of failure modes following flexural testing in the GE/Dry specimens

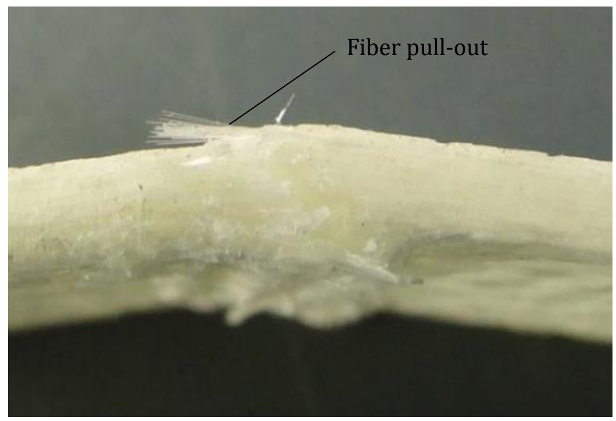

Figure 7. Photographs of failure modes following flexural testing in the GE/Aged specimens

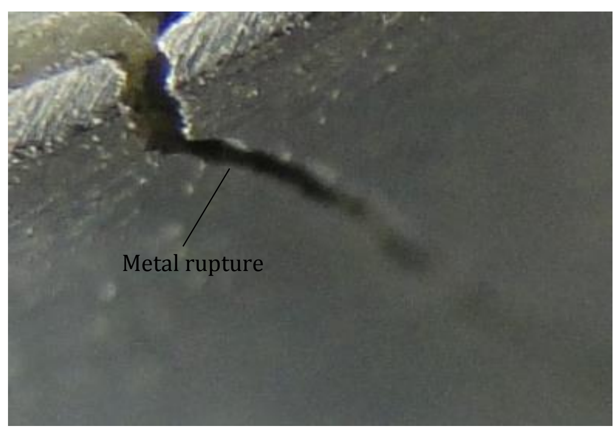

Figure 8. Photographs of failure modes following flexural testing in the FML/Dry specimens

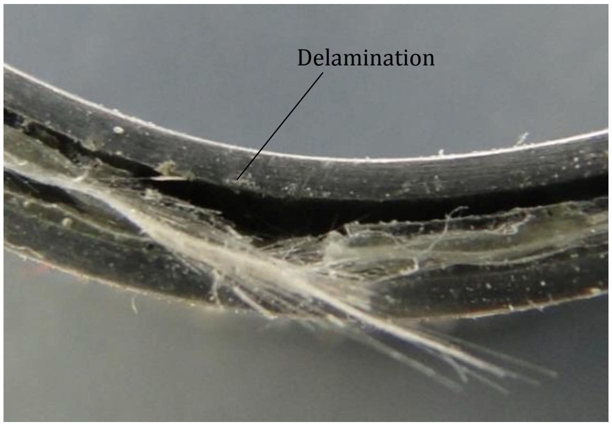

Figure 9. Photographs of failure modes following flexural testing in the FML/Aged specimens

3.3 Impact Properties

Table 5 shows the measured impact strength of FML and GE specimens before and after hygrothermal aging. It is noted that the impact properties of both types of conditioned materials were not affected as much as the flexural properties were by hygrothermal aging. The results also indicate that the impact strength and flexural properties of the aged GE composites were both decreased more than those of the FMLs; . The impact strength of the FML/Aged and GE/Aged composites decreased by 3.76% and 9.53% compared to the dry specimens, respectively.

This different impact to material properties from hygrothermal conditioning may be explained by an increase in the ductility of the matrix. Water absorption can increase the ductility of polymers through plasticization. However, this ductility is restricted by the confining factors such as stiff and brittle fibers. On the other hand, water may weaken the fiber/matrix interfacial bond and induce osmotic pressures which would augment the activation of crack growth, thereby decreasing the toughness of PMCs [30]. Consequently, these contradictory effects may cancel each other, leading to the degraded toughness of PMCs.

Table 5. Summary of the Charpy impact strength values for FML specimens before and after exposure to the hygrothermal aging

Material	Charpy impact Strength (kJ/m^2)	Specific Charpy impact strength (J.m/kg)
FML/Dry	941.41	381.13
FML/Aged	906.02	362.76
GE/Dry	396.55	206.53
GE/Aged	358.76	183.97

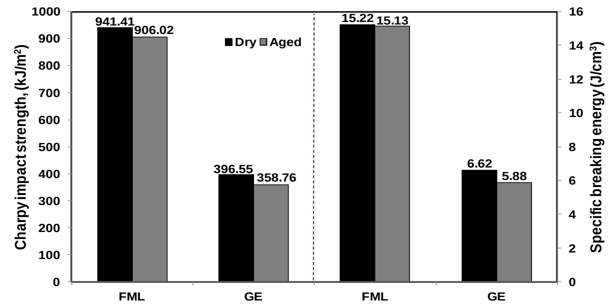

Figure 14. Charpy impact strength and specific breaking energy of the FMLs and GE composites before and hygrothermal aging

By comparing the values of specific breaking energy and the Charpy impact strength of the specimens (Fig. 10), it is clear that a good correlation between impact strength and specific breaking energy is governed, whether before or after hygrothermal aging.

Figs. 11-14 illustrate fracture photographs of specimens after Charpy impact testing. The GE/Dry composite fracture appearance shows the brittle nature of the material characterized by severe glass fibers breaking predominantly in the failure plane, and limited fiber pullout as shown in Fig. 11. As can be seen, significant matrix cracking and extensive fiber breakage occurred at the location of impact on the specimen and no delamination was apparent. Note that the GE/Dry specimens fractured into two completely separated pieces.

In contrast, different failure patterns were observed following Charpy impact tests on GE/Aged specimens as shown in Fig. 12. In this figure GE/Aged specimens show a ductile behavior. In fact, as clearly shown in Fig. 12, no obvious brittle damage was observed in the aged GE/Aged specimens. Thus, the change of material behavior from brittle to ductile can be ascribed to moisture and temperature [31]. Once water infused the interfaces of the composites, the water acted as a plasticizer, spacing the polymer chains apart. Therefore adhesion between the two phases was destroyed and PMC laminates probably became softer [32]. Kinking, which is the predominant form of compression failure, can be observed in the fractured surfaces of GE/Aged specimens (Fig. 12). This failure mode is often associated with an irregular and stepped fracture surface as shown in Fig. 12. In this failure mode, the fibers are rotated by a large amount and the matrix is usually subjected to a large shear deformation [33].

Examination of the fracture surfaces of specimens revealed considerable plastic deformation, rupture of aluminum layers, and fracture of the composite plies in FML/Dry and FML/Aged (Figs. 13, 14). However, plastic deformation and delamination were more significant in FML/Aged specimens. Extended delamination in FML/Aged specimens initiated close to the point of impact and progressively failed toward the specimens end in the presence of a peeling force (Fig. 14). The delamination allows FMLs to deform and fracture in a more efficient membrane-like way, and it contributes to total energy absorption [34, 35]. In FML/Aged specimens, an extended region of delamination was evident in the vicinity of the impact zone, indicating that the aluminum/composite interface most likely had been weakened because of water absorption. Moreover, it appears that hygrothermal mismatching of the aluminum layers and composite plies played an important role in the formation of possible interfacial cracks as the origins of delamination in FML/Aged specimens.

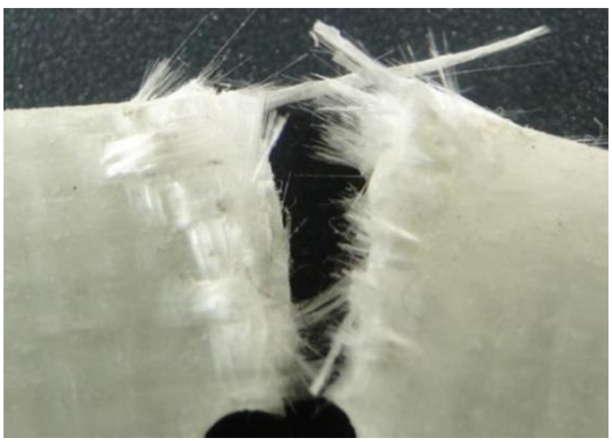

Figure 11. Photographs of failure modes following impact testing in the GE/Dry specimens

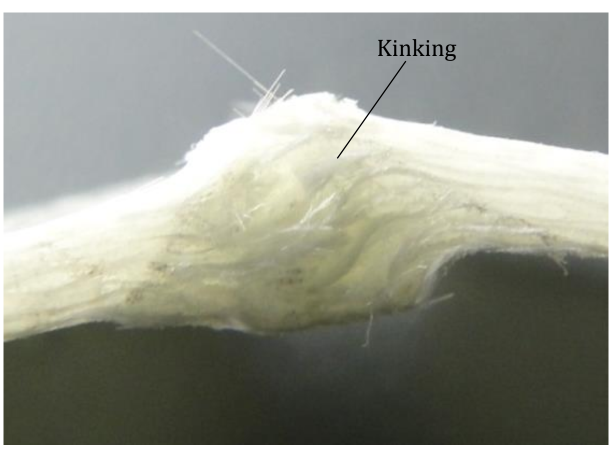

Figure 12. Photographs of failure modes following impact testing in the GE/Aged specimens

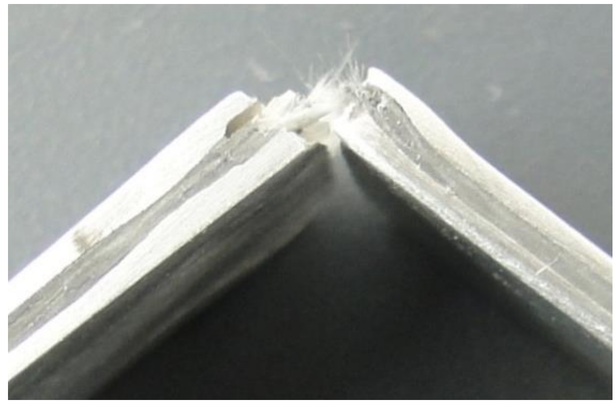

Figure 13. Photographs of failure modes following impact testing in the FML/Dry specimens

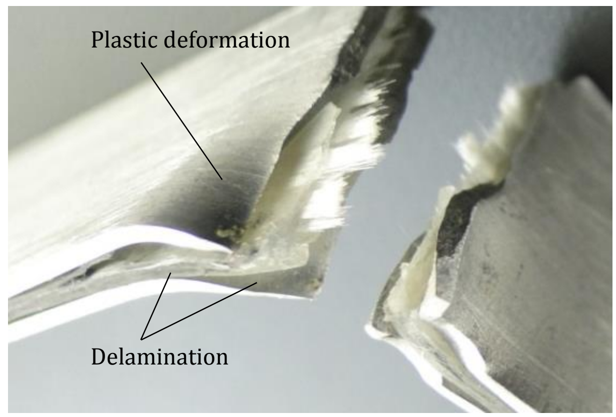

Figure 14. Photographs of failure modes following impact testing in the FML/Aged specimens

4. Conclusions

In this paper, the effects of hygrothermal aging on the flexural and impact behaviors of glass/epoxy (GE) composites and fiber metal laminates (FMLs) were investigated, providing valuable information about the durability of these materials in harsh environments. From the obtained results, the following conclusions are drawn:

1. The water absorption of GE composites during hygrothermal aging quickly increases at first, then gradually the rate of increase slows down. For FML specimens the water absorption gradually increased over the total period of time. The final level of water absorption recorded for FML and GE specimens were 0.212% and 1.751%, respectively. Lower water absorption in FMLs is attributed to the excellent protection by aluminium barrier layers against water penetration.

2. Hygrothermal conditioning for 5 weeks resulted in a considerable reduction of 18.93% in flexural strength and 14.01% in flexural stiffness of GE specimens. As expected, the same conditioning exhibited a negligible decrease in flexural strength and stiffness of FML specimens: 6.57% and 1.95%, respec-

tively. The impact strength of the hygrothermal aged FML and GE composites decreased by 3.76% and 9.53%, respectively. Hence, the impact performance of both FML and GE composites suffers less than the flexural performance under hygrothermal conditions. The increased ductility of polymeric matrix because of water absorption is responsible for reduced sensitivity of impact properties to hygrothermal aging.

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