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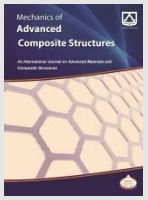

Environmental effects on mechanical properties of glass/epoxy and fiber metal laminates, Part II: Isothermal aging

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ABSTRACT

The aim of this study is to investigate effects of isothermal aging on mechanical properties of fiber metal laminates (FMLs) and glass/epoxy composites. For this purpose, both materials were fabricated using the wet lay-up manufacturing technique under vacuum pressure. Both the glass/epoxy composites and the FML specimens were then subjected to isothermal aging (130°C, dried air) for up to 5 weeks. After the isothermal aging, the specimens' weight loss, caused by thermo-oxidative conditioning, was evaluated. Bending and Charpy impact tests were conducted on both the unaged and aged specimens to examine the isothermal conditioning's effect on the mechanical properties of studied materials. Experimental results revealed that isothermal aging severely affected impact strength in the form of embrittlement and reduced ductility. However, no significant reduction was found in the flexural stiffness of isothermally aged FML and glass/epoxy specimens.

1. Introduction

Advanced materials are essential for product development in many fields, including aerospace, the military, sporting goods design and the automotive industry. These advanced materials must maintain high performance and unique properties [1-3]. A most promising engineering materials is polymer matrix composite (PMC), which is used in aircraft and marine structures as primary load carrying members as well as secondary members [4]. It is well known that PMCs offer many advantages compared with conventional metallic materials, because of PMC's high strength and stiffness-to-weight ratio, superior fatigue performance, and corrosion resistance [5]. In aeronautical applications, structural parts made of composite materials are usually exposed to harsh conditions for extended durations while in service. These harsh environments can be thermal influences, load cycling, ultraviolet radiation, and temperature variations ranging from -50°C to 130°C in aircraft service conditions. Unfortunately, in spite of their numerous advantages, PMCs are very vulnerable to these environmental conditions. Therefore, environmental aging is garnering a great deal of attention regarding long-term performance of composite materials during the intended service life.

One widely used strategy for environmental protection of PMC structures within the aerospace industry is application of protective coatings. However, because of the gradual erosion of coating or crack propagation in severe friction applications, as well as formation of microcracks/scratches, use of these protective coatings is not considered a permanent solution.

The idea of combining aluminum alloys and PMCs to create a hybrid structural material was born in the 1970s to overcome most of the disadvantages of both materials, such as high sensitivity of PMCs to environmental conditions and poor fatigue properties of aluminum alloys [6]. These hybrid materials were primarily developed at Delft University of Technology in the early 1980s. Fiber metal laminates (FMLs) was a new category of hybrid composite materials consisting of stacked plies of PMC laminates and thin metal sheets. It has been known for some time that FMLs can provide improved weight reduction, fatigue performance, and damage–tolerance characteristics compared with monolithic aluminum alloys [7, 8].

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Recently, FMLs have been introduced as structural hybrid materials for many potential applications in aircraft structures. One such application can be found on the upper fuselage skin structure of the Airbus A380 [9]. Most previous studies have focused on FMLs' primary advantages, such as impact resistance and fatigue tolerance [10, 11]. Notably, other interesting aspects of FMLs, like their durability against high-temperature and other environmental factors has been less investigated. Because of the lay-up pattern of FMLs, the thermo-oxidative process is quite distinct from that of PMCs. Only the outer aluminum layers of FMLs are exposed to environmental conditions.

It has been found that isothermal aging can decrease the mechanical properties of PMCs due to several mechanisms, including degradation of the fiber-resin interface and oxidation of the matrix. It also has been proven that the mechanical properties of PMCs decrease with the passage of time and isothermal aging.

Thermal degradation due to elevated temperature exposure is also another important issue when considering composites for long-term applications. Elevated temperatures can accelerate the oxygen diffusion rate and oxidation reactions that result in several degradation mechanisms, including weight loss, surface degradation, the creation of microcracks and resin-fiber debonding [12-14]. Ozcelik et al. [15] studied the effect of thermo-oxidative aging on aerospace-grade graphite/epoxy composite laminates. Results showed that this material was highly stable at 100°C, because no noticeable weight loss or strength reduction was observed during 5,000 hours (h) of aging at this temperature. However, after 5,000 h, the laminates experienced 0.81%, 2.31%, and 5.84% weight loss coupled with 10.87%, 33.07%, and 49.86% short beam strength reduction at 150°C, 175°C, and 200°C, respectively.

Lafarie-Frenot et al. [16] investigated the damage processes caused by isothermal aging in carbon/epoxy laminates. They showed that isothermal aging results in oxidation of the matrix on the surfaces directly exposed to the atmosphere and induces visible matrix shrinkage and fiber–matrix debonding on the sample surfaces. Due to elevated temperatures in FMLs, several damage mechanisms may occur, including matrix microcracking, interfacial debonding in the fiber-matrix interface and delamination between composite and metal plies resulting from prevented thermal expansions of the plies. Unfortunately, there is little in the literature despite the significant effect of isothermal aging on the mechanical properties of FMLs. Zhu et al. [17] conducted studies and compared the decomposition behavior of epoxy resins Br-127 and FM 94 in FML, which provided valuable information about the thermal delamination of FMLs. The results indicated that the decomposition temperature of Br-127 and FM 94 was 188°C and 255°C, respectively. It was also concluded that an oxidative atmosphere is more conducive to epoxy resin decomposition compared with inert atmosphere.

In the present study, a series of experimental tests were carried out to evaluate the effect of isothermal aging on the mechanical behaviors of FMLs and conventional glass/epoxy composites. Thermo-oxidative aging (130°C, dried air) for a 5-week duration was used as an accelerated degradation test. Following the thermo-oxidative conditioning, weight loss analysis, impact resistance, and flexural properties of thermally conditioned and pristine specimens were measured experimentally to provide comparative data for understanding FML and PMC behavior under thermo-oxidative conditions.

2. Experimental Procedure

2.1 Materials

Two types of materials were prepared in this study. The first was FML 2/1 (2 layers of metal sheet and 1 layer of composite) consisting of 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. The second material was E-glass/epoxy (GE) composite, consisting of 10 plies of E-glass fabrics and epoxy resin.

2.2 Manufacturing Process

For manufacturing the FMLs, square sheets of aluminum alloy 2024-T3 with dimensions of 400 mm × 400 mm were first abraded using Al2O3-sandpaper and then washed with acetone and degreased using methyl ethyl ketone (MEK) for the duration of 10 minutes (min) at a temperature of 50°C. After dipping the degreased aluminum (Al) sheets in a water tray and rinsing them, the Al sheets were etched with alkaline through immersion in a 5% NaOH solution for the duration of 10 min at room temperature. The Al sheets then were rinsed again, thoroughly with hot water, to remove any traces of NaOH. To ensure thorough cleaning of NaOH particles, acid etching of the Al sheets was carried out through immersion in a solution of ferric sulphate and sulphuric acid, followed by rinsing in hot distilled water and air-drying. Chromate conversion treatment was performed by immersing the Al sheets in a liquid coating bath composed of CrO_3, $Na_2Cr_2O_7$, and NaF at room temperature [18]. The final step in the Al sheets' surface treatment was a water rinse followed by air drying. The FMLs and GE laminates used in this study were manufactured by stacking the specified number of Al sheets and GE

layers in a simple mold. In this regard, the top face of the first layer placed in the mold (Al sheet in FMLs and E-Glass fabric in GE laminates) was coated uniformly with a layer of epoxy resin that had been premixed with its hardener. Similarly, other layers were sequentially impregnated with resin and stacked in the mold. After the wet lay-up process, the FMLs and GE laminates were cured under a vacuum of -60 kPa for one day at room temperature. To minimize the effect of thermal expansion mismatch among the various laminate components, a lower maximum temperature was selected to post-cure the laminates. Therefore, the set was put into the autoclave for curing under a pressure of 400 kPa and a temperature of 100°C for 4 h, as recommended by the resin manufacturer.

2.3 Specimen Preparation

Specimens were cut from FML and GE plates in desired dimensions using a water-jet cutting machine according to ASTM standard recommendations. Each specimen was polished using a milling machine and soft emery sandpaper; potential surface cracks and local material inhomogeneities were removed through this method. After being cut to the specified dimensions, the specimens were coded and their weights were measured accurately by a digital balance. Specimens in this study were divided into 12 groups, based on their constituents, environmental conditioning, and mechanical-testing types. F and I symbols were used as specimen codes to represent flexural or impact—the two types of mechanical testing conducted on the specimens. In addition, the T symbol indicated that the sample had undergone isothermal aging. For example, FML-impact specimens exposed to isothermal aging were coded as FML/TI, and GE flexural specimens exposed to isothermal aging were coded as GE/TF. It should be noted that these are not the T symbol used in other studies to identify virgin specimens that were not exposed to any environmental conditioning.

2.4 Isothermal Aging

Because the temperature of structural parts of supersonic aircrafts can reach 130°C based on fly stages [16], the temperature of 130°C was selected as the aging temperature for the materials considered in this study. Therefore, isothermal aging was conducted on FMLs and GE specimens by placing them in an air-circulating oven (Model Heraeus, Germany) at a temperature of 130°C, as shown in Fig. 1. After each week of aging, certain specimens of each material were removed from the oven for weighing on a digital balance with a 0.0001 g resolution.

Figure 1. Circulating air oven

2.5 Flexural Testing

Flexural properties were measured using three-point bending tests according to ASTM D 790, using a universal testing machine (STM-150 20kN, Santam Engineering Design Co., Iran). The nominal dimension for FML and GE flexural test specimens was 100 × 12.7 mm² (length × width). The test was conducted at crosshead speed at 1 mm/min. Ultimate flexural strength (σ_f), flexural stiffness (E_f), and strain at ultimate strength (ε_f) are estimated as follows [19, 20]:

σ_f: Determined from the maximum load (P_{max}) of bending test:

$$\sigma_f = \frac{3P_{max}L}{2bh^2} \quad (1)$$

where L is the support span length in the three-point bending test, b and h are the width and thickness of the specimen, respectively.

E_f: Flexural stiffness is determined from the slope (m) of the initial region of the load-deflection curve:

$$E_f = \frac{mL^3}{4bh^3} \quad (2)$$

ε_f: Strain at maximum strength is calculated using the maximum deflection (D) of the center of the beam as follows:

$$\varepsilon_f = \frac{6Dh}{L^2} \quad (3)$$

2.6 Charpy Impact Testing

Charpy impact specimens were tested in a pendulum machine (Torsee, MFG. CO., Ltd., Japan) according to ASTM D 6110 [21]. This machine was equipped with a 750 mm-long Charpy hammer with a maximum impact velocity of 5.1 m/s. The dimensions of the specimens were 127 × 12.7 mm². A 45°-angle V-notch

with a root depth of 2.54 mm was made on one side of the specimens. Experiments were conducted at an ambient temperature of 24±1°C and relative humidity of 38%. A schematic Charpy impact specimen is shown in Fig. 2.

3. Results and Discussion

3.1 Weight Loss Due to Isothermal Aging

Fig. 3 shows the percentage of weight loss experienced by the FML and GE specimens against the number of hours of isothermal aging. Each point presented on the figures is an average value obtained by weighing the three specimens.

Fig. 3 indicates that FML specimens demonstrated less than 0.12% weight loss, which remained relatively constant after 840 h of isothermal aging. This slight, finite weight loss was possibly due to degassing and/or desorption of moisture during elevated temperature exposure, which conformed well to the shielding effect of the FML Al layers [15].

Unlike the FML specimens, the isothermal aging induced more significant weight loss in GE composites. Meanwhile, a rapid preliminary mass loss of up to 500 h aging can also be observed on the curve obtained for GE specimens. However, after approximately 500 h, the mass of the isothermally aged GE specimens decreased by a lower rate and gradually moved toward stability. When PMCs are subjected to isothermal aging under oxidizing atmospheres, high temperatures can possibly lead to thermo-oxidation and/or thermolysis of the matrix. The phenomenon of thermo-oxidation affects primarily the external surfaces of the composites and leads to weight loss in the specimens [16, 22]. Thus, as expected, because of having a larger surface area and a generally higher matrix content, the GE specimens subjected to elevated temperatures are more prone to weight loss than are FMLs. In spite of all this, it is notable that none of the isothermally aged GE and FML specimens showed a weight variation greater than 0.53% and 0.11%, respectively.

3.2 Flexural Properties

Typical flexural stress-strain curves for the isothermally aged FML and GE specimens, as well as virgin specimens, are presented in Figs. 4 and 5. It is interesting to note that the general flexural behavior of isothermally aged FML and GE specimens was somewhat similar. For example, FML/F specimens, like isothermally aged specimens, initially demonstrated a linear elastic behavior, but with an increasing bending load, plastic deformation was observed up to the ultimate stress. Notably, the flexural behavior of both isothermally aged and virgin FML specimens in the linear elastic region is almost identical.

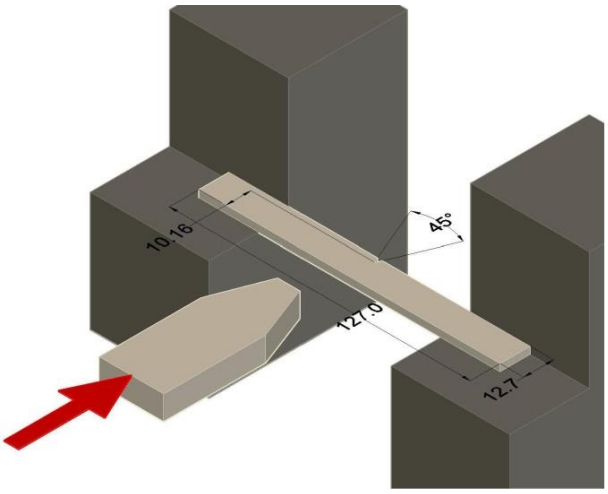


Figure 2. Schematic Charpy test specimen prepared according to ASTM D 6110 (All dimensions are in mm)

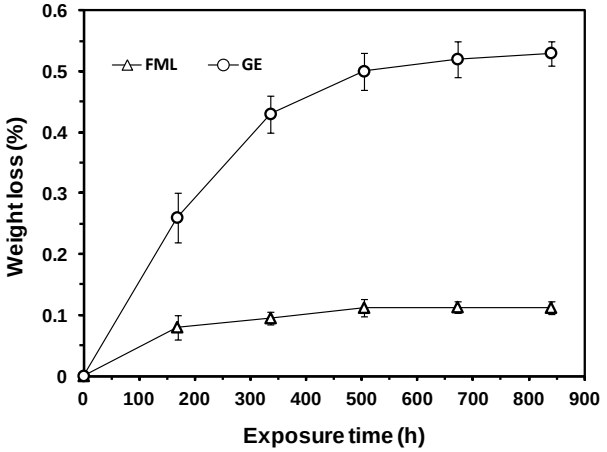


Figure 3. Percentage weight loss of GE and FML specimens subjected to isothermal aging at 130°C

However, the results indicate that the FML/TF specimens did not exhibit a plateau region in their stress-strain curve, which can be attributed to the progressive failure of the Al–composite interface that continued delaminating during loading. On the other hand, a relatively similar trend for FML specimens in the linear elastic region was observed for GE specimens. Both the GE/F and GE/TF specimens manifested similar behavior, showing a steady increase in linear stress with increasing strain, progressing up to quite an instantaneous failure.

The flexural properties of each material, which were derived from average values of the three replicate tests for virgin and isothermally aged specimens, is presented in Tables 1 and 2.

To further substantiate the effect of isothermal aging, Fig. 6 shows the normalized flexural properties of isothermally aged FML and GE compared to the as-received condition.

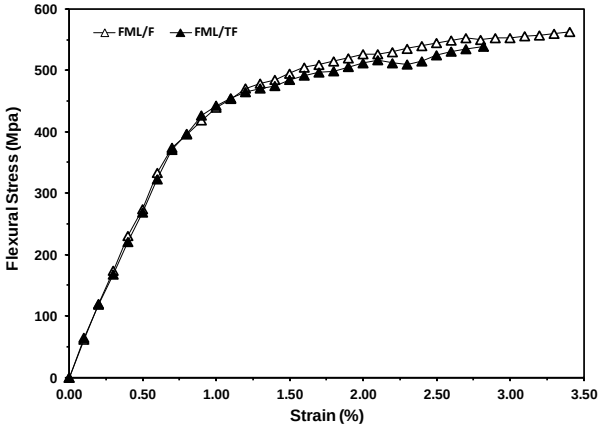


Figure 4. Typical flexural stress-strain curves for FML laminates before and after exposure to isothermal aging

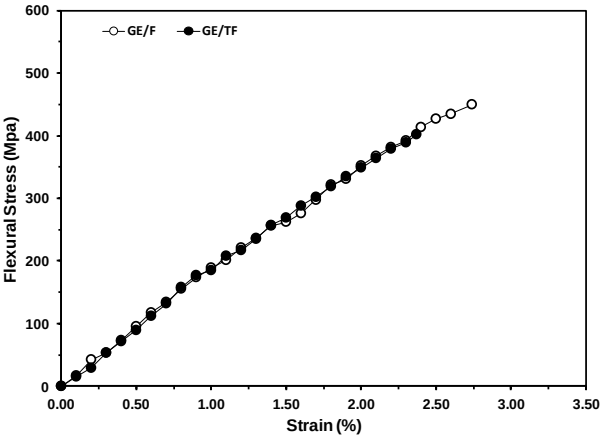


Figure 5. Typical flexural stress-strain curves for GE laminates before and after exposure to isothermal aging

Table 1. Three-point bending test results for FML specimens before and after exposure to the isothermal aging

Property	FML/F	FML/TF
Flexural stiffness (GPa)	49.52	49.44
Ultimate flexural strength (MPa)	563.00	539.24
Strain at ultimate strength (%)	3.41	2.82
Specific breaking energy (J/cm^3)	15.22	12.79
Density (g/cm^3)	2.47	2.47
Specific flexural stiffness (GPa/(g/cm^3))	20.05	20.02
Specific flexural strength (MPa/(g/cm^3))	227.94	218.31

Table 2. Three-point bending test results for GE specimens before and after exposure to isothermal aging

Property	GE/F	GE/TF
Flexural stiffness (GPa)	18.91	18.04
Ultimate flexural strength (MPa)	449.75	402.06
Strain at ultimate strength (%)	2.74	2.37
Specific breaking energy (J/cm^3)	6.62	5.01
Density (g/cm^3)	1.92	1.93
Specific flexural stiffness (GPa/(g/cm^3))	9.85	9.35
Specific flexural strength (MPa/(g/cm^3))	234.24	208.32

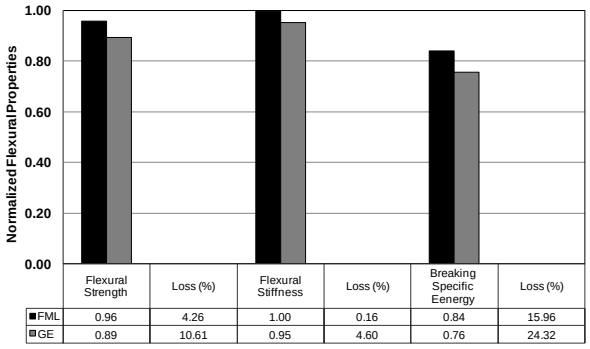


Figure 6. Degradation of flexural properties of FML and GE laminates after exposure to the isothermal aging

It can be seen in Fig. 6 that the flexural stiffness of GE/TF and FML/TF specimens decreased by 4.60% and 0.16%, respectively, compared to the virgin specimens. Therefore, it seems that flexural stiffness of the FML and GE laminates was nearly independent of isothermal aging. In contrast, the same isothermal condition was very different in the case of flexural strength. Fig. 6 reveals that isothermal aging negatively affected the flexural strength of both the FML and the GE specimens. A significant drop of 10.61% and 4.26% in flexural strength of GE/TF and FML/TF, respectively, can be observed. When PMCs are exposed to thermal aging, because of higher coefficients of thermal expansion of the matrix compared to the fibers, matrix shrinkage can occur. Following this, it seems likely that the presence of high strain gradients in the matrix adjacent to the fibers leads to induce high thermal stresses. This situation can be conducive to debonding of the fibers and the matrix and/or to crack initiation [16]. The fiber/matrix interface also becomes more prone to aggressive reactions from exposure to high temperatures, which can lead to additional fibers and matrix degradation [23]. Meanwhile, degassing of water vapor and organic volatiles may increase the formation of micro and macro cracks in the matrix [24]. These cracks can cause significant reductions in the strength properties of composites to even below the glass transition temperature(Tg). Moreover, some chemical reactions, such as cross-linking and chain scission, may occur during isothermal aging [25]. The best explanation for deterioration in flexural strength of GE/TF composites, as well as relative stability in flexural stiffness, may be attributed to the post-curing effect and degradation of the matrix by causing increased cross-linking or chain scission, respectively [26]. Isothermal aging under oxidizing atmospheres can create additional cross-linking, causing the polymer matrix to become more brittle and stiff. On the other hand, chain scission breaks the polymer chains, resulting in microcracks.

Chain scission begins on exposed composite surfaces and moves inward; thus, it can affect surface properties like flexural strength [27]. Therefore, despite the enhancement of stiffness due to further cross-linking reactions, the weakening effect of chain scission reactions might dominate over the stiffening effect of post-curing and, consequently, lead to a reduction in flexural strength in the GE/TF specimens.

In FML specimens, as a result of the mismatch in coefficients of thermal expansion and higher stiffness of Al layers relative to composite plies, the Al layers want to shrink more than the composite plies as they cooling down. This creates tensile residual stresses in the Al layers and compressive residual stresses in the composite plies [28]. Thus, elevated temperatures have a favorable effect by reversing the residual stresses and decreasing the tensile residual stresses in the Al layers. However, a slight degradation of flexural properties of FML/TF specimens revealed that, similar to the weakening effect of chain-scission reactions, it has overcome the further cross-linking that occurred in the matrix as well as the stress-restoring effect caused by elevated temperatures.

Figs. 7 and 8 show the fracture surface of the FML/TF and GE/TF composites following flexural testing. Both matrix cracking and fiber breakage, as well as a reduction in deformation that is characteristic of brittle fracture, were observed in fracture surfaces of GE/TF composites. The GE/TF composite brittleness can emerge as a result of densification of the material during isothermal aging [29]. In the case of the FML/TF specimens, closer examination of the fracture appearance revealed that no rupture or tearing occurred in AL layers of the FML/TF specimens. As shown in Fig. 8, the failure mode of the FML/TF specimens was mostly due to delamination buckling away from the loading points, with some fiber bridging involvement.

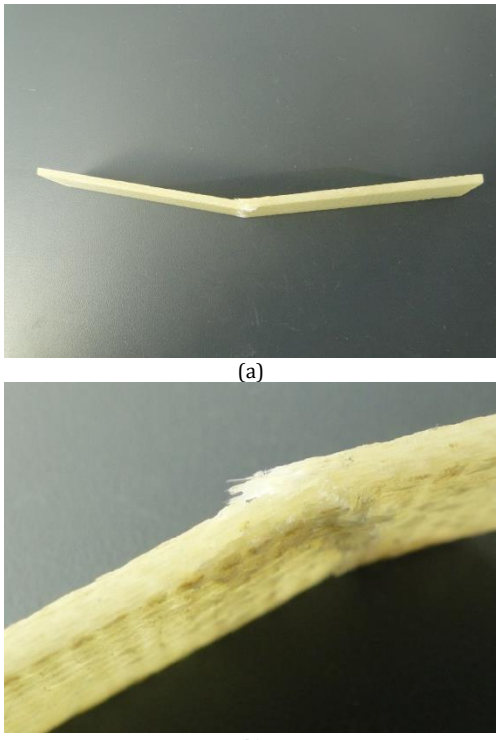
In addition, broken glass fibers inside the composite layer of the FML/TF adjacent to delamination position were also apparent. This specific type of buckling is usually associated with an extended delamination in the composite–metal interface as well as the Al layer buckling. During bending, the inside of the laminate is compressed.

When compression stresses in the inside layer increase, notwithstanding the adhesion and the stabilizing curvature of the bend, the inside layer buckles as the peel stresses on the composite layer exceed their ultimate values [30].

Greenhalgh et al. [31], in their research, emphasized that the amount of destruction of composite layers in the compression zone is larger under specific flexural loading conditions such as buckling.

3.3 Impact properties

In order to determine the influence of elevated temperatures on the impact properties of FMLs and composite materials, isothermal aging was conducted on FMLs and GE specimens at a temperature of 130°C. The results of Charpy impact tests on FMLs and GE composites before and after isothermal aging are shown in Table 3. It is noteworthy that the impact properties of both material types were considerably affected by isothermal aging. The results also indicated that the impact strength of the isothermally aged GE composites was decreased by a greater amount than were the FMLs. This, in some ways, was similar to the observed trend in the flexural properties results. As is shown in Table 3, the GE/TI composites showed the Charpy impact strength of 294.86 kJ/m^2, which is about 25% lower than that of the virgin specimens. This value for FML/TI was 806.02 kJ/m^2, which is equivalent to 85.61% of the virgin specimen strength.



(a)

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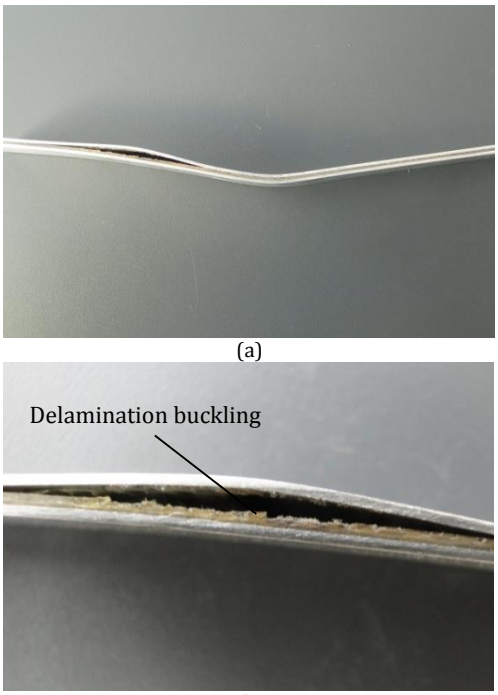
Figure 7. (a) Photographs of failure modes following flexural testing in the GE/TF specimens, (b) magnified at fracture surface



(a)

(b)

Figure 8. (a) Photographs of failure modes following flexural testing in the FML/TF specimens, (b) magnified at fracture surface

Table 3. Charpy impact strength values for FML specimens before and ***after*** exposure to isothermal aging

Material	Charpy impact Strength (kJ/m²)	Specific Charpy impact strength (J.m/kg)
FML/I	941.41	381.13
FML/TI (after)	**806.02 (-14.39%)**	326.32
GE/I	396.55	206.53
GE/TI (after)	**294.86 (-25.7%)**	152.77

The decrease in impact strength associated with a reduction in specimen deformation under impact test can be explained by several degradation phenomena caused by thermal aging, which is discussed in detail earlier in this report.

The weakening of the fiber–matrix interface, deterioration of composite–Al layer interfacial bond, transverse matrix cracks in the external surface, and oxidation of the matrix are considered plausible reasons for the degradation in impact properties of FMLs and GE composites. Furthermore, since the resin used in this study was brittle, and deformation was decreased after thermal aging, the embrittlement phenomenon has likely occurred in the matrix which, in turn, can contribute to reductions in impact properties.

To illustrate the relationship between Charpy impact strength and specific breaking energy as a function of deformation and stiffness, a comparative diagram is presented as Fig. 9, which depicts a good correlation between impact strength and specific breaking energy of both materials, whether before or after isothermal aging.

The fracture surfaces of the isothermally aged specimens after impact testing are shown in Figs. 10 and 11.

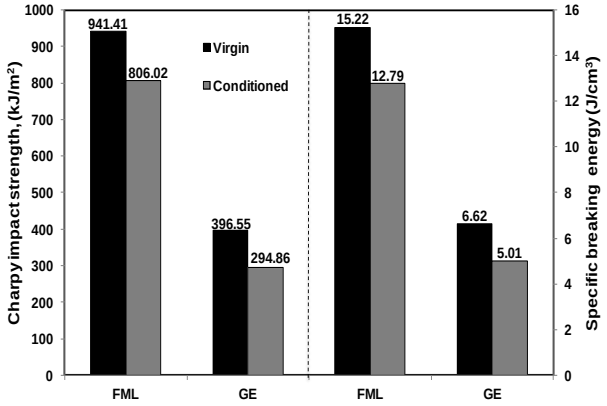
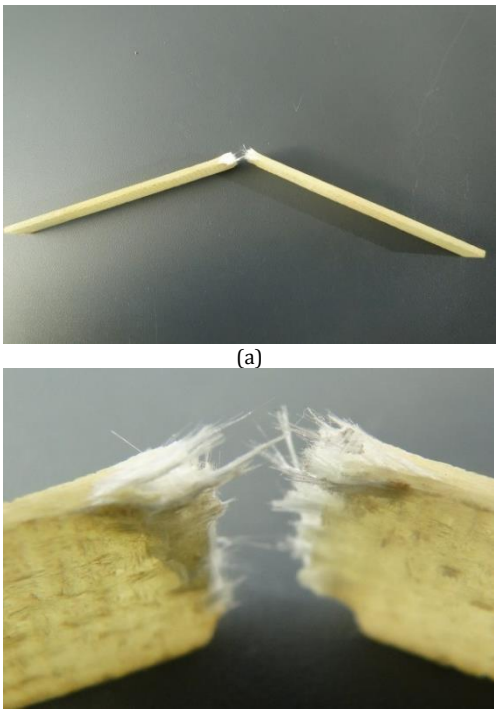


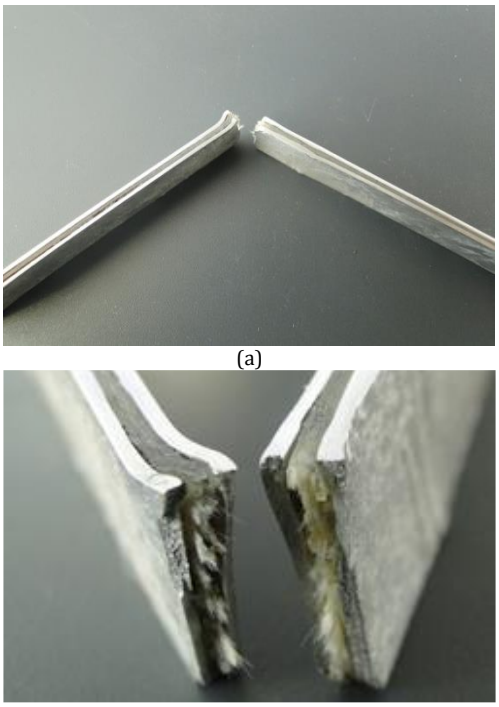
Figure 9. Charpy impact strength and specific breaking energy of the FMLs and GE composites before and isothermal aging



(a)

(b)

Figure 10. (a) Failure modes following impact testing in the GE/TI specimens, (b) magnified at fracture surface



(a)

(b)

Figure 11. (a) Failure modes following impact testing in the FML/TI specimens, (b) magnified at fracture surface

Fracture surfaces of both the GE/TI and FML/TI specimens are characterized by catastrophic failure associated with the considerable amount of fiber breakage, which is typical for the brittle composites.

Moreover, no evidence of interfacial debonding and delamination phenomena was present for GE/TI specimens, although there was a slight delamination in the FML/TI specimens in the location of the impact point only.

The origins of these failure modes are likely the cracks that occurred during the isothermal aging process. Decelle, et al. [32] reported that because of matrix shrinkage that takes place during thermal aging, high strain gradients are generated in matrix areas close to fibers, resulting in the initiation of these cracks. The reduction of impact properties due to thermal aging can be explained by the cumulative damage effect of these cracks.

4. Conclusions

An experimental investigation of the flexural and impact properties of glass/epoxy (GE) composites and fiber metal laminates (FMLs) after 840 h of exposure at 130°C was presented. From the experiment's results, the following conclusions were made:

1. Isothermal conditioning of GE composites at 130°C can reduce flexural strength by 10.61%, coupled with a weight loss of 0.53%. A similar, though relatively significant, loss of 4.26% in aged FML flexural strength was observed. Microcracks caused by scission of polymer chains and matrix loss during thermal aging are believed to be responsible for the GE composite specimens' decrease in flexural strength. However, despite the favorable effect of elevated temperatures in reducing aluminum–layer residual stress in FML specimens, the same weakening effect of chain scission reactions can probably overcome the further cross-linking that occurred in the matrix. Moreover, no significant weight loss was observed during isothermal aging at 130°C, indicating that FML specimen weight is highly stable.

2. Experiment results also indicated that no discernible effect was found in flexural stiffness in either GE or FML specimens after isothermal aging. This was mainly due to the fact that isothermal aging can probably create additional cross-linking, causing the polymer matrix to become more brittle and stiff.

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