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Effect of Thermal Cycling on Hardness and Impact Properties of Polymer Composites Reinforced by Basalt and Carbon Fibers

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Abstract

The polymer-matrix composites may be significantly affected by cyclic temperature changes. This study investigates the effects of thermal cycles on hardness and impact resistance of three types of phenolic-matrix composites, that is, phenolic resin reinforced with (1) woven basalt fibers, (2) woven carbon fibers and (3) hybrid of basalt and carbon fibers. The effect of thermal cycling on hardness and impact resistance was material-dependent. While, the thermal cycling rapidly decreased the hardness of composites reinforced by carbon fibers, it gradually decreased the hardness of composites reinforced by basalt fibers. While, the Charpy impact energy of carbon/phenolic (CFP) and basalt/carbon/phenolic (BCFP) composites was not significantly affected by thermal cycles, the Charpy impact energy of basalt/phenolic (BFP) composites shows a sharp decline with increasing thermal cycling, and reaches a plateau after a certain cycle. Based on the results, the BFP composites was significantly harder than CFP and the composites containing carbon fibers in spite of demonstration of low impact resistance at primal cycles, possessed very gradual decline in impact resistance compared to BFP composites after the thermal cycling.

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Keywords: Phenolic resin, Basalt fiber, Carbon fiber, Thermal cycling, Hardness, Charpy impact

1- Introduction

In the last two decades, polymer composite materials have generated considerable interest in various industries due to their unique and beneficial properties. With the increased use of polymer composites in many areas, it becomes imperative that engineers and scientists understand the mechanical behavior of various material systems under all service environments. One of the critical service environments for composite material usage is cyclic temperature changes. Any material to be used in such an environmental condition must be capable of handling various combinations of loading (static and dynamic), thermal cycling, heat, ice, and snow.

Several studies are conducted in the last decade to characterize the dynamic properties and hardness of traditional structural composite laminates, especially those of carbon fiber reinforced polymers [1–3]. Traditional carbon fiber reinforced polymers, in which continuous carbon fibers strengthen brittle or toughened polymer matrices, still meet the basic design requisites for aircraft construction. However, these structures (e.g., High Speed Civil Transport Project), may encounter temperature excursions during use, whether due to exhaust gas, de-icing, ambient temperature variations or engine operation [4].

It is found that the carbon fiber reinforced polymers are very vulnerable to microcracking caused by thermal cycling [5]. Because of the large difference between the coefficient of thermal expansion of carbon fibers (typically around -6×10^{-6} C^{-1}) in the longitudinal direction and that of the polymer matrix (typically ranging from 20×10^{-6} C^{-1} to 120×10^{-6} C^{-1}),

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temperature excursions result in changes in the amount of thermal stress in the composite. This means that for example, as the temperature drops, the matrix wants to contract more than the fibers, which induces stresses in the matrix and the fiber, as well as in the interface between the two materials. Such thermal stress variations may cause thermal fatigue. Damage degrades the modulus of the composite, thus enhancing vibrations and aggravating further damage [4–6]. Thermal cycling slightly reduces the tensile strength and modulus of the composite, but has a more serious effect on matrix-dominated mechanical properties of the composite [7,8].

Toshio Nakamura et al [9] studied thermal cycling crack growth of fiber-reinforced composite laminate under a combined experimental and computational investigation. Examination of the fractured surface at the conclusion of experiment revealed complex crack propagation pattern.

Paillous and Pailler [10] examined the degradation induced by exposure to a space environment on multi-ply polymer matrix composites. They exposed various graphite/epoxy laminate specimens to thermal cycling. Their study showed that thermal cycling degraded the matrix by chain scission, crosslinking and microcrack damage, altering the composite properties.

Lafarie-Frenot and Rouquie [11] compared the damage developed in carbon/epoxy laminates during isothermal ageing or thermal cycling. They showed that the oxidation phenomenon induces matrix shrinkage which is visible on the surfaces of the samples. This shrinkage, which is non-existent on the specimens tested in neutral atmospheres, seems to be favourable to fiber–matrix debonding and matrix crack onset. In this case, a coupling between oxidation and cyclic thermo-mechanical stresses due to temperature variations,

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accelerates all the phases of the damage process.

In composite materials, the oxidation mechanism strongly depends on temperature and oxygen pressure [12], specimen geometry and anisotropy [13–15] and matrix–fiber bonding [16]. In oxidative atmospheres, the coupling between oxidation, occurring at high temperatures and thermomechanical cyclic stresses due to temperature variations, accelerate the damage process [17].

Seferis and Hayes [18] showed that the properties and fiber modulus have a significant effect on the response of the carbon/epoxy composites to thermal cycling. It was found that the higher glass transition temperatures of the laminates decreased the microcracking propensity of these laminates. Higher modulus and linear coefficients of thermal expansion of the fibers were found to increase the microcrack density in the laminates.

It is known that the basalt fibres have high chemical stability [19,20], they are non-toxic, non-combustible [21] and freely withstands temperature differences of -200 ºC to +700 ºC [22]. Moreover, their specific mechanical properties are comparable with, or better than, those of E-glass ones.

Therefore, a woven hybrid composite of basalt and carbon fibers is reasonably priced with adequate mechanical, thermal and impact properties. However, it is not clear as how the impact properties of the hybrid composites are changed as temperature fluctuations occurred.

In general, the purpose of hybridization is to achieve a composite architecture which synergizes the properties of both materials and/or lowers the cost, since one type of the fibers could be too expensive [23]. Hybrid composites typically meet the requisites of high stiffness, high mechanical resistance (especially to fatigue crack growth), high static fracture toughness, good capacity for absorbing transverse impact energies (trans-thickness impact),

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high flame resistance and hardness [24,25].

Very little work is done to evaluate the thermal cycling influence on the hardness and impact behavior of fibers reinforced polymer composites, especially for textile composites. The objective of this paper is to develop durable, hard and tough polymer matrix composites that are capable of withstanding extreme thermal cycling and impact loads.

In the present study, the effect of thermal cycling on the Charpy impact behavior of carbon, basalt and basalt/carbon hybrid fiber reinforced composites, and also the effect of these thermal cycles on the hardness of carbon and basalt fiber reinforced composites are investigated. The composite specimens are prepared according to ASTM standards and then subjected to different number of thermal cycles between -30 ºC to +220 ºC. The thermal cycled specimens are tested in Charpy impact and also by Barcol impresser to study the effect of thermal cycles on their mechanical behavior.

2- Experimental Procedure

2-1- Materials and Fabrication

Three kinds of materials were prepared. The first material was a composite laminate consisted of 8 plies of carbon fibers (AC220, Colan Products, Australia) and a phenolic resin (Phenlam ® CL2000T, Huntsman Chemical Company, Australia). The second material consisted of 4 plies of basalt fibers (BAS 630, Basaltex™, Belgium) and the above said phenolic resin. The third material consisted of alternatively 3 plies of basalt and 3 plies of carbon fibers and the above mentioned phenolic resin. Specification of the resin and the fibers used in this work are presented in Table 1.

The materials were fabricated in the form of plates with dimensions of 400 mm × 400

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mm × 2.5 mm. The fiber volume fraction in all the composite specimens was approximately 35%. The materials were combined and the plates were fabricated by hand lay-up technique.

Table 1- Specification of the fibers and the resin

Specification	Basalt fibers	Carbon fibers	Phenolic resin
Weave type	Twill 1/3	Plain	…
Thickness (mm)	0.56	0.25	…
Specific surface weight (g/m^2)	630	198	…
Density (g/m^3)	2.70	1.75	1.2
Tensile strength (MPa)	3000	4210	24 - 45
Tensile modulus (GPa)	89	230	4
Elongation at break (%)	3.15	1.5	1 - 2
Coef. of thermal expansion (10^{-6}/ºC)	8	-6	20 - 31
Gel time (min)	…	…	25

2-2- Specimen Preparation

Specimens were cut from the plates in the desired dimensions of 75 mm × 12 mm × 2.5 mm, using a water jet cutting machine. Each specimen was polished using emery sand paper. In this way, the local surface cracks and local surface material heterogeneities were removed. After cutting to the specified dimensions, the specimens were coded and their dimensions were measured. Specimens were divided into three groups: (1) basalt/phenolic composites (BFP), (2) carbon/phenolic composites (CFP), (3) basalt/carbon/phenolic composites (BCFP). Specimens in each group were stored at room temperature until impact and hardness testing. A specimen of CFP is shown for testing in Figure 1.

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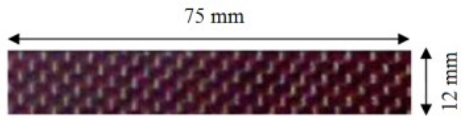

Figure 1- Specimen of CFP prepared according to ASTM D6110.

2-3- Thermal Cycling

To investigate the thermal cycling effect, the specimens were put into a temperature shock test chamber (Model TS^{130}, Weiss Umwelttechnik GmbH, Germany) as shown in Figure 2.

Specimens were exposed to -30 ºC for 3 min and then they were immediately exposed to +220 ºC for the same duration (3 min). The general temperature profile applied on the specimens is shown in Figure 3. According to this profile, one cycle of thermal variation was designated as the sequence from -30 ºC to +220 ºC, so that the total duration of each cycle was 6 min. The thermal cycling was repeated for 5, 10, 15, 20, 25 and 30 cycles.

(a) (b)

Figure 2- (a) Temperature shock test chamber, (b) Front view of designed aluminum stand with composite specimens inside the chamber.

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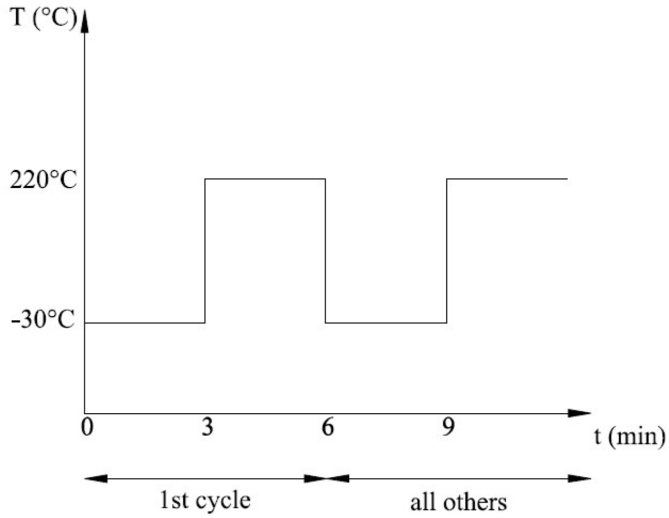

Figure 3- Thermal cycle profile.

The specimen codes, type of fiber reinforcement and no. of thermal cycles for each specimen are listed in Table 2. The numbers used in the specimen codes represent the thermal cycle numbers. Also, "H" and "I" symbols represent the test types, i.e., hardness and impact tests, respectively.

Table 2- The composition of the materials and number of thermal cycling exposure of specimens

Specimen code	Reinforcement fibers	Numbers of thermal cycling	Test type
BFP- H0, 5, ..., 30	Basalt	0, 5, 10, …, 30	Hardness
CFP- H0, 5, ..., 30	Carbon	0, 5, 10, …, 30	Hardness
BFP- I0, 5, ..., 30	Basalt	0, 5, 10, …, 30	Impact
BCFP- I0, 5, ..., 30	Basalt/Carbon	0, 5, 10, …, 30	Impact
CFP- I0, 5, ..., 30	Carbon	0, 5, 10, …, 30	Impact

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2-4- Testing Procedure

The specimens prepared in section 2.3 were tested in Barcol hardness and Charpy impact tests according to the ASTM standards. Three specimens were tested for Charpy impact tests and the reading is according to the average value of the three readings. For hardness test, eight readings were taken and the final result is according to the average value of these readings.

2-4-1- Hardness Testing

Hardness tests were performed according to ASTM D 2583 by a Barber Colman Barcol impressor hardness tester (Model GYZJ 934.1, USA) as shown in Figure 4. The tests were conducted at ambient temperature (25 ºC) and relative humidity of 50%. The hardness tests were conducted on BFP and CFP specimens.

Figure 4- Impression made by Barcol impressor hardness tester.

2-4-2- Charpy Impact Testing

Charpy impact specimens were tested in a pendulum machine (Tester MT220, TERCO, Sweden) equipped with Charpy hammers according to ASTM D 6110, as shown in Figure 5. The experiments were conducted at ambient temperature (25 ºC) and relative humidity of 50%.

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Figure 5- EMIC pendulum Charpy impact tester.

3- Results and Discussion

3-1- Hardness Test Results

This section describes the results obtained from the hardness test. First, the variation of hardness values obtained is modeled using normal distribution. Statistics analyses are performed by the software program (SPSS^{TM}) with statistical abilities. Eight hardness tests were performed on each specimen and using the test data, the corresponding standard deviation and variance for each specimen are determined and shown in Tables 3 and 4 for BFP and CFP, respectively. Notice that due to the hardness is the property that strongly related to the surface of specimens, and there was not a surface that contained both basalt and carbon fibers, therefore this test is not reasonable and not performed.

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Table 3- Statistics results of Barcol hardness tests on BFP composites

Statistical parameters	BFP-H0	BFP-H5	BFP-H10	BFP-H15	BFP-H20	BFP-H25	BFP-H30
Mean (Barcol)	69.125	68.8750	68.0000	66.3750	65.5000	65.1250	65.0000
Standard Error of Mean (Barcol)	0.37806	0.35038	0.37796	0.46049	0.32733	0.39810	0.53452
Standard Deviation (Barcol)	1.07824	0.99103	1.06904	1.30247	0.92582	1.12599	1.51186
Variance ($Barcol^2$)	1.16260	0.98214	1.14286	1.69643	0.85714	1.26786	2.28571

Table 4- Statistics results of Barcol hardness tests on CFP composites

Statistical parameters	CFP-H0	CFP-H5	CFP-H10	CFP-H15	CFP-H20	CFP-H25	CFP-H30
Mean (Barcol)	64.125	63.7500	62.1250	61.0000	58.6250	56.5000	54.5000
Standard Error of Mean (Barcol)	0.31403	0.83986	0.63913	0.42258	0.46049	0.32733	0.80178
Standard Deviation (Barcol)	0.94792	2.37547	1.80772	1.19523	1.30247	.92582	2.26779
Variance ($Barcol^2$)	.89855	5.64286	3.26786	1.42857	1.69643	.85714	5.14286

Figure 6 shows the effect of thermal cycling on the hardness of thermally conditioned BFP and CFP composites. These conditions result different effects on BFP and CFP composites. The stability of hardness values is noticeable for the BFP composites after 20

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cycles, but there is a continuous reduction in hardness values for the CFP composites after 20 cycles. The gradual stability of hardness in BFP composites implies that some kind of permanent stiffening occurs in the aged material.

As shown in Figure 7, it is obvious that the CFP composites are more susceptible to surface damages by thermal cycling as compared to the BFP composites.

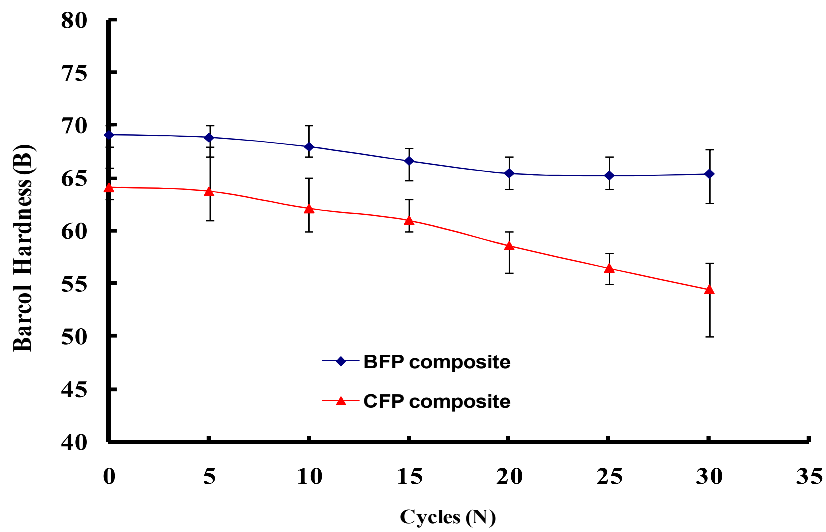

Figure 6- Variation of hardness values for composites after exposure to thermal cycling.

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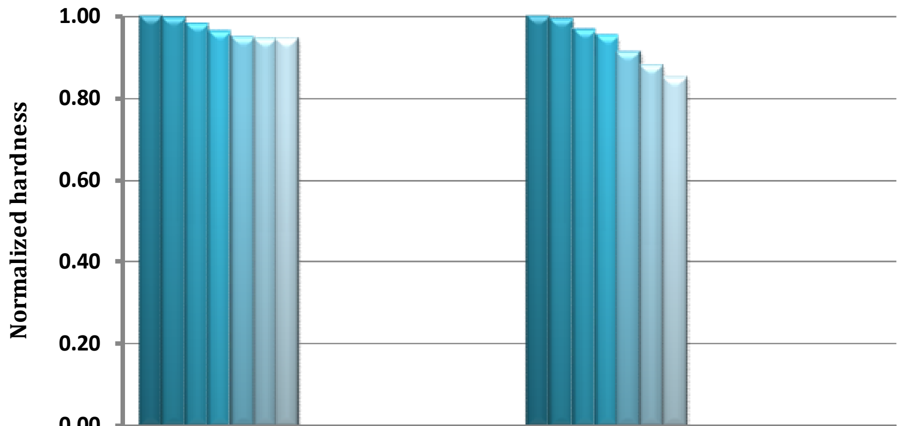

	BFP	Loss (%)	CFP	Loss (%)
Original	1.00	0.00	1.00	0.00
5 cycles	1.00	-0.36	0.99	-0.58
10 cycles	0.98	-1.63	0.97	-3.12
15 cycles	0.96	-3.56	0.95	-4.87
20 cycles	0.95	-5.24	0.91	-8.58
25 cycles	0.94	-5.55	0.88	-11.89
30 cycles	0.95	-5.35	0.85	-15.01

Figure 7- Degradation of hardness values for BFP and CFP composites after exposure to the thermal cycling.

The effect of thermal cycling definitely causes debonding and/or weakening of the interface because of different coefficients of thermal expansion and/or contraction for the polymer matrix and the fiber reinforcements [26]. Large differences in the coefficients of thermal expansion and modulus between the fibers and the matrix, increased the thermal stresses at low temperatures and caused a corresponding increase in micro-cracking [27].

In this study, the polymer matrix is a material with high thermal expansion (coefficient of thermal expansion of 20-31×10^{-6}/ºC). The differences between the thermal expansion coefficients of basalt and carbon fibers and polymer matrix, lead to high interfacial strains during a thermal cycling process between +220 ºC and -30 ºC. The stresses caused by strains can lead to create some micro-cracks on the surface of the specimens and thereby decreasing

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the hardness and impact properties of the composites. When the composite specimens are subjected to thermal cycling under oxidizing atmospheres, the high temperatures create a thermo-oxidation of the matrix. This phenomenon of thermo-oxidation affects primarily the external surfaces of the specimens [11]. In this case, a coupling between oxidation and cyclic thermo-mechanical stresses due to temperature variations can accelerate all the phases of the damage process particularly in the CFP composites.

The overall conclusion drawn here is that there are a number of mechanisms which can be responsible for causing a stiffening of the material during the thermal cycling procedure. The most acceptable explanation of the stability in hardness properties that occurred in the BFP composites is a possible post-curing that took place during the thermal cycling procedure. This happened due to a high temperature, which were not severe enough to break the chemical bonds of the polymer [28–32], but in contrast, it contributed to the creation of free radicals to the molecules of phenolic which had not already reacted, and thus, to a further cross-linking [32-36]. Several mechanisms may contribute to the fiber/matrix adhesion, i.e., covalent bonding, physical interactions, and mechanical interactions [26]. These post-curing phenomena could dominate over the de-adhesion effect of thermal cycling. Thus, the stability in hardness is reflected for the BFP composites. The cryogenic conditioning at -30 ºC for different cycles may impart different degrees of shrinkage compressive strength at the interface. This could help in strengthening the adhesion. But, the weakening effect of thermal cycling might dominate over the post-curing hardening effect in the CFP composites and consequently, it may result in the reduction of hardness.

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3-2- Impact Test Results

In this section, the specimens prepared for Charpy impact were tested. BCFP specimens were also tested. The results of Charpy impact tests are shown in Figure 8.

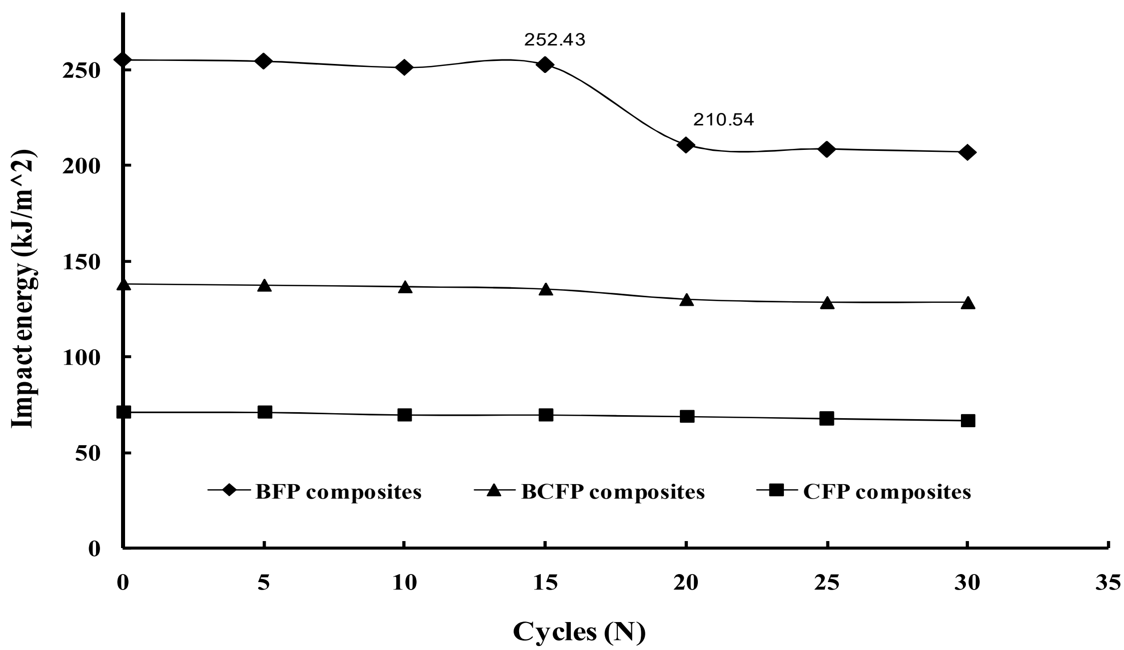

Figure 8- Variations of Charpy impact energy of composites after exposure to the thermal cycling.

The CFP composites show a good stability at different thermal cycles. But, the BFP composites show a region over which the impact energy is reduced by 19%. As can be seen in Figure 8, before 20 thermal cycles, the average impact energy of the BFP composites was approximately constant and 350% and 180% higher than that of the CFP and BCFP composites, respectively. After 20 thermal cycles, the CFP and BCFP composites showed almost negligible decrease in impact properties, while the average impact energy of the BFP composites was dropped by nearly 19%. Note that, there is not appeared significant decrease

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in the average impact energy of the CFP and BCFP composites relative to the BFP specimens.

The low-velocity impact causes many types of damages within the composites, depending on the amount of impact energy imparted to the specimen. Matrix cracking, delamination, fiber/matrix debonding, fiber pullout, and fiber rupture are some of the damage modes caused by low-velocity impact [37].

Initially, when a composite is impacted with enough energy, cracks form in the matrix material. Since with the polymer matrix composites, the matrix is much more ductile than the fibers, upon impact, the adhesion between the stiff fibers and the more ductile matrix restricts the matrix from fully deforming as it would be with the support of the fibers [38].

The next mode of the damage is delamination between plies of the composite. This type of damage occurs with higher impact energies than those which caused matrix micro-cracking alone [39]. Much of the energy absorbed within a composite upon impact is dissipated through the formation of delaminations.

The next mode of damage is fiber rupture, which is occurred following the matrix micro-cracking and delaminations. Fiber rupture occurs on the back face of the specimen, exactly opposite to the location of impact on the top face of the specimen. Upon impacting with a high enough energy, the delaminations progress due to high shear stresses ahead of the crack tip which in turn cause more delaminations. The specimen continues to carry load until the fibers in the next layer begin to fail in tension. This is the manner in which the composite laminates can fail. Fibers may rupture on the bottom surface of the specimen due to the increase in tension caused by the deformation of the specimen, while fibers on the top surface may remain intact [40].

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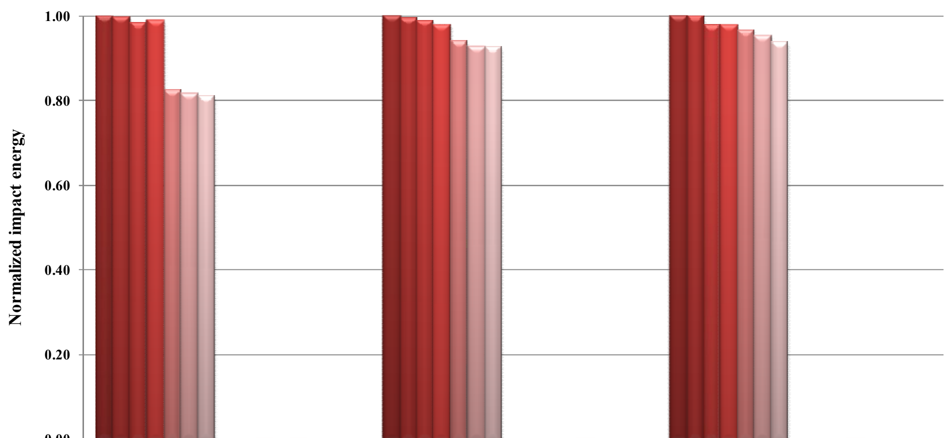
Figure 9 shows the effect of thermal cycling on Charpy impact energy, in the form of normalized values compared with the original untreated specimen for the CFP, BFP and BCFP specimens. As a general rule, the trend of decreasing in impact energy in all the composite specimens with increasing thermal cycles could be observed, as shown in Figure 9.



	BFP	Loss (%)	BCFP	Loss (%)	CFP	Loss (%)
Original	1.00	0.00	1.00	0.00	1.00	0.00
5 cycles	1.00	-0.28	0.99	-0.51	1.00	-0.07
10 cycles	0.98	-1.57	0.99	-1.12	0.98	-1.99
15 cycles	0.99	-1.01	0.98	-2.01	0.98	-2.04
20 cycles	0.83	-17.44	0.94	-6.00	0.97	-3.22
25 cycles	0.82	-18.32	0.93	-7.13	0.95	-4.74
30 cycles	0.81	-18.94	0.93	-7.13	0.94	-6.26

Figure 9- Degradation of Charpy impact energy of composites after exposure to the thermal cycling.

Notice that during the initial 15 thermal cycles, the rate of degradation of the Charpy impact energy is not as greater as the rate during the rest of thermal cycles. This can be attributed to the fact that the size and/or number of cracks increases.

As shown in Figure 9, After 30 thermal cycles of exposure, the Charpy impact energy of the BFP, BCFP and CFP composites reduces by almost 19%, 7% and 6%, respectively. However, the Charpy impact energy of the BFP, BCFP and CFP composites after 15 cycles remain relatively constant. Also, it is obvious that in spite of the BFP composites are much

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stronger than CFP composites; they are more suseptible to impact damages by thermal cycling as compared to the CFP and BCFP composites. The combination of basalt and carbon fibers added both the advantages of stability of various thermal cycles and higher impact energy, for hybrid BCFP composites.

The fracture surfaces of the impact specimens are shown in Figs. 10-12. Fracture surfaces of CFP specimens are dominated by brittle fracture, which is typical for these types of composites. Also, no evidence of interfacial debonding and delamination phenomena is present. Fracture mode of BFP composites and BCFP composites are fiber pull out, fiber rupture and delamination at low thermal cycles and matrix cracking at higher thermal cycles.

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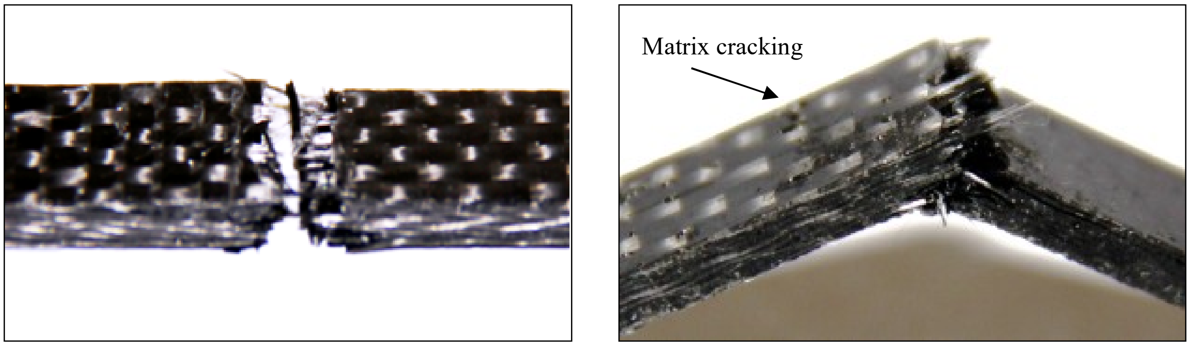
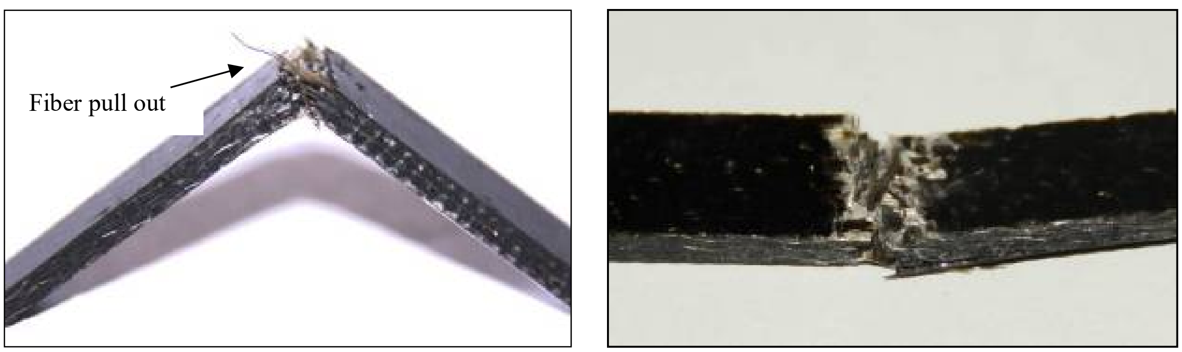


(a) (b)

Figure 10- Typical photograph taken from CFP composite exposed to (a) 5 cycles, (b) 30 cycles, after Charpy impact tests.



(a) (b)

Figure 11- Typical photograph taken from BCFP composite exposed to (a) 5 cycles, (b) 30 cycles, after Charpy impact tests.

(a) (b)

Figure 12- Typical photograph taken from BFP composite exposed to (a) 5 cycles, (b) 30 cycles, after Charpy impact tests.

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4- Conclusions

In the present work, the effect of thermal cycling on hardness and Charpy impact resistance of polymer-matrix composites (phenolic resin reinforced with woven basalt fibers (BFP) and woven carbon fibers (CFP)) is investigated. The thermal cycles manifest similar kinds of debonding effects for thermally conditioned specimens for CFP and BFP composites.

In conclusion, the thermal cycling results in severe degradation effects in hardness for the CFP composites. The same conditioning causes no extensive weakening response for the BFP composites. It is also reasonable to conclude that the CFP composites are more susceptible to surface damage by this type of thermal cycling exposure as compared to the BFP composites. The reduction of hardness after thermal cycling was less than 6% of that of the initial condition for BFP composites; while the hardness of the CFP was reduced by more than 15% as compared to its initial untreated specimens.

The mean Charpy impact energy of the BFP specimens in this study is higher than that of the other specimens at all thermal cycles. However, after 20 thermal cycles, the Charpy impact energy of BFP composites dropped by approximately 19%. Finally, it was found that the BCFP composites (hybrid of basalt and carbon fibers composite layers) as the impact resistant materials present an impact energy of approximately 190% higher than that of CFP composites and a negligible decrease in impact properties caused by thermal shock cycles at all the cycle levels.

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