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An Experimental Investigation on Fracture Analysis of Polymer Matrix Composite under Different Thermal Cycling Conditions

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ABSTRACT

Fracture analysis of glass/epoxy composites under different thermal cycling conditions is considered. Temperature difference, stacking sequence, fiber volume fraction and number of thermal cycles are selected as the experimental design factors. The Taguchi method is implemented to design of the experiment and an apparatus is developed for automatic thermal cycling tests. The tensile tests are done to study mechanical behavior of the specimens after the thermal cycling. The results show that the stacking sequence is the main effective factor on the fracture surface behavior of the specimens. Also, long splitting, lateral and angled breakage are the dominate failure mode of $[0]_8$, $[0_2/90_2]_s$ and $[0/\pm45/90]_s$ layups, respectively. It's found that the thermal cycling and temperature difference cause to increase the surface matrix loss significantly. This surface matrix loss can be an initial region to matrix debonding and crack propagation. Also, when the angle difference between lamina is increased the mechanical properties are reduced under the thermal cycling, significantly.

Keywords : Thermal cycling; Fracture of composite; Polymer matrix composites (PMCs) ; Taguchi method.

1 INTRODUCTION

POLYMER matrix composites (PMCs) commonly used as structural components in aerospace applications. In addition to mechanical or structural loads, they are often subjected to repeated temperature fluctuations during their service life. For example, composite materials of a satellite structure would undergo periods of solar eclipse and sun illumination within a service temperature range from -150 to 150˚C [1]. Also, containment structures of single stage to orbit (SSTO) launch vehicles, used for future space transportation systems, manufactured by new, high performance polymeric matrix fibrous composites. These structures were required to sustain an extensive combination of cryogenic and elevated temperature during testing and flights [2]. Furthermore, environmental conditions may cause mismatched expansions and generate additional stresses within composite laminates [3]. These stresses, at various conditions, can generate matrix cracking and promote laminate delaminations, mass loss and fiber-matrix debonding under thermal cycling. Many studies have focused on the fracture of the PMCs under different thermal cycling and thermal aging conditions [4-13]. Most of these works focused on one or two effective factors [4-11], but some of them consider more than two parameters [12-13]. These works always had studied different effective parameters on fracture behaviors (mainly resin, fiber and curing agent) [4-5, 13], number of

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thermal cycling [6-7, 12], stacking sequence and layer orientation [8-10, 12-13], environmental atmospheres (oxidative or neutral) [11-12] and exposure (ultraviolet or electron radiation) [13] conditions. Eselun et al. [4] noted influences of resin micro cracks, generated by thermal cycling, on fatigue life, tensile strength and interlaminar shear strength. Also, Rinaldi and Maura [5] used glass/epoxy specimens were cycled up to 60 times between 0 and 110˚C. Also, they used different curing agent to study effects of them on tensile strength. The results showed that using polyaminophenolic curing agent the tensile strength increased from 30 to 50 MPa, after 30 cycles. But these values increased slightly for the long chain amine and then fell back to a constant value. It was concluded the polyaminophenolic curing agent was superior. Cohen et al. [6] found that during thermal cycling, cracks accumulated up to a brief number of cycles, remained constant, and later increased. Also, Ghasemi et al. [7], used glass/epoxy specimens were cycled up to 90 times between 0 and 75˚C. The results of these studies demonstrated that the non-linear behavior of composites influences the residual stresses significantly. Besides, if the sample becomes more brittle, the residual strains remain constant; while the elasticity modulus and residual stresses decrease.

An attempt to emulate the effect of a space environment on T300/5208 carbon-epoxy specimens were made by Adams et al. [8]. Various symmetrical layups were used as $[0/90_3]_s$, $[0_2/90_2]_s$, $[0_3/90]_s$, $[90/0_3]_s$, $[90_2/0_2]_s$ and $[90_3/0]_s$ and were subjected up to 500 cycles between -157 and 121˚C at a rate of 5.68˚C/min. Transverse cracks were first detected at -46˚C though many specimens remained crack free at the lowest temperature. In no case was crack saturation observed even after 500 cycles. Rouquie et al. [9] pointed out the influence of layer orientation on the transverse cracking damage of carbon fiber reinforced plastic (CFRP) laminates. They concluded the damage observed on the polished edges of the samples highly depends on the stacking sequence. Also, Scida et al. [10] described the influence of hygrothermal aging on mechanical properties and damage behavior of quasi-unidirectional flax-fiber reinforced epoxy (FFRE) composite. Young's modulus and tensile strength are clearly affected by the hygrothermal aging because a significant reduction in Young's modulus is shown while tensile strength decreases much less for water-saturated composites. Lafarie-Frenot et al. [11] highlighted the accelerating effect of an oxidative atmosphere. They concluded that the higher oxygen concentration cause to the more significant acceleration of cracking development. Also, Lafarie-Frenot et al. [12] studied the damage mechanisms of CFRP laminate subjected to repeated thermo-mechanical loads. The experiments showed that the cyclic thermal stresses lead to many matrix cracks. As the number of thermal cycles increased, transverse matrix cracks increased steadily, but the kinetics of their development was observed much faster in oxygen than in nitrogen. Also, the results highlighted that the matrix cracks of the external layer spontaneously appear longer, compared to those in the inner layer, and their increase in number and length is a little faster when the free edge is orthogonal to the fiber direction. Mouzakis et al. [13] had been assessed the failure of PMCs upon exposure to the environment. The accelerated environmental aging was based on two kinds of alternating cycles, which provided humidity, temperature and ultraviolet radiation. Pure polyester and polyester materials reinforced with a glass fiber random mat onto which glass fibers were knitted at 0°/90° examined. The results revealed that the aged materials gained in stiffness, whereas a small deterioration in strength was found. Also, some microcracks have occurred on the surface of the specimens.

The literature review showed that multi-variable thermal cycling experiment to find and quantify the effective factors on fracture of the PMCs is necessary. Design of experiment (DOE) is a suitable method to reduce number of test where problem contains multiple and many interacting variables [14]. Ju [15] tests 2^k as the factorial design for the four factors to provide their quantitative primary and interaction effects of thermal cycling on crack density. The test involved four controlling parameters named, average in-plane mechanical strains, thermal cycling temperature amplitudes, number of thermal cycles, and heating rate. All the parameters involved two levels. Taguchi method as another DOE method reduces the number of experiments that are required to model the response function compared with the full factorial DOEs, effectively [16]. Vankanti et al. [17] optimized the drilling process of glass fiber reinforced polymer (GFRP) composites using Taguchi experimental design and an L9 orthogonal array. The used parameters named, cutting speed, feed, point angle and chisel edge width. This method is a suitable method to study the effects of different parameters on thermal cycling degradation of the PMCs using minimum test.

The current study is undertaken to study the composite breakage behavior under different thermal cycling conditions. Specifically, it reveals how temperature difference, fiber volume fraction, stacking sequence and number of thermal cycles can effect the mechanical properties of the PMCs. Using the Taguchi method experiment scheme is extracted for three level factors. Required specimens are prepared using E glass fiber and epoxy. A two chamber thermal cycling apparatus is developed for the thermal cycling tests and a tension test machine is used for the tension tests on the specimens. Using these apparatus, tensile modulus and strength stress are obtained and tensile failure behavior of glass/epoxy laminated composite is investigated. A statistical approach is utilized to identify contribution of the effective factors on mechanical degradation of the specimens. Based on this approach, the effects of these four factors on fracture surface are investigated.

2 DESIGN OF EXPERIMENT:TAGUCHI METHOD

Effective parameters on the PMCs resistance to thermal cycling can be categorized to the three branches:

- Geometrical parameters (stacking sequence, specimen form, …)
- Material parameters (fiber and matrix properties, …)
- Thermal cycling parameters (number of cycles, heating rate, …)

For designing an experiment, its required to consider all the effective parameters is impossible. Among these parameters the material properties depend on chemical structures of constituents; while the thermal cycling parameters and geometrical parameters depend on the apparatus specification and sample fabrication, respectively. Composite designer always don't change the chemical structures of material and they change the fiber volume fraction and stacking sequence occasionally. The Taguchi method is effective method to deal with responses which was influenced by multi-variables. As a result, the experiment design of this research is accomplished according to the composite designers view point and it considers the four effective parameters simultaneously. For this purpose, the flat geometry without any initial crack or stress concentration is selected as the sample geometry. Also, the stacking sequence and fiber volume fraction are selected as the geometrical and material parameters, respectively. Besides, number of thermal cycling and temperature difference are chosen to consider the thermal cycling parameters. Finally, four essential parameters namely the number of thermal cycles; temperature difference, fiber volume fraction and stacking sequence are selected as the main experimental factors.

Here, the orthogonal array of L9 type was used to design the experiment. This scheme requires nine experiments with four parameters that each parameter has three levels. If three levels were assigned to each of these factors, number of permutations would be 3^4 or 81 experiments. The Taguchi L9 method reduced the number of experiments to nine. Table 1. shows the four factors and three levels used in the experiments. Also, the orthogonal array of L9 type is presented in Table 2. As shown in this Table, three different stacking sequences are selected as $[0]_8$, $[0_2/90_2]_s$ and $[0/\pm45/90]_s$ that considers unidirectional, cross-ply and quasi-isotropic sequence, respectively. The minimum temperature of the all cycles were selected 0˚C. For the study different temperature effects, three different cycles are considered that have the maximum temperature of 50˚C, 75˚C and 100˚C, respectively (Fig. 1). The maximum temperature of the thermal cycle was selected 100˚C to accelerate the damage processes. This temperature is much higher than that supported by this material in the real applications. Also, the fiber volume fractions of the composites were 45, 55 and 60% and the specimens are cycled up to 1, 50 and 100 cycles.

Table1
Level of experiment parameters.

Symbol	Factor	Level 1	Level 2	Level 3
A	Number of thermal cycles	1	50	100
B	Stacking sequence	$[0]_8$	$[0_2/90_2]_s$	$[0/\pm45/90]_s$
C	Temperature difference (˚C)	50	75	100
D	Fiber volume fraction (%)	45	55	60

Table 2
Taguchi´s L9 (3^4) orthogonal array.

Test Group	Number of Thermal Cycles	Stacking Sequence	Temperature Difference (˚C)	Fiber Volume Fraction (%)
Run-1	1	$[0]_8$	50	45
Run-2	1	$[0_2/90_2]_s$	75	55
Run-3	1	$[0/\pm45/90]_s$	100	60
Run-4	50	$[0]_8$	75	60
Run-5	50	$[0_2/90_2]_s$	100	45
Run-6	50	$[0/\pm45/90]_s$	50	55
Run-7	100	$[0]_8$	100	55
Run-8	100	$[0_2/90_2]_s$	50	60
Run-9	100	$[0/\pm45/90]_s$	75	45

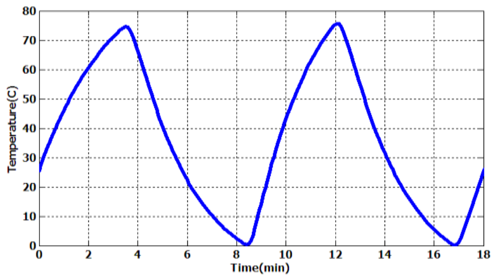

Fig.1
Experimental profile of thermal cycling.

3 EXPERIMENTS

3.1 Specimens preparation

The ML506 epoxy resin and polyamine hardener (HA-11) are used as the matrix of composite. This resin has good mechanical properties and low viscosity that makes it a suitable material for the composite applications (Table 3.). Also, unidirectional *E* glass fibers (supplied by Gurit™) have been used as the reinforcing material in this investigation (Table 3.). The composite thin laminates are fabricated using hand lay-up method and are allowed to cure for seven days at room temperature. The test specimens were cut from laminates according to the standard ASTM D3039 [18].

Table 3
Mechanical and physical properties of epoxy resin and *E*-Glass fiber.

Material properties	ML506 Epoxy	E-Glass
Tensile modulus (*GPa*)	2.79	41
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*)	62	4.9

3.2 Thermal cycling test apparatus

A thermal cycling apparatus consisting of heating and cooling chambers and a rail system for specimen motion was assembled to provide the temperature cycle of the specimens. Mechanism of this three sensors-actuators system is shown in Fig. 2. Heating and cooling of composite samples were achieved by using double 1000 watt heating element systems and a refrigerant system fed with R12, respectively. Environmental chamber was equipped with a fan to perform the uniform temperature interior of the chambers. Temperature operating range of this apparatus is between -20 to 150˚C and the interior dimension of each chamber is 420×420×350 *mm*.

The rail system is used to move specimens between the cold and hot region. The temperature of the specimen was monitored with a Delta controller and PT100 sensors. Temperatures were measured with four thermocouple gauges; two sensors for measurement of the heating and cooling chamber temperature and two sensors for two sides of the specimen [19].

(a) (b)

Fig.2
Thermal cycling two chambers apparatus a) picture of the apparatus, b) schematic diagram of the apparatus.

3.3 Tensile test

The mechanical tests were conducted on a 50 *kN* hydraulic universal material tester (Fig. 3), and each data point indicated in the results is an average of three separate measurements. The specimens were tested in accordance with the ASTM D3039 standard at a crosshead speed of 2 *mm/min*.

Fig.3
Hydraulic universal material tester.

4 RESULTS
4.1 Results of tensile test

The nine different experiment groups according to the test plan (Table 2.) were performed using the design parameters combinations in the specified orthogonal array. Three specimens were fabricated for each of the orthogonal array. After thermal cycling, tensile tests were used to obtain the elasticity modulus and tensile strength. The average results and standard deviations values (SDV) of the tensile tests are reported in Table 4. It is observed that the specimens with $[0]_8$ layups have the maximum elastic modulus and tensile strength. Also, the specimens with the $[0/\pm45/90]_s$ layups have the minimum results.

Table 4
Average elastic modulus and tensile strength of the various test groups designed based on the Taguchi method.

Test Group	Elastic Modulus (*GPa*) (±SVD)	Tensile Strength (*MPa*) (±SVD)
Run-1	22.33 (0.74)	691.32 (0.83)
Run-2	13.71 (1.49)	400.83 (14.31)
Run-3	9.25 (0.41)	299.65 (12.61)
Run-4	23.16 (0.20)	711.36 (31.47)
Run-5	12.78 (0.12)	354.18 (6.10)
Run-6	11.91 (0.49)	253.89 (10.8)
Run-7	25.97 (0.13)	733.85 (6.67)
Run-8	13.04 (0.34)	385.01 (7.11)
Run-9	11.45 (0.08)	271.83 (4.61)

4.2 Analysis of variance (ANOVA)

Statistical analysis of the elastic modulus based on the Taguchi method is obtained using “MINITAB R17” [20] as the statistical software and ANOVA results are shown in Table 5. As shown in this Table, the stacking sequence is the main effective factor on the elastic modulus with 96% contribution. Also, the fiber volume fraction and number of thermal cycling are the next effective factors.

Similar analysis can be done on the tensile strength. Table 6 shows the variance analysis and the impact factor of each level of factors. Also, degree of freedom (DOF) and difference of the maximum and minimum effect value of the factors (Delta) are indicated in this Table. As shown in Table 6., the stacking sequence has the most contribution on the tensile strength.

Table 5
ANOVA for elastic modulus.

Factors	Level 1	Level 2	Level 3	Delta	DOF	Contribution (%)	Rank
Number of Cycles	15.09	15.95	16.82	1.73	2	1.49	3
Stacking Sequence	23.82	13.18	10.87	12.95	2	96.05	1
Temperature Difference (˚C)	15.76	16.10	16.00	0.34	2	0.06	4
Fiber Volume Fraction (%)	15.52	17.19	15.15	2.04	2	2.40	2
Total					8	100	

Table 6
ANOVA for tensile strength.

Factors	Level 1	Level 2	Level 3	Delta	DOF	Contribution (%)	Rank
Number of Cycles	463.9	439.8	463.6	24.1	2	0.36	3
Stacking Sequence	712.2	380.1	275.1	437.1	2	99.02	1
Temperature Difference (˚C)	443.4	461.3	462.6	19.2	2	0.22	4
Fiber Volume Fraction (%)	439.1	462.9	465.3	26.2	2	0.40	2
Total					8	100	

4.3 Stress-strain curves

The stress-strain curves of the layups under different thermal cycling conditions are shown in Fig. 4-6. The stress-strain curve in longitudinal direction was linear elastic until breakage.

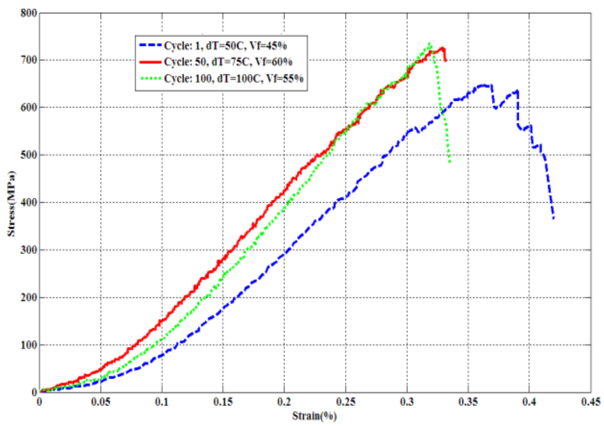

Fig.4
Stress-strain curves of the $[0]_8$ layups specimens under tensile test.

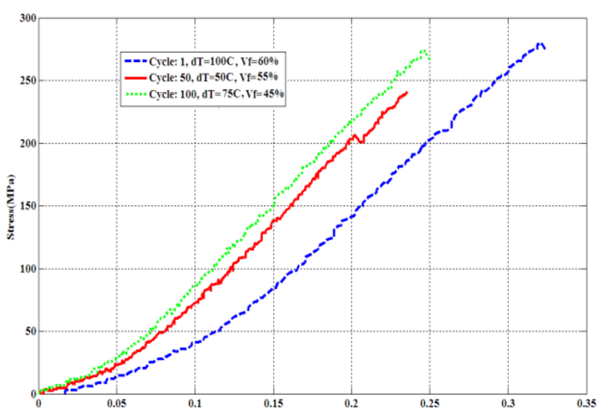

Fig.5
Stress-strain curves of the $[0/\pm45/90]_s$ layups specimens under tensile test.

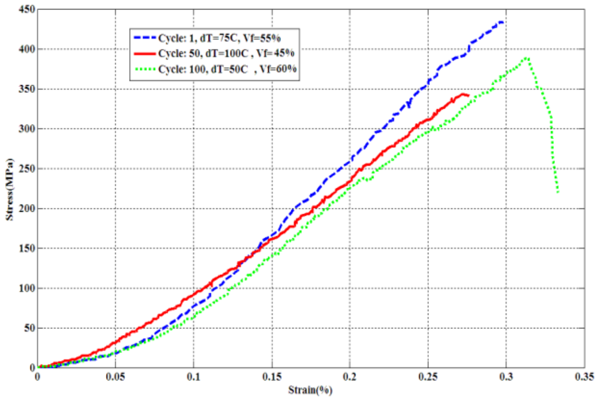

Fig.6
Stress-strain curves of the $[0_2/90_2]_s$ layups specimens under tensile test.

As shown in these figures, increment of the number of thermal cycles has been increased the tensile strength and elastic modulus of the $[0]_8$ layup. Despite the $[0]_8$ layup, the tensile strength and elastic modulus decreased at the $[0_2/90_2]_s$ layups. At the $[0/\pm45/90]_s$ layup the elastic modulus increased and tensile strength decreased, consequently. When the angle difference between lamina increased, the effect of thermal cycling is significant. As shown in Fig. 4, there is no angle difference at $[0]_8$ lamina. So, thermal cycling caused to curing the laminate and strength it. On the other hand, there is 45˚ angle difference between lamina at the $[0/\pm45/90]_s$ layup and this difference caused the crack between the plies and reduced restraint on the zero plies. Cracking in the inner plies had significant influence on performance of the laminates. So, the tensile strength decreased at this layup. Also, thermal cycling increased the modulus, probably because of the relief of internal stresses.

The obtained results are compared with available results. Lafarie-Frenot et al. [11] compared damages according to the location of the plies in the stacking sequence of the laminate. Microscopic observations conducted on two perpendicular sides of the samples. Throughout thermal cycling tests, the observations of the free edges have shown three types of damages: permanent deformation of the matrix due to its shrinkage, debonding between fibers and matrix and matrix cracking.

The largest decrease in properties occurred for $[0_2/90_2]_s$ layups with the highest proportion of angle difference because of the reduced restraint on the zero plies when the cross-ply layer is cracked. As the number of 90 layers decreased the temperature at which cracking began and the crack density increased, though the ply distribution did not have much effect on the results.

Moreover, the observations of the transverse cracks in the various laminates using *X*-ray technique [9] showed that the angle orientations of the cracked ply as well as of the adjacent ones are involved in the crack path, the crack opening and the fracture topography. Indeed, the transverse cracks observed in the central layer of the cross ply laminates are rectilinear; whereas those observed on the 45˚ laminates, present a more tortuous aspect. Such a tortuous path might lead to a higher length of a transverse crack in a 45˚ ply than in a 90˚ one that could explain the delay of transverse cracking observed on the curves of crack density.

4.4 Analysis of failure modes

Thermal cycling tests induce various types of damages, depending to a certain extent on the orientation and the fiber volume fraction of the composite, but mainly on the environment of the specimen. Depending on the experimental conditions (stacking sequence, environment, etc.) and the number of cycles, either none, or some, or all these types of damages could be observed. Failure region study of the specimens showed that stacking sequence is the main effective factor on the failure mode changes. Significant change could be observed in the fracture surface of the $[0]_8$, $[0_2/90_2]_s$ and $[0/\pm45/90]_s$ layups. As shown in Fig. 7, long splitting fiber breakage is the dominated failure mode in the $[0]_8$ layups. Also, lateral fiber breakage and fiber matrix debonding are the two dominate failure modes of the $[0_2/90_2]_s$ layups. There is a large amount of matrix-fiber de-bonding near the gauge area of this layup. Furthermore, angled breakage is occurred in the $[0/\pm45/90]_s$ layups. As shown in other works [11, 19], transverse cracks are visible in the plies where the fiber direction is not parallel to the observation plane. These cracks are propagating through the matrix but with a direction guided by the fibers. Transverse cracks in 90˚ plies surrounded by 0˚ plies appear globally perpendicular to the layer interfaces. It is found from a visual inspection of the damage surfaces that the failure regions (consist of debonding area and fiber breakage area) decrease when the fiber volume fraction is decreased (Fig. 8). The major difference between different fiber volumes fractions is that bigger failure region of which is obvious at high volume fraction.

Studying the failures regions of the composite specimens at different thermal cycling condition is another interesting subject that is studied. As is shown in Fig. 9, the surface matrix loss is observed at the high thermal cycles. This increase was also observed and studied by Lafarie-Frenot [13] on both the unidirectional and angle ply of the carbon/epoxy composite material. As shown in this work [13], the number of thermal cycles increased, transverse matrix cracks increased steadily in number and length. Whereas on the edges cut at ±45˚ to fibers, matrix cracking was detected only in the external layers. Compared to the cracks observed on the edges perpendicular to fibers, those appear slightly slanted, much less opened and, especially, more sinuous.

Temperature difference increment makes the surface matrix loss more significant. This surface matrix loss can be an initial region for the matrix debonding and crack propagation. The results showed that the mode of failure is not changed under thermal cycling.

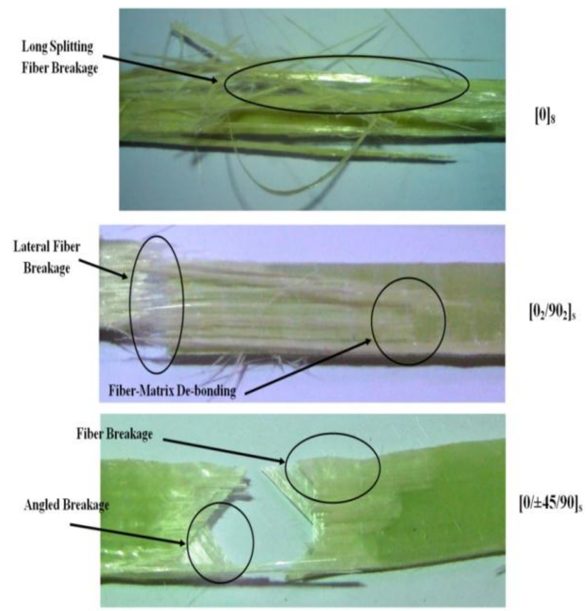

Fig.7
Failure regions of the $[0]_8$, $[0_2/90_2]_s$, $[0/\pm45/90]_s$ layups.

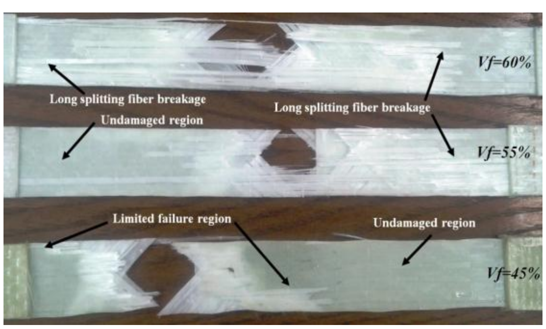

Fig.8
Failure regions of the $[0/\pm45/90]_s$ specimens at different fiber volume fraction.

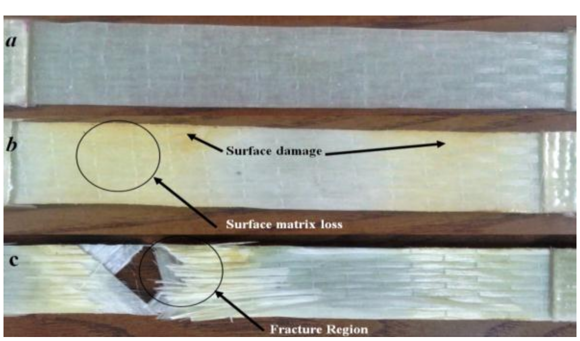

Fig.9
Surface of the $[0/\pm45/90]_s$ layup under thermal cycling condition (V_f=45%, dT=100˚C, N=100): a) before thermal cycling, b) after thermal cycling, c) after tensile test.

5 CONCLUSIONS

In this article, fracture behavior of glass/epoxy laminated composite subjected to thermal cycling at different temperature differences, fiber volume fraction and stacking sequences were investigated experimentally. A statistical approach was utilized to identify the main effective factors and contribution of them on the mechanical degradation of the PMCs. The orthogonal array of L9 type Taguchi method was used to reduce the number of the experiments to the nine run. Thermal cycling test are done using a developed apparatus. Based on the experiment design table, elastic modulus and tensile strength are obtained using the tensile test. The ANOVA is applied to obtain the contribution of the each factor. The following conclusions can be drawn:

- The Taguchi method is suitable approach to reduce the number of experiment especially for phenomena like thermal cycling with different effective factors.
- Long splitting fiber breakage, lateral fiber breakage and angled breakage are dominate failure mode of the $[0]_8$, $[0_2/90_2]_s$ and $[0/\pm 45/90]_s$ layups, respectively.
- The $[0]_8$ layups is strongest than other layups under thermal cycling degradation. The maximum elastic modulus and tensile strength change observed in the $[0/\pm 45/90]_s$ layup.
- Thermal cycling and fiber volume fraction are the next effective factors on the elastic modulus and tensile strength, respectively.
- The visual inspections showed when the fiber volume fraction is decreases the damage surfaces of failure regions (consist of debonding area and fiber breakage area) decreases. So, the wider failure region of matrix is observed at the high fiber volume fraction.
- The surface matrix loss is observed at the high thermal cycles. Also, temperature difference increment makes the surface matrix loss more significant. This surface matrix loss can be an initial region for matrix debonding and crack propagation.
- When the angle difference between lamina increased the effect of thermal cycling is significant. Thermal cycling caused to curing and stress relieving the laminate and strength of the $[0]_8$ laminate (no angle difference).
- On the other hand, thermal cycling caused the crack between the plies and reduced restraint on the zero plies at the $[0/\pm 45/90]_s$ layup (45˚ angle difference). So, the tensile strength decreased at this layup. Also, the elastic modulus increased because of the relief of internal stresses.
- The largest decrease in properties occurred for $[0_2/90_2]_s$ layups with the highest proportion of angle difference because of the reduced restraint on the zero plies when the cross-ply layer is cracked.

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