International Journal of Emerging Technology and Advanced Engineering
Website: www.ijetae.com (ISSN 2250-2459, ISO 9001:2008 Certified Journal, Volume 6, Issue 9, September 2016)

Fatigue Behavior of Carbon/Epoxy Composites Produced by VARTM: Thermography, Frequency and Stress Level

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***Abstract* — Carbon fiber/epoxy composites have grown significantly for industrial applications, especially in the structural elements. In this work, laminated composites were fabricated by VARTM – Vacuum Assisted Resin Transfer Molding. This process complies with the requirements for structural application and presents a significant cost reduction when compared to the usual processing technique using autoclave. This paper aims to study the influence of the frequency and stress level on the fatigue life for in-plane shear properties of a carbon fiber/epoxy composite produced by VARTM. Thermograph monitoring techniques were used to characterize the increase of temperature during the fatigue tests. The results provide information about composite fatigue behavior using frequencies of 12, 24 and 50 Hz. The residual strength was determined after cyclic tests up to 10^6 cycles. Results showed that an increase in frequency could lead into overheating and premature failures, reducing the fatigue life.**

***Keywords* — Composites, Carbon/epoxy, Fatigue frequency, Thermography, VARTM**

I. INTRODUCTION

The development of new materials has marked the advances of society throughout history. Composite materials are a group of materials, which has been widely used and is continuing developed for a broad range of present-day industrial applications[1]. They are lightweight and strong, providing a winning combination of properties that propels composites into new applications in the manufacturing industry[2], especially on aeronautical industries.

A current trend in advanced composite materials, especially carbon fiber reinforced polymer matrix composites, is to extent their use from aeronautic and defense industries to other industry segments, such as automotive[3], wind turbine blade[4] and civil infrastructure[5]. However, it is necessary significant improvements in processing efficiency and a sharp cost reduction of composites in order to foment their usage in large scale. This addresses the development of new processing technologies and materials[6].

Among the various processing technologies for composites, the Vacuum Assisted Resin Transfer Moulding (VARTM) is being widely used for moulding complex composite structures because at relative low cost[7,8]. VARTM can also be an alternative to high cost prepreg technology if it is able to achieve lower than 2% volume of void defects in composites, which matches aeronautic standards[9]. The presence of voids in composites is deleterious to its mechanical properties such as tensile, flexural, and fatigue strengths[10-14].

The VARTM is a composite manufacturing process technique in which the surface of the mould gives the structure or component geometry. The reinforcement material is layered over the mould surface, and the top layer is the flexible vacuum bag sheet. Vacuum is applied in order to compact the layered reinforcement material and to allow resin to flow along the reinforcement[15].

Figure 1 shows the auxiliary materials required for resin infusion, and the processing stages[16]. Initially, layers of fibrous reinforcement are laid up on the mould surface to form a preformed reinforcement. A peel ply is then laid over the preform, avoiding the contact of the consumables with the part that will be moulded, as well as providing a near flat regular surface on the side of the part not in contact with the mould. A distribution media can be laid over the peel ply. This enhance resin flow throughout the preform regardless the in-plane permeability. Once inlet and vent tubes are right placed, the mould is closed using a vacuum bag sheet and sealant tape. Once that cavity is sealed, the inlet tube is clamped and vacuum is applied at the vents, which is referred in this paper as ‘pre-filling.’

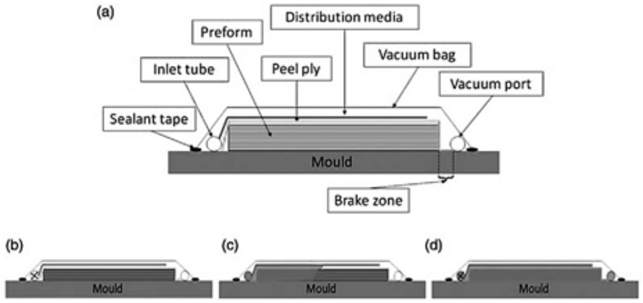

Figure 1 - Stages in the resin infusion process. (a) Lay up (b) Pre-filling (c) Filling (d) Post-filling[16]

At the end of pre-filling, the inlet is opened and the resin flows through the preform. During the 'filling stage', resin pressure in the vacuum bag cavity varies in position and time. From the vent to the flow front, pressure is equal to the vacuum pressure applied at the vent. Within the impregnated portion of the preform, the resin pressure varies from vacuum at the flow front to near atmospheric pressure at the inlet. Once the resin front reaches the end of the preform, the inlet is clamp off. However, the inlet can also be turned into a vent. The 'post-filling' stage involves removal of excess resin, and allows resin pressure to equilibrate within the vacuum bag cavity[16-19]. Once the resin is fully cured, the vacuum is released and the part is removed from the mould and consumables are discharged.

An important issue for product designers and material developers is the long-term behavior of composite materials. Therefore, fatigue life of composite materials plays the role on the characterization grid schedule[20]. Composite materials are, in general, less sensitive to fatigue than metallic materials. On the other hand, due to their anisotropic and non-homogeneous characteristics, the fatigue behavior of composites is more complex. In composite materials, fatigue life is dependent on materials design features such as fiber and matrix selection, fiber orientation, fiber/matrix interface, fiber volume content and porosity[21].

Testing parameters may also affect the fatigue life of composites. Testing frequency, stress ratio and stress level may cause overheating in the material and lead to premature failures, decreasing the fatigue life[21].

Temperature rises during static mechanical tests but is more evident during dynamical fatigue tests, mainly due friction and hysteresis of matrix material. Viscoelastic behavior of matrix material also has a great influence on temperature rise during fatigue tests[22]. Geometric parameters, size or shape of testing samples also can lead to different temperature rise, which justify some different published results. Consequently, the correct definition of testing parameters and specimen geometry is critical for fatigue analysis of the material.

Despite these drawbacks, the fatigue performance of composite materials is a topic of very high scientific and industrial interest. Many interesting works in the field of fatigue for composite materials can be found in the literature. For instance, Case and Reifsnider[23] and Talreja[24] provided an excellent overview of the issue of fatigue in composite materials. Both references explain the fatigue life diagrams and analyze the damage and failure mechanisms in different types of composite materials, i.e., laminate composites, woven composites, unidirectional, etc. They present in detail various fatigue life prediction concepts and models, giving a parallel with the selected experimental data. Good predictions for fatigue life were obtained by several authors[25-27] by relating hysteresis loop responses and thermographic monitoring techniques. Therefore, for a successful fatigue life or residual stress prediction models, the test parameters must be accurately defined to avoid misleading results.

The goal of this paper is the study of the influence of frequency and stress levels in fatigue life of carbon/epoxy composites fabricated by VARTM. Fatigue tests were performed on in-plane shear mode, which is highly influenced by matrix, and monitored by an infrared camera.

II. EXPERIMENTAL

In this work an epoxy resin system Araldite/Aradur 5052 from Huntsman® was use as matrix. A GA045 (6K, 149 g/m2) unidirectional dry carbon fiber tape, from Hexcel®, was used as reinforcement. The composite laminates $[+45/-45]_{3S}$ were produced by VARTM (Vacuum Assisted Resin Transfer Moulding) process. The 12 layers with dimensions of 30 cm by 30 cm were placed on the tool. A layer of peel-ply is laid over the preform, allowing for easy separation of the part from the consumables (include vacuum bag, distribution medium, resin and vacuum tubes, etc.). The distribution medium is positioned over the peel-ply to enhance resin flow. Once resin inlet and vacuum vent tubes are in place, the mold is closed using a vacuum bag sealed with sealant tape. A vacuum is applied (89,74 kPa) to compact the reinforcement material and to drive the resin through the porous fabric preform. The preform was infused with the resin system with viscosity between 550-650 mPa.s. The injection line is closed after the fabric was fully saturated. The implemented curing process was 4 hours at 80 ° C. Once the resin is fully cured, the vacuum is released and the part is lifted off the mold and separated from the consumables.

Sixteen points for measuring thickness over the composite plate were set at regular intervals using a Mitutoyo Coordinate Measure Machine model B 251. The fiber volume fraction and void volume content of the resulting molded composite was determined using the ASTM 3171 Standard. The samples for measuring volume fractions (fiber, resin and voids) were taken from the composite plates at specific locations, near the resin inlet, in the center, and near the vacuum outlet.

Quasi-static and fatigue tests were carried out using in-plane shear test specimens, prepared according to guidelines from the ASTM D3518 Standard. Five specimens were tested in order to obtain the maximum in-plane shear strength value. Self-heating measurements were conducted on the carbon fiber/epoxy composites. The specimens were monitored by an infrared camera, FLIR® E30, during the fatigue cycling to monitor the heating profile caused by hysteretic heating.

All fatigue tests were performed under load control condition, by a sinusoidal waveform and tension-tension cyclic loading, at frequencies of 12 Hz, 24 Hz and 50 Hz. A stress ratio of $R = 0.1$ was used and tests were done up to a maximum of 1.000.000 cycles. The stress levels were set at 40%, 45% and 50% from the maximum in-plane shear strength value.

The testing parameters were chosen based on literature and preliminary tests. As several authors use frequencies between 10 and 15 Hz in laboratory experimental tests[28-30], it has been selected the initial value of 12 Hz. Preliminary tests for frequencies above 50 Hz showed that the sample heating is too fast and the fail occurs within a few seconds. Thus, it was defined 50 Hz as a limit value and 24 Hz as a middle value. The stress levels used were also determined from preliminary tests, showing that the values around 45% of the maximum in-plane shear strength value led to a sample failure.

Two samples were tested for each testing condition. The residual strength was determined after cyclic tests up to 10^6 cycles on surviving coupons. The maximum number of cycles 10^6 covers many applications areas including aeronautical field. Typical composite parts, like pressure bulkhead, wings tub, or landing gear doors for commercial aircraft are designed for two or three lives, no more than 10^6 cycles[21].

All tests were carried out under controlled environmental conditions, 23±5 °C and 50±10% relative humidity. A computer-controlled servo-hydraulic testing machine INSTRON® 8874 with hydraulic jaw grips was used.

III. RESULTS AND DISCUSSION

The measurement of thickness variation throughout the composite specimen was conducted in order to evaluate the material homogeneity, process robustness and quality of the VARTM process. Resulting averages and standard errors for all measurements are reported in Table I for the infusion processes.

Due to the pressure gradient used during the VARTM process, there is a trend on thickness variation on the composite plate. This drawback can cause low dimensional tolerances due to the inherent thickness gradient[31,32].

TABLE I
THICKNESS MEASUREMENTS OF VARTM LAMINATES

Infusion	Thicknes Average [mm]	Standard Error [mm]	Standard Error [%]
1	2,267	0,039	1,72
2	2,245	0,069	3,07
3	2,368	0,074	3,12
4	2,223	0,076	3,42
5	2,193	0,047	2,14

The higher standard error found was 3.42 % of the average value in infusion 4. The measured thickness results are in agreement with the literature data. Li et al.[18] obtained standard error values corresponding to 2.65 %; 2.98 % and 3.64 % in relation to the average value for composite laminates produced by VARTM. No significant differences were observed between the points near the resin inlet and the points near vacuum outlet.

The average value of fiber, matrix and void volume fraction in the composites are shown in Table II.

TABLE II
FIBER, MATRIX AND VOIDS VOLUME FRACTION

Infusion	Fiber Volume (%)	Matrix Volume (%)	Voids Volume (%)
1	52,2	46,3	1,5
2	52,2	46,5	1,2
3	50,2	48,5	1,4
4	53,7	44,8	1,5
5	53,9	44,7	1,4

The values obtained are in agreement with the structural requirements[33,34], which are fiber volume greater than 50 % and voids below 2 %, demonstrating the good quality of the VARTM process.

The quasi-static test results shows that the average of maximum in-plane shear strength value obtained was 65,45 ±1,04 MPa, corresponding to a range 185.500 – 190.500 με. The shear stress/shear strain curve is shown in Figure 2.

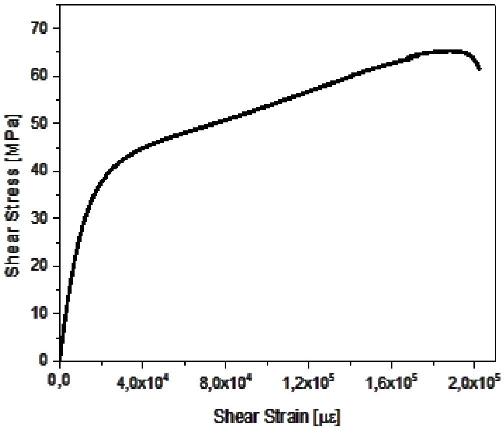

Figure 2 - Shear stress/shear strain average curve

All results of the fatigue tests are summarized in Table III, including the temperature values at rupture point and number of cycles for each coupon.

Analyzing the fatigue tests performed at frequency of 12 Hz, it was observed that the samples with 45 and 40 % of tension level did not fail at 10^6 cycles and presented maximum temperature of 45 °C during the fatigue tests. In order to evaluate the fatigue process in the materials, a residual strength test was performed, and the results are shown in Table IV.

TABLE III
TEMPERATURE AND NUMBER OF CYCLES FOR EACH FREQUENCY TEST AT RUPTURE POINT

Frequency Test of 12 Hz		
Stress Level [%]	Temperature [°C]	Number of Cycles
40	-	-
40	-	-
45	-	-
45	-	-
50	101,5	15.000
50	114,5	26.000
Frequency Test of 24 Hz		
Stress Level [%]	Temperature [°C]	Number of Cycles
40	114,8	26.000
40	118,0	61.000
45	116,0	5.893
45	114,8	5.922
50	104,4	2.794
50	113,3	2.960
Frequency Test of 50 Hz		
Stress Level [%]	Temperature [°C]	Number of Cycles
40	131,4	4.193
40	129,4	5.340
45	124,9	2.385
45	127,8	3.147
50	114,4	1.944
50	113,8	2.096

TABLE IV
RESIDUAL STRENGTH FOR SURVIVAL SAMPLES UP TO 10^6 CYCLES

% Stress	**Residual Strength (MPa)**
40	70,69
40	66,62
45	69,82
45	68,20

According to the residual strength results, the fatigue process in lower frequency (12 Hz) has a minimum effect on the material when submitted to lower stress levels even after being loaded for an extended period.

The residual strength values were slightly higher than the average results in static tests. This behavior was also found by Shokrieh[35], Ancelotti[36] and Awerbuch[37]. Shokrieh[35] noted an increase between 8% and 13% of residual strength in relation to the static tensile strength for a unidirectional carbon/epoxy composite when subjected to fatigue with stress values between 60% and 80% of the rupture tension. Ancelotti[32] showed similar residual strength behavior for composites with porosity below 2% for tensile and in plane shear tests, wherein the behavior was more significant for in plane shear. The increase in residual strength values can be explained by the tensions redistribution in the composite, which generates a final mechanical strength dominated by the carbon fiber and no longer by the fiber/matrix system, increasing the residual strength[37-38].

Samples tested under stress level of 50% have failed at low number of cycles (<26.000 cycles). The temperature and cycles (log) curves for 12 Hz and 50 % of stress level are shown in Figure 3.

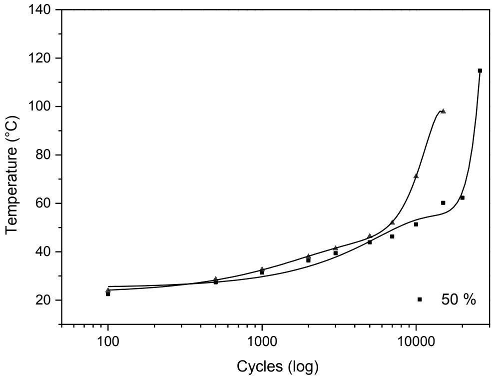

Figure 3 - Temperature monitoring in function of number of cycles at frequency of 12 Hz (50% stress level)

As noted, after a short number of cycles (15.000 and 26.000) the temperature increases abruptly (from 23 to 101,1 and 114,5 °C), indicating the damage process and final rupture of samples. This result shows that in-plane shear strength failure in composites can lead to a catastrophic failure and that temperature measurement can be a parameter for fatigue failures.

The Figures 4 and 5 present the evolution of sample temperature at three different stress levels along fatigue cycling performed at 24 Hz and 50 Hz, respectively.

It can be noted a relation among temperature increase, stress level and fatigue life. Increases in tension level leads to a reduction of fatigue life and causes overheating. The same behavior was noted for 24 and 50 Hz fatigue frequencies. The number of cycles tolerated by the material before fail, as expected, reduces when stress gradually increases.

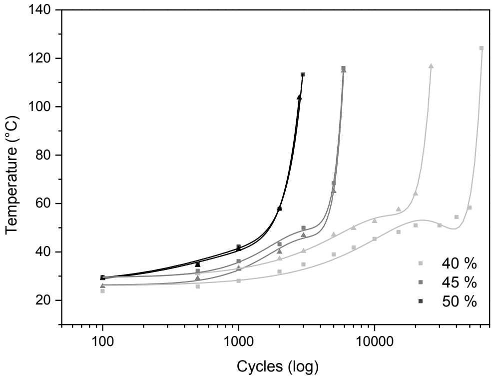

Figure 4 - Temperature monitoring in function of number of cycles at frequency of 24 Hz

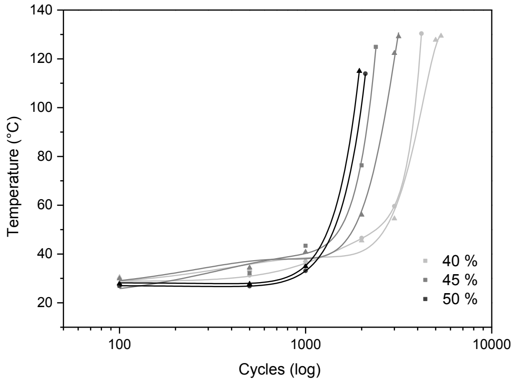

Figure 5 - Temperature monitoring in function of number of cycles at frequency of 50 Hz

As observed in Figures 4 and 5, for the same groups of tension and the same frequencies; even if minor, is possible to see how the temperature, when the rupture of the sample material occurs, increases while number of cycles raises too. This fact might be associated to the growth and expansion of micro cracks into samples structure while the material is loaded. For this reason, in the samples with longer life the crash temperature should be higher than the samples with short life.

In the samples tested on frequencies of 50 Hz, in Fig. 5, is noted a premature rupture of the specimen, which may be related to worst heat dissipation in a shorter period.

The Figure 6 shows clearly the relation between each tension level at frequencies of 12, 24 and 50 Hz in fatigue life. The tendency is decreasing the fatigue life as the frequency test and tension level increases.

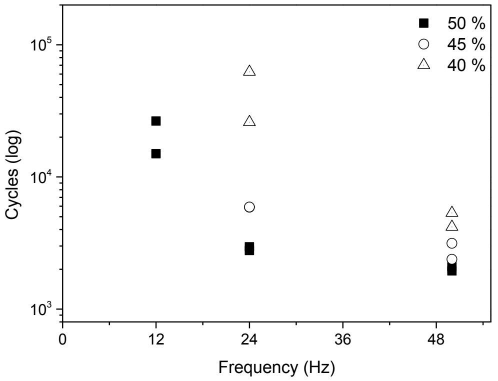

Figure 6 - Number of cycles in function of frequency tests

The rise of temperature can be attributed to a number of heat sources including friction and the viscoelastic behavior of the matrix[39]. Thermographic images were obtained in order to evaluate the frequency test influence on the composite fatigue life, including the correlation between the temperature and number of cycles. The Figure 7 shows the thermographic images for the coupons tested at 12, 24 and 50 Hz, with 50 % of stress level, since for 40 and 45 % the composites did not fail in the tests with 12 Hz of frequency.

Two measurement points (the center and the warmest region) were positioned on each coupon. For the analysis of the heating process were chosen three temperature values to obtain the thermographic images, 35° C, 70° C and at the rupture point (approximately 100° C). It was adjusted the starting point zero when the specimen reaches 35° C. The time to reach 70° C and the rupture point were calculated and are shown in Figure 7.

Significant hysteresis effect occurs more sensitively at 50 Hz. Temperature monitoring during fatigue cycling can be used to predict failures in composites. In the fatigue test at 12 Hz frequency the time required for the specimen to break was 14 minutes and 38 seconds; while at 24 and 50 Hz the rupture times were 2 minutes and 56 seconds respectively.

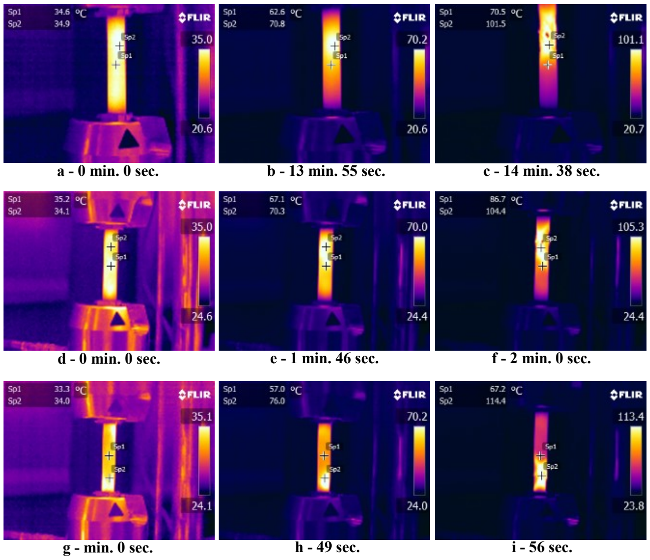
a - 0 min. 0 sec.
b - 13 min. 55 sec.
c - 14 min. 38 sec.
d - 0 min. 0 sec.
e - 1 min. 46 sec.
f - 2 min. 0 sec.
g - min. 0 sec.
h - 49 sec.
i - 56 sec.

Figure 7 - Thermographic images for the coupons tested at 12 (a, b, c), 24 (d, e, f) and 50 Hz (g, h, i), with 50 % of stress level at different temperatures

IV. CONCLUSIONS

The VARTM process was satisfactory, manufactured composites reached the requirements for structural application, fiber volume greater than 50 % and voids below 2 %.

The establishment of fatigue testing parameters, such as testing frequency and stress levels are fundamental to avoid incorrect results interpretation. While adoption of lower frequencies are time consuming, higher frequencies at high stress levels can lead to overheating and premature failures.

Residual strength results at lower stress (40 and 45% from the maximum in-plane shear strength value) and frequency of 12 Hz indicate a minimum fatigue effect on the material. According to the analyzed parameters, it is possible to conclude that the frequency value of 12 Hz did not cause overheating.

Increases in tension level lead to a reduction of fatigue life and cause overheating. The same behavior was noted for 24 and 50 Hz fatigue frequency. Significant hysteresis effect occurs more sensitively at 50 Hz. Temperature monitoring during fatigue cycling can be used to predict failures in composites.

Acknowledgment

The authors gratefully acknowledge FINEP (Project 01.13.0169.00 315/12) by financial support.

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