Available online at www.sciencedirect.com
ScienceDirect
Procedia Engineering 2 (2010) 341–348
Procedia Engineering
www.elsevier.com/locate/procedia

Fatigue 2010

Carbon fiber non-crimp multi-axial reinforcement and epoxy mono-component system composite: fatigue behavior

M.O.H. Cioffi[a], G. Volpiano, H.J.C. Voorwald[a]*, M.C. Rezende[b],

[a]*Fatigue and Aeronautical Materials Research Group*-DMT/FEG/UNESP,
Av. Ariberto Pereira da Cunha, 333 – CEP 12516 410, Guaratinguetá /SP – Brasil.
[b]Divisão de Materiais /IAE/CTA. Praça Marechal Eduardo Gomes, 50 Campus do CTA - Vila das Acácias 12228-904
São José dos Campos/SP - Brazil

Received 28 February 2010; revised 9 March 2010; accepted 15 March 2010

Abstract

Fiber reinforced polymer composites have been widely applied in the aeronautical field. However, composite processing, which uses unlocked molds, should be avoided in view of the tight requirements and also due to possible environmental contamination. To produce high performance structural frames meeting aeronautical reproducibility and low cost criteria, the Brazilian industry has shown interest to investigate the resin transfer molding process (RTM) considering being a closed-mold pressure injection system which allows faster gel and cure times. Due to the fibrous composite anisotropic and non homogeneity characteristics, the fatigue behavior is a complex phenomenon quite different from to metals materials crucial to be investigated considering the aeronautical application. Fatigue sub-scale specimens of intermediate modulus carbon fiber non-crimp multi-axial reinforcement and epoxy mono-component system composite were produced according to the ASTM 3039 D. Axial fatigue tests were carried out according to ASTM D 3479. A sinusoidal load of 10 Hz frequency and load ratio R = 0.1. It was observed a high fatigue interval obtained for NCF/RTM6 composites. Weibull statistical analysis was applied to describe the failure probability of materials under cyclic loads and fractures pattern was observed by scanning electron microscopy.

Keywords: Shot peening, Residual stress;4340 Steel; Fatigue, HVOF process

1. Introduction

Fiber reinforced polymer introduces specific better properties, typically, mechanical behavior, however, in many cases, the wear resistance should be improved. It is known that polymers present low surface friction coefficient during dry sliding, which confer for this material the role of solid lubricant [1].

Polymer matrix composites have relatively lower thermal resistance when compared to the metals and ceramics [1], which might be considered a problem in applications where high surface friction resistance is required. On the other hand, polymer composites reinforced with carbon fibers provide a good thermal conductivity and, therefore, the surface friction coefficient is less affected during the sliding process [2].

Due to the crosslinking characteristic of epoxy resin, thermosetting polymer matrix is mechanically stronger than thermoplastic polymer matrix. Considering that epoxy presents relatively low impact resistance, it should not be

* Corresponding author.
E-mail address: voorwald@feg.unesp.br

1877-7058 © 2010 Published by Elsevier Ltd.
doi:10.1016/j.proeng.2010.03.038

used as a sliding part, where the high wear resistance is required. The use of reinforcements effectively increases the wear capability of the epoxy resin [2].

Woven fabric advanced fiber reinforcements present fairly large advantages in applications where the material is subjected to sliding and surface friction loads due to fibers complexity, which can impose adequate characteristics to the performed structure [3]. Friction characteristics of carbon fiber reinforced polymer composites are strongly correlated with the type and resistance of interfacial bonds, the shear and the rupture of rubbed materials inside and around the contact region, and finally of the real contact area [4].

Mathew et al. [2] show a comparison between biaxial, triaxial and quadriaxial preforms using some types of thermosetting resins as matrices. To identify the best reinforcement/matrix combination tribological properties are analyzed. It was observed that epoxy resin reinforced with quadriaxial carbon fabric presents low thermal expansion with a friction coefficient against steel equal to 0.60 in dry condition and 0.30 in lubricated condition respectively, which indicates for this material high load bearing capability [3].

Fatigue is a very critical loading mode for laminates, because under cyclic loads failure occurs by the initiation and growth of a crack (delamination) at loads far away from the strength of the material. Generally, conventional ''Wöhler'' curves or S–N (S = stress amplitude or maximum stress, N = load cycle number) curves or hysteresis measurements are used to characterize the behavior of laminates under cyclic loads [5]. The generation of S–N-curves is very time-consuming and a large number of test specimens is needed; nevertheless, they only allow failure criteria for cyclically loaded components to be inferred and therefore enable the definition of allowable stresses as a function of the number of load cycles.

1.2. Weibull distribution

The presence of voids presence is a problem that occurs during composites fabrication process. These voids can decrease the composites strength whereas act as stress concentrators [3]. Composites defects are inherent to these materials and make difficult the mechanical behavior study if compared to the metals. In such case, a fracture event will depend on defect size and orientation [4]. Fatigue evaluation data through Weibull distribution can estimate safe life while the material is submitted cyclic load besides defects types and distribution present in the composite [5-7]. The obtainments of

Weibull parameters allow knowing the fatigue behavior before the use of material. The Weibull statistic is based on the fact that the current is not stronger than the weakest link. In composites, the weakest links are defects like voids, dry wide and twist fibers. Waloddi Weibull (1951) proposes the distribution to describe the failure probability of materials under cyclic loads, according to the equation 1 [8]:

$$F(x) = 1 - \exp\left[-\left(\frac{x-\gamma}{\eta}\right)^{\beta}\right] \qquad x, \beta, \eta \rangle 0 \tag{1}$$

where:

β = shape parameter or slope; γ = location parameter;

η = scale parameter; x = cycle number.

In fatigue tests it is usual to consider that g = 0, because the material is subject to failure since the beginning of the test. Mathematically, it can be written as Equation 2:

$$F(x) = 1 - \exp\left[\left(\frac{-x}{\eta}\right)^{\beta}\right] \tag{2}$$

From Equation 1 the scale parameter h indicates the 63% failure probability. For *x* = h, Equation 2 becomes:

$$F(x) = 1 - \exp\left[-\left(\frac{-\eta}{\eta}\right)^{\beta}\right] \quad \text{or} \quad F(x) = 1 - \exp[-1] \cong 0{,}63 \tag{3}$$

A mathematical modeling is required to produce a Weibull parameter. From equation 2:

$$\frac{1}{1-F(x)} = \exp\left[\left(\frac{x}{\eta}\right)^{\beta}\right] \quad (4)$$

Applying the function ln twice in Equation 5:

$$\ln\frac{1}{1-F(x)} = \left(\frac{x}{\eta}\right)^{\beta} \quad (5)$$

$$\ln\ln\frac{1}{1-F(x)} = \beta\ln\left(\frac{x}{\eta}\right) \quad (6)$$

$$\ln\ln\frac{1}{1-F(x)} = \beta\ln x - \beta\ln\eta \quad (7)$$

Considering the equation 8 as straight-line equation, y = ax + b:

$$y = \ln\ln\frac{1}{1\text{ - }F(x)}$$

$$a = \beta, \quad x = \ln(x), \quad b = \quad \beta\ln(\eta)$$

Calculated by n measured data. Using Wilks Methods (1942) [9] estimation of F(x) can be calculated according to Equation 4:

$$F(x) = \frac{i \quad 0.3}{n+0.4} \quad (7)$$

Where:
n = number of specimens measured;
i = failure element ranking (i = 1, 2, ..., n).

On case of a complete experimental data, F(x) can be expressed as like Method of Least Squares.

2. Experimental Procedure

Carbon fiber non-crimp quadriaxial orientated +45/0/-45/90 with a mirror 90/-45/0/+45, stitched by a polyester yarn, was used. This reinforcement is a combination of Hexcel intermediate modulus IM7-12k with a real weight 772g/m². Characteristically, this dry reinforcement presents a good flexibility due to the fiber architecture, which allows the production of a dense substantial material in thin plys and with modulus that can stand heavy tows. This reinforcement was impregnated with RTM6 that is a mono-component liquid epoxy resin system developed by Hexcel Composites, which can be used in service temperature range from –60°C to 180°C.

Fig. 1a shows the Carbon Fiber/RTM6 composite laminate produced by RTM technique and provided by Hexcel Composites and Fig. 1b is represents the impregnation map from C-san analysis, which shows a good impregnation of the reinforcement by the epoxy matrix.

Fig. 2a represents the axial fatigue specimen produced from the Carbon Fiber/RTM6 composite laminate based on ASTM D 3039 test method [23], where is indicated the length of gripping as twice than the width of the specimen. Tabs were considered unnecessary based on the valid failure mode obtained; in this case, it was observed that the failure location occurred in the middle part of the specimen with delamination and in an explosive form, as observed in Fig. 2b.

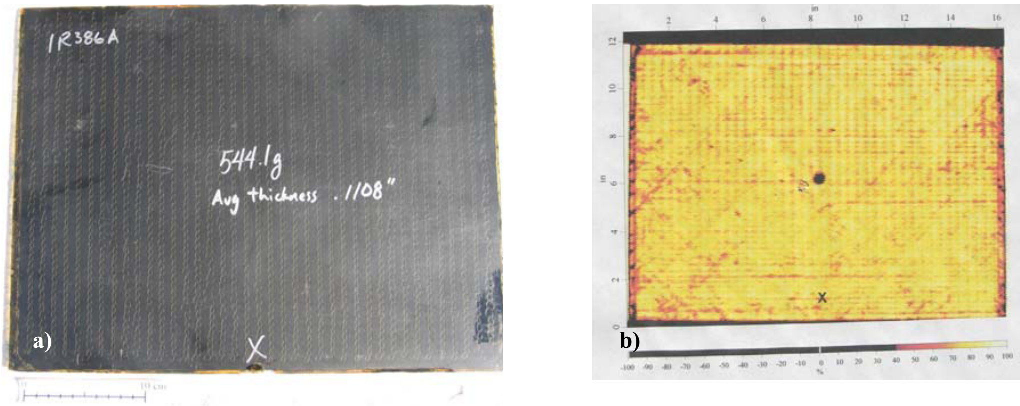

Fig. 1. Carbon NCF-RTM6 Composite a) Laminate; b) C-scan analysis of the laminate.

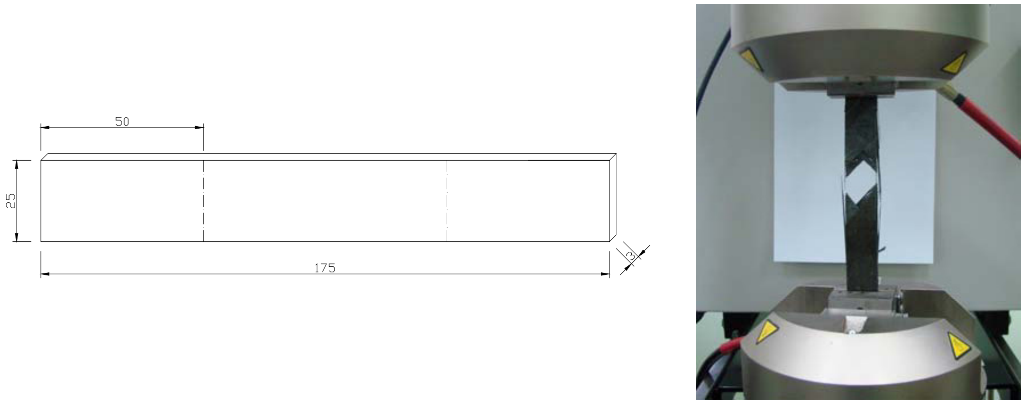

Fig. 2. Composite tensile test.

Axial fatigue tests were conducted according to the ASTM D 3479 [24]. Experimental tests were performed with a sinusoidal load of 10 Hz frequency and load ratio R = 0.1 in a universal testing machine INSTRON 8801. Herein, R denotes the ratio of the minimum (S_{min}) and the maximum (S_{max}) nominal axial stress components in one load cycle [16].

Fractures surfaces obtained from fatigue tests were analyzed in JEOL JSM 5310 scanning electron microscopy with tungsten filament operating at 15 kV, employing low vacuum technique in which images were obtained by scattering electron method. Samples were covered with gold layer to be observed in the MEV

3. Results and discussion

Axial fatigue tests results for NC2/RTM6 composite are shown in S-N curve indicated in Figure 3. This figure presents normalized SxN curves with the number of fatigue cycles represent as a function of the ratio between maximum applied stress and the ultimate tensile strength of the tested materials.

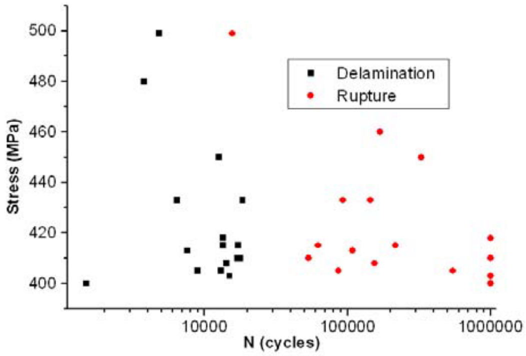

Fig. 3. Axial SxN Curve for the Carbon NCF

It is absolutely clear the high fatigue strength for the NC2/RTM6 composites. For this composite the applicable stress ratio belongs to the interval 72-60% during the fatigue. The SxN graph indicates the used maximum stress values between 499 and 400 MPa. A significant data scattering is also observed in this case, especially in the range from 400 to 420 MPa, lateral delamination process was observed for the specimens. This phenomenon occurs when the energy exceeds the matrix absorption capacity and the delamination process adjusts to the new system, which minimizes the accumulated stress until the fracture, this phenomenon is associated to the low toughness characteristic of the matrix.

Table 1. Weibull parameters of specimen IR 384 A

Stress (MPa)	%UTS	Cycles (N)	Shape Parameter, β	Scale Parameter, α (N), 63,2%
410	57	110639 216389 575016	0,86	2.082.204
482	67	1593 5676 8933	0,82	41.425
518	72	2410	0,63	101.653

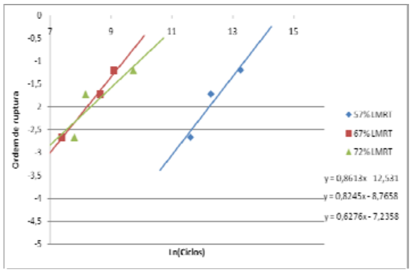

Fig. 4. Weibull parameters Curve – different tension

Shape parameter results showed values lower than 1, which mathematically means that this material present a failure ratio decreasing and should be associated to the higher tendency of material to fail at initial fatigue life phenomena called burn in, typical development of premature material dead. Low values of shape parameter put in evidence the high scattering of results in consequence of materials heterogeneity

Table 2. Weibull parameters of specimen IR 386 A

Stress (MPa)	%UTS	Cycles (N)	Shape Parameter, β	Scale Parameter, α (N), 63,2%
		217363		
412	57	256366	0,73	4.874.500
		1006370		
		25321		
445	67	36672	0,42	4.595.000
		38713		
479	72	17348	3,11	60.030

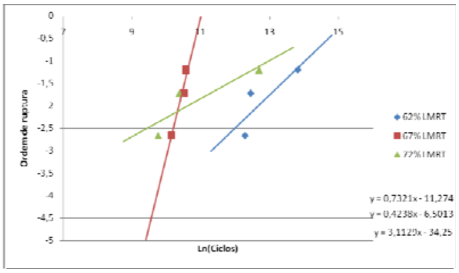

Fig. 5. Weibull parameters Curve – different tension

Table 3. Weibull parameters of specimen IR 388 A

Stress (MPa)	%UTS	Cycles (N)	Shape Parameter, β	Scale Parameter, α (N), 63,2%
		110639		
410	57	216389	0,86	2.082.204
		575016		
		1593		
482	67	5676	0,82	41.425
		8933		
518	72	2410	0,63	101.653

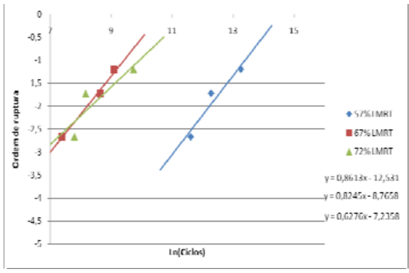

Fig. 6. Weibull parameters Curve – different tension

Table 3. Weibull parameters of specimen IR 390

Stress (MPa)	%UTS	Cycles (N)	Shape Parameter, β	Scale Parameter, α (N), 63,2%
		71405		
390	62	85341	5,40	116.000
		93544		
		17663		
420	67	31135	1,46	107.500
		48370		
450	72	16800	0,41	3.583.000

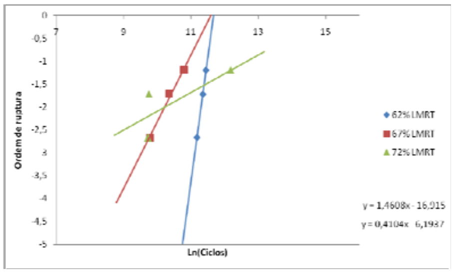

Fig. 7. Weibull parameters Curve – different tension

Some samples presented also β higher than 1, which is characterized like an ideal behavior for materials.

The figure 8 represent the impregnation map of composite material obtained by the C-scan ultrasound. The impregnation is acceptable considering that in aeronautical field 12% of attenuation is the limit. It can be observed also the heterogeneity of material associated to the red and yellow points, barriers to the wave during the passing through the laminate composites. Blue regions are associated to the satisfactory wave absorption and refraction.

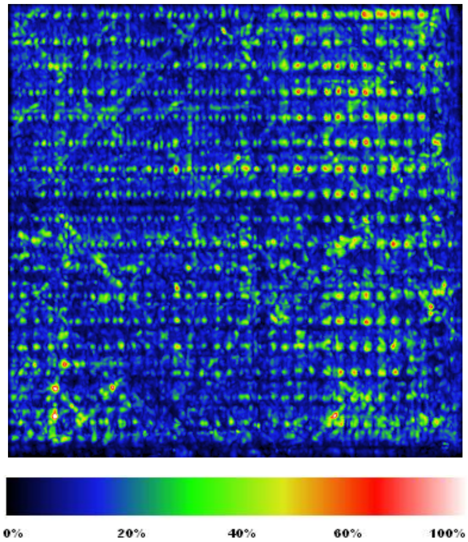

Fig. 8. Composite C-scan impregnation map

Polymer failure modes are associated to the molecular structure, specimen geometry, processing methods, tests temperature, loading ratio, stress state, reinforcement and plastification degree [11-12]. With respect to the polymeric matrix composites laminates, matrix behavior shows also load dependence. Tensile load in the transversal direction produces ruptures as observed in Fig. 9a and shear load introduces bands as cusps observed in Fig. 9b, similar those found in literature [11, 28- 29].

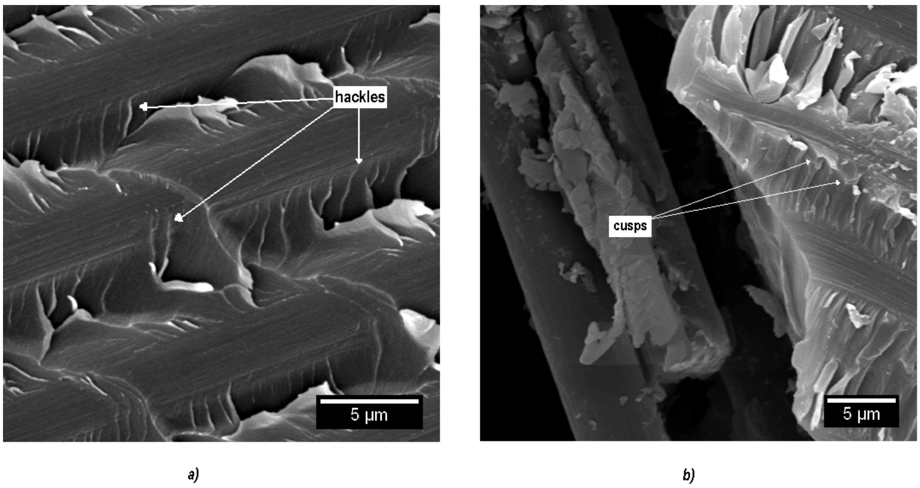

Fig. 9. Fatigue Fracture Surfaces. a) Matrix Rupture. b) Cusps formation

Fig. 9a also indicates high fiber/matrix adhesion and, at the same time, resin cohesive failure [15], associated to the low toughness of the matrix. Fig. 9b shows deformation of matrix and the presence of cusps, which were attributed to the shear load acting in composites during the fatigue test [16].

4. Conclusions

For maximum applied stress in the range of 405 MPa to 480 MPa, the ratio delamination cycles/fractures cycles presented values from 2 to 6%, with a maximum applied equal to 8% for 415 MPa.

Despite of the delamination occurrence, the number of cycles to fracture is, for each stress level, significantly higher than that associated to the delamination.

Weibull parameters show the tendency of premature failure of NC2/RTM6 composite material, confirmed to the heterogeneity of material in the impregnation map.

Fatigue fracture from scanning electron microscopy analysis showed typical events as fiber/matrix debonding, hackles and cusps characteristics of cyclically loaded polymer. It was also observed cohesive failure of resin, associated to the low toughness of the matrix

Author Artwork

Maria Odila Hilário Cioffi is an associated professor of DMT/FEG/UNESP -Univ Estadual Paulista, Guaratinguetá, Brazil and member of Fatigue and Aeronautical Materials Research Group.

Acknowledgements

Authors wish to express their gratitude to the Brazilian funding agencies CNPq through the processes numbers 472570/2006-4, 300233/2006-0, 301528/2006-0 and Fapesp through the process number 2006/02121-6 for the financial support. The collaboration of AMR/IAE/CTA and Hexcel Composites is also recognized.

References

[1] Larsen, T. O., Andersen, T. L., Thorning, B., Vigild, M. E., 2008.. *Wear* 2008; **264**: 857-68.
[2] M.T. Mathew, N. V. Padaki, L.A. Rocha, J.R. Gomes, R. Alagirusamy, B.L. Deopura, R. Fangueiro. *Wear* 2007;**263:**930–8.
[3] Myshkin, N.K., Petrokovets, M.I., Kovalev A.V., 2006*Tribol. Inter.* 2006;**38** (11-12): 910-21.
[4] Pandita, S. D., Huysmans, G., Wevers, M., Verpoest, I., 2007.. *Wear* 2008;**264**:638–47.
[5] G. Pinter; E. Ladstätter; W. Billinger; R.W. Lang. *Int J Fatigue* 2006; ***28***:1277
[6] J. Lee; C. Soutis. *Composites Science and Technology*, 2007;***67***:2015.
[7] B. Birgoren; M. H. Dirikolu. *Composites: Part B*, 2004;***35***: 263.
[8] A. B. Morais. *Composites Science and Technology*, 2006, ***66***, 2990.
[9] Y. Zhou; F. Pervin; V. K. Rangari; S. Jeelani. *Materials Science and Engineering*: *A*, 2006, ***426***, 221.
[10] E. V. Larve; R. Kim; D. Mollenhauer. *Composites Part A: Applied Science and Manufacturing*, 2007, ***38***, 174.
[11] W. Weibull. *Journal of Applied Mechanics*, 1951, ***18***, 292.
[10] S. S. Wilks. In Mathematical Statistics, 1942, **12**,91.
[11] M. Kashtalyan, C. Soutis. Comp.: Part A 2007: **38**:1262–9.
[12] S. Suresh. Fatigue of Materials. 2.ed. Cambridge: Cambridge University Press, 1998, 679p.
[13] E. Totry, C. González, J. Llorca. Comp. Sci. Tech. 2008:**68**:829-39.
[14] T. J. Vogler, S. Y. Hsu, S. Kyriakides. Inter. J. Sol. Struc. 2000;**37**:1765–91.
[15] P. Chalkley, A. Rider. Comp.: Part A 2003:**34**:341–8.
[16] A. Sjögren, L. E. Asp. Inter. J. Fat. 2002;**24:**179–84.