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STRAIN ENERGY RELEASE OF QUASI-STATIC DELAMINATION GROWTH RELATION WITH FIBRE VOLUME FRACTION, AND ACOUSTIC EMISSION

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Abstract. *Composite laminates have been widely employed in aeronautics and aerospace industry as a consequence of its high mechanical properties with a low specific weight. However, due to the anisotropic nature of composites, this material presents low strength to out-of-plane stresses, which makes them susceptible to delamination. Composites have specific features that present a great influence over its mechanical properties, such as fiber volume fraction (FVF). The FVF can be varied in order to comply with design concerns and reach desired mechanical properties or as a consequence of some manufacturing event that originates regions with different FVF. This research work aims to study the influence of FVF variation over the strain energy released during delamination growth. The tests were conducted in carbon fiber reinforced polymers matrix under mode I quasi-static and low rate cyclic loading to analyse the potential of acoustic emission (AE) technique in quantifying physical damage. The low FVF decreased the strain energy released, and the number of acoustic signals detected during crack propagation. AE technique presented a strong relation with the strain energy released during delamination growth. Therefore, AE technique showed a great potential to quantify physical damage in composites.*

Keywords: *Carbon fiber reinforced polymer, fiber volume fraction, delamination, acoustic emission, strain energy release.*

1. INTRODUCTION

Composite laminates have been widely employed in aeronautics and aerospace industry as a consequence of its high mechanical properties and low specific weight. The replacement of metals by composites, in general, reduces the structural weight, leading to operational cost and fuel consumption reductions, and moreover contributes to environmental impact reduction. However, composites present poor interlaminar strength as a consequence of lack of through-to-thickness reinforcement, making them susceptible to delamination process, which is the most observed damage mode in composites (Amaral; Alderliestes; Benedictus, 2018). Among all damage types of composites, delamination is the most severe, reducing the material strength and stiffness, which may lead to structural failure (Khan;

Alderliesten; Benedictus, 2014). Therefore, several researchers have worked to gain knowledge about delamination growth in composites (Amaral et al., 2015; Jones et al., 2017; Khan et al., 2014; Shahkhosravi et al., 2019; Yao et al., 2018).

Aiming to fully understand of damage propagation mechanisms in composite structures, numerous health monitoring techniques have been studied, among them is the acoustic emission (AE) technique. AE is a technique capable of detect and locate the damage in progress. Every time that some damage mechanism is developed in the material a certain amount of elastic energy is released as ultrasonic waves. These waves propagate through the object until reach a piezoelectric sensor attached to the materials surface, where a correspondent voltage output is produced (MCCRORY et al., 2015).

Some authors have also been used AE technique to quantify fatigue damage. Amaral et. al. (Alderliesten; Brunner; Pascoe, 2018; Amaral et al., 2015) verified whether "crack length" as a dimension represents correctly the physical damage created in fatigue fracture. Pascoe et. al. (Pascoe, 2018; Amaral et al., 2015) used the AE technique to study damage evolution within a single load cycle. Therefore, as AE technique is directly related to damage evolution, would it be a better option to capture the physical damage created in delamination growth?

Composite laminates have several specific features that present a huge influence over its mechanical properties, such as fiber volume fraction (FVF), stacking order, void content, and so on. The laminate FVF is extremely important for material properties, and this material feature can be variated as a matter of some design concern to reach the mechanical properties wanted or a consequence of some manufacturing defects that originates restrict regions of FVF variation, such as resin pockets or regions of poor resin impregnation (Swapnil et al., 2017). Thus, if the FVF variation changes the composite mechanical properties, which is the influence of FVF over the energy released during crack propagation? And, is there any relation between the strain energy released and AE?

Therefore, this research work aims to study the influence of FVF variation over the strain energy released during delamination growth in carbon fiber reinforced polymers (CFRP) under mode I quasi-static and low rate cyclic loading and examine the potential of AE technique quantifying physical damage.

1.1 Hypothesis – Analysis of strain energy release in crack propagation under quasi-static mode I loading

During delamination, a crack propagates in between adjacent plies creating new fracture surfaces. According to Griffith (Griffith, 1920), the process of crack propagation is explained by an energy balance based on the thermodynamic first law. Every time that a body is deformed elastically, the material assumes a state of thermodynamic unbalance, in which there is an elastic strain energy accumulation. To restore the energy balance, part of this strain energy accumulated must be released according to the minimum potential energy theorem. The energy release occurs through the creation of new fracture surfaces. However, new fracture surfaces will only be created when enough strain energy is accumulated.

Figure 1 presents a scheme with load (P) x displacement (δ) curves of quasi-static mode I tests under displacement control, conducted according to ASTM D5528-13 with double cantilever beam (DCB) specimens. The area under these curves (E_1 and E_2) corresponds to the elastic strain energy accumulated in the specimen and E_{1-2} is the difference between E_1 and E_2. The assessment of these areas enables an illustration of the energy balance described by Griffith. E_1 is the amount of strain energy just before the crack growth, which means that the material is in a critical state of energy. After this moment, the crack propagates, and new fracture surfaces are created releasing part of the strain energy accumulated (E_{1-2}). Consequently, the specimen reaches to a lower energy state represented by E_2.

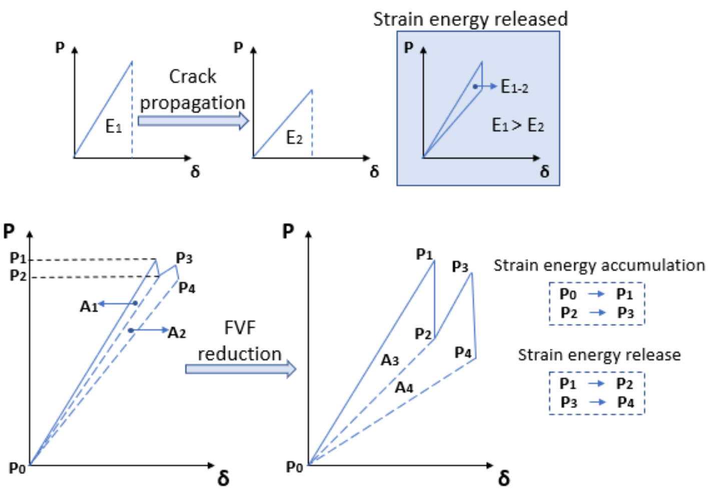

Figure 1. Strain energy release scheme of crack propagation under mode I loading for composite laminates with FVF variation.

Therefore, although specimens are under quasi-static loading, the process of crack propagation is cyclic (Amaral et al., 2015). Firstly, the cycle starts when the specimen is deformed until the strain energy accumulated reaches a level high enough to create new fracture surfaces and, consequently, release part of its energy reducing the load and completing one propagation cycle. After that, the displacement keeps increasing and a new cycle is started. The areas A_1, A_2, A_3, and A_4 represents the energies released by these propagation cycles.

The FVF presents a clear influence over the mechanical properties of composite laminates. Thus, it could be affirmed that the FVF influences the amount of energy released during delamination. Low FVF leads to high amount of polymer in the layer between the reinforcement plies where the crack propagates. This means that the delamination will be mainly governed by the thermoset matrix, which is brittle by nature.

As a consequence of high matrix content, a lower formation of fiber bridging is expected, facilitating the delamination through the specimen.

Therefore, considering a brittle behavior of matrix and low influence of fiber bridging, large delamination increments are expected per propagation cycle, since more instability of delamination is expected. Hence, more energy is released per cycle for materials with low FVF (A_1, A_2 < A_3, A_4). However, this fact does not mean that these materials release more energy during delamination, since large delamination increments result in fewer cycles of propagation. Thus, it is expected that materials with high FVF (> 50%) need more cycles to delaminate, resulting in more energy released during delamination.

2. EXPERIMENTAL PROCEDURES

2.1 Materials and theoretical FVF calculation

The experimental work was carried out on CFRPs with different FVF. The composite matrix was a monocomponent PRISM™ EP2400 epoxy system supplied by SOLVAY. The laminates were reinforced with eight plies of bidiagonal stitched fabrics supplied by SAERTEX, comprised of intermediated carbon fibers (Hexcel IM7 GP) and stitched with PES SC yarns.

Aiming to obtain specimens with different FVF, three laminates were produced by resin transfer molding (RTM) with different thickness but the same lay-up. The preform was assembled in an orthotropic $[0°/90°]_{4S}$ lay-up with an inserted film of polytetrafluoroethylene with 25 µm thickness between 0°/0° interface to produce an artificial pre-crack of approximately 50 mm (measured from the load application point until the end of specimens), as specified in ASTM D5528-13.

After the production of the laminate, DCB specimens were produced according to ASTM D5528-13 with nominal dimensions of 25 mm (width) and 150 mm (length). The theoretical FVF of each specimen was calculated by Eq. (1) to enable further correlations.

$$V_f = \frac{A_w \cdot n_p}{\rho_f \cdot t} \tag{1}$$

Where V_f is the FVF, A_w is the fabric areal weight (408 g/m^2), n_p is the number of fabric layers, ρ_f is the fiber density (1.78 g/cm^3) and t is the measured specimen thickness.

2.2 Determination of mode I interlaminar fracture toughness and strain energy released (SER)

Tests of mode I interlaminar fracture toughness were conducted according to ASTM D5529-13 standard under constant displacement rate of 1mm/min in a MTS 15 kN servo-hydraulic fatigue machine equipped with a 1 kN load-cell. The crack propagation was monitored by a camera with an image acquisition rate of one picture per second positioned in the specimen's side as shown in Fig. 2.

Acoustic emission measurements were carried out using an AMSY-6 Vallen, 8-channel AE system with 4 parametric input channels. One wide-band piezoelectric sensor (AE1045S) with an external 34 dB pre-amplifier and a band-pass filter of 20-1200 kHz was clamped on the specimens as shown in Fig. 2.

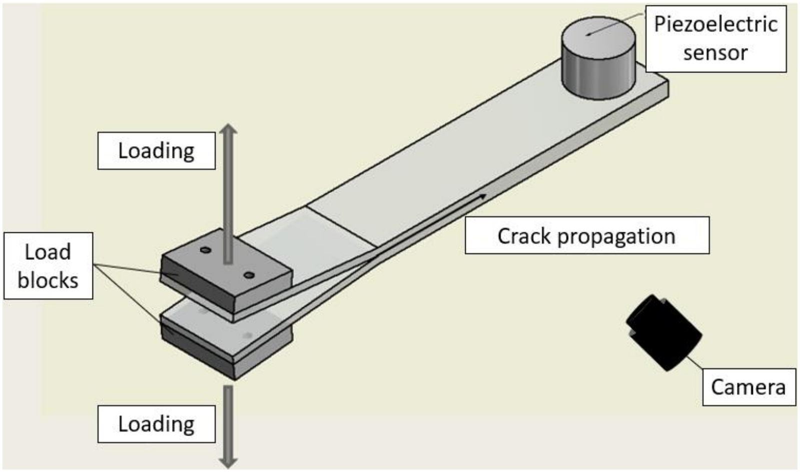

Figure 2. Camera and piezoelectric sensor position in mode I interlaminar fracture toughness tests.

The strain energy released during crack propagation was calculated by the area bellow load (N) x displacement (m) curve as presented in Fig.3.

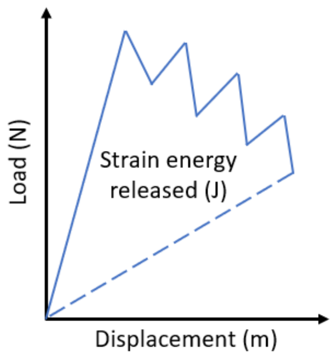

Figure 3. Correlation between the area bellow load (N) x displacement (m) curve and strain energy dissipation.

Aiming to correlate the strain energy dissipated with FVF and AE, the crack surface areas must be considered. Equation 2 was used to calculate the amount of strain energy released per area of fracture surface created.

$$\Delta U = \frac{A_{LD}}{b\ .\ \Delta a} \tag{2}$$

Where ΔU (J/m^2) in the strain energy released, A_{LD} (J) is the area bellow the load – displacement curve, b (m) is the width of the specimen and Δa (m) is the crack length propagated during the test.

2.3 Correlating strain energy released and AE

Considering section 1.1, delamination under quasi-static loading occurs through a cyclic process. However, for small delamination increments these cycles become close to each other, making difficult to associate each delamination increment to the energy released per cycle. Therefore, to accomplish it, quasi-static tests were conducted under cyclic loading with same displacement rate under displacement control. The maximum displacement of the total cycles was defined as 70% of the displacement.

Within one load cycle, the elastic strain energy stored in the specimen (U_{total}) can be measured by the area bellow the load-displacement curve (Fig. 4) (Pascoe; Alderliesten; Benedictus, 2014). However, U_{total} does not regard to crack growth. U_{total} instead is comprised by the energy released, as a consequence of damage development (ΔU), and the residual elastic energy recovered ($U_{recovered}$) by the specimen during unloading, as shown in Fig. 4.

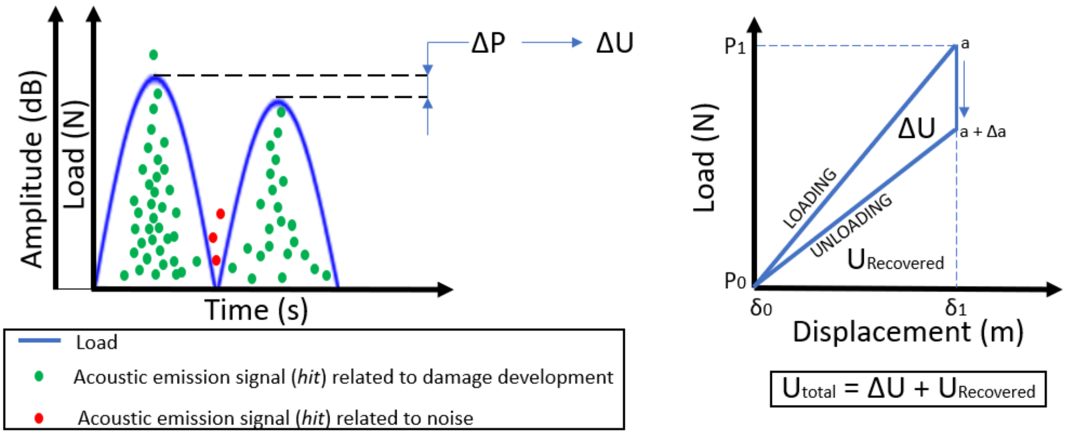

Figure 4. Energy released within one load cycle and behavior of *hits* during cyclic loading.

Therefore, ΔU must be calculated aiming to enable correlations between crack growth and strain energy released. However, the energy released for a delamination increment within one single cycle could be time demanding. A straightforward method consists in calculate ΔU by the difference between the U_{total} of two consecutive cycles. Which makes sense since the damage developed during a cycle causes a reduction of U_{total} in the subsequent cycle as depicted in Fig. 4.

As can be observed in Fig. 4, *hits* regarded to each cycle can be easily determined since load and AE signals were correlated by the time, enabling comparisons of experimental data such as AE, strain energy released and FVF (specimens with different FVF were tested under cyclic loading). Finally, *hits* presented in Fig. 4 in red are regarded as noise as a consequence of crack closure and they were not considered in quantitative results.

3. RESULTS AND DISCUSSION

Results were obtained for six DCB specimens, namely: QST-1, QST-2, QST-3, FT-1, FT-2 and FT-3. Each of these specimens presents a different value of FVF to verify its influence over the strain energy dissipation and AE during crack propagation.

3.1 FVF influence over strain energy released and AE under quasi-static loading

According to the hypotheses presented in section 1.1, it was expected that specimens with low FVF exhibit a brittle behavior as a consequence of crack propagation process controlled mainly by matrix properties.

The load-displacement curves with the FVF of each specimen can be observed in Fig. 5. From the assessment of the curves, the behavior of the specimens during delamination was quite different. As expected, the reduction of FVF leads to large instantaneous load reductions as a consequence of large increments in delamination length, in other words, crack tip jumps were observed. These crack tip jumps, observed mainly in specimen QST-3, result in large strain energy dissipation followed by large periods of strain energy accumulation, until reaches a critical energy value for new crack growth. Therefore, the number of crack growth cycles needed for the propagation of the specimen QST-3 was smaller when compared to the other specimens tested for the same final crack length.

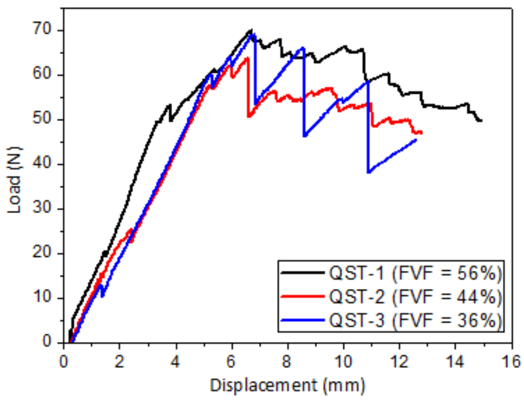

Figure 5. Diagram of load (N) x displacement (mm) of the specimens QST-1, QST-2, and QST-3.

Considering that FVF variation changes the amount of matrix in between layers, the FVF can be considered a parameter that governs the delamination. From another perspective, delamination governed by matrix properties means less fiber constraining the delamination, leading to a reduction of fiber bridging content, which contributes for large delamination increments.

Fiber bridging consists of detached fibers from the delamination interface that link the beam arms providing resistance and constraining the delamination. Therefore, the delamination needs more energy to detach or break these fibers to advance. Figure 6 shows a decreasing FVF that led to a reduction of fiber bridging formation until the total absence of this mechanism in specimen QST-3.

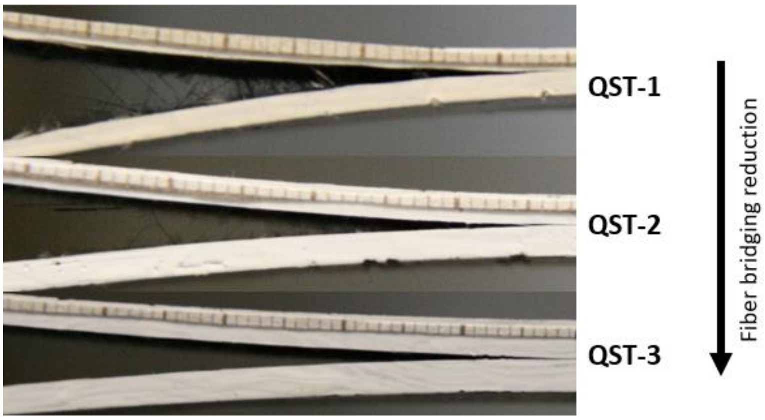

Figure 6. Development of fiber bridging during crack propagation of the specimens QST-1, QST-2, and QST-3.

Thus, from the analysis of Figs. 5 and 6, and based on the hypotheses presented in section 1.1, it is clear that the reduction of FVF leads to a reduction of the energy released during delamination since the propagation cycles are large resulting in increasing periods of strain energy accumulation in which no damage is developed and, consequently, no energy is released. Besides, the fiber bridging formation is reduced leading to less energy associated to delamination. These statements can be proved by the results presented in Table 1.

Table 1. Experimental results of FVF, strain energy released and AE average rate of the specimens QST-1, QST-2, and QST-3.

Specimens	FVF (%)	ΔU (J/m²)	AE average rate (hit/s)
QST-1	56	547	58
QST-2	44	358	33
QST-3	36	330	7

Table 1 presents results of FVF, strain energy released per area of delamination and AE average rate of the specimens QST-1, QST-2 and QST-3.

A strong correlation of ΔU and FVF is observed by verifying that a FVF reduction led to a reduction of the strain energy released to create new fracture surfaces. However, they were not linear correlated.

When the FVF was lower than 50% (QST-2 and QST-3) the amount of energy released was similar, while for the specimen QST-1 with FVF higher than 50% the amount of strain energy released exhibit a higher level. Therefore, CFRP with FVF higher than 50% presented better fracture toughness properties, which means that aiming structural applications, composites with FVF lower than 50% must not be used.

Regarding the AE average rate and the FVF, a strong correlation can be verified as well. Though, this correlation is approximately linear, which means proportional reduction of both: AE average rate and FVF.

Considering results of ΔU and AE average rate, another correlation was established. The reduction of the strain energy released leads to an AE average rate reduction. However, why the emission of hits correlates with the energy dissipated during crack growth?

The AE technique is directly related to damage evolution, each micro mechanism of damage, such as matrix cracking, fiber breakage and fiber/matrix debonding developed during crack propagation is a source of AE signal or *hit.* All these micro mechanisms of damage are associated with some amount of energy; thus, it seems reasonable to correlate AE with strain energy released.

When FVF is reduced, the number of propagation cycles is reduced because of the crack tip jumps, which reduces the AE average rate as well. During a crack propagation cycle, the damage is developed only during delamination growth, while no damage is developed during the process of strain energy accumulation. Therefore, the emission of acoustic signal only happens during crack propagation, which means that large periods of energy accumulation leads to large periods of no AE leading to a reduction of the AE average rate. Besides, the absence of fiber bridging might have had some influence over the AE rate since this damage mechanism was not observed during crack propagation of specimen QST-3.

Aiming to enable better correlations among strain energy released, AE and FVF, the next section will present the results and discussions about the tests conducted under cyclic loading with the same displacement rate of the quasi-static tests.

3.2 Correlation among FVF, strain energy released and AE within a single load cycle

Three specimens with different values of FVF were submitted to cyclic load at very low rate aiming to reduce the noise generated by the machine and mechanical joints and enable comparations with quasi-static tests since the same displacement rate was used. Each specimen was submitted to twenty load cycles as depicted in Fig. 7.

The distribution of *hits* detected during damage evolution can be observed in Fig. 7. Firstly, a qualitative analysis indicates that the quantity of *hits* detected reduces with FVF reduction, which agrees with quasi-static results presented in section 3.1. However, this also suggests that less damage was developed leading to less delamination. Moreover, an AE reduction can be observed in the course of successive load cycles, a reduction that was already expected as the tests were conducted under displacement control. The displacement control leads to a reduction of the strain energy input into the specimen per cycle as a consequence of the energy released during damage development. Thus, if less energy is input into the specimen, less damage development is expected.

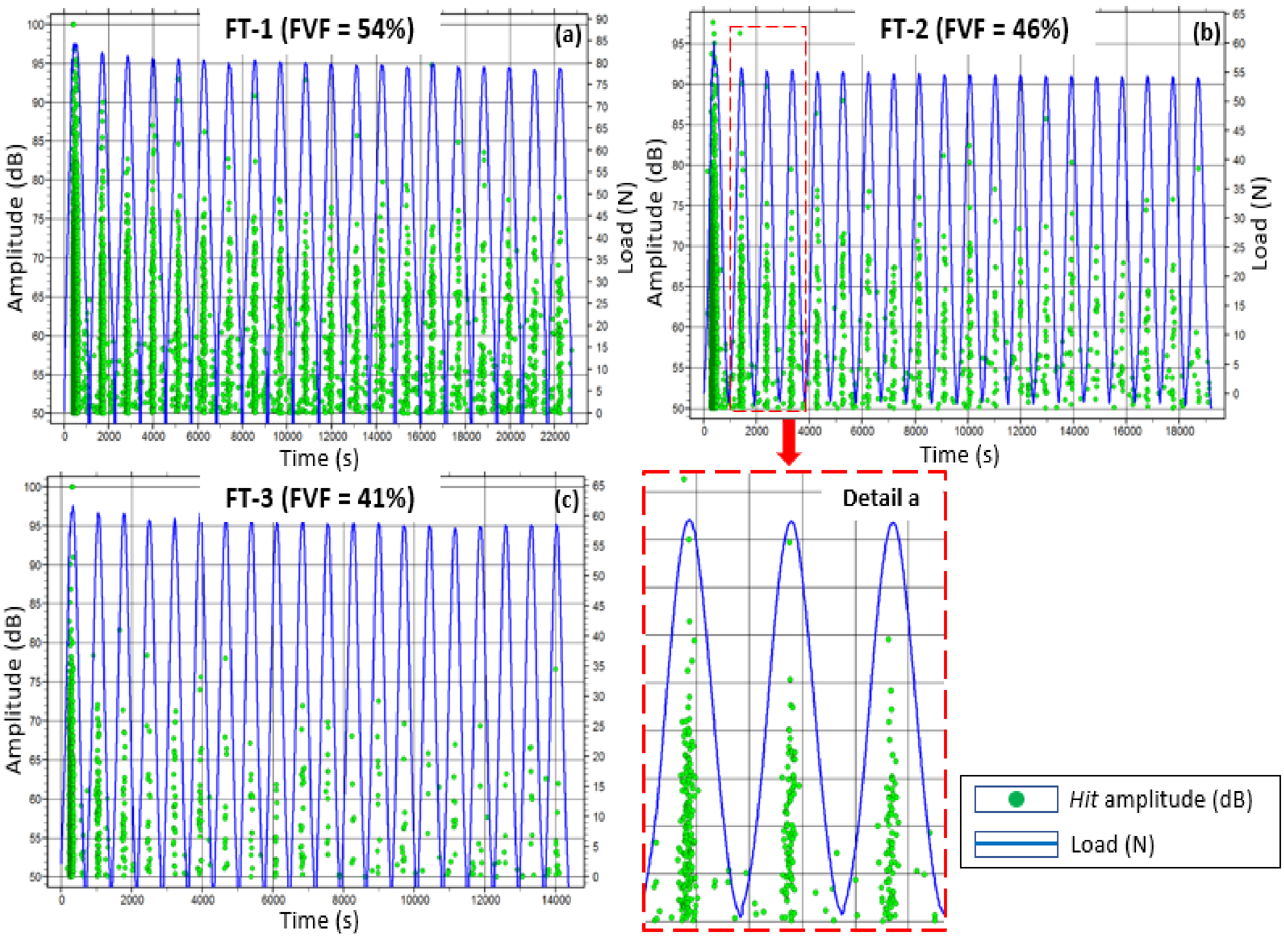

Figure 7. Diagram of the amplitude (dB) of the *hits* and load (N) x time (s) of the specimens FT-1, FT-2, and FT-3.

Focusing on a single loading cycle analyses, the distribution of *hits* detected within a single loading cycle can be observed in Fig. 7 (detail a). A loading cycle presents a variation of the load applied to the specimen, which leads to a variation of the energy applied as well. Besides, according to Griffith (Griffith, 1920), new fracture surfaces will only be created if an energy threshold is reached. Therefore, damage evolution within a single loading cycle will only happen if a critical value of energy is reached. However, these statements do not mean that new fracture surfaces will only be created at the cycle peak. From the analysis of Fig. 7 (detail a), it is possible to affirm that the main damage is developed at the cycle peak, however, it is not restricted only to this moment.

Until now, the discussion developed in this section were mainly based on qualitative observations. Figure 8 shows quantitative results of the number of *hits* detected per cycle and the delamination growth. The data relative to the first loading cycle was not considered in Fig. 8 diagrams because the number of acoustic signals in the first cycle was much higher than in the subsequent cycles, as a consequence of large delaminations (about 8.5 mm, 8 mm and 3.5 mm for the specimens FT-1, FT-2, and FT-3, respectively).

The results depicted in Fig. 8 are consistent with the qualitative analysis performed based on Fig. 7. A reduction of the number of acoustic signals emitted per cycle was observed for both, the FVF reduction and over the development of successive load cycles. However, from the assessment of Fig. 8, the crack propagation was smaller for specimens with low FVF (FT-2 and FT-3), which explains the AE rate reduction as the *hits* are directly related to damage evolution. Therefore, the FVF influence over AE rate cannot be confirmed or measured.

As already mentioned, in this work, the AE is directly related to damage. However, the capacity of the AE technique quantifying physical damage is not fully understood. The parameter that has been used to quantify damage evolution is the delamination length. Figure 8 presents both, the emission of acoustic signal per cycle and the delamination length variation.

From Fig. 8 results, it is possible to observe that no delamination length increment was observed during a several number of cycles for all the three specimens, mainly during the last ten cycles. However, *hits* were detected for all load cycles of the three specimens, as shown in Fig.7. Thus, damage was developed and detected by AE technique, but it was not enough to increment the crack length.

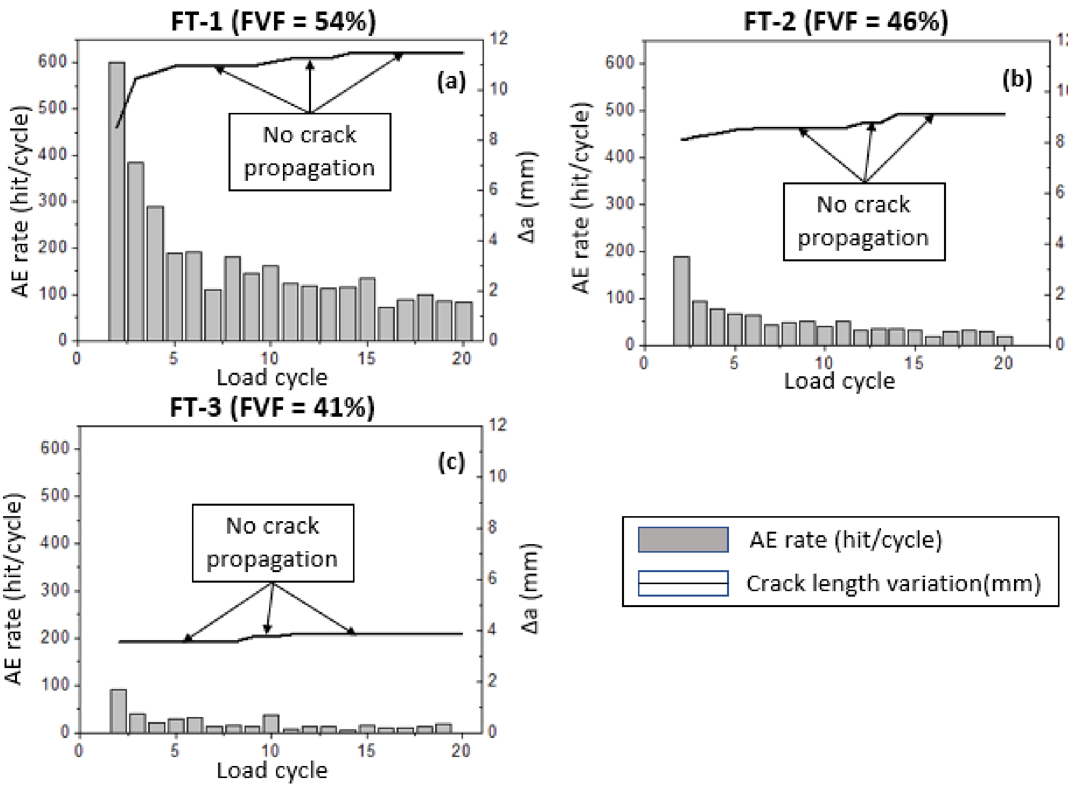

Figure 8. Diagram of the AE rate (hit/cycle) and Δa (mm) x load cycle of the specimens FT-1, FT-2, and FT-3.

This fact proves that AE has a great potential to quantify physical damage since is directly related to damage development and proved to be more sensitive than crack length to detect damage progression.

Finally, Fig. 9 presents the results of the strain energy released per cycle. ΔU was calculated following the methodology described in section 2.3. However, it was only possible to determinate the energy released during the initial cycles in which more energy was released as consequence of more damage development. For cycles with low energy released, ΔU showed to be extremely hard to be quantified, since the test machine does not have enough accuracy.

In general, results presented in Fig. 9 are coherent with results presented so far. Firstly, a reduction of the energy released per cycle correlates with the FVF reduction, following what was observed in quasi-static tests. However, the area of the fracture surfaces created was not considered, which might have overestimated the FVF influence over energy dissipation. A reduction of the energy released over the development of successive cycles was observed as well. Which is a consequence of the displacement control as already discussed in this section.

Lastly, from the assessment of Fig. 8 and Fig. 9 together, it is possible to establish a correlation between the strain energy released and the number of *hits* detected within a single load cycle. High energy dissipation correlates with high AE rates, which makes sense since the acoustic signals are originated by damage development. Damage, on the other hand, needs of energy to be developed. Therefore, the correlation between AE and energy proves that AE has a great potential to be used to quantify physical damage.

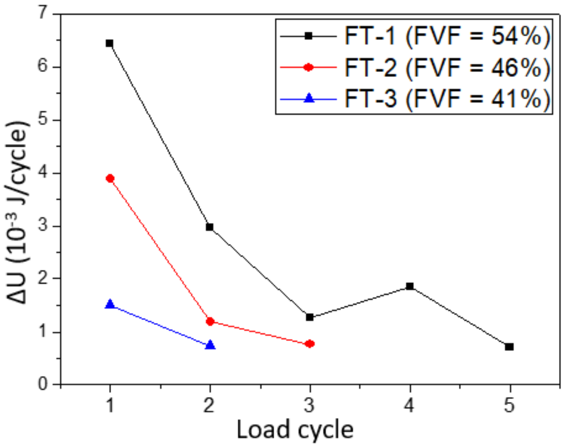

Figure 9. Diagram of ΔU (10^{-3} J/cycle) x load cycle of the specimens FT-1, FT-2, and FT-3.

4. CONCLUSIONS

The acoustic emission technique was used to investigate crack growth behavior under quasi-static and low rate cyclic loading in CFRP with different values of FVF.

Based on the results presented, it was possible to verify that materials with low FVF (< 50%) resulted in materials with brittle behavior, demanding less energy for delamination. Therefore, laminates with low FVF must not be employed in structural applications. Besides, FVF variation caused by manufacturing defects might reduce the operational life of components since regions with high FVF could leads the material to a brittle behavior increasing the crack growth rate for both, quasi-static and cyclic loading. Finally, AE technique presented a strong correlation with the strain energy released during delamination as well as FVF variation. Therefore, AE technique showed a great potential to quantify physical damage in composites.

5. ACKNOWLEDGEMENTS

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6. REFERENCES

Alderliesten, R. C.; Brunner, A. J.; Pascoe, J. A. "Cyclic fatigue fracture of composites: What has testing revealed about the physics of the processes so far?". *Engineering Fracture Mechanic*, v. 203, p. 186–196, 2018.

Amaral, L. et al. "The relation between the strain energy release in fatigue and quasi-static crack growth". *Engineering Fracture Mechanics*, v. 145, p. 86–97, 2015.

Amaral, L.; Alderliesten, R.; Benedictus, R. "Towards a physics-based relationship for crack growth under different loading modes". *Engineering Fracture Mechanics*, v. 195, n. August 2017, p. 222–241, 2018.

Griffith, A. "The Phenomena o f Rupture and Flow in Solid".i. By. v. C, p. 163–198, 1920.

Jones, R. et al. "Delamination growth in polymer-matrix fibre composites and the use of fracture mechanics data for material characterisation and life prediction". *Composite Structures*, v. 180, p. 316–333, 2017.

Khan, R. et al. "Crack closure and fibre bridging during delamination growth in carbon fibre / epoxy laminates under mode I fatigue loading". *Composites : Part A*, v. 67, p. 201–211, 2014.

Khan, R.; Alderliesten, R.; Benedictus, R. "Two-parameter model for delamination growth under mode I fatigue loading (Part B : Model development)". *Composites : Part A*, v. 65, p. 201–210, 2014.

Mccrory, J. P. et al. "Damage classification in carbon fibre composites using acoustic emission: A comparison of three techniques". *Composites Part B: Engineering*, v. 68, p. 424–430, 2015.

Pascoe, J. A.; Alderliesten, R. C.; Benedictus, R. "Towards Understanding Fatigue Disbonding Growth via Cyclic Strain Energy". *20th European Conference of Fracture*, p. 610–615, 2014.

Shahkhosravi, N. A. et al. "Fatigue life reduction of GFRP composites due to delamination associated with the introduction of functional discontinuities". *Composites Part B: Engineering*, v. 163, p. 536–547, 2019.

Swapnil, A. S. et al. "Experimental Investigation of Mechanical Properties of Glass Fibre/Epoxy Composites with variable volume fraction". *Materials Today: Proceedings*, v. 4, n. 9, p. 9487–9490, 2017.

Yao, L. et al. "Delamination fatigue growth in polymer-matrix fibre composites: A methodology for determining the design and lifing allowables". *Composite Structures*, v. 196, n. May, p. 8–20, 2018.

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