

DIFFERENTIATION OF MICRO-FAILURES IN GLASS POLYPROPYLENE COMPOSITES BY MEANS OF ACOUSTIC EMISSIONS TECHNOLOGIES

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ABSTRACT

Currently there is no complete knowledge of the behaviour of composite materials subjected to unpredictable conditions such as those related with wind energy technologies. It is the intention of the article to provide a starting point on characterization of composite materials based on the frequency obtained from their acoustic emissions.

Tensile test were carried out on glass/polypropylene specimens made in-home, and acoustic emissions were recorded from these tests. The fibre orientation of each specimen was different in order to obtain preferred failure modes. The hypothesis is that each micro mechanical event will have one distinctive waveform as fingerprint. In order to differentiate the waveforms the primarily frequency is plotted on a power spectrum graph by means of a Fast Fourier Transformation. The primarily frequency from each power spectrum is then plotted against progress of test. The resulting graph showed clusters around well-defined frequencies. From the results it can be observed the existence of a relation between micro mechanical events and specific frequencies.

1. INTRODUCTION

Despite having complex inner structures, composite materials usually have well defined mechanical characteristics. And in the last decade the use of composite materials has embraced several applications. This phenomenon has lead to an increase on the complexity of shapes and the different types of loads under which they work. Several applications such as wind energy technologies have used composite materials for many years, but as they grow in size and move offshore they require more reliable materials. Therefore a deeper understanding of the behaviour of composite materials is demanded.

Several researches on this matter have been carried out in late days; most of them had used non-destructive tests in order to obtain information from the composite material without damaging the piece [ⁱ, ⁱⁱ, ⁱⁱⁱ, ^{iv}]. Acoustic emission technologies provide a wider understanding on the micro mechanical events occurring within the test piece. When a micro mechanical event occurs, energy is released through the material as a wave. This wave will be picked by a transducer and then processed in order to obtain information about the source of it. From the information available the feature holding more information about the source is the frequency of the signal.

2. THEORETICAL BACKGROUND

Wide ranges of technologies are available to monitor the progression of damage within composite materials; these are grouped under the term Non Destructive Testing (NDT). Examples of NDT technology are: thermal emission (SPATE), optical transference (using embedded optical fibres), ultrasonic scanning (C-Scan), and acoustic emission. Acoustic emission (AE) monitoring is the primary technology used for the identification of different types of failure in composite materials for in-service monitoring [^v].

According to Wolters & Bardenheir [^{vi}], the term Acoustic Emission (AE) relates to any material system under test where mechanical vibrations with no external excitation are detected and evaluated. The range of these AE events extends across events with low frequency such as earthquakes, in the audible sound range the breaking of wood and in the ultrasonic range (high frequencies) micro cracking in solids. When critical stress values are exceeded in a material, it fails locally (on a micro-scale) and the strain energy stored is

suddenly released; this creates mechanical stress waves, which are spread concentrically around the place of origin. The energy released in this way can be detected with suitable sensors: the mechanical information picked up from the material is then converted into an electrical signal.

Each of these signals contains amplitude and frequency spectrum information sufficient for each signal to be clarified as originating from a defined micro-mechanical event. Each signal is defined as a wave having several parameters conforming it. According to Pollock [vii] the most commonly used are: amplitude, duration, counts, rise time and MARSE (measured area under the rectified signal envelope). It is interesting to note that by the time Pollock's document was published, frequency was not considered significant for AE analysis, but nowadays it is one of the defining parameters for signal differentiation [viii].

2. TESTING AND RESULTS

The manufacturing technique used was membrane forming. This technique employs two flexible silicon layers fixed to a frame that closes and holds Plytron™ layers between them. The side of the frame has an entrance from where a vacuum can be applied and both membranes flatten the material within them by applying one atmosphere (0.1 MPa) of uniaxial pressure. When the material is flattened and under pressure, the whole frame is placed in an oven, which increases the temperature of the polypropylene matrix and melts it. Plytron™ is a prepreg which holds polypropylene along with glass fibres in a thin tape ready to conform, it is cut in layers or plies, which are arranged one on top of the other configuring different fibre orientations (0°, 90°, 0-90°, +/- 45°, etc...). This is done to obtain preferred failure modes in the specimens. Plytron™ is fully laminated after 2-5 minutes in the oven and then it needs 10 minutes to cool down and be ready for cutting the test specimens.

The composite plate is marked and cut on a band saw with a fine-toothed blade to obtain tensile test specimens. The specimen dimensions are 200 x 25 x 1.7mm. 25mm width was essential to provide the AE sensor with 18 mm diameter with enough area to create a good coupling. Specimens were tensile loaded on an Instron 4550 universal test machine. The crosshead speed was set to 5 mm/min and the minimum load for the operation to stop was 30 N, complying with ASTM D3039 standard.

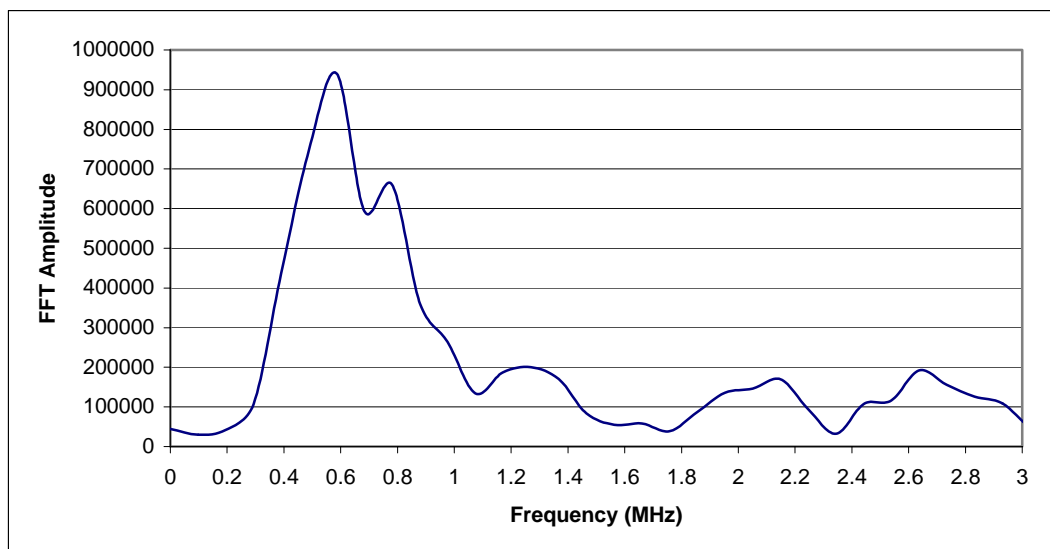


Figure 1 Frequency response of the WD sensor

A broadband WD piezoelectric sensor by Physical Acoustic Corporation was used to capture the stress waves (AE events). After capturing the event, the piezoelectric sensor transforms the mechanical deformation of the material surface into an electric field. This electric field is

transferred to a sound card with a high sampling rate, which processes it and displays it as a wave. The software used with this soundcard can display on screen the transient waveform for each event. Each event can then be individually exported into an ASCII file, which is processed by a program written in 'R' programming language (open source code language). By using this program is possible to conduct and visualize the Fast Fourier Transformation for each event and select the most dominant frequency, which is to be used as the descriptor of the event. According to Qi [ix], 90% of AE activities for glass fibre reinforced (GFR) composite materials are concentrated in the frequency range 10-550KHz. The sensor used has an operation range of frequencies between 100 to 1000 kHz with a resonant frequency at 125 kHz. The wide bandwidth response of this sensor is shown in Figure 1. The sensor was attached to the specimen by means of a G aluminium clamp with a plastic screw. The surface of the sensor was covered with silicon grease in order to provide a good acoustic coupling between the specimen and the sensor. The signal was detected by the sensor and enhanced by a 2/4/6-AST pre-amplifier. The gain selector of the pre-amplifier was set to 40 dB, as the material used was not considered 'loud'.

The acoustic emissions software was the TRA (transient recorder package) from Physical Acoustics Corporation 2001. The hardware was a sound card AEDSP-32 (Acoustic Emissions Digital Signal Processor) with a digital signal processor by Texas Instruments (TMS320C40). It has a maximum sampling speed of 8 MHz and 16 bits resolution, between 20 dB to 100dB. Using an empirical approach the following settings were selected for the succeeding tests (Table 1):

Settings	Description
<i>Rate: 4 MHz</i>	Sampling rate, from the literature review it was seen that frequencies are rarely above 1 MHz, therefore it is recommended to sample at twice the speed of the highest signal recorded. The options from the TRA provide 2, 4 and 8 MHz sampling.
<i>Low: 10 kHz</i>	High pass on-board filter, the frequencies found on composite materials are usually low.
<i>High: 1200 kHz</i>	Low pass on-board filter, this is the highest option available on the TRA system.
<i>Mode: IND</i>	Trigger mode, set to independent to each sensor.
<i>Source: DIG</i>	Trigger source, set to digital.
<i>Level: 54 Db</i>	Trigger level, within the software an AE event is recorded whenever the signal exceeds the preset threshold. Based on the sensitivity and the loudness of the material this level might vary.
<i>Delay: -20.00µs</i>	Trigger delay, when negative indicates that the recording of the signal starts before the trigger is activated. e.g. the software retrieves data from prior to trigger.
<i>Length: 1 k</i>	Hit length in time, when sampling at 4MHz, 1 k = 0.250 µs
<i>Points: 1023</i>	Data points, each sample, when turned into an ASCII file, it will contain 1023 points.
<i>Preamp: 40 Db</i>	Gain selection, defines the gain leading for the software, 40 Db gain from the pre-amplifier equals to 10 volts at the output.
<i>HLT: 1000µs</i>	Hit lockout time, this is the time between recordings. It prevents the system recording signals which are part of a bigger signal

Table 1 Recording settings for the acquisition software.

From the literature review, it appears that there are several different approaches to the event recognition analysis, from simple hit analysis to amplitude-based and frequency-based studies [x, xi, xii, xiii]. The amplitude of an acoustic wave is attenuated as the wave passes through a material. Thus the amplitude recorded at a sensor depends upon the distance the sensor is from the source of the emission. Based on the previous, for the present study the dominant frequency of the signals was considered the most relevant characteristic.

When the specimen is subjected to a tensile test, micro mechanical events occur and generate acoustic emissions. The TRA software records these signals as waveform events. A waveform event contains several component frequencies, and it is not possible to determine them visually, therefore a program that calculates the Fast Fourier Transformation (FFT) processes each waveform. The procedure produces a power vs. frequency graph of a waveform (power spectrum). Higher powers contained at particular frequencies are then shown as peaks on this

representation of the waveform. According to Qi [^{xiv}] these frequencies are useful to “distinguish different AE signals from various possible failure modes in fibre reinforced composites”. If more than one frequency is super-imposed on the waveform each component appears as secondary frequency and so on. Currently the program that generates the FFT identifies the 5 frequencies from each signal with the highest power peaks as shown in Figure 2: (a) Transient event waveform; (b) FFT power spectrum. In several cases the power difference between the highest power frequency and the subsequent lower power frequencies is such that it can be assumed that the single frequency constitutes the definition of that waveform. Based on the data, it was assumed that the event primary frequency (1°EF) alone was enough to characterize each acoustic event.

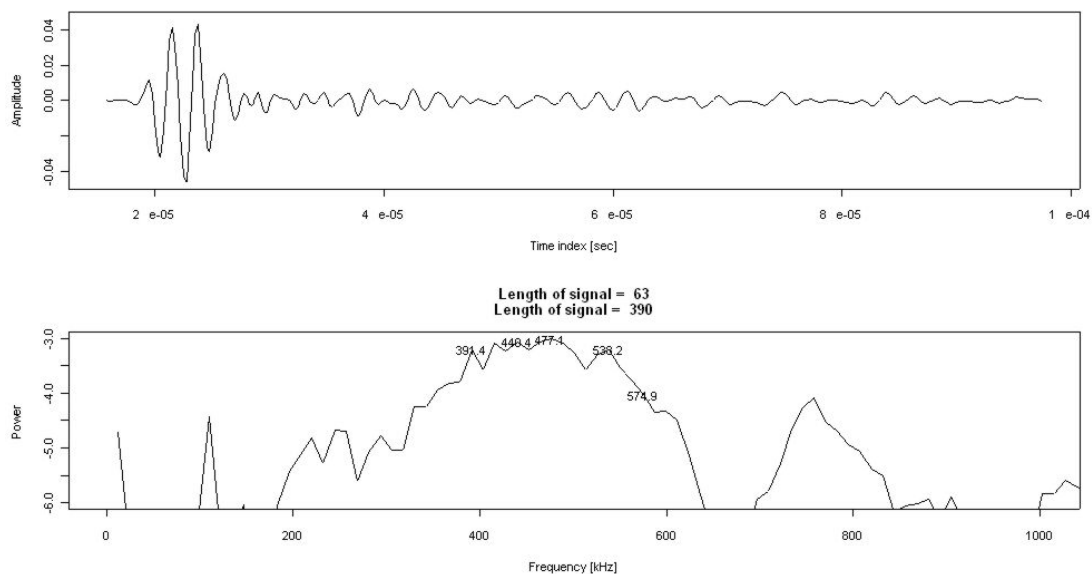


Figure 2: (a) Transient event waveform; (b) FFT power spectrum

Figure 2 shows a typical AE event recorded by the system, Figure 2a shows the original signal and Figure 2b presents its FFT power spectrum. The FFT power spectrum in this specific case shows several peaks, the higher one being 477 KHz and having a second higher peak at 440 KHz. The FFT power spectrum can provide enough information to ‘fingerprint’ each event and therefore may be used as means of distinguishing them.

The laminate stacking sequence with respect to the direction of the applied force defines the potential predominant failure modes: fibre breakage, fibre-matrix debonding or matrix cracking. In the case of PP thermoplastics, the matrix is rather ductile, resulting in few acoustic events occurring in the deformed matrix. This characteristic provides a starting point for the identification of the event frequencies with their respective micro mechanical events.

Transverse orientated glass/polypropylene (90°)

The resulting frequencies from these tests provided clear evidence of at least one micro event: fibre/matrix debonding. The fibres for 90° specimens when tested in tension tend to get separated from the matrix (fibre/matrix debonding without fibre breaking). In these tests many primary event frequencies found in following tests were not present. This is the result of certain micro failures not being present. As for those frequencies that were present, they could be related with failure modes which are present on the specimens: fibre/matrix debonding and some matrix cracking. Figure 3 shows the 1°EF vs. time results of one tensile test for 90° UD glass/polypropylene. In order to identify which frequency is related with each type of

microfailure, the progress-based distribution of 1° EF for each test was plotted. As stated, each point on the graph is one acoustic emission event, which 1°EF falls on the vertical axis of the graph and its horizontal position is related to time. The density of points is directly related to the amount of micro-mechanical events happening within the specimen.

UD (90°) plytron progress of test

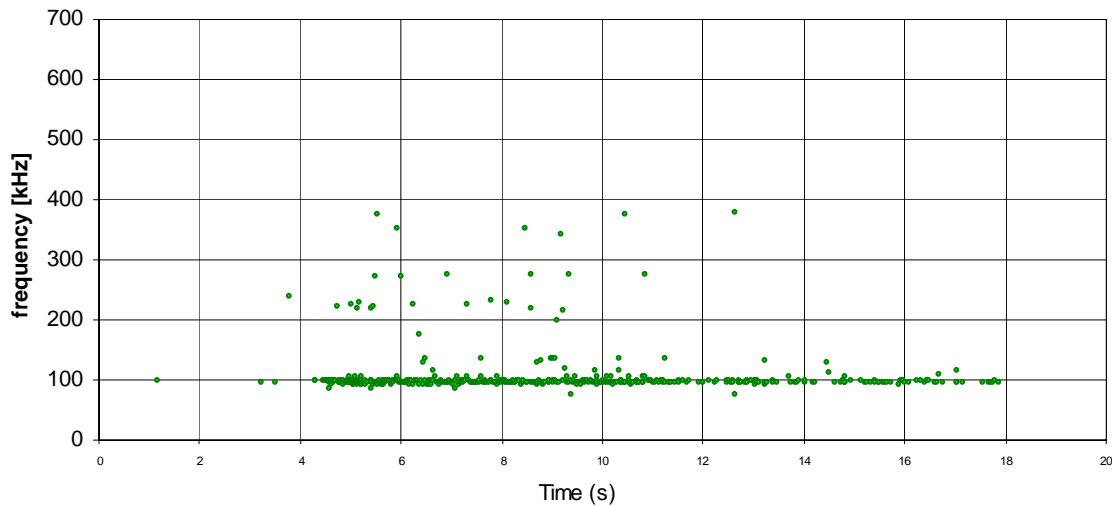


Figure 3 1°EF test for transverse Plytron™.

The previous figure showed mainly 1°EF around 100 kHz, few dots appeared above 200 kHz and none above 400 kHz. Figure 4 illustrates the fact that almost no fibres broke during this test; therefore all the events encountered can be related solely to micro failure occurring between the matrix and the fibres.

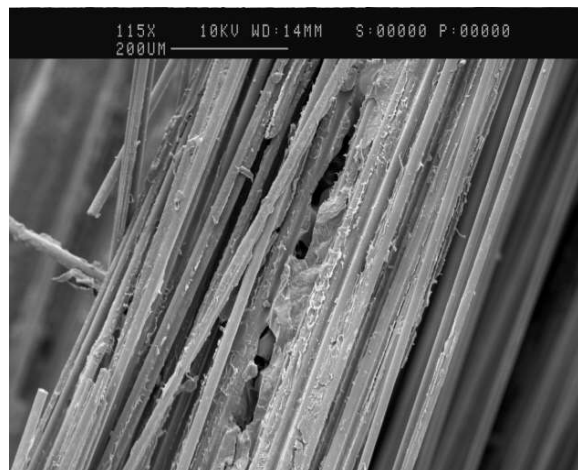


Figure 4 SEM micrograph of typical fracture damage for 90° configuration.

Unidirectional glass/polypropylene (0°)

The specimens were manufactured with 8 layers of unidirectional Plytron™ orientated parallel to the direction of the force [0]₈. Twelve tensile tests were carried out on unidirectional Plytron™ at 0°.

Due to extensive damage in the 0° test specimens, post-test analyses of them would probably not yield enough information about the damage initiation and progress. To obtain failure in a localized area another set of tests was carried out with a 1 mm hole drilled in the middle of the specimen. It was expected that transverse failure would be initiated near the hole, in fact the

specimen failed in shear along the specimen length (longitudinally), initiating at the hole. Figure 5 shows the resulting primary event frequencies from the hole test, a band pattern at certain frequencies can be observed. This behaviour was considered to be closely related to the failure events occurring during the test and it was different to the UD test when the hole is not present.

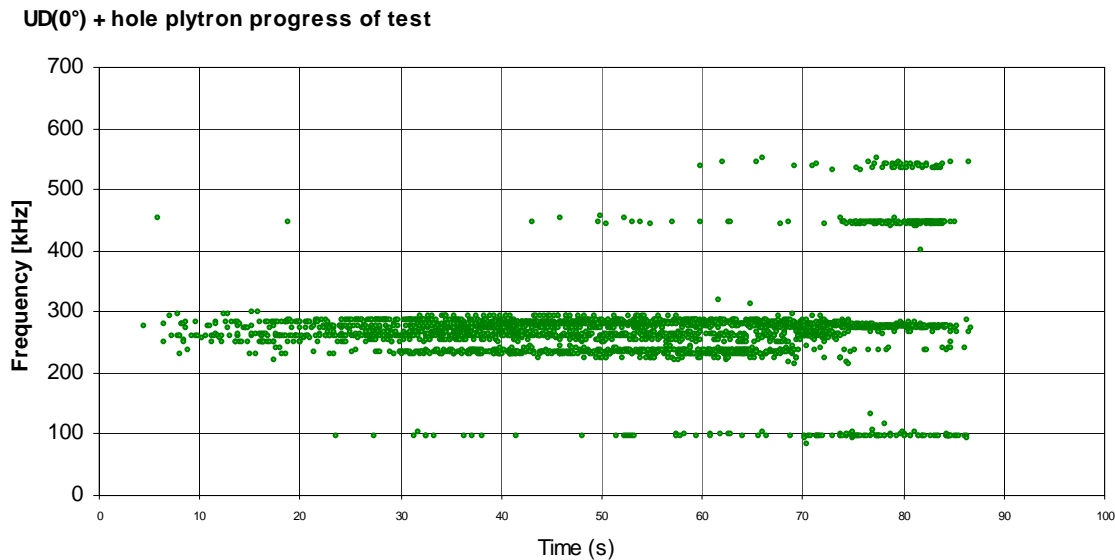


Figure 5 1°EF for unidirectional Plytron™ + drilled hole.

The information shown in Figure 5 is remarkably important. Events are occurring towards the end of the test, prior to failure, with 1°EF of 450 kHz and 550 kHz. The events occurring at frequencies between 220 and 300 kHz also showed variation as the time progressed, with time-dependent (and so strain-dependent) transitions between primary event frequencies. The inclusion of the drilled hole appeared to change the micro-mechanical events occurring in the specimen. Figure 6 shows a micrograph of the glass fibres and the state of the polypropylene matrix from the 0° hole-drilled specimen fracture surface.

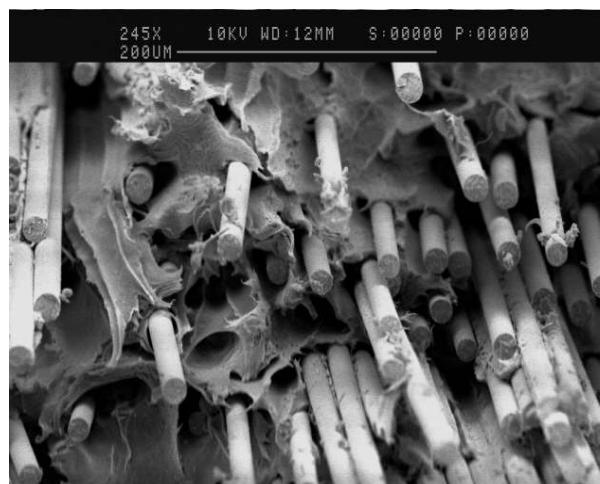


Figure 6 SEM micrograph of typical fracture damage for 0° configuration.

Note the fluid behaviour of the polypropylene when subjected to stress. The separation between the glass-fibre and the polypropylene is also seen, which effectively illustrates fibre/matrix pull out.

Angle-ply glass/polypropylene (+/- 45°)

Interlaminar shear is the primary mechanical event occurring within the $[\pm 45^\circ]_s$ specimen. The group of 1°EF observed in Figure 7 just above 300 kHz are not found in any other previous tests. These events appear at the beginning of the test and disappear after 30% progress of test; therefore it is believed that these frequencies represent interlaminar shear micro-level events. The cluster of 1°EF above 500 kHz appears to shift from higher frequency to a slightly lower frequency as test time progresses. Nevertheless, the cluster may be related with one micro-failure, and the fact that it reduces its frequency throughout the test might be because of an interference with another type of micro-failure. In such cases, the mechanical properties of the specimens vary during the test as new micro-failure types are triggered.

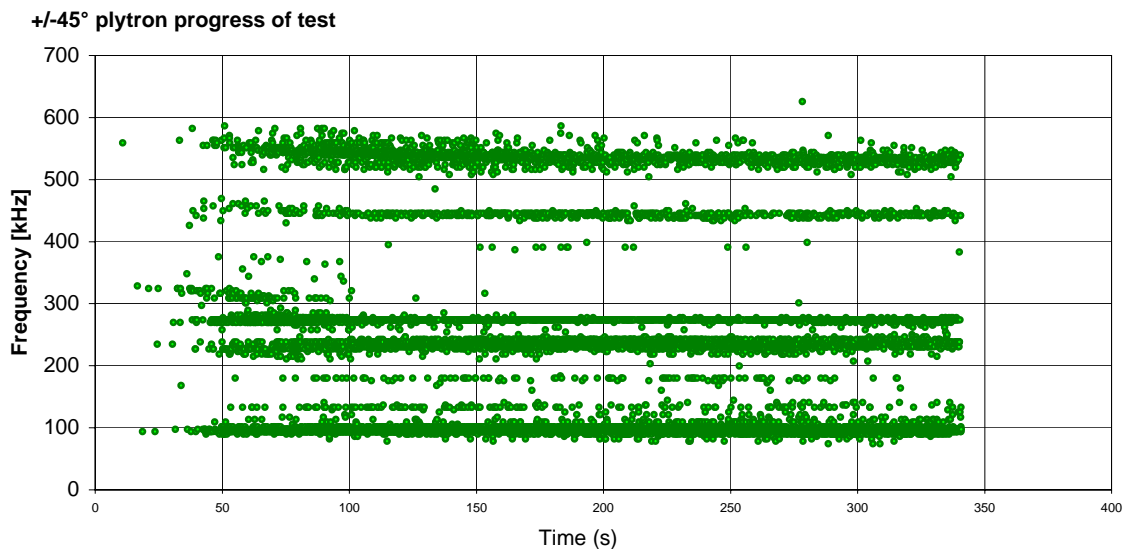


Figure 7 1°EF test for +/- 45° Plytron™.

It is clear that these tests have a longer duration (go to higher strains) than the other tests, and hence more events are recorded. The unidirectional tensile test at 0° usually results in 4000 event points compared with 14000 recorded from the (+/- 45°) test.

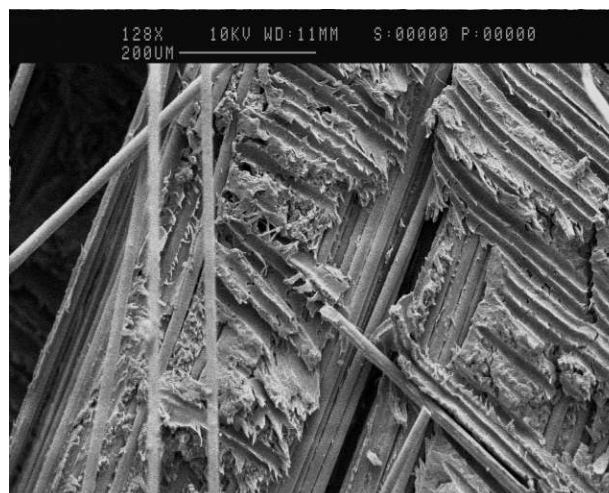


Figure 8 SEM micrograph of typical fracture damage for +/-45° configuration.

Figure 8 illustrates the polypropylene ductile character as the fibres draw out fibrils when subjected to tensile test.

Cross-ply glass/polypropylene (0/90°)

Figure 9 shows the 1°EF band for a tensile test of a 0/90° specimen. In this case the failure was more localized. For this fibre orientation, some fibre debonding just after the beginning of the test was expected; followed by shear and matrix cracking. Certainly the number of fibres actually breaking for the cross-ply material is less than for unidirectional Plytron™ tested at 0°. The cross-ply test pieces were made with 4 layers of Plytron™ at [0,90]_s. Here, fibre/matrix debonding is expected from the beginning of the test.

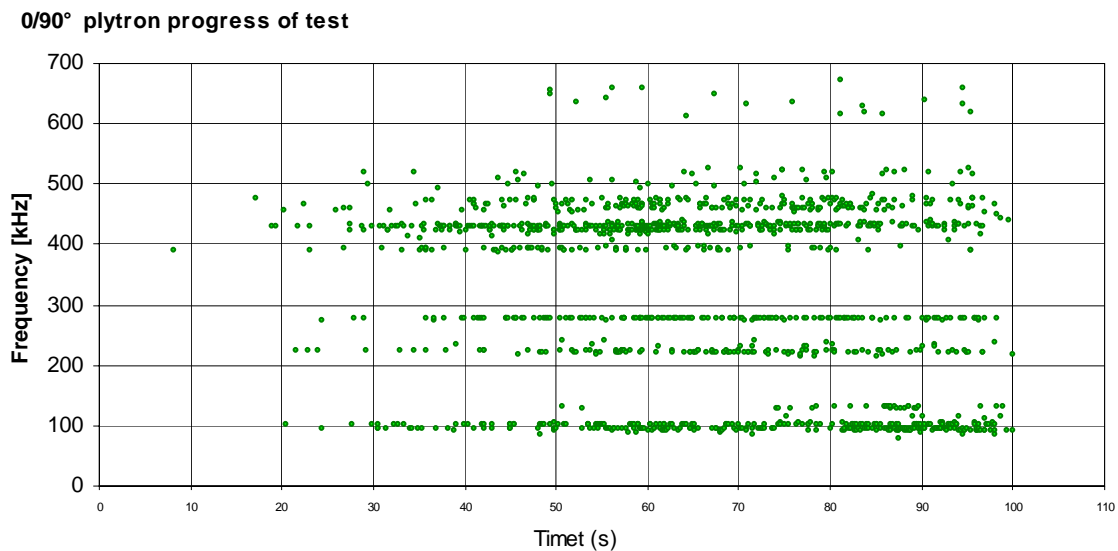


Figure 9 1° EF test for 0/90° Plytron™.

Figure 10 shows a micrograph of the fracture surface for 0/90° tested specimen. Due to fibre debonding and pull out, the polypropylene showed a crossed pattern illustrating grooves where glass fibres were held.

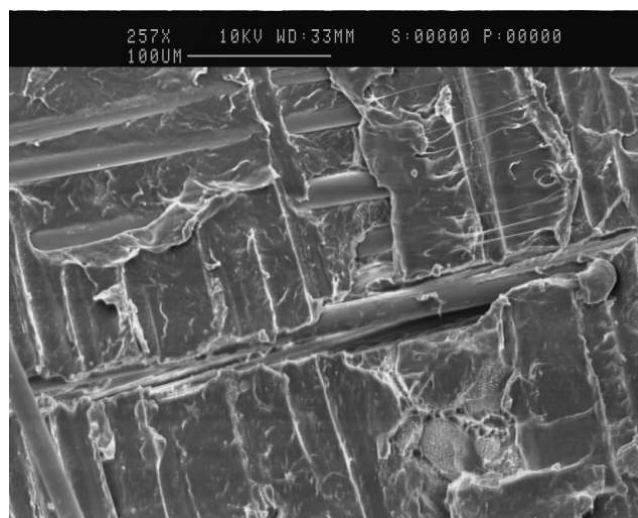


Figure 10 SEM micrograph of typical fracture damage for 0/90° configuration.

3. CONCLUSIONS

During the testing and analysing the graphs obtained from each different fibre orientation, patterns in primary event frequency distribution were found. It is believed that these patterns are related with the specific micro failure within the material.

The behaviour of a specimen manufactured only with polypropylene under tension loading is known from previous tests undertaken in the department. Randle [^{xv}] carried out several tests on pure polypropylene specimens and reported that the material shows a comparatively low number of acoustic emissions when loaded due its ductile behaviour. These few events are found at the beginning of the test, because of the gripping jaws, and at the end of the test because of ultimate fracture. Considering the 'quiet' behaviour of polypropylene, matrix cracking as a separate event was not considered for the allocation of a distinctive frequency. Nevertheless, it is believed that cracking of the matrix on glass fibre reinforced materials occurs when the fibres break and release energy to the matrix. In this case the micro event is considered the result of the behaviour of both the matrix and the fibres.

Figure 3 shows the results for the Plytron™ unidirectional tensile test with a 90° fibre orientation. It is seen that there is a cluster of events appearing at 25% of the test with a primary frequency of around 100 kHz. It is possible to relate these events with the fibre-matrix debonding occurring within the test piece. Due the orientation of the fibres there are no other micro mechanical events that could possibly occur during the test.

Figure 5 shows unidirectional 0° hole-in-plate test results, where the hole in the middle of the piece generated different stresses when subjected to force. This behaviour leads to several events occurring between the matrix and the fibres before the actual breaking of the fibres. These events have 1°EF between the 200 and 300 kHz range. The combination of different micro mechanical failure modes might lead to clusters with wider frequency range. These events do not appear to occur in the 90° test; therefore they can be related to fibre pull out and fibre/matrix slippage. Also in Figure 5, some events with primary frequencies at 450 and 550 kHz towards the end of the test can be seen. According to the literature fibre breaking events always appear at higher frequencies, so it is believed that those frequencies correspond to the fibre breaking within the test piece [^{xvi}, ^{xvii}, ^{xviii}].

Figure 7 shows the primary event frequency results for +/- 45° orientated fibre tests. The failure strain of the specimen was five times larger than the unidirectional specimens due to the high elongation ability of the lay-up configuration. The high complexity of the behaviour of the specimen when tested (high amounts of interlaminar shear) resulted in a higher density of 1°EF points. It was found that the same 1°EF banding was present as in previous tests over the 100, 240, 280, 450 and 550 kHz frequencies. Previously unseen 1°EF appeared (albeit sporadically) between 100 and 200 kHz, but currently there is uncertainty as to which events they are related to.

Figure 9 shows the results for 0/90° cross-ply Plytron™. It was tested in order to differentiate between those events appearing in unidirectional 0° Plytron™ and those appearing on the UD 90°. The appearance of new 1°EF was observed; one cluster just below 400 kHz and another one around 480 kHz. The known clusters around 240 and 280 kHz appeared well defined in this test, as well as the 100 kHz cluster. It was also found that there were some isolated dots above 600 kHz that haven't been defined yet. This test did not establish the differences between the sources of the 1°EF between 200 and 300 kHz. Added to the fibre/matrix pull out, fibre/matrix debonding and fibre breaking, this configuration of plies also creates inter-laminar shear.

The 100 kHz primarily event frequency range (1°EF) in glass polypropylene pieces tested, is due to fibre/matrix debonding. The 1°EF occurring between 200 and 300 kHz are due to fibre slippage and fibre pull out, but it has not yet been defined which frequency is related with each event. The two higher 1°EF appearing in all tests (except those at 90°) are related with fibre breaking. The range of these 1°EF varies from test to test the highest being at around 520

kHz and the second one at around 420 kHz and. Nevertheless further work is needed for defining the natural frequency of the material.

It is relevant to analyse the relationship between the highest (primary) and the second highest (secondary) frequency appearing in each event, because it might provide enough information to differentiate combined events. These two frequencies might be related to the same event. An in-depth study of the relationship between the two highest power frequencies or even the three highest power frequencies might provide a more accurate way to define each event. Testing brittle materials from the thermosetting polymer matrix family combined with glass will also illustrate the values for matrix cracking (if any). Similarly testing polypropylene with carbon fibres might also provide different patterns of clustering.

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